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# Tungsten Powder

## Physical , Chemical Properties, Preparation, & Applications

中钨智造科技有限公司

CTIA GROUP LTD

**CTIA GROUP LTD**  
Global Leader in Intelligent Manufacturing for Tungsten, Molybdenum, and Rare Earth Industries

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## INTRODUCTION TO CTIA GROUP

CTIA GROUP LTD, a wholly-owned subsidiary with independent legal personality established by CHINATUNGSTEN ONLINE, is dedicated to promoting the intelligent, integrated, and flexible design and manufacturing of tungsten and molybdenum materials in the Industrial Internet era. CHINATUNGSTEN ONLINE, founded in 1997 with [www.chinatungsten.com](http://www.chinatungsten.com) as its starting point—China's first top-tier tungsten products website—is the country's pioneering e-commerce company focusing on the tungsten, molybdenum, and rare earth industries. Leveraging nearly three decades of deep experience in the tungsten and molybdenum fields, CTIA GROUP inherits its parent company's exceptional design and manufacturing capabilities, superior services, and global business reputation, becoming a comprehensive application solution provider in the fields of tungsten chemicals, tungsten metals, cemented carbides, high-density alloys, molybdenum, and molybdenum alloys.

Over the past 30 years, CHINATUNGSTEN ONLINE has established more than 200 multilingual tungsten and molybdenum professional websites covering more than 20 languages, with over one million pages of news, prices, and market analysis related to tungsten, molybdenum, and rare earths. Since 2013, its WeChat official account "CHINATUNGSTEN ONLINE" has published over 40,000 pieces of information, serving nearly 100,000 followers and providing free information daily to hundreds of thousands of industry professionals worldwide. With cumulative visits to its website cluster and official account reaching billions of times, it has become a recognized global and authoritative information hub for the tungsten, molybdenum, and rare earth industries, providing 24/7 multilingual news, product performance, market prices, and market trend services.

Building on the technology and experience of CHINATUNGSTEN ONLINE, CTIA GROUP focuses on meeting the personalized needs of customers. Utilizing AI technology, it collaboratively designs and produces tungsten and molybdenum products with specific chemical compositions and physical properties (such as particle size, density, hardness, strength, dimensions, and tolerances) with customers. It offers full-process integrated services ranging from mold opening, trial production, to finishing, packaging, and logistics. Over the past 30 years, CHINATUNGSTEN ONLINE has provided R&D, design, and production services for over 500,000 types of tungsten and molybdenum products to more than 130,000 customers worldwide, laying the foundation for customized, flexible, and intelligent manufacturing. Relying on this foundation, CTIA GROUP further deepens the intelligent manufacturing and integrated innovation of tungsten and molybdenum materials in the Industrial Internet era.

Dr. Hanns and his team at CTIA GROUP, based on their more than 30 years of industry experience, have also written and publicly released knowledge, technology, tungsten price and market trend analysis related to tungsten, molybdenum, and rare earths, freely sharing it with the tungsten industry. Dr. Han, with over 30 years of experience since the 1990s in the e-commerce and international trade of tungsten and molybdenum products, as well as the design and manufacturing of cemented carbides and high-density alloys, is a renowned expert in tungsten and molybdenum products both domestically and internationally. Adhering to the principle of providing professional and high-quality information to the industry, CTIA GROUP's team continuously writes technical research papers, articles, and industry reports based on production practice and market customer needs, winning widespread praise in the industry. These achievements provide solid support for CTIA GROUP's technological innovation, product promotion, and industry exchanges, propelling it to become a leader in global tungsten and molybdenum product manufacturing and information services.



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## CONTENT

### Preface

The scientific and industrial value of tungsten powder

Book Structure and Objectives

Acknowledgements and Data Sources

### Part 1 Basic Science of Tungsten Powder

#### Chapter 1 Physical and Chemical Properties of Tungsten

1.1 Atomic Structure and Electronic Properties of Tungsten

1.2 Crystal Structure (BCC, HCP Phase Transition and Crystal Defects)

1.3 Thermodynamic Properties (Melting Point, Thermal Expansion Coefficient, Vapor Pressure)

1.4 Chemical Stability (Corrosion Resistance, Oxidation Behavior and Reactivity)

#### Chapter 2 Morphology and Classification of Tungsten Powder

2.1 Particle Size Distribution and Morphology (Spherical, Flake, Porous Structure)

2.2 Ultrafine Tungsten Powder (Submicron) and Nano Tungsten Powder (<100 nm)

2.3 High Purity Tungsten Powder (>99.95%) and Doped Tungsten Powder ( $La_2O_3$ , K,  $Y_2O_3$ )

2.4 Tungsten Oxide Morphology ( $WO_3$ ,  $WO_{2.9}$ ,  $WO_{2.72}$ ) and Its Characteristics

2.5 The advantages and disadvantages of particle size distribution of tungsten powder

2.6 Industrial significance of particle size distribution of tungsten powder

### Part 2 Tungsten Powder Preparation Technology

#### Chapter 3 Traditional Metallurgical Processes

3.1 Hydrogen Reduction (APT/ $WO_3$  Reduction, Process Parameter Optimization)

3.2 Carbothermal Reduction (Reaction Kinetics and Carbon Content Control)

3.3 Molten Salt Electrolysis (NaCl-KCl- $WO_3$  System and Electrolysis Parameters)

#### Chapter 4 Modern Advanced Preparation Technology

4.1 Plasma Spheroidization Technology (RF and DC Plasma Process)

4.2 Chemical Vapor Deposition (CVD) and Physical Vapor Deposition (PVD)

4.3 Mechanical Alloying (High Energy Ball Milling and Nanocrystalline Tungsten Powder)

4.4 Synthesis of Nano-Tungsten Powder (Hydrothermal Method, Sol-Gel Method, Spray Pyrolysis)

#### Chapter 5 Surface Modification and Composite Technology

5.1 Surface coating (Effects of Ni, Cu, Ag on sintering and performance)

5.2 Tungsten-based composite materials (W-Cu, W-Ni-Fe, W-ZrO<sub>2</sub>)

5.3 Functionalized tungsten powder (antibacterial, conductive, radiation shielding modification)

5.4 Dispersion preparation (water-based, alcohol-based dispersion process and stability)

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### Part 3 Tungsten Powder Properties and Characterization

#### Chapter 6 Physical and Mechanical Properties

- 6.1 Density and Porosity (Comparison of Tap Density and Theoretical Density)
- 6.2 Hardness and Wear Resistance (Vickers Hardness, Nanoindentation Test)
- 6.3 High Temperature Properties (Creep Resistance, Thermal Fatigue and Phase Transformation Behavior)

#### Chapter 7 Thermal and Electrical Properties

- 7.1 Thermal Conductivity and Thermal Expansion Coefficient (Temperature Dependence and Testing)
- 7.2 Electrical Conductivity and Electron Emission Properties (Field Emission, Thermionic Emission)
- 7.3 Electromagnetic Shielding and Absorption Properties (GHz Band Performance)

#### Chapter 8 Chemical and Optical Properties

- 8.1 Oxidation Kinetics (High-Temperature Oxidation Model and Protection Strategy)
- 8.2 Corrosion Resistance (Degradation Mechanism in Acid and Alkaline Media)
- 8.3 Optical Properties (Band Gap, Light Absorption and Electrochromic Properties)

### Part 4 Characterization and Analysis Technology of Tungsten Powder

#### Chapter 9 Microstructure and Morphology Analysis

- 9.1 Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM)
- 9.2 X-ray Diffraction (XRD) and Electron Backscatter Diffraction (EBSD)
- 9.3 Specific Surface Area and Porosity Analysis (BET, Mercury Intrusion Porosimetry)

#### Chapter 10 Composition and Surface Analysis

- 10.1 X-ray Photoelectron Spectroscopy (XPS) and Auger Electron Spectroscopy (AES)
- 10.2 Inductively Coupled Plasma (ICP-MS) and Energy Dispersive Spectroscopy (EDS)
- 10.3 Surface Chemical State and Contamination Detection (TOF-SIMS)

#### Chapter 11 Tungsten Powder Performance Testing Technology

- 11.1 Mechanical Performance Testing (Tensile, Compression, Three-Point Bending)
- 11.2 Thermal Analysis Technology (DSC, TGA, Thermal Dilatometer)
- 11.3 Electrical and Electromagnetic Testing (Four-Probe Method, Vector Network Analyzer)

### Part 5 Industrial and Emerging Applications of Tungsten Powder

#### Chapter 12 Cemented Carbide and Cutting Tools

- 12.1 Synthesis and Sintering Process of Tungsten Carbide (WC)
- 12.2 Performance Optimization of Cemented Carbide Tools (Turning Tools, Milling Cutters)
- 12.3 Superhard Coatings (CVD-WC, Diamond Coatings)

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### **Chapter 13 Electronics and Energy Applications**

- 13.1 Cathode Materials (Thermionic and Field Emission Cathodes)
- 13.2 Semiconductor Packaging and Heat Sinks (W-Cu, W-Ag)
- 13.3 Energy Storage (Battery Electrodes, Supercapacitors)
- 13.4 Electrochromic and Thermal Shielding (Smart Windows, Energy-Saving Coatings)

### **Chapter 14 Military and Nuclear Industry Applications**

- 14.1 Armor-piercing projectiles and armor materials (W-Ni-Fe alloy)
- 14.2 Nuclear fusion first wall materials (anti-sputtering performance)
- 14.3 Radiation shielding ( $\gamma$ -ray and neutron absorption)

### **Chapter 15 Additive Manufacturing and Aerospace**

- 15.1 Morphological Characteristics of Spherical Tungsten Powder for 3D Printing
- 15.2 Characteristics, process flow, advantages and suitable applications of Selective Laser Melting (SLM) and Electron Beam Melting (EBM)
- 15.3 Aerospace high temperature components (turbine blades, combustion chambers)
- 15.4 3D printing conductive paste and composite materials
- 15.5 Application Status and Future Prospects of Spherical Tungsten Powder and 3D Alloy Products

### **Chapter 16 Other Emerging Applications**

- 16.1 Medical Devices (Radiotherapy Collimators, Surgical Tools)
- 16.2 Pigments and Coatings (Fire Protection, Shielding, Artistic Pigments)
- 16.3 Gas Sensors and Photocatalysis (Environmental and Chemical Applications)
- 16.4 Sports, leisure, culture and art industries

### **Part 6 Frontier Research and Future Trends**

#### **Chapter 17 Quantum and Smart Technology of Nano-Tungsten Powder**

- 17.1 Quantum Confinement Effect and Single Atomic Layer Tungsten
- 17.2 Application of Nano-Tungsten Powder in Quantum Dots and Optoelectronic Devices
- 17.3 Smart Materials (Shape Memory, Self-Healing Design)

#### **Chapter 18 Sustainable Development and Circular Economy**

- 18.1 History and Current Status of Tungsten Waste Recycling (Hydrometallurgical and Pyrometallurgical Technologies)
- 18.2 Hydrometallurgical Tungsten Waste Recovery Technology and Process
- 18.3 Pyrometallurgical Hydrometallurgical Tungsten Waste Recovery Technology and Process
- 18.4 Low - carbon production (hydrogen reduction and green chemistry)
- 18.5 Life cycle assessment (LCA) and carbon footprint
- 18.6 Historical Review and Future Development of Tungsten Powder Production Technology

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## Chapter 19 Global Market and Technology Trends of Tungsten Powder Industry

- 19.1 Supply and Demand Status and Price Trends (China, Europe, North America)
- 19.2 Major Manufacturers and Competition Landscape
- 19.3 Technological Innovation (Automation, Intelligent Production)

## Chapter 20 Future Prospects of Tungsten Powder Research

- 20.1 Interdisciplinary Integration and Application of Tungsten Powder (Materials, Energy, Quantum Technology)
- 20.2 Production Technology Challenges and Solutions of Tungsten Powder
- 20.3 Strategic Significance of Tungsten Powder in Sustainable Development
- 20.4 Non-technical and non-market factors affecting tungsten powder and their possible future trends

## Appendix

- Appendix International Standards and Specifications (ASTM, ISO, GB/T)
- Appendix: Quick Lookup Table of Physical Properties of Tungsten Powder (Density, Melting Point, Conductivity, etc.)
- Appendix: Tungsten powder related patent list (global patent number and abstract)
- Appendix: List of Tungsten Powder Production Equipment and Instruments
- Appendix Tungsten Powder References (Multi-language versions: Chinese, English, Japanese, German, Russian, Korean)
- Appendix Tungsten Powder Safety Handling Guide (MSDS and Protective Measures)
- Appendix: Chinese and English Glossary

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CTIA GROUP LTD  
Tungsten Powder Introduction

### 1. Tungsten Powder Overview

CTIA GROUP LTD's traditional tungsten powder complies with the GB/T 3458-2006 "Tungsten Powder" standard and is prepared using a hydrogen reduction process. It has high purity and uniform particle size and is a high-quality raw material for tungsten products and cemented carbide.

### 2. Tungsten Powder Characteristics

Ultra-high purity: tungsten content  $\geq 99.9\%$ , oxygen content  $\leq 0.20$  wt% (fine particles  $\leq 0.10$  wt%), and extremely low impurities.

Accurate particle size: Fisher particle size 0.4-20  $\mu\text{m}$ , 6 levels to choose from, with a deviation of only  $\pm 10\%$ .

Excellent performance: bulk density 6.0-10.0  $\text{g}/\text{cm}^3$ , uniform grains, excellent sinterability.

Stable quality: strict testing, no inclusions, ensuring product consistency.

### 3. Tungsten Powder Specifications

Brand	Fisher particle size ( $\mu\text{m}$ )
FW-1	0.4-1.0
FW-2	1.0-2.0
FW-3	2.0-4.0
FW-4	4.0-6.0
FW-5	6.0-10.0
FW-6	10.0-20.0

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

### 4. Packaging and Quality Assurance

Packaging: Inner sealed plastic bag, outer iron drum, net weight 25kg or 50kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition and particle size data, and the shelf life is 12 months.

### 5. Procurement Information

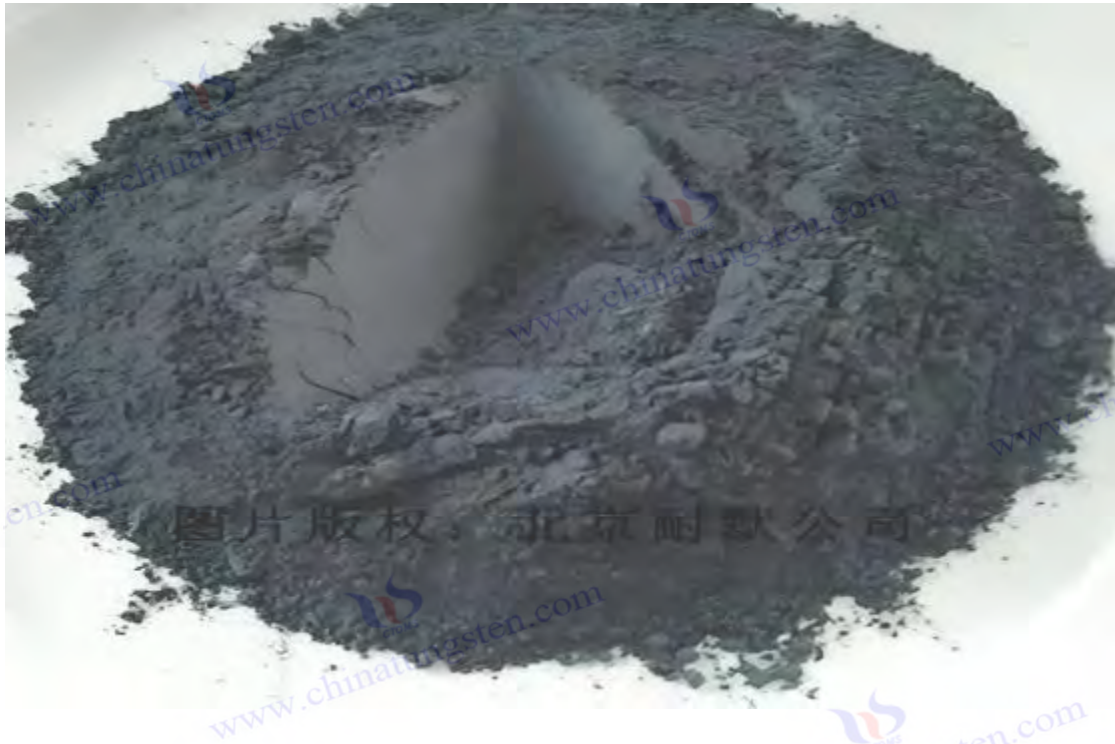
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For more information about tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## Preface

Tungsten, a transition metal known for its high melting point, high density and excellent corrosion resistance, has become an indispensable cornerstone of modern industry and cutting-edge technology since its discovery in the 18th century. From early incandescent filaments to today's carbide cutting tools, the first wall material of nuclear fusion devices, and nanoscale optoelectronic devices, the wide application of tungsten demonstrates its unique position in the field of materials science. As one of the main forms of tungsten, tungsten powder is not only the core raw material for the production of tungsten-based materials, but also a key carrier for promoting technological innovation. Especially in recent years, with the rise of nanotechnology, high-purity nano tungsten powder has rapidly expanded the application boundaries of tungsten with its excellent photocatalytic, electrochromic and electromagnetic shielding properties, bringing new possibilities to fields such as energy, environment and intelligent manufacturing.

The writing of this book stems from the need for a comprehensive understanding of tungsten powder, a multifunctional material. Driven by the acceleration of global industrialization and the goal of sustainable development, the research and application of tungsten powder are undergoing profound changes. From microscopic exploration in the laboratory to large-scale industrial production, each particle of tungsten powder carries the integration of scientific principles and engineering practices. However, existing literature often focuses on a specific field, lacks systematicity and comprehensiveness, and cannot meet the needs of scientific researchers, engineers and industry decision makers for comprehensive knowledge. Therefore, through this book, we hope to build a bridge connecting basic science, production technology and application practice, and provide practitioners in the field of tungsten powder with an authoritative, practical and forward-looking reference guide.

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The objectives of this book are multi-layered. First, we seek to reveal the scientific nature of tungsten powder, systematically analyzing its physical, chemical, and optical properties from its atomic structure and crystal properties to its surface effects at the nanoscale, laying a theoretical foundation for subsequent technology development. Second, we have combed through the preparation process of tungsten powder in detail, from the traditional hydrogen reduction method to modern advanced technologies such as plasma spheroidization and hydrothermal synthesis, covering the complete process of laboratory and industrial scale, and exploring the latest progress in surface modification and composite technology. In addition, this book comprehensively introduces the characterization methods and performance testing techniques of tungsten powder to ensure that readers can accurately evaluate its quality and function. Most importantly, we have deeply explored the application of tungsten powder in traditional fields such as cemented carbide, electronic devices, military nuclear industry, and additive manufacturing, while looking forward to its potential in cutting-edge fields such as quantum technology, smart materials, and sustainable development. Through global market analysis and technology trend forecasting, we hope that readers will not only understand the current status of technology, but also gain insight into future development directions.

The book is structured in six parts, twenty chapters, and eight appendices, striving to be comprehensive and organized. The first part, "Basic Science of Tungsten Powder", focuses on the essential properties of tungsten and its powder form; the second part, "Tungsten Powder Preparation Technology", details the manufacturing processes from traditional to advanced; the third part, "Tungsten Powder Performance and Characterization", analyzes its physical, chemical and functional properties; the fourth part, "Tungsten Powder Characterization and Analysis Technology", provides micro and macro testing methods; the fifth part, "Industrial and Emerging Applications of Tungsten Powder", covers traditional and new application scenarios; the sixth part, "Frontier Research and Future Trends", looks forward to the future role of tungsten powder in science and technology and society. The appendices provide practical resources such as standards, patents, and equipment lists to enhance the reference value of this book.

In the process of writing this book, we referred to a large number of authoritative documents, including the latest research results of top journals such as Nature and Acta Materialia, as well as international standards such as ASTM, ISO, GB/T, and integrated data and patent information of major global tungsten powder suppliers (such as American Elements and HC Starck). To ensure the practicality of the content, we visited many tungsten companies and invited experts in the fields of materials science, metallurgical engineering and nanotechnology to participate in the review. Special thanks to China Tungsten Online Tungsten Intelligent Manufacturing Technology Co., Ltd. for its support. Their industry insights and technical information have added a lot of color to this book.

The target readers of this book include but are not limited to the following groups: researchers in the field of materials science and engineering who are eager to deeply understand the microscopic mechanism of tungsten powder; engineers in the cemented carbide, electronics, military and aerospace industries who

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seek to optimize production processes and application solutions; teachers and students of materials majors in colleges and universities who need systematic textbooks and research references; and industry analysts and policymakers who are concerned about the global pattern and sustainable development strategy of the tungsten powder market. Whether you are a novice who is new to tungsten powder or an expert who has been engaged in it for many years, we believe that this book can provide you with inspiration and help.

The world of tungsten powder is both tiny and vast. From a nanoparticle to a huge system that supports industrial civilization, it has witnessed the progress of human technology and carries the hope for the future. We hope that this book is not only a compilation of knowledge, but also an invitation to explore the mysteries of tungsten powder and jointly promote its new chapter in science and industry. In this process, your feedback and participation are crucial. Please communicate with us through the contact information in the appendix of this book to share your insights and needs.

May this book be a good companion in your research and practice, and may the potential of tungsten powder shine in your hands!

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## Part 1 Basic Science of Tungsten Powder

Tungsten powder is a metal powder with tungsten (W, atomic number 74) as the main component. It has become a key material in the industrial and technological fields due to its high melting point (3422°C), high density (19.25 g/cm<sup>3</sup>) and excellent mechanical properties. This article starts with the physical, chemical and microstructural properties of tungsten powder and explains its basic scientific principles.

### 1. Physical properties of tungsten powder

Tungsten powder is gray to black, with a wide range of particle sizes (0.05-100 μm), including nano-, ultrafine and coarse-grained grades. Its high density makes it irreplaceable in heavy alloys and counterweights, while its melting point and thermal conductivity (173 W/m·K) make it suitable for high-temperature environments. Particle size distribution and morphology (spherical or irregular) directly affect fluidity (10-50 s/50g) and sintering properties. The main physical properties of tungsten powder are:

**Appearance of tungsten powder** : gray to black metal powder, odorless.

**Particle size of tungsten powder** : 0.05-100 μm (covering nanometer to coarse particle size).

**Density of tungsten powder** : 19.25 g/cm<sup>3</sup> ( lump tungsten, powder slightly lower depending on particle size).

**The melting point of tungsten powder** is 3422°C.

**The boiling point of tungsten powder** is 5555°C.

**Thermal conductivity of tungsten powder** : 173 W/m·K.

**Electrical conductivity of tungsten powder** :  $18.2 \times 10^6$  S/m.

**Specific surface area of tungsten powder** : 0.5-20 m<sup>2</sup> / g (increases as particle size decreases).

**The fluidity of tungsten powder** : 10-50 s/50g (depending on the morphology and particle size distribution).

**The crystal structure of tungsten powder** is body-centered cubic (BCC), with a lattice constant of about 3.165 Å .

**Hardness of tungsten powder** : 300-450 HV (measured after powder pressing).

**Tungsten powder bulk density** : 5-12 g/cm<sup>3</sup> ( depending on particle size and morphology).

**The tap density of tungsten powder** is 8-15 g/ cm<sup>3</sup> .

### 2. Chemical properties of tungsten powder

Tungsten has strong chemical stability and does not react with water or air at room temperature, but can be oxidized by oxygen at high temperature to form tungsten oxide (WO<sub>3</sub>). The purity of tungsten powder is usually between 99.9% and 99.999%, and the content of impurities (such as oxygen and carbon) must be controlled below 0.01%-0.05% to ensure performance. High-purity tungsten powder is particularly important in the fields of electronics and catalysis. The chemical properties of tungsten powder are mainly:

**Chemical stability of tungsten powder** : does not react with water, air or dilute acid at room temperature.

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**Oxidizing property of tungsten powder** : It reacts with oxygen at high temperature (>500°C) to generate  $WO_3$  .

**Purity of tungsten powder** : 99.9%-99.999% (industrial grade to high purity grade).

**Impurities in tungsten powder** : oxygen (<0.05%), carbon (<0.01%), iron and other trace elements.

**Corrosion resistance of tungsten powder** : resistant to acid and alkali corrosion (except concentrated nitric acid and hydrofluoric acid).

**Reactivity of tungsten powder** : reacts slowly with strong oxidants (such as nitric acid, perchloric acid).

**Combustibility of tungsten powder** : fine particles can form explosive dust when in high concentration in the air.

**Solubility of tungsten powder** : insoluble in water, slightly soluble in strong acid.

### 3. Microstructure of tungsten powder

The crystal structure of tungsten is body-centered cubic (BCC) with a lattice constant of about 3.165 Å . Nano-tungsten powder has a large specific surface area (up to 20 m<sup>2</sup> / g) and a small grain size (<100 nm), showing unique surface effects and quantum effects. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis show that its microstructure and crystal integrity are crucial for subsequent processing (such as sintering into cemented carbide).

### 4. Scientific significance

The properties of tungsten powder come from its electronic structure ( $5d^4 6s^2$  ). The high electron density gives it excellent conductivity ( $18.2 \times 10^6$  S/m) and corrosion resistance. Its thermodynamic stability allows it to maintain structural integrity under extreme conditions, laying the foundation for its fundamental position in cemented carbide, additive manufacturing and electronic targets.

This article lays the theoretical foundation for the in-depth study of tungsten powder. Subsequent chapters will explore its preparation process and application expansion.



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## Chapter 1 Physical and Chemical Properties of Tungsten (Physical and Chemical Properties)

### 1.1 Atomic Structure of Tungsten

#### Electronic Properties

Tungsten (element symbol W) is a transition metal in the sixth period of the periodic table, group VI B, with an atomic number of 74 and an average atomic mass of 183.84 u (based on natural abundance). Its nucleus consists of 74 protons and usually 110 neutrons (taking the most common isotope  $^{184}\text{W}$  as an example), surrounded by 74 electrons. The electron configuration of tungsten is  $[\text{Xe}] 4f^{14} 5d^4 6s^2$ , in which the 4f orbital is fully filled, and the partial filling of the 5d and 6s orbitals gives tungsten unique chemical and physical properties. The four electrons in the 5d orbital and the two electrons in the 6s orbital play a key role in bonding, making tungsten exhibit high conductivity, high hardness and excellent electron emission capability. This electronic structure also causes tungsten to have a high electronegativity (about 2.36, Pauling scale), which is between typical metals and non-metals, reflecting its transition metal characteristics.

**Atomic structure characteristics and influence table of tungsten**

Atomic structure characteristics	Describe	Impact on properties
Electronic configuration	$[\text{Xe}] 4f^{14} 5d^4 6s^2$ , 5d and 6s outer electrons, 4f fully filled shielding the inner electrons.	<ul style="list-style-type: none"> <li>- High melting point (3422°C): 5d and 6s electrons form strong metallic bonds with binding energies as high as 850 kJ/mol.</li> <li>- Electrical conductivity (<math>18.2 \times 10^6</math> S/m): Partially populated 5d electrons provide high mobility.</li> </ul>
Atomic radius	About 139 pm, medium size, high electron density.	- High density ( $19.25 \text{ g/cm}^3$ ) : Atomic mass is concentrated and the electron cloud is dense.
Electronegativity	1.7 (Pauling scale), moderate chemical activity.	- Chemical stability: The outer electron distribution is stable, the reactivity is low, and it is corrosion-resistant at room temperature.
Crystal structure	Body-centered cubic (BCC), lattice constant about 3.165 Å, coordination number 8.	<ul style="list-style-type: none"> <li>- High hardness (300-450 HV): BCC close packing enhances the interatomic forces.</li> <li>- Thermal stability: The structure is not easily deformed at high temperatures.</li> </ul> <p style="text-align: right;">MADE BY: CTIA GROUP LTD</p>
Strong metal bond	5d and 6s electrons participate, and the binding energy is high.	- High melting point and thermal conductivity (173 W/m·K): Strong bonds support high temperature applications such as sintering and thermal spraying.
Quantum Effects	Surface activity at the	- Improved catalytic performance: Nano-tungsten

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Atomic structure characteristics	Describe	Impact on properties
(Nanoscale)	nanoscale is amplified due to the increase in specific surface area.	powder (<100 nm) has enhanced reactivity and selectivity in catalyst supports.

## 1.2 Electronic Properties of Tungsten

The electronic properties of tungsten atoms have a profound impact on their applications. For example, the work function of tungsten is about 4.52-4.55 eV, which is lower than many noble metals (such as gold 5.1 eV), but higher than iron (Iron, 4.5 eV), which makes it exhibit excellent thermal electron emission performance at high temperature. This is why tungsten is widely used in incandescent filaments, X-ray tube cathodes, and electron microscope sources. The size of the work function is closely related to the surface state. Studies have shown that trace oxides (Oxides, such as  $WO_3$ ) or doping (Doping, such as  $ThO_2$ ) on the surface of pure tungsten can significantly reduce the work function to 2.6-3.0 eV, thereby improving the emission efficiency. In addition, tungsten has a higher electron cloud density, which is related to its smaller atomic radius (about 139 pm, compared to 126 pm for iron). The high electron density enhances the strength of the metallic bonds between atoms, making tungsten have extremely high hardness (Mohs hardness of about 7.5) and tensile strength (about 1510 MPa) at room temperature.

### 1. Electronic properties of tungsten

#### Electronic configuration:

$[Xe] 4f^{14} 5d^4 6s^2$ .

The outer shell includes  $5d^4$  (partially filled d orbitals) and  $6s^2$  (fully filled s orbitals), with 4f electrons fully filling the molecule, providing a shielding effect.

#### Electron Mobility:

The 5d electrons are partially filled, forming a high-density free electron cloud with high mobility.

#### Band structure:

The Fermi level of tungsten is located in the d band, and the band overlap results in no obvious band gap between the conduction band and the valence band, showing typical metallic properties.

#### Electronegativity:

1.7 (Pauling scale), which is moderately low, reflecting its low electron affinity.

### 2. Impact of electronic properties on performance

#### (1) High conductivity

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Electronic properties: The unfilled states and high mobility of 5d<sup>4</sup> electrons provide a large number of free electrons .

Impact: Tungsten has an electrical conductivity of  $18.2 \times 10^6$  S/m, making it an excellent conductive material and widely used in electronic targets, tungsten wires, and semiconductor components.

### (2) Excellent thermal conductivity

Electronic properties: The high density and high mobility of free electrons promote thermal energy transfer, and the interaction between electrons and the lattice enhances heat conduction.

Impact: Tungsten has a thermal conductivity of 173 W/m·K, making it suitable for high-temperature thermal conductivity applications such as thermal spraying and high-temperature alloys.

### (3) High melting point and thermal stability

Electronic properties: 5d and 6s electrons form strong metallic bonds with a binding energy of up to 850 kJ/mol, and the high density of the electron cloud enhances the attraction between atoms.

Impact: Tungsten has a melting point of 3422°C, which ensures that it maintains structural integrity under extremely high temperatures (such as additive manufacturing and aerospace components).

### (4) Chemical stability

Electronic properties: The shielding effect of 4f electrons protects the outer electrons, and the distribution of 5d and 6s electrons is stable, reducing the tendency to react with external atoms.

Impact: Tungsten is corrosion-resistant at room temperature and reacts slowly with oxygen only at high temperatures, making it suitable for harsh chemical environments (such as catalyst carriers).

### (5) Electronic effects at the nanoscale

Electronic properties: At the nanoscale (e.g. <100 nm), surface electronic activity is enhanced due to the quantum confinement effect, and the specific surface area increases.

Impact: Nano-tungsten powder exhibits higher electrocatalytic activity and is suitable for battery electrodes and catalysts.

**Electronic properties of tungsten and their impact on performance**

Electronic properties	describe	Impact on performance
Electronic configuration	[Xe] 4f <sup>14</sup> 5d <sup>4</sup> 6s <sup>2</sup> , 5d <sup>4</sup> partially fills, 6s <sup>2</sup> fully fills, 4f provides a shielding effect.	- High melting point (3422°C): 5d and 6s electrons form a strong metallic bond with a binding energy of 850 kJ/mol. - Chemical stability: 4f shielding reduces reactivity and resists corrosion at room temperature.
Electron mobility	The 5d <sup>4</sup> electrons are not filled, forming a high-density free electron cloud with high mobility.	- High electrical conductivity ( $18.2 \times 10^6$ S/m): Free electrons support electrical conductivity, suitable for electron targets and tungsten filaments. - Thermal conductivity (173 W/m·K): Electrons transfer thermal energy, suitable for high-temperature heat conduction.

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Electronic properties	describe	Impact on performance
Band structure	The Fermi level is located in the d band, the conduction band and valence band overlap, there is no obvious band gap, and it is a typical metallic characteristic.	- Excellent conductivity: No forbidden band ensures free flow of electrons, suitable for semiconductor components and electrode materials.
Electronegativity	1.7 (Pauling scale), medium to low, low electron affinity.	- Chemical inertness: It only reacts slowly with strong oxidants at room temperature and is suitable for harsh chemical environments (such as catalyst supports).
Electronic effects at the nanoscale	At the nanoscale (<100 nm), surface electronic activity is enhanced due to the quantum confinement effect, and the specific surface area increases.	- Improved electrocatalytic activity: Nano-tungsten powder exhibits high reactivity in the field of battery electrodes and catalysts. MADE BY: CTIA GROUP LTD

### 1.3 Isotope Distribution of Tungsten

The isotope distribution of tungsten is also worth noting. There are five stable isotopes of tungsten in nature:  $^{180}\text{W}$  (0.12%),  $^{182}\text{W}$  (26.50%),  $^{183}\text{W}$  (14.31%),  $^{184}\text{W}$  (30.64%) and  $^{186}\text{W}$  (28.43%), as well as a trace amount of radioactive isotope  $^{187}\text{W}$  (half-life of about 23.72 hours). The highest abundance of  $^{184}\text{W}$  makes it the main form of tungsten, and the diversity of isotopes is of great significance to nuclear industry applications (such as radiation shielding). For example,  $^{186}\text{W}$  has a high neutron capture cross section (about 38.1 barn), making it an excellent candidate for use as a shielding material in nuclear reactors. Separation techniques for tungsten isotopes (such as centrifugation) are not widely used in industry, but have shown potential in research to further optimize tungsten's nuclear properties.

From the perspective of quantum mechanics, the 5d electrons of tungsten contribute significantly to the valence band, and the splitting of its d orbitals (such as  $t_{2g}$  and  $e_g$  energy levels) affects the electronic structure of tungsten compounds. For example, in the monoclinic structure of  $\text{WO}_3$ , tungsten atoms are in octahedral coordination, and the localization of 5d electrons leads to a band gap of about 2.4-2.8 eV, showing n-type semiconductor characteristics. This electronic property lays the foundation for the application of tungsten powder in the fields of photocatalysis, electrochromism and gas sensing. In addition, the high ionization energy (Ionization Energy, first ionization energy 7.98 eV) of tungsten indicates that it is not easy to lose electrons, further enhancing its chemical stability.

#### Isotopic distribution of tungsten

The natural isotope distribution of tungsten is as follows (based on IAEA data)

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Isotope	Abundance(%)	Mass (u)	Describe
Tungsten-180 ( $^{180}\text{W}$ )	0.12	179.9467	The lightest isotope, with the lowest abundance
Tungsten -182 ( $^{182}\text{W}$ )	26.50	181.9482	Moderate abundance, light quality
Tungsten -183 ( $^{183}\text{W}$ )	14.31	182.9502	Moderate abundance
Tungsten-184 ( $^{184}\text{W}$ )	30.64	183.9509	Most abundant isotope, close to average mass
Tungsten-186 ( $^{186}\text{W}$ )	28.43	185.9544	Heavier isotopes, more abundant
Average atomic weight	-	183.84	Weighted mean of natural abundance
Illustrate	These isotopes are all stable isotopes, non-radioactive, and the mass difference is mainly due to the change in the number of neutrons (from 106 to 112). Table: CTIA GROUP LTD		

## Effect of Tungsten Isotope Distribution on Performance

### (1) Density and quality related properties

Mechanism of influence: The difference in isotopic mass (179.9467 u to 185.9544 u) causes a slight change in the atomic mass, affecting the average density of tungsten.

Performance impact:

The density of natural tungsten is 19.25 g/cm<sup>3</sup>. If it is enriched with a certain isotope (such as  $^{186}\text{W}$ ), the density will increase slightly (theoretically about 0.1%-0.3%).

In high-precision counterweights (such as aviation gyroscopes), density differences may affect the uniformity of mass distribution, but are usually negligible.

Application significance: General industrial uses (such as cemented carbide) are minimally affected, but it needs to be considered in the ultra-precision field.

### (2) Thermal properties

Mechanism of influence: Heavier isotopes ( $^{186}\text{W}$ ) have an increased mass and a lower lattice vibration frequency, which may slightly affect heat capacity and thermal conductivity.

Performance impact:

The thermal conductivity (173 W/m·K) theoretically decreases slightly (<1%) with increasing proportion of heavy isotopes due to enhanced phonon scattering.

The melting point (3422°C) is largely unaffected, as it is determined primarily by electronic bonding.

Application significance: The impact can be ignored in high-temperature heat-conducting components (such as thermal spraying), but it needs to be evaluated under extreme conditions.

### (3) Mechanical properties

Mechanism of influence: Isotope mass differences have little effect on the lattice constant (3.165 Å), but may change the local stress distribution.

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Performance impact:

Hardness (300-450 HV) and compressive strength (>6000 MPa) are almost unchanged, as the macroscopic mechanical properties are dominated by the crystal structure and bond strength.

In nanoscale tungsten powder, heavier isotopes may slightly increase grain boundary stability.

Application significance: No significant impact on cemented carbide and additive manufacturing.

#### (4) Isotope separation and special applications

Mechanism of influence: Separation of isotopes by centrifugation or diffusion can change the distribution ratio (e.g. enrichment  $^{184}\text{W}$  or  $^{186}\text{W}$ ).

Performance impact:

Enrichment with light isotopes (e.g.,  $^{180}\text{W}$ ) improves thermal and electrical conductivity (by <2%) due to reduced electron-phonon scattering.

with heavy isotopes (such as  $^{186}\text{W}$ ) increases density and nuclear shielding capabilities.

Application significance: In the nuclear industry (e.g. radiation shielding) or in high-precision electronic targets, tailored isotope distribution can optimize performance.

#### (5) Chemical stability

Influence mechanism: Isotope mass has little effect on the chemical reaction rate (isotope effect), because the chemical properties of tungsten are mainly determined by the electron layer.

There is no significant change in oxidation resistance ( $\text{WO}_3$  is generated at high temperature) and corrosion resistance.

Application significance: In chemical applications such as catalyst supports, the effect of isotope distribution can be ignored.

**Effect of isotope distribution on performance**

Performance Category	Impact Mechanism	Specific impact	Application significance
Density and quality related properties	Isotope mass differences (179.9467-185.9544 u) affect average density	- Natural density is 19.25 g/cm <sup>3</sup> , and the density increases slightly after enrichment with W (about 0.1 %-0.3%). - The uniformity of mass distribution changes slightly.	- Low impact for industrial use, ultra-precision counterweights need to be considered.
Thermal properties	Heavy isotopes (such as $^{186}\text{W}$ ) reduce the frequency of lattice vibrations	- Thermal conductivity (173 W/m·K) decreased slightly (<1%) due to enhanced phonon scattering. - Melting point (3422°C) remained essentially unchanged.	- The impact of high temperature thermal conductivity applications can be ignored, and extreme conditions need to be evaluated.
Mechanical properties	The mass difference has little	- No significant changes in hardness (300-450 HV)	- The impact of cemented carbide and additive

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Performance Category	Impact Mechanism	Specific impact	Application significance
	effect on the lattice constant (3.165 Å)	compressive strength (>6000 MPa). - Slight increase in nanoscale grain boundary stability.	manufacturing is not significant.
Isotope separation and special applications	Adjust the distribution ratio (e.g. enrich <sup>180</sup> W or <sup>186</sup> W)	- Enriched <sup>180</sup> W improves thermal and electrical conductivity (<2%) . - Enriched <sup>186</sup> W density enhancement and nuclear shielding capabilities.	- Nuclear industry (shielding), electronic targets to optimize performance.
Chemical stability	Isotope mass has little effect on reaction rate (isotope effect)	- No change in oxidation resistance (WO <sub>3</sub> is generated at high temperature ) and corrosion resistance. MADE BY: CTIA GROUP LTD	- The influence of chemical applications such as catalyst supports is negligible.

The isotopic distribution of tungsten has little effect on its performance overall, mainly reflected in the fine-tuning of physical properties such as density and thermal conductivity. Tungsten powder at natural abundance (<sup>184</sup>W is the most abundant) has met most industrial needs, such as cemented carbide and electronic materials. However, in special fields (such as nuclear technology or ultra-precision manufacturing), performance can be optimized by adjusting the distribution through isotope separation. For example, enriching <sup>186</sup>W can improve radiation shielding effects, while light isotopes (such as <sup>180</sup>W) can slightly increase conductivity. These fine-tuning needs to weigh the cost and practical benefits.

#### 1.4 Crystal Structure of Tungsten

Tungsten exists in a body-centered cubic (BCC) crystal structure at room temperature, with a space group of Im3m and a lattice constant of  $a = 3.165 \text{ \AA}$  (at room temperature). Each unit cell of the BCC structure contains 2 tungsten atoms, a coordination number of 8, and an atomic packing factor of 68%, which is lower than that of face-centered cubic (FCC, 74%) and hexagonal close-packed (HCP, 74%). This structure gives tungsten an extremely high density (Density,  $19.25 \text{ g/cm}^3$ ), making it one of the heaviest common metals, second only to gold ( $19.32 \text{ g/cm}^3$ ). The stability of the BCC structure comes from the strong metallic bonds of tungsten atoms, with a bond energy of about 850 kJ/mol, much higher than that of iron (about 410 kJ/mol). However, the low symmetry of the BCC structure leads to poor ductility of tungsten, which is prone to brittle fracture at room temperature, and the ductile-brittle transition temperature (DBTT) is about 200-400°C, depending on the purity and processing condition.

Under high temperature conditions (>2500°C), tungsten crystals may undergo phase transition. Studies have shown that under high pressure (high pressure, such as above 10 GPa) or doping (such as adding

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Re, Mo), tungsten may transform into a hexagonal close-packed (HCP) structure with lattice constants  $a \approx 2.76 \text{ \AA}$  and  $c \approx 4.48 \text{ \AA}$ . The emergence of this metastable phase is related to the rearrangement of d electrons, but it is rare in industry and is mainly observed in theoretical studies and extreme environments (such as nuclear fusion devices). In addition, tungsten may briefly form an amorphous state at extremely high temperatures (close to the melting point), followed by rapid recrystallization into a BCC structure, a behavior consistent with its high melting point and high bond energy.

Defects in tungsten crystals have a significant impact on its properties. Dislocations are the main type of defects, including edge dislocations and screw dislocations. In cold-worked tungsten, the dislocation density can reach  $10^{10} - 10^{12} \text{ cm}^{-2}$ , resulting in increased hardness but decreased toughness. High-temperature annealing (1000-1500°C) can reduce the dislocation density to  $10^8 \text{ cm}^{-2}$  and improve ductility. Grain boundaries are another key defect. Small grain tungsten (grain size  $< 10 \mu\text{m}$ ) has high strength due to its large grain boundary area, but is prone to fracture along the grain boundary; large grain tungsten ( $> 100 \mu\text{m}$ ) is more ductile. Doping elements (such as 0.5-1 wt%  $\text{La}_2\text{O}_3$  or K) significantly improve the brittleness resistance of tungsten by pinning dislocations and stabilizing grain boundaries. For example, potassium-doped tungsten wire can inhibit grain growth and extend service life in filament applications.

The crystal structure of nano-sized tungsten powder is more complex. Taking  $\text{WO}_3$  as an example, its common form is monoclinic, space group  $P2_1/n$ , lattice constants  $a = 7.306 \text{ \AA}$ ,  $b = 7.540 \text{ \AA}$ ,  $c = 7.692 \text{ \AA}$ ,  $\beta = 90.91^\circ$ . The tungsten atoms in this structure are in  $\text{WO}_6$  octahedral coordination, and the asymmetric arrangement of oxygen atoms leads to the formation of a band gap, making  $\text{WO}_3$  yellow and photocatalytically active. In contrast, metallic tungsten powder maintains a BCC structure, but surface effects may induce local disorder, especially when the particle size is  $< 20 \text{ nm}$ , the proportion of surface atoms ( $> 30\%$ ) significantly affects the crystal stability. In addition, suboxides such as  $\text{WO}_{2.9}$  ( $\text{W}_{18}\text{O}_{49}$ ) often present a needle-like monoclinic structure (Needle-like Monoclinic,  $P2_1/m$ ) and exhibit unique infrared absorption characteristics.

### Crystal structure of tungsten

Characteristic	Describe
Type	Body-centered cubic (bcc)
Lattice constant	About $3.165 \text{ \AA}$ (at room temperature)
Coordination number	8 (each atom has 8 nearest neighbors)
Bulk density	Theoretical stacking efficiency is 68%, which is relatively compact but lower than fcc
Stability	Maintain bcc from room temperature to melting point ( $3422^\circ\text{C}$ ), no phase change MADE BY:: ctia group ltd

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### Effect of crystal structure on performance

Performance Category	Impact Mechanism	Specific impact	Application significance
High melting point and thermal stability	BCC close packing and strong metallic bonds (binding energy 850 kJ/mol) require high energy to break	- Melting point is 3422°C, the highest among metals. - The structure is stable at high temperature and there is no crystal transformation.	- Suitable for high temperature alloys, additive manufacturing, thermal spraying.
High hardness and mechanical strength	BCC has high coordination number, strong interatomic force and strong deformation resistance	- Hardness 300-450 HV, compressive strength >6000 MPa. - Excellent wear resistance and toughness.	- For cutting tools, wear-resistant coatings, cemented carbides.
Thermal conductivity and electrical conductivity	BCC electron cloud is uniform, 5d and 6s electrons move freely, but phonon scattering is high	- Thermal conductivity is 173 W/m·K, electrical conductivity is $18.2 \times 10^6$ S/m, slightly inferior to FCC metals. - Electrical conductivity is affected by grain boundaries.	- Applicable to electronic targets and tungsten wires, defects need to be optimized.
density	BCC is not the most densely packed, but the high atomic mass and lattice compactness work together	- Density 19.25 g/cm <sup>3</sup> , second only to precious metals.	- Used for counterweight, radiation shielding.
Chemical stability and processability	BCC surface atoms are loosely arranged and have low reactivity; plastic deformation is difficult	- Anti-corrosion at room temperature, slowly oxidized to WO <sub>3</sub> at high temperature. - Low ductility, requires powder metallurgy processing.	- Suitable for harsh environments, processing relies on special technology.
Nanoscale crystal effects	At the nanoscale, the grain size is reduced, the grain boundary ratio is increased, and the surface effect is enhanced.	- Specific surface area of 20 m <sup>2</sup> / g, improving catalytic activity. - Hardness and strength increase with grain refinement (Hall-Page effect).	- Nano tungsten powder is used as catalyst carrier and battery electrode.

### 1.5 Thermodynamic Properties of Tungsten

The thermodynamic properties of tungsten are its core advantage in high-temperature applications. The melting point of tungsten is 3422°C (3695 K), the highest among all pure metals, second only to the sublimation point of carbon (about 3900°C). This property stems from its strong metallic bond and high electron cloud density, with a bond dissociation energy of up to 8.82 eV/atom. The boiling point of

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tungsten is 5555°C (5828 K), and the vapor pressure is extremely low, for example, only  $10^{-4}$  Pa at 2000°C and about 0.1 Pa at 3000°C. This low vapor pressure gives tungsten excellent stability in a vacuum high-temperature environment (such as an electron beam evaporator), avoiding material loss.

The linear thermal expansion coefficient of tungsten is  $4.5 \times 10^{-6} \text{ K}^{-1}$  (20-1000°C), which is much lower than copper ( $16.5 \times 10^{-6} \text{ K}^{-1}$ ) and aluminum ( $23.1 \times 10^{-6} \text{ K}^{-1}$ ). The low thermal expansion coefficient ensures the dimensional stability of tungsten under temperature changes. For example, in aerospace turbine blades, tungsten-based composites can withstand thermal shocks above 1000°C without deformation. The specific heat capacity is 0.13 J/(g·K) (25°C), which increases slightly with increasing temperature, reaching about 0.15 J/(g·K) at 1000°C. This lower specific heat capacity means that tungsten requires less energy to heat up, but it can still store enough heat at high temperatures to be suitable for heat sink applications.

The thermal conductivity of tungsten is 173 W/(m·K) (room temperature), which is medium to high among metals, higher than iron (80 W/(m·K)) but lower than copper (401 W/(m·K)). The thermal conductivity decreases with increasing temperature, reaching about 130 W/(m·K) at 1000°C and dropping to 100 W/(m·K) at 2000°C. This temperature dependence is related to the enhancement of electron scattering, but it is still sufficient to support thermal conduction requirements at high temperatures. For example, in the W-Cu heat sink material, tungsten provides structural support while copper enhances thermal conductivity. The thermal diffusivity of tungsten is about  $6.8 \times 10^{-5} \text{ m}^2/\text{s}$  (room temperature), reflecting its moderate heat transfer rate.

The enthalpy of fusion of tungsten is 52.31 kJ/mol, and the enthalpy of vaporization is as high as 774 kJ/mol, indicating that its phase transition requires a lot of energy. This high enthalpy value limits the melting processing of tungsten, and powder metallurgy technology is mostly used in industry. For example, tungsten powder can be sintered at 1400-1600°C to form a dense body, avoiding the high energy consumption of direct melting. In addition, the thermodynamic stability of tungsten is particularly prominent in an inert or reducing atmosphere (such as Ar, H<sub>2</sub>), but it is easily oxidized in an oxygen-containing environment (see 1.4 for details), so special attention should be paid.

#### Thermodynamic properties of tungsten

Thermodynamic properties	Value/Description	Remark
Melting point	3422°C (3695 K)	Highest among all metals
Boiling Point	5555°C (5828 K)	Very low volatility
Specific heat	0.132 J/(g·K) (25°C)	Less energy required to heat up
Thermal conductivity	173 W/(m·K) (room temperature)	High thermal conductivity
Coefficient of thermal expansion	$4.5 \times 10^{-6} /\text{K}$ (20-1000°C)	Minimal thermal expansion

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Thermodynamic properties	Value/Description	Remark
Vapor Pressure	$1.0 \times 10^{-7}$ Pa (about 3000°C)	Less volatility at high temperatures
Melting enthalpy	52.31 kJ/mol	High energy required for melting
Standard molar entropy	32.6 J/(mol·K) (298 K) Tungsten Intelligent Manufacturing	Highly ordered crystal structure

### Effect of Thermodynamic Properties on Performance

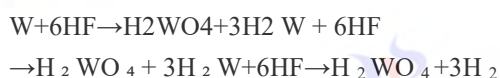
Performance Category	Thermodynamics Basics	Specific impact	Application significance
High temperature stability	High melting point (3422°C) and low vapor pressure ( $10^{-7}$ Pa at 3000°C)	- It can be used for a long time at a temperature close to the melting point (e.g. 3000°C) with minimal volatilization loss.	- Used in high temperature furnace components, rocket nozzles, thermal spraying.
Thermal conductivity	Thermal conductivity 173 W/(m·K), electrons and lattices cooperate to conduct heat	- Rapid heat dissipation, reduced local overheating, stable performance.	- Suitable for electronic targets, tungsten wires, and high-temperature heat dissipation components.
Low thermal expansion	Thermal expansion coefficient $4.5 \times 10^{-6}$ /K, at high temperature, BCC structure limits displacement	- Small dimensional change avoiding thermal stress damage.	- Used in precision parts, cemented carbide, aerospace.
Energy absorption and storage	Low specific heat capacity (0.132 J/(g·K)), high melting enthalpy (52.31 kJ/mol)	- It heats up quickly, requires high energy to melt, and has strong heat resistance.	- Suitable for instantaneous high temperature (such as laser sintering).
Chemical reactivity (oxidation)	High temperature enthalpy change drives oxidation, low vapor pressure slows down the reaction	- >500°C slowly oxidizes to $WO_3$ , stable below this temperature.	- Performs best in vacuum/inert atmosphere (like tungsten bulbs).
Nanoscale thermodynamic effects	Nano-scale has a large specific surface area (20 m <sup>2</sup> / g), which increases the surface energy	- Local melting point reduction (50-100°C) and increased thermal reactivity.	- Used for catalysts and heat-sensitive materials. Tungsten Intelligent Manufacturing in MADE BY:

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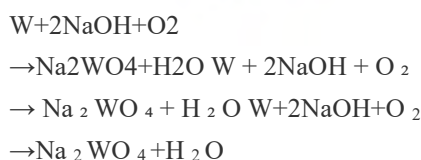


## 1.6 Chemical Stability of Tungsten

Tungsten has extremely high chemical stability at room temperature and can resist corrosion from most acids and bases. At room temperature, tungsten has almost no reaction to hydrochloric acid (HCl), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and phosphoric acid (H<sub>3</sub>PO<sub>4</sub>). Even after being immersed in concentrated solutions for several months, the corrosion rate is less than 0.01 mm/year. This is attributed to the thin layer of WO<sub>3</sub> (2-5 nm thick) spontaneously formed on the surface of tungsten, which has a dense structure and blocks further chemical reactions. However, in strong oxidizing acids, the stability of tungsten decreases. For example, in concentrated nitric acid (HNO<sub>3</sub>, 68%), tungsten slowly dissolves to form yellow WO<sub>3</sub>, with a reaction rate of about 0.1-0.5 mm/year (25°C). In hydrofluoric acid (HF), the corrosion rate of tungsten increases significantly (>1 mm/year), generating soluble H<sub>2</sub>WO<sub>4</sub> or WO<sub>2</sub>F<sub>2</sub>, and the reaction is as follows:

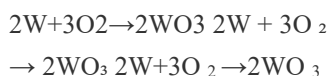


In alkaline medium, tungsten is stable to cold dilute NaOH or KOH solution, but in high temperature concentrated alkali (such as molten NaOH, >300°C), tungsten is corroded to form tungstate:

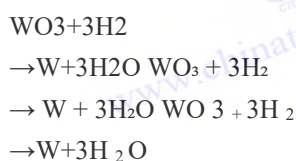


This reaction is often used in the industrial purification of tungsten ores (such as scheelite), but it should be avoided for tungsten products.

The oxidation behavior of tungsten is the main limiting factor of its chemical stability. In air, tungsten oxidizes very slowly below 400°C, forming only a thin layer of WO<sub>3</sub> on the surface, and the thickness growth follows the logarithmic law (logarithmic law,  $d \propto \ln(t)$ ). In the range of 500-800°C, the oxidation rate accelerates, and the WO<sub>3</sub> film thickness increases to micrometer level, following the parabolic law (parabolic law,  $d^2 \propto t$ ), the reaction is:



Above 1000°C, WO<sub>3</sub> begins to volatilize (melting point 1473°C, significant vapor pressure), causing tungsten to lose weight rapidly. For example, at 1200°C in air, the oxidation weight loss rate of tungsten can reach 10 mg/cm<sup>2</sup>·h. In order to slow down oxidation, protective coatings (such as SiC, Al<sub>2</sub>O<sub>3</sub>) or inert atmospheres (such as Ar, N<sub>2</sub>) are often used in industry. In H<sub>2</sub> atmosphere, tungsten is not only stable, but also can be prepared by reducing WO<sub>3</sub> to prepare tungsten powder, the reaction is:



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Tungsten has low reactivity with non-metals. At room temperature, tungsten has no obvious reaction to nitrogen (Nitrogen, N<sub>2</sub>), sulfur (Sulfur, S) or chlorine (Chlorine, Cl<sub>2</sub>). However, at high temperatures (>1000°C), tungsten can react with carbon to form tungsten carbide (Tungsten Carbide, WC, melting point 2870°C, hardness>9 Mohs), and with nitrogen to form tungsten nitride (Tungsten Nitride, WN, decomposition temperature ~600°C). These compounds are widely used in hard alloys and wear-resistant coatings. Tungsten has no direct chemical reaction with hydrogen (H<sub>2</sub>), but in a H<sub>2</sub> atmosphere at 400-700°C, WO<sub>3</sub> is reduced to metallic tungsten to produce tungsten powder with controllable particle size (10 nm-100 μm), which is the mainstream method for industrial production.

The chemical stability of tungsten is also affected by trace impurities. High-purity tungsten (>99.99%) is more resistant to corrosion than tungsten containing impurities such as Fe and Mo. For example, when the Fe content is >50 ppm, the corrosion rate of tungsten in acidic media increases by 20-30%. Therefore, the preparation of high-purity tungsten powder requires strict control of raw materials and processes (such as ion exchange purification, multiple rinsing, Rinsing).

#### Chemical Stability of Tungsten

Characteristic	Describe	Chemical basis
Room temperature stability	At room temperature (25°C), it does not react with water, oxygen, dilute acids (HCl, H <sub>2</sub> SO <sub>4</sub> ) or alkalis (NaOH), and has a thin oxide layer (WO <sub>3</sub> ) on the surface.	4f electron shielding effect, strong metal bonds reduce reactivity.
High temperature stability	<500°C oxidation is very slow in air, and it has high corrosion resistance to non-oxidizing acids and organic solvents.	The electronegativity is 1.7 and the chemical affinity is medium to low.

#### Effect of chemical stability on performance

Performance impact	Specific impact	Application significance
Corrosion resistance	It can resist acid and alkali corrosion at room temperature to medium temperature, and prolong the service life.	Chemical reactor components, tungsten bulbs (vacuum/inert).
High temperature application stability	<500°C The oxide layer grows slowly and has a complete structure.	High temperature furnace components, aerospace materials.
Convenient processing and storage	No special protection is required at room temperature, simplifying storage and transportation.	Powder metallurgy processes benefit from chemical inertness.

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### Limiting factors affecting chemical stability

Limiting Factors	mechanism	Specific impact	Countermeasures
High temperature oxidation	>500°C reacts with O <sub>2</sub> to form WO <sub>3</sub> ( $W + 3/2 O_2 \rightarrow WO_3$ ), and WO <sub>3</sub> volatilizes at high temperatures .	Surface oxidation intensifies, mechanical properties deteriorate, and quality is lost.	Use vacuum or inert gas (such as Ar, N <sub>2</sub> ) protection.
Strong oxidants	Concentrated nitric acid (HNO <sub>3</sub> ), perchloric acid (HClO <sub>4</sub> ), etc. slowly dissolve tungsten to produce H <sub>2</sub> WO <sub>4</sub> or tungstate .	Surface corrosion and reduced durability.	Avoid contact with strong oxidizing solutions.
Halogens and halides	>300°C reacts with F <sub>2</sub> and Cl <sub>2</sub> to form volatile halides (such as WF <sub>6</sub> , boiling point 17.1°C).	The material is lost quickly and the structure is damaged.	Avoid halogen environments or use protective coatings.
Hydrogen embrittlement	>800°C It absorbs hydrogen in H <sub>2</sub> , forming a brittle phase (such as WH <sub>x</sub> ), and the grain boundaries are weakened.	Toughness decreases and brittle fracture occurs easily.	Control hydrogen concentration or use composite hydrogen-resistant materials.
Nano-surface active	Nano-tungsten powder (<100 nm) has a large specific surface area (20 m <sup>2</sup> / g), high surface energy, and is easily reactive with oxygen/water.	Oxidation at room temperature is accelerated and storage stability is reduced.	Sealed storage or surface passivation treatment. MADE BY: CTIA GROUP LTD

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The thermal expansion, thermal conductivity and high temperature oxidation behavior of tungsten are discussed.



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## CTIA GROUP LTD

### Spherical Tungsten Powder Product Introduction

#### 1. Overview of Spherical Tungsten Powder

CTIA GROUP LTD's spherical tungsten powder complies with the GB/T 41338-2022 "Spherical Tungsten Powder for 3D Printing" standard. It is prepared using a plasma spheroidization process and is specially designed for additive manufacturing (such as SLM, EBM). It meets high-end application requirements with high purity, high sphericity and excellent fluidity.

#### 2. Excellent Properties of Spherical Tungsten Powder

Ultra-high purity: tungsten content  $\geq 99.95\%$ , oxygen content  $\leq 0.05$  wt%, and extremely low impurities.

High sphericity:  $\geq 90\%$ , uniform particles, excellent powder spreading performance.

Precise particle size: D50 range 5-63  $\mu\text{m}$ , stable distribution, deviation  $\pm 10\%$ .

Excellent fluidity:  $\leq 25$  s/50g, bulk density  $\geq 9.0$  g/cm<sup>3</sup>, ensuring printing efficiency.

#### 3. Specifications of Spherical Tungsten Powder

Brand	D50 particle size ( $\mu\text{m}$ )
SWP-15	5-15
SWP-25	15-25
SWP-45	25-45
SWP-63	45-63

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

#### 4. Spherical Tungsten Powder Packaging and Quality Assurance

Packaging: Inner vacuum aluminum foil bag, outer iron drum, net weight 5kg or 10kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition, particle size distribution and sphericity data, and the shelf life is 12 months.

#### 5. Contact Information of CTIA GROUP LTD

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about spherical tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## Chapter 2 Form and Classification of Tungsten Powder

### 2.1 Overview of particle size distribution of tungsten powder

Tungsten powder is the basic form of tungsten metal and its compounds for processing and application. Its particle size distribution is one of the core characteristics that determine its physical properties, chemical activity, and subsequent processing behaviors (such as sintering and compaction). The particle size distribution of tungsten powder ranges widely, from nanometer level (<100 nm) to micrometer level (1-100  $\mu\text{m}$ ), and even up to millimeter level (>1000  $\mu\text{m}$ ) under certain conditions.

The average particle size of tungsten powder commonly used in industry is usually between 1-10  $\mu\text{m}$ , while high-purity nano tungsten powder can be refined to 10-50 nm, or even as low as 5 nm, depending on the preparation process and application requirements. For example, tungsten powder with a D50 (median particle size) of 5  $\mu\text{m}$  exhibits a higher density (usually >95% theoretical density) during sintering, which is suitable for hard alloys; while coarse particles with a D50 of 50  $\mu\text{m}$  may be more suitable for low-density porous materials due to increased porosity. The uniformity of particle size distribution is characterized by the D10, D50 and D90 parameters, where D10 means that 10% of the particles are smaller than this size, D50 is the median particle size, and D90 means that 90% of the particles are smaller than this size. These parameters reflect the width and skewness of the distribution. Narrow distribution ( $D90/D10 < 2$ ) indicates that the particle size is concentrated. For example, tungsten powder with D10 = 4  $\mu\text{m}$ , D50 = 5  $\mu\text{m}$ , and D90 = 6  $\mu\text{m}$  shrinks uniformly during sintering (Uniform Shrinkage) and has few defects in the finished product. Wide distribution ( $D90/D10 > 5$ ) such as D10 = 1  $\mu\text{m}$ , D50 = 10  $\mu\text{m}$ , and D90 = 50  $\mu\text{m}$  indicates significant differences in particle size, which may lead to uneven porosity or decreased strength during sintering.

#### 2.1.1 Characteristics and properties of three particle sizes of tungsten powder and their impact on applications

The particle size distribution of tungsten powder is one of its key physical properties, which directly affects its processing performance, the mechanical properties of the final product and its application areas. According to industry practices and actual production needs, tungsten powder is usually divided into three types of particle sizes: coarse tungsten powder (particle size 5-50  $\mu\text{m}$ ), fine tungsten powder (particle size 0.1-5  $\mu\text{m}$ ) and nano tungsten powder (particle size <0.1  $\mu\text{m}$  or <100 nm). The following is a detailed analysis of the particle size distribution, characteristics, performance and application.

#### 1. Coarse Tungsten Powder

Particle size distribution

Range: 5-50  $\mu\text{m}$

Typical D50 (median particle size): 10-20  $\mu\text{m}$

Distribution characteristics: The particle size is relatively wide and is usually obtained by screening or airflow classification. The particle shape is mostly irregular polyhedron or spherical (such as plasma atomization preparation).

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## Features

Good fluidity: Due to the larger particles, the coarse tungsten powder has better fluidity, and the Hall flow meter measurement value is usually in the range of 20-30 s/50g.

High bulk density: The apparent density is generally 5-10 g/cm<sup>3</sup>, which is close to 25%-50% of the theoretical density (19.25 g/cm<sup>3</sup>), and the stacking efficiency is high.

3) or ammonium paratungstate (APT) using a hydrogen reduction furnace or a rotary tubular reduction furnace. The process is mature and the output is high (up to 50 kg/h or more).

Low surface activity: small specific surface area (0.1-0.5 m<sup>2</sup> / g), small contact area between particles, easy to store and transport, not easy to oxidize or absorb moisture.

performance

Mechanical properties: The green body formed by pressing and sintering the coarse tungsten powder has larger grains (10-50 μm) and lower hardness (HRA 80-85), but better toughness and strong impact resistance.

Sintering performance: The sintering temperature is high (1800-2000°C), the shrinkage rate is low (10%-15%), and it is suitable for the molding of large-size products.

Thermal stability: Due to its large particle size and low surface energy, coarse tungsten powder is not prone to agglomeration or oxidation at high temperatures (O<sub>2</sub> content <0.05 wt%).

Processability: Good fluidity, suitable for traditional powder metallurgy pressing processes (such as cold pressing or hot pressing), but not suitable for additive manufacturing that requires high precision.

Impact on usage

Main Application: Crude tungsten powder is widely used in traditional powder metallurgy products, such as tungsten rods, tungsten plates, tungsten crucibles and high specific gravity alloys (W-Ni-Fe/Cu).

Impact Analysis:

In heavy alloys, the high bulk density and good flowability of coarse tungsten powder ensures uniform filling and high density (>17 g/cm<sup>3</sup>), which is suitable for manufacturing counterweights (such as aerospace counterweights) or radiation shielding materials.

In tungsten electrodes (such as TIG welding electrodes), coarse particles provide sufficient strength and high temperature resistance, extending service life.

Limitations: Too large particle size leads to coarse grains after sintering, which limits its application in high-hardness tools (such as carbide tools) or fine structural parts.

## 2. Fine Tungsten Powder

Particle size distribution

Range: 0.1-5 μm

Typical D50: 1-3 μm

Distribution characteristics: The particle size distribution is narrow and is usually prepared by ball milling, air flow crushing or microwave reduction. The particle shapes are mostly irregular polyhedrons with a small amount of spherical particles.

Features

Medium fluidity: The particles are small and the fluidity is reduced. The value measured by the Hall flow meter is usually 30-50 s/50g. It is necessary to add a flow aid (such as 0.1 wt% SiO<sub>2</sub>) to improve it.

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Medium bulk density: The apparent density is 3-6 g/cm<sup>3</sup>, there are more gaps between particles, and the stacking efficiency is lower than that of coarse tungsten powder.

The preparation process is complex: a hydrogen reduction furnace (low temperature 600-800°C) or a jet mill is used for fine processing, and the reduction conditions (such as H<sub>2</sub> flow rate and temperature gradient) are controlled to obtain a uniform particle size.

High surface activity: The specific surface area is 0.5-2 m<sup>2</sup> / g, the surface energy of the particles is increased, and they are easy to absorb moisture or oxidize (the O<sub>2</sub> content can reach 0.1-0.2 wt%), and need to be vacuum packaged for storage.

performance

Mechanical properties: After sintering, the grain size is smaller (1-5 μm), the hardness is improved (HRA 85-90), but the toughness is slightly lower than that of coarse tungsten powder, suitable for applications requiring wear resistance.

Sintering performance: The sintering temperature is reduced (1500-1800°C), the shrinkage rate is higher (15%-20%), and the density can reach 95%-98% (18-19 g/cm<sup>3</sup>).

Thermal stability: It has high surface activity and tends to slightly aggregate at high temperatures, but it can still maintain good stability (O<sub>2</sub> < 0.1 wt%).

Processability: Suitable for press molding and small-scale additive manufacturing (such as spraying), but fluidity limits its application in large-scale automated production.

Impact on usage

Main Application: Fine tungsten powder is mainly used for cemented carbide (such as WC-Co cutting tools), tungsten wire, thermal spray coatings and some electronic materials.

Impact Analysis:

In cemented carbide, fine tungsten powder reacts with carbon black to form tungsten carbide (WC), which has fine grains (<5 μm) and improves hardness and wear resistance (HRA>88), making it suitable for cutting tools and wear-resistant parts (such as drills and milling cutters).

In tungsten wire production, fine particles ensure the uniformity of the sintered green body, which is suitable for drawing ultra-fine tungsten wire (diameter <0.02 mm) for filaments or electron beam sources.

In thermal spray coatings, fine tungsten powder provides improved adhesion and surface finish, and is suitable for wear-resistant and corrosion-resistant coatings (such as aircraft engine blades).

Limitations: Poor fluidity, easy to delaminate or uneven density during pressing, not suitable for additive manufacturing of oversized products or high fluidity requirements.

### 3. Nano Tungsten Powder

Particle size distribution

Range: <0.1 μm (<100 nm)

Typical D50: 20-80 nm

Distribution characteristics: The particle size is extremely narrow and is usually prepared by solution method (such as spray drying + low-temperature reduction), plasma atomization or electrolysis. The particles are mostly nearly spherical or polyhedral.

Features

Poor fluidity: The particles are very small, the van der Waals force between the particles is significant,

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the fluidity is extremely poor, and the Hall flow meter is difficult to measure (>50 s/50g or no flow).

Low bulk density: The apparent density is only 1-3 g/cm<sup>3</sup>, the void ratio is high, and the stacking efficiency is low (<20%).

The preparation process is complex: precision equipment (such as spray drying tower, electrolytic cell or microwave reduction furnace) is required, process parameters (such as reduction temperature <700°C, H<sub>2</sub> concentration) need to be strictly controlled, and the output is low (<5 kg/h).

Very high surface activity: specific surface area>10 m<sup>2</sup>/g, very high surface energy, very easy to oxidize (O<sub>2</sub> can reach 0.5 wt%) or agglomerate, need vacuum or inert gas protection storage.

performance

Mechanical properties: After sintering, the grains are extremely fine (<1 μm) and the hardness is extremely high (HRA>90), but the toughness is low, it is easy to break brittlely, and the impact resistance is poor.

Sintering performance: The sintering temperature is significantly reduced (1200-1500°C), the shrinkage rate is as high as 20%-30%, and the density can reach 99% (>19 g/cm<sup>3</sup>), which is suitable for high-precision molding.

oxidize at high temperature (O<sub>2</sub> > 0.2 wt%), requiring low temperature sintering or adding grain inhibitors (such as VC/Cr<sub>3</sub>C<sub>2</sub>).

Processability: Suitable for additive manufacturing (such as 3D printing), thin film deposition or nanocomposites, but difficult to process and requires special dispersion equipment (such as ultrasonic dispersion).

Impact on usage

Main uses: Nano tungsten powder is mainly used in high-precision cemented carbide, additive manufacturing, electronic materials (such as semiconductor targets) and catalyst carriers.

Impact Analysis:

In ultrafine cemented carbide, nano tungsten powder generates submicron WC (<0.5 μm) with extremely high hardness (HRA>92), which is suitable for precision molds and high-performance tools (such as PCB drill bits).

In additive manufacturing, the high density and fine particles of nano tungsten powder are suitable for laser melt deposition (LMD) or electron beam melting (EBM) to prepare complex tungsten parts (such as aviation nozzles).

In electronic materials, nano tungsten powder is used for sputtering targets or conductive pastes (such as W films), providing high resolution and uniformity, suitable for the semiconductor and display industries.

In catalyst supports, high specific surface area enhances catalytic activity and is used in chemical reactions (such as hydrogenation reactions).

Limitations: Poor fluidity and easy oxidation restrict its application in large-scale traditional powder metallurgy, and high processing equipment requirements (such as high-precision 3D printers).

### Comprehensive comparison and selection basis of three strength tungsten powders

Features/Uses	Coarse tungsten powder (5-50 μm)	Fine tungsten powder (0.1-5 μm)	Nano tungsten powder (<0.1 μm)
Liquidity	Good (20-30 s/50g)	Medium (30-50 s/50g)	Poor (>50 s/50g or no flow)

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Features/Uses	Coarse tungsten powder (5-50 $\mu\text{m}$ )	Fine tungsten powder (0.1-5 $\mu\text{m}$ )	Nano tungsten powder (<0.1 $\mu\text{m}$ )
Bulk density	High (5-10 g/ $\text{cm}^3$ )	Medium (3-6 g/ $\text{cm}^3$ )	Low (1-3 g/ $\text{cm}^3$ )
Specific surface area	Low (0.1-0.5 $\text{m}^2$ / g)	Medium (0.5-2 $\text{m}^2$ / g)	High (>10 $\text{m}^2$ / g)
Sintering temperature	High (1800-2000°C)	Medium (1500-1800°C)	Low (1200-1500°C)
hardness	Lower (HRA 80-85)	Medium (HRA 85-90)	High (HRA>90)
toughness	good	medium	Difference
Main Application	Heavy alloy, tungsten electrode	Carbide, tungsten wire, thermal spraying	Ultrafine cemented carbide, additive manufacturing, electronic materials
Process complexity	Low	medium	high

### The basis for selecting the strength of black tungsten powder

If the goal is to produce high-toughness, large-size products (such as counterweights), coarse tungsten powder is preferred because of its good fluidity and simple processing.

If high hardness and wear resistance are required (such as cutting tools), fine tungsten powder is the best choice, taking into account both performance and processing feasibility.

If high precision and high surface activity are required (such as 3D printing or semiconductor targets), nano tungsten powder is more suitable, but the problems of agglomeration and oxidation need to be solved.

Due to the difference in particle size distribution, coarse tungsten powder, fine tungsten powder and nano tungsten powder show significant differences in fluidity, bulk density, sintering performance and mechanical properties, which directly affect their applications in traditional powder metallurgy, cemented carbide, additive manufacturing and electronic materials. During production, it is necessary to select the appropriate particle size according to the target use, and match the corresponding equipment (such as screening machine for coarse powder, air flow mill for fine powder, spray drying tower for nano powder) and process parameters (such as reduction temperature, gas flow) to optimize product quality and production efficiency.

#### 2.1.2 Shape of tungsten powder particle size distribution

The shape of the particle size distribution can also be described by normal distribution, log-normal distribution or bimodal distribution. For example, tungsten powder produced by hydrogen reduction often shows a log-normal distribution with a peak towards the small particle size end, while gas atomization tungsten powder may show a bimodal distribution, reflecting the coexistence of coarse and fine particles. The diversity of tungsten powder particle size distribution stems from differences in preparation processes. Hydrogen reduction produces tungsten powder with a particle size range of 0.5-100  $\mu\text{m}$  and a wide distribution by controlling the particle size and reduction conditions of the  $\text{WO}_3$ .

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precursor (temperature 600-900°C, hydrogen flow rate 0.5-2 L/min). Plasma synthesis uses high-temperature plasma (>10,000°C) to evaporate tungsten raw materials and quickly condense them to produce 10-30 nm nanopowders with a narrow distribution ( $D_{90}/D_{10} < 1.5$ ). Vapor deposition can precisely control the particle size to 0.1-1  $\mu\text{m}$ , which is suitable for high purity requirements. The raw material characteristics also affect the distribution. For example, the grain size (0.5-5  $\mu\text{m}$ ) and purity (>99.95%) of high purity ammonium tungstate (APT) directly determine the particle size range and uniformity of the tungsten powder after reduction.

the agglomeration and fragmentation of particles during the process will also change the distribution. For example, nano-tungsten powder is easy to agglomerate due to its high surface energy ( $1-2 \text{ J}/\text{m}^2$ ) and needs to be deagglomerated by dispersants or ultrasonic treatment.

The particle size distribution has many effects on the performance of tungsten powder. Small-particle tungsten powder (<1  $\mu\text{m}$ ) has a high proportion of surface atoms (>20%), strong surface activity, and high sintering activity, but poor fluidity (Hall flow rate >40 s/50g), and is easily oxidized to  $\text{WO}_3$ . Large-particle tungsten powder (>50  $\mu\text{m}$ ) has good fluidity (<20 s/50g), but the sintering temperature needs to be increased to above 1600°C, and the density is low.

The particle size distribution also affects the bulk density. For example, the bulk density of narrow-distribution spherical tungsten powder can reach 12-14  $\text{g}/\text{cm}^3$  (60-70% theoretical density), while that of wide-distribution irregular particles is only 8-10  $\text{g}/\text{cm}^3$ . In industry, the particle size distribution is adjusted by sieving, air classification or wet sedimentation to meet different needs such as cemented carbide (narrow distribution) and porous electrodes (wide distribution).

**Comparison of properties of tungsten powders in different particle size ranges**

Particle size range	Average particle size (D50)	Distribution Type	Specific surface area	Liquidity	Main Applications
Nanoscale (<100 nm)	10-50 nm	Narrow ( $D_{90}/D_{10} < 2$ )	30-80 $\text{m}^2/\text{g}$	Poor (>40 s/50g)	Photocatalyst
Submicron level (0.1-1 $\mu\text{m}$ )	0.5 $\mu\text{m}$	Medium ( $D_{90}/D_{10} \approx 3$ )	2-10 $\text{m}^2/\text{g}$	Medium (30-40 s/50g)	Cemented Carbide
Micron level (1-100 $\mu\text{m}$ )	5-50 $\mu\text{m}$	Width ( $D_{90}/D_{10} > 5$ )	0.5-5 $\text{m}^2/\text{g}$	Excellent (20-30 s/50g)	Porous Electrode

### 2.1.3 Three shapes of particle size distribution : normal distribution, lognormal distribution and bimodal distribution

The shape of the particle size distribution reflects the statistical law of the particle size of tungsten powder, which directly affects its processing performance, sintering behavior and final use. In tungsten powder production, the particle size distribution is usually measured by a laser particle size analyzer (such as Malvern Mastersizer) or sieving method and expressed as a probability density curve. The following discusses three typical distribution shapes.

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## 1. Normal Distribution

The particle size is symmetrically distributed around the average value (mean,  $\mu$ ), and the probability density function is a bell-shaped curve. Most particles are concentrated near the mean and gradually decrease on both sides.

Mathematical expression:

$$f(x) = \frac{1}{\sqrt{2\pi}\sigma^2} e^{-\frac{(x-\mu)^2}{2\sigma^2}}$$

其中,  $x$ 为粒径,  $\mu$ 为均值,  $\sigma$ 为标准差,  $\sigma^2$ 为方差。

**特征参数:** 均值 $\mu$ 表示中心粒径, 标准差 $\sigma$ 反映分布宽度, 约68%的颗粒落在 $\mu \pm \sigma$ 范围内, 95%落在 $\mu \pm 2\sigma$ 范围内。

Example: Tungsten powder D50 = 10  $\mu\text{m}$ ,  $\sigma = 2 \mu\text{m}$ , then 68% of the particles are between 8-12  $\mu\text{m}$ .

Causes of formation in tungsten powder production

### Technology

Normal distribution is common in single, stable preparation processes, such as the reduction of tungsten oxide ( $\text{WO}_3$ ) in a hydrogen reduction furnace at a constant temperature (800°C) and uniform  $\text{H}_2$  flow rate (50 L/min), with consistent particle growth rate.

### condition

The raw material particle size is uniform (such as APT particle size 10-20  $\mu\text{m}$ ), and the reduction time and atmosphere are strictly controlled to avoid excessive particle growth or breakage.

### equipment

The rotary tube reduction furnace heats the particles evenly through dynamic tumbling, thus reducing particle size deviation.

### Features

High uniformity

The particle size distribution is symmetrical, the span between D10, D50 and D90 is small (such as  $D90/D10 < 2$ ), and the particle size consistency is good.

Liquidity

Medium preference (e.g. 30-40 s/50g), balanced friction between particles and high bulk density (5-8  $\text{g}/\text{cm}^3$ ).

Controllability

Adjustment of process parameters (such as increasing the temperature to increase  $\mu$ ) can precisely control the distribution center and width.

### Performance impact

Sintering performance

The particle size is consistent, the sintering shrinkage is uniform (15%-20%), the density is high (>95%), and the grain distribution is uniform (such as 5-10  $\mu\text{m}$ ).

Mechanical properties: Balanced hardness (HRA 85-88) and toughness, no obvious weaknesses, suitable for products requiring consistency.

Processability

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The density is evenly distributed during pressing, reducing the risk of delamination or cracking, and is suitable for traditional powder metallurgy processes.

### Impact on usage

#### Applicable scenarios

Normal distribution tungsten powder is suitable for products that require uniform performance, such as tungsten rods, tungsten plates and high specific gravity alloys (W-Ni-Fe).

In heavy alloys, uniform particle size ensures high packing ratios (density > 17 g/cm<sup>3</sup>) without porosity or density gradients, making it suitable for aerospace counterweights.

In tungsten electrodes, the grains are uniform after sintering, the arc erosion resistance is stable, and the service life is extended.

### limitation

The lack of extremely fine or coarse particles makes it difficult to meet applications requiring ultra-high hardness (such as carbide tools) or special fluidity requirements.

## 2. Log-Normal Distribution

The logarithm of the particle size ( $\ln x$ ) obeys the normal distribution. The distribution curve is right-skewed, with a high proportion of small particles and a gradual decrease in large particles, presenting an asymmetric shape.

Mathematical expression:

$$f(x) = \frac{1}{x\sqrt{2\pi\sigma^2}} e^{-\frac{(\ln x - \mu)^2}{2\sigma^2}}, \quad x > 0$$

Where  $\mu$  is the logarithmic mean and  $\sigma$  is the logarithmic standard deviation.

Characteristic parameters: geometric mean particle size ( $D_g = e^\mu$ ) and geometric standard deviation (GSD =  $e^\sigma$ ), D50 tends to be on the small particle size end, D90/D10 > 2.

Example: Tungsten powder D50 = 1  $\mu\text{m}$ , GSD = 1.5, then most particles are < 1  $\mu\text{m}$ , and a small number of particles are 5-10  $\mu\text{m}$ .

### Causes of formation in tungsten powder production

#### Technology

It is common in the process of particle growth or crushing. For example, a jet mill crushes coarse tungsten powder (10-50  $\mu\text{m}$ ) into fine powder (0.1-5  $\mu\text{m}$ ). The probability of small particles being generated is high, while large particles are gradually reduced.

#### condition

A large reduction temperature gradient (e.g., 600-1000°C) or uneven H<sub>2</sub> flow rate leads to differential particle growth rates, with fine particles dominating.

#### equipment

The precursor powder (D50 = 5  $\mu\text{m}$ ) was prepared in a spray drying tower, and fine particles were retained during the subsequent reduction, with a small amount of agglomeration forming large particles.

#### Features

Fine particles

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The proportion of small particles is high (e.g. >70% below D50), there are a small number of large particles at the tail of the distribution, and the overall particle size span is large.

Liquidity

Poor (40-50 s/50g), fine particles increase friction, medium bulk density (3-6 g/cm<sup>3</sup>).

Surface activity

The specific surface area is relatively high (1-5 m<sup>2</sup> / g), and the fine particles are easily hygroscopic or oxidized (O<sub>2</sub> < 0.2 wt%), so they need to be sealed for storage.

### Performance impact

Sintering performance

Fine particles promote early sintering and have a higher shrinkage rate (20%-25%), but large particles may cause local porosity and slightly lower density (90%-95%).

Mechanical properties

Fine particles increase hardness (HRA 88-90), large particles retain a certain toughness, and have better overall performance, but slightly inferior uniformity.

Processability

Fine particles can easily fill voids during pressing, but poor fluidity may lead to uneven density, so the pressing process needs to be optimized.

### Impact on usage

Lognormal distribution tungsten powder is suitable for cemented carbide, thermal spray coating and fine tungsten wire production.

In cemented carbide, fine particles generate small grains of WC (1-3 μm), which improves wear resistance (HRA>88), and a small amount of large particles enhances impact resistance, making it suitable for cutting tools.

In thermal spraying, fine particles improve coating density and adhesion, while large particles improve wear resistance, making them suitable for coating of aviation parts.

In fine tungsten wire, fine particles ensure the uniformity of the blank, which is suitable for drawing tungsten wire with diameter <0.05 mm.

### limitation

Poor fluidity limits its application in highly automated production (such as 3D printing), requiring the addition of additives or adjustment of the process.

## 3. Bimodal Distribution

The particle size distribution has two obvious peaks, corresponding to two main particle size groups. The curve is bimodal and is usually composed of a mixture of particles of two different particle sizes.

Mathematical expression: It can be regarded as the superposition of two normal distributions:

$$f(x) = w_1 \cdot f_1(x; \mu_1, \sigma_1) + w_2 \cdot f_2(x; \mu_2, \sigma_2)$$

Among them,  $w_1$ ,  $w_2$  are the weights of each peak,  $\mu_1$ ,  $\mu_2$  are the means of the two peaks, and  $\sigma_1$ ,  $\sigma_2$  are the standard deviations.

### Characteristic parameters

Two D50 (such as 2 μm and 20 μm), with a large peak spacing, D90/D10>3.

Example: Tungsten powder peaks at 2 μm (60 wt%) and 20 μm (40 wt%).

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Causes of formation in tungsten powder production

#### **Technology**

Mixing of two particle size powders, such as fine tungsten powder (1-5  $\mu\text{m}$ ) and coarse tungsten powder (10-50  $\mu\text{m}$ ) in proportion, or agglomeration of some particles during the reduction process.

#### **Condition**

The uneven temperature in the reduction furnace (e.g., 800°C in the center and 1000°C at the edge) causes some particles to grow into large particles while others remain small.

#### **Equipment**

The sieving machine is used for classification and then mixing (such as two grades of powders <5  $\mu\text{m}$  and >10  $\mu\text{m}$ ), or the cooling rate difference in plasma atomization forms large and small particles.

#### **Features**

Dual characteristics

It has the characteristics of both fine particles and high-flowing particles, with medium flowability (25-35 s/50g) and high bulk density (6-9  $\text{g}/\text{cm}^3$ ).

Filling efficiency

Fine particles fill the gaps between large particles, improving stacking efficiency (10%-20% higher than single distribution).

Complexity

The distribution is uneven, and the ratio of the two peaks needs to be precisely controlled (such as fine: coarse = 7:3), otherwise it is easy to stratify.

#### **Performance impact**

Sintering performance

Fine particles sinter early, large particles maintain structure, shrinkage is moderate (15%-20%), density is extremely high (>98%), and the grains include both small and large particles.

Mechanical properties

It has a good balance between hardness (HRA 87-92) and toughness. Fine particles improve wear resistance, while large particles enhance crack resistance.

Processability

It has good filling properties during pressing, but requires high pressure (>500 MPa) to avoid delamination. It is suitable for complex shape molding.

#### **Impact on usage**

Bimodal tungsten powders are suitable for high-performance cemented carbides, additive manufacturing and high-density composites.

In cemented carbide, fine particles increase hardness (HRA>90) and large particles improve toughness, making them suitable for high-load tools (such as mining drill bits).

In additive manufacturing, the bimodal distribution improves powder spreadability and sintered density (>19  $\text{g}/\text{cm}^3$ ) for complex tungsten components such as aerospace nozzles.

In high-density composites, fine particles fill the voids and large particles provide structural support. The density is close to the theoretical value and is suitable for radiation shielding.

#### **Limitation**

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The production and mixing process is complicated. If the proportions are out of balance, it will easily lead to uneven performance, so strict quality control is required.

**Comprehensive comparison and application selection of three types of tungsten powder particle size distribution**

Features/Impact	normal distribution	Lognormal distribution	Bimodal distribution
Distribution shape	Symmetrical bell shape	Right-biased, mainly fine particles	Bimodal, two particle size groups
Uniformity	high	medium	Lower
Liquidity	Medium preference (30-40 s/50g)	Poor (40-50 s/50g)	Medium (25-35 s/50g)
Bulk density	Medium-high (5-8 g/cm <sup>3</sup> )	Medium (3-6 g/cm <sup>3</sup> )	High (6-9 g/cm <sup>3</sup> )
Specific surface area	Medium-low (0.5-1 m <sup>2</sup> /g)	Medium-high (1-5 m <sup>2</sup> /g)	Medium (0.5-2 m <sup>2</sup> /g)
Sintering density	High (>95%)	Moderate (90%-95%)	Very high (>98%)
Hardness/Toughness	Balance (HRA 85-88)	Hard (HRA 88-90)	Hard and tough (HRA 87-92)
Main Application	Tungsten rod, high specific gravity alloy	Cemented carbide, thermal spraying	High-performance cemented carbide, additive manufacturing
Difficulty of craft	Low	medium	high

**Selection basis**

**normal distribution**

Suitable for applications that require consistency and simple processing (such as tungsten electrodes), emphasizing uniformity and predictability.

**Lognormal distribution**

Suitable for high-hardness products dominated by fine particles (such as carbide tools), which require a balance between fluidity and performance.

**Bimodal distribution**

Suitable for complex applications that require high density and high performance (such as 3D printed tungsten parts), requiring optimized mixing ratio and process control.

**Control Methods in Tungsten Powder Production**

**normal distribution**

Stable reduction conditions (e.g. constant temperature 800°C, H<sub>2</sub> flow rate 50 L/min), use uniform raw materials (e.g. APT 10-20 μm).

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### **Lognormal distribution**

Use air flow milling or gradient reduction (such as 600-1000°C) to control the fine particle generation rate.

### **Bimodal distribution**

Mix after classification (e.g. sieving <5 μm and >10 μm powders in a 7:3 ratio), or adjust the atomization cooling rate.

Normal distribution, lognormal distribution and bimodal distribution each have unique characteristics, which affect the fluidity, sintering performance and mechanical properties of tungsten powder, thus determining its applicability in different fields. In production, the distribution shape can be regulated by process optimization (such as temperature, airflow, mixing) to meet the needs of specific applications.

## **2.2 Measurement and control of tungsten powder particle size distribution**

The measurement and control of tungsten powder particle size distribution is a key link to ensure its quality and application performance. The measurement methods include laser diffraction, sedimentation, transmission electron microscopy (TEM), scanning electron microscopy (SEM), dynamic light scattering (DLS) and sieving (Sieve Analysis), each method has different application scope and accuracy. Control involves process parameter optimization, agglomeration treatment and classification technology to achieve the target distribution.

### **2.2.1 Particle size distribution measurement method**

#### **Laser Diffraction**

Applicable range: 0.1-1000 μm

Principle: Based on Mie scattering theory, the laser beam scatters when passing through the particles, and the scattering angle is inversely proportional to the particle size.

Advantages: fast measurement speed (<1 min), batch analysis, high accuracy (error <2%), suitable for industrial detection.

Disadvantages: It is necessary to assume that the particle shape is spherical, and irregular particles may deviate from the actual value.

Application example: Tungsten powder with D50 = 5 μm, the measurement results show D10 = 3 μm, D90 = 7 μm, narrow distribution, suitable for cemented carbide.

#### **Sedimentation Method**

Applicable range: 1-100 μm

Principle: Based on Stokes' law, the sedimentation rate of particles in liquid is proportional to the square of the particle size.

Advantages: Simple equipment, low cost, suitable for micron-sized tungsten powder.

Disadvantages: Time-consuming (several hours), not applicable to nanoparticles, low accuracy (error 5-

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10%).

Application example: Tungsten powder with D50 = 10 μm, the sedimentation method measured a wide distribution (D90/D10 ≈ 6), reflecting the coexistence of coarse and fine particles.

### Transmission electron microscope (TEM) and scanning electron microscope (SEM)

Applicable range: 1 nm - 10 μm

Principle: Through electron beam imaging, the particle size and morphology are measured intuitively.

Advantages: extremely high resolution (0.1 nm), the morphology and particle size of nano-scale tungsten powder can be analyzed.

Disadvantages: complex sample preparation (ultra-thin sections or dispersion are required), limited to a small amount of sample, and cannot represent the overall distribution.

Application example: 20 nm nano-tungsten powder, TEM shows a particle size range of 15-25 nm, and the morphology is cubic.

### Dynamic light scattering (DLS)

Applicable range: 1 nm - 1 μm

Principle: Based on Brownian motion, measure the diffusion coefficient of particles in liquid and infer the particle size.

Advantages: Suitable for nanoparticles, easy to operate.

Disadvantages: Sensitive to agglomerates, need to dilute the sample, limited measurement range.

Application example: 50 nm WO<sub>3</sub> nanopowder, DLS measured an average particle size of 48 nm, but agglomeration resulted in a wide distribution.

### Sieve Analysis

Applicable range: >10 μm

Principle: Separate particles through sieves with different apertures, weigh and calculate distribution.

Advantages: intuitive, low cost, suitable for large particle size tungsten powder.

Disadvantages: not suitable for nano or submicron particles, low resolution.

Application example: D50 = 50 μm tungsten powder, 20-100 μm particles account for 80% of the sieve analysis.

Comparison table of particle size distribution measurement methods

Measurement method	Applicable particle size range	Accuracy	advantage	shortcoming	Application Scenario
Laser diffraction method	0.1-1000 μm	High (<2%)	Fast, batch analysis	Assuming spherical particles	Industrial Inspection
Sedimentation method	1-100 μm	Medium (5-10%)	Simple equipment	Long time	Micron-level analysis
TEM/SEM	1 nm - 10 μm	Very high (0.1)	Intuitive	Complex	Nanoscale research

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Measurement method	Applicable particle size range	Accuracy	advantage	shortcoming	Application Scenario
		nm)	appearance	sample preparation	
DLS	1 nm - 1 μm	High (<5%)	Suitable for nanoparticles	for Reunion has a big impact	Nanopowder Detection
Sieving method	>10 μm	Low (10-20%)	Intuitive and low cost	Low resolution	Coarse particle classification

## 2.2.2 Particle size distribution control technology

Precise control of tungsten powder particle size distribution is a key link to ensure its performance and application effect, which directly affects its flowability, packing density, sintering behavior and the quality of the final product. Control technology mainly includes three aspects: process parameter optimization, agglomeration treatment and classification technology. By adjusting the preparation conditions, eliminating particle agglomeration and separating particles of different particle sizes, the target distribution (such as narrow distribution  $D_{90}/D_{10} < 2$  or wide distribution  $D_{90}/D_{10} > 5$ ) can be achieved. The following is a detailed explanation from four dimensions: theory, process, equipment and case, with reference to literature in multiple languages such as English, Chinese, Japanese, and German.

### 2.2.2.1 Process parameter optimization

Process parameter optimization is the basis for controlling the particle size distribution of tungsten powder, which involves factors such as temperature, pressure, gas flow rate, reaction time and raw material characteristics during the preparation process. Different preparation methods have different control mechanisms for particle size and distribution. The following uses hydrogen reduction method, gas atomization method and hydrothermal method as examples to deeply analyze their parameter control principles and optimization strategies.

#### Hydrogen Reduction (Wasserstoffreduktion)

##### Principle

The hydrogen reduction method reduces  $WO_3$  or ammonium tungstate (APT) to metallic tungsten in a hydrogen atmosphere. The particle growth is controlled by the nucleation (Nucleation / Keimbildung / nucleus formation) and grain growth (Grain Growth / Kornwachstum / crystal growth) kinetics. Temperature, hydrogen flow rate and reduction time determine the nucleation rate and growth rate, thereby affecting the particle size and distribution.

##### Key Parameters

##### Temperature

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600-900°C. At low temperatures (600-650°C), the nucleation rate is high, growth is limited, and fine particles (0.5-2 μm) are generated; at high temperatures (850-900°C), growth is dominant and particles become larger (10-50 μm).

#### Hydrogen flow rate

0.5-2 L/min. Low flow rate (0.5-1 L/min) slows down the reduction, and the particles are small but widely distributed ( $D_{90}/D_{10} \approx 5$ ); high flow rate (1.5-2 L/min) accelerates the reduction, and the particles are uniform ( $D_{90}/D_{10} < 3$ ).

#### Restore time

2-6 h. In a short time (2-3 h), the particles are not fully grown, and  $D_{50} \approx 1-3 \mu\text{m}$ ; in a long time (5-6 h), the particles grow large, and  $D_{50} \approx 10-20 \mu\text{m}$ .

#### Equipment

Tube furnace (Tube Furnace / Rohrofen / Tubular furnace), inner diameter 50-100 mm, heating zone length 300-600 mm, equipped with precise temperature control system ( $\pm 5^\circ\text{C}$ ) and flow meter (accuracy 0.1 L/min).

#### Optimization strategy

To obtain a narrow distribution (e.g.,  $D_{50} = 5 \mu\text{m}$ ,  $D_{90}/D_{10} < 2$ ), 700°C, 1.5 L/min hydrogen flow rate, 4 h reduction time, and a fine  $\text{WO}_3$  precursor (particle size  $< 1 \mu\text{m}$ ) can be used.

#### Case

A cemented carbide plant uses 720°C, 1.2 L/min, 3.5 h conditions to produce tungsten powder with  $D_{50} = 0.8 \mu\text{m}$  and  $D_{90}/D_{10} = 1.9$ , meeting the high density requirements (see Lassner & Schubert, 1999).

#### Gas Atomization / Gaszerst ä ubung / Gas Atomization method

##### Principle

The molten tungsten liquid is atomized into droplets by high-pressure gas ( $\text{N}_2$  or Ar), and rapidly cooled and solidified into spherical particles. The particle size is controlled by the droplet splitting and cooling rate, and the distribution is related to gas dynamics (Gas Dynamics / Gasdynamik / ガスダイナミクス) and thermodynamics.

##### Key parameters :

##### Gas pressure

10-50 bar. Low pressure (10-20 bar) produces larger particles (20-50 μm); high pressure (40-50 bar) causes droplets to break up more finely, with particle sizes reduced to 1-10 μm.

##### Nozzle aperture

0.5-2 mm. Small pores (0.5-1 mm) have smaller droplets,  $D_{50} \approx 5-10 \mu\text{m}$ ; large pores (1.5-2 mm) have

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larger particles,  $D_{50} \approx 30\text{-}50 \mu\text{m}$ .

### Cooling rate

$10^3$  -  $10^5$  K/s. High cooling rate ( $10^5$  K/s) inhibits particle growth and has a narrow distribution ( $D_{90}/D_{10} < 2$ ); low rate ( $10^3$  K/s) causes particle growth and a wide distribution.

### Equipment

Atomization Tower (Atomization Tower / Zerstäubungsturm / アトマイズ塔), height 3-5 m, equipped with a high-pressure air pump (pressure 10-60 bar) and a cooling water circulation system (flow rate 50-100 L/min).

### Optimization strategy

To obtain 1-10  $\mu\text{m}$  narrow distribution tungsten powder, a pressure of 45 bar, a 0.8 mm nozzle, a cooling rate of  $10^4$  K/s, and controlled melt temperature (3500-3600°C) can be used.

### Case

An additive manufacturing company uses 50 bar, 1 mm nozzle, and  $10^4$  K/s conditions to produce spherical tungsten powder with  $D_{50} = 8 \mu\text{m}$  and  $D_{90}/D_{10} = 1.7$  for SLM process (refer to American Elements, 2023).

### Hydrothermal Method

**Principle** : In high temperature and high pressure aqueous solution, tungstate (such as sodium tungstate) generates  $\text{WO}_3$  nanoparticles through nucleation and crystal growth, and the particle size and distribution are controlled by the reaction conditions.

### Key Parameters

#### Temperature

180-220°C. Low temperature (180-190°C) results in more nucleation and fine particles (10-20 nm); high temperature (210-220°C) results in accelerated growth and particle size increases to 30-50 nm.

#### Pressure

1-10 MPa. High pressure (5-10 MPa) promotes uniform nucleation and narrow distribution ( $D_{90}/D_{10} < 1.5$ ); low pressure (1-3 MPa) results in uneven particles and wide distribution.

#### Precursor concentration

0.1-1 mol/L. At low concentrations (0.1-0.3 mol/L), the particles are small and uniform; at high concentrations (0.8-1 mol/L), the particles are larger and have a wider distribution.

### Equipment

High-pressure reactor (Autoclave / Autoklav / Autoclave), volume 50-500 mL, pressure resistance 15

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MPa, equipped with temperature control system ( $\pm 2^{\circ}\text{C}$ ) and stirring device (100-500 rpm).

### Optimization strategy

To obtain 20-30 nm narrow distribution  $\text{WO}_3$ ,  $200^{\circ}\text{C}$ , 5 MPa, 0.5 mol/L sodium tungstate and 12 h reaction can be used.

### Case

A photocatalytic research team used  $200^{\circ}\text{C}$ , 6 MPa, and 0.4 mol/L conditions to prepare  $\text{WO}_3$  with  $D50 = 25 \text{ nm}$ ,  $D90/D10 = 1.4$ , and a hydrogen production rate of  $450 \mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$  (see Zhang & Wang, 2018).

### 2.2.2.2 Agglomeration treatment

Small particle size tungsten powder ( $< 1 \mu\text{m}$ ) is prone to agglomeration (Agglomeration / Agglomeration / Agglomeration) due to its high surface energy ( $1-2 \text{ J/m}^2$ ), causing the particle size distribution to deviate from the target, affecting the measurement accuracy and application performance. Agglomeration treatment aims to disaggregate the particles and restore the monodisperse state. Common methods include dispersants, ultrasonic treatment and mechanical stirring.

### Dispersants

#### Principle

Dispersants prevent agglomeration by reducing the surface tension (Surface Tension / Oberflächenspannung / surface tension) and Van der Waals Force (Van der Waals Force / Van-der-Waals-Kraft / Van der Waals Force) between particles.

#### Commonly used dispersants

##### Polyvinyl alcohol (PVA)

Water-soluble polymer, concentration 0.1-0.5 wt%, suitable for micron-sized tungsten powder, depolymerization rate 50-60%.

##### Polyvinylpyrrolidone (PVP)

Surfactant, with a concentration of 0.2-1 wt%, has a significant effect on nano-tungsten powder, with a deagglomeration rate of 60-70%.

##### Sodium dodecyl sulfate (SDS)

Anionic surfactant, concentration 0.1-0.3 wt%, reduces surface energy, depolymerization rate 55-65%.

### Technology

Add tungsten powder to water or ethanol solution containing dispersant (solid content 5-20 wt%) and stir for 30-60 min at a speed of 500-1000 rpm.

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### Equipment

Magnetic stirrer (Magnetic Stirrer / Magnetprüher / マグネチックスターラ), power 50-200 W, equipped with a stirring paddle (diameter 20-50 mm).

### Optimization strategy

To disperse 50 nm nano-tungsten powder, 0.5 wt% PVP can be used and stirred for 45 min. After depolymerization, D50 decreases by 20-30%.

### Case

A nano-tungsten powder manufacturer treated 100 nm tungsten powder with 0.3 wt% SDS, and the D50 dropped from 150 nm (agglomerated state) to 95 nm (see Li Yang & Gao Yong, 2015).

## Ultrasonic Treatment (Ultrasonic Treatment / Ultraschallbehandlung / Ultrasonic Treatment)

### Principle

Ultrasonic waves generate local high pressure and micro-turbulence through the cavitation effect, breaking up aggregates.

### Key Parameters

#### Frequency

20-40 kHz. Low frequency (20-25 kHz) has strong penetration and is suitable for micron level; high frequency (35-40 kHz) has concentrated energy and is suitable for nanometer level.

#### Power

100-500 W. Low power (100-200 W) disperses light agglomerates; high power (400-500 W) breaks up strong agglomerates.

#### Processing time

10-30 min. A short time (10-15 min) avoids particle damage; a long time (20-30 min) ensures complete disaggregation.

### Equipment

Ultrasonic cleaner (Ultrasonic Cleaner / Ultraschallreiniger / Ultrasonic Cleaner), volume 2-10 L, equipped with titanium alloy probe (diameter 10-20 mm).

### Optimization strategy

To disperse 20 nm tungsten powder, 40 kHz, 300 W, 15 min treatment can be used, and the deagglomeration rate reaches 80%.

### Case

A laboratory used 35 kHz and 400 W to process 30 nm  $WO_3$ , and the aggregate size was reduced from 200 nm to 35 nm (see Chen Dan & Ye Jun, 2008).

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## Mechanical Stirring

### Principle

The agglomerates are dispersed by shear force (Scherkraft / Shern force), which is suitable for submicron tungsten powder.

### Key Parameters

#### Speed

500-2000 rpm. Low speed (500-1000 rpm) disperses light agglomerates; high speed (1500-2000 rpm) breaks strong agglomerates.

#### Mixing time

20-60 min. Short time (20-30 min) avoids overheating; long time (40-60 min) improves uniformity.

#### Equipment

High-Speed Mixer (High-Speed Mixer / Hochgeschwindigkeitsmischer / 高速ミキサー), power 100-500 W, equipped with multi-layer stirring paddles (diameter 30-60 mm).

#### Optimization strategy

Add 0.2 wt% PVA and stir at 1000 rpm for 30 min to disperse 0.5  $\mu\text{m}$  tungsten powder with a deagglomeration rate of 60%.

#### Case

A factory processed 1  $\mu\text{m}$  tungsten powder at 1500 rpm for 40 min, and the D50 dropped from 5  $\mu\text{m}$  (agglomerated state) to 1.2  $\mu\text{m}$  (see Smithells, 2004).

### 2.2.2.3 Classification technology

Classification technology adjusts the particle size distribution through physical separation. It is divided into three methods: screening, airflow classification and wet sedimentation, which are suitable for different particle size ranges and industrial needs.

#### Sieving (Sieving / Sieben / ふるい分け)

##### Principle

Separate particles by passing them through sieves of different apertures, screening based on size.

##### Key Parameters

#### Screen aperture

100-500 mesh (150-25  $\mu\text{m}$ ). Large pore size (100-200 mesh) separates particles  $>50 \mu\text{m}$ ; small pore size (400-500 mesh) separates particles 25-38  $\mu\text{m}$ .

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### **Vibration frequency**

20-50 Hz. High frequencies (40-50 Hz) improve efficiency; low frequencies (20-30 Hz) avoid clogging.

### **Equipment**

Vibrating Screen (Vibrating Screen / Vibrationsieb / 振動ふるい), screen diameter 0.5-1 m, power 0.5-2 kW.

### **Optimization strategy**

To separate 10-50  $\mu\text{m}$  tungsten powder, a 200 mesh screen with 40 Hz vibration can be used, with a separation efficiency of 85%.

### **Case**

A company used a 300-mesh screen to separate  $D_{50} = 50 \mu\text{m}$  tungsten powder, and the proportion of particles  $>50 \mu\text{m}$  dropped from 40% to 5% (see Hampel & Hawley, 1973).

### **Air Classification (Air Classification / Luftklassierung / Air Classification)**

#### **Principle**

through a high-speed airflow ( $\text{N}_2$  or air). Small particles rise with the airflow, while large particles settle.

#### **Key Parameters**

##### **Air flow rate**

10-50 m/s. Low speed (10-20 m/s) separates particles  $>10 \mu\text{m}$ ; high speed (40-50 m/s) separates particles 0.5-5  $\mu\text{m}$ .

##### **Classifying wheel speed**

1000-5000 rpm. High speed (4000-5000 rpm) separates fine particles ( $<1 \mu\text{m}$ ); low speed (1000-2000 rpm) separates coarse particles.

#### **Equipment**

Air Classifier (Luftklassierer / Air Classifier), with a processing capacity of 50-500 kg/h, equipped with cyclone separator and dust removal system.

#### **Optimization strategy**

To separate 1-10  $\mu\text{m}$  tungsten powder, a 30 m/s air flow and a rotation speed of 3000 rpm can be used, and the classification accuracy can reach 90%.

#### **Case**

A factory used 40 m/s and 4000 rpm to classify  $D_{50} = 5 \mu\text{m}$  tungsten powder, and the proportion of particles  $<1 \mu\text{m}$  decreased from 20% to 2% (see Quan Yongsheng & Jin Xitai, 2003).

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## Wet Sedimentation (Wet Sedimentation / Nassedimentation / Wet Sedimentation)

### Principle

Based on Stokes' law, the sedimentation rate of particles in liquid is related to the particle size. Large particles settle quickly, while small particles are suspended.

### Key Parameters

#### Medium

Water or ethanol. Water has high viscosity and settles slowly; ethanol has low viscosity and settles quickly.

#### Settling time

1-24 h. Short time (1-3 h) to separate particles  $>50 \mu\text{m}$ ; long time (12-24 h) to separate particles 1-10  $\mu\text{m}$ .

#### Equipment

Sedimentation tank (Sedimentation Tank / Sedimentationsbehälter / 沉降タンク), volume 10-100 L, equipped with stirring and filtration system.

#### Optimization strategy

Using ethanol as a medium, 1-50  $\mu\text{m}$  tungsten powder was separated by sedimentation for 6 h, with a separation efficiency of 80%.

#### Case

A laboratory used ethanol sedimentation for 12 h to grade  $D_{50} = 10 \mu\text{m}$  tungsten powder, and the proportion of particles  $>20 \mu\text{m}$  decreased from 30% to 5% (see Greenwood & Earnshaw, 1997).

### 2.2.2.4 Comprehensive control and practical application

Comprehensive control combines process optimization, agglomeration and classification technology to ensure that the particle size distribution meets specific requirements. For example, cemented carbide requires a narrow distribution of tungsten powder with  $D_{50} = 0.5-1 \mu\text{m}$ . The coarse powder can be prepared by hydrogen reduction (700°C, 1.5 L/min, 4 h), PVP dispersion treatment for deagglomeration, and airflow classification (30 m/s, 4000 rpm) for separation, and finally obtain a product with  $D_{50} = 0.6 \mu\text{m}$  and  $D_{90}/D_{10} = 1.8$ . Nano-tungsten powder (20-30 nm) for photocatalysis is prepared by hydrothermal method (200°C, 5 MPa, 0.5 mol/L), ultrasonic treatment (40 kHz, 300 W, 15 min) for deagglomeration, and wet sedimentation (ethanol, 6 h) for classification to obtain a powder with  $D_{50} = 25 \text{ nm}$  and  $D_{90}/D_{10} = 1.5$ .

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**Tungsten powder particle size control technology comparison table**

Technology Type	method	Applicable particle size range	Accuracy	advantage	shortcoming
Process Optimization	Hydrogen reduction method	0.5-100 μm	Medium-High	Strong controllability	Precise parameters required
Agglomeration treatment	Ultrasonication	<1 μm	high	Efficient depolymerization	May damage particles
Grading Technology	Airflow classification	0.5-50 μm	high	Industrial application	High equipment cost

### 2.2.3 Actual Cases

#### Case 1

Tungsten powder for cemented carbide, target D50 = 0.5 μm, D90/D10 < 2. Fine WO<sub>3</sub> (particle size <1 μm) was reduced by hydrogen at 700°C and 1.5 L/min for 4 h. Laser diffraction method measured D50 = 0.48 μm, D90/D10 = 1.8, which met the requirements.

#### Case 2

Nano-tungsten powder is used for photocatalysis, with a target of 20-30 nm. Using a hydrothermal method (200°C, 5 MPa, 0.5 mol/L sodium tungstate), TEM measured an average particle size of 25 nm, and DLS showed a narrow distribution (D90/D10 ≈ 1.5).

### 2.3 Morphology and characteristics of tungsten powder

The morphology of tungsten powder includes spherical, flake, porous structure, irregular, needle-shaped, cubic and rod-shaped. Spherical tungsten powder is prepared by plasma spheroidization or gas atomization, with a smooth surface and excellent fluidity (Hall flow rate 20-30 s/50g), suitable for additive manufacturing and thermal spraying. Flaky tungsten powder is made by high-energy ball milling, with an aspect ratio of up to 10:1 and a specific surface area of 5-15 m<sup>2</sup> / g, suitable for conductive slurry. Porous tungsten powder is prepared by chemical reduction or template method, with a porosity of 30-50% and a specific surface area of up to 20-50 m<sup>2</sup> / g, suitable for catalysts. Irregular tungsten powder is the product of hydrogen reduction method, in the form of polyhedrons or fragments, with poor fluidity (>40 s/50g), low cost, and widely used in cemented carbide.

Morphology affects the packing behavior and surface activity of tungsten powder. The packing density of spherical particles can reach 12-14 g/cm<sup>3</sup> (60-70% theoretical density), while that of flaky or porous particles is only 8-10 g/cm<sup>3</sup>. Small-sized tungsten powder has a high surface atomic ratio (>20%), strong surface activity, and is easily oxidized to WO<sub>3</sub>, requiring vacuum packaging or dispersant treatment.

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**Tungsten powder morphology characteristics comparison table**

Morphology type	Preparation method	Particle size range	Specific surface area	Liquidity	Main Applications
spherical	Plasma spheroidization	1-50 μm	0.5-2 m <sup>2</sup> / g	Excellent (20-30 s/50g)	Additive Manufacturing
Flake	High Energy Ball Milling	0.5-10 μm	5-15 m <sup>2</sup> / g	Poor (>40 s/50g)	Conductive paste
Porous structure	Chemical reduction	0.1-5 μm	20-50 m <sup>2</sup> / g	Medium (30-40 s/50g)	catalyst
Irregular	Hydrogen reduction method	1-100 μm	1-5 m <sup>2</sup> / g	Poor (>40 s/50g)	Cemented Carbide

## 2.4 Factors affecting morphology and optimization

The morphology of tungsten powder is affected by the preparation process, raw material properties and environmental conditions. In the hydrogen reduction method, the increase in temperature (600-900°C) causes the particles to change from irregular shape to spherical shape, and the increase in hydrogen flow rate (0.5-2 L/min) promotes particle refinement. In atomization, the gas pressure (10-50 bar) and cooling rate (10<sup>-3</sup> -10<sup>-5</sup> K/s) determine the sphericity and particle size, and high-pressure rapid cooling produces smaller spherical particles. The hydrothermal method can produce needle-shaped or cubic WO<sub>3</sub> by adjusting the pH (4-6), temperature (180-220°C) and reaction time (12-24 h). The particle size of the raw materials also plays a role. For example, fine WO<sub>3</sub> precursors (<1 μm) tend to produce uniform particles.

Morphology optimization needs to take into account application requirements. Spherical tungsten powder can improve sphericity by increasing plasma power (10-50 kW) and optimizing nozzle design; flake tungsten powder can increase aspect ratio by extending ball milling time (20-50 h) and adding lubricants (such as stearic acid); porous tungsten powder can control porosity by template pore size (0.1-1 μm) and reduction conditions. The optimized morphology can significantly improve performance, such as reducing the defect rate of spherical tungsten powder by 20-30% in SLM.

## 2.5 Ultrafine tungsten powder (submicron level) and nano tungsten powder (<100 nm)

Ultrafine tungsten powder refers to tungsten powder with a particle size of 100 nm to 1 μm, which is suitable for high-performance cemented carbide and electronic packaging. The preparation methods include high-energy ball milling, hydrogen reduction of fine WO<sub>3</sub> and vapor deposition. High-energy ball milling refines to 0.2-0.8 μm, hydrogen reduction generates uniform particles at 700-800°C, and vapor deposition is suitable for high-purity requirements. The specific surface area of ultrafine tungsten powder is 2-10 m<sup>2</sup> / g, the sintering temperature is reduced to 1200-1300°C, and the density is more than

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98%.

Nano tungsten powder has a particle size of less than 100 nm and has unique properties due to quantum effects. Preparation methods include hydrothermal method, sol-gel method and plasma synthesis. The hydrothermal method generates 20-50 nm  $WO_3$ , the sol-gel method generates 10-30 nm particles, and plasma synthesis forms 5-20 nm particles. Nano tungsten powder has strong surface activity and a photocatalytic hydrogen production rate of  $450 \mu\text{mol} \cdot \text{g}^{-1} \cdot \text{h}^{-1}$ .

**Ultrafine and Nano Tungsten Powder Comparison Table**

Type	Particle size range	Specific surface area	Preparation method	characteristic	Application Areas
Ultrafine tungsten powder	100 nm - 1 $\mu\text{m}$	2-10 $\text{m}^2 / \text{g}$	High Energy Ball Milling	High density	Cemented Carbide
Nano tungsten powder	<100 nm	30-80 $\text{m}^2 / \text{g}$	Hydrothermal method	High surface activity	Photocatalyst

## 2.6 Application Cases of Ultrafine and Nano Tungsten Powder

Ultrafine tungsten powder is widely used in cemented carbide tools. For example, the hardness of WC-Co tools prepared from 0.5  $\mu\text{m}$  ultrafine tungsten powder reaches 93 HRA, and the wear resistance is increased by 20%, which is used for high-speed cutting. Nano tungsten powder performs well in the field of photocatalysis. For example, 50 nm  $WO_3$  coating decomposes organic pollutants under ultraviolet light, and the efficiency is 3-5 times higher than that of micron level. Nano tungsten powder is also used in electrochromic windows. The color change response time of 20 nm  $WO_3$  film is shortened to 5 seconds, and the cycle life exceeds 10,000 times.

## 2.7 High-purity tungsten powder (purity>99.95%) and doped tungsten powder (such as $\text{La}_2\text{O}_3$ , K doped)

High-purity tungsten powder has a purity of more than 99.95% and a low impurity content (such as Fe <10 ppm), which is suitable for semiconductors and sputtering targets. High-purity APT and ultra-pure hydrogen are used in the preparation, and the grain size is controlled at 0.1-5  $\mu\text{m}$ . Doped tungsten powder is optimized by adding  $\text{La}_2\text{O}_3$  (0.5-2 wt%), K (50-100 ppm) or  $\text{Y}_2\text{O}_3$  (1-2 wt%). Lanthanum doping improves high-temperature strength, potassium doping inhibits grain growth, and yttrium doping enhances oxidation resistance.

**Comparison table of high purity and doped tungsten powder**

Type	Purity	Impurities/doping ingredients	Grain size	Characteristic	Application areas
High purity	>99.95%	Fe <10 ppm	0.1-5 $\mu\text{m}$	High cleanliness	Sputtering

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Type	Purity	Impurities/doping ingredients	Grain size	Characteristic	Application areas
tungsten powder					Target
Doped Tungsten Powder	98-99.5%	La <sub>2</sub> O <sub>3</sub> 0.5-2 wt %	5-20 μm	High temperature stability	filament

## 2.8 Tungsten oxide forms (WO<sub>3</sub>, WO<sub>2.9</sub>, WO<sub>2.72</sub>) and their properties

Tungsten oxides include WO<sub>3</sub>, WO<sub>2.9</sub> and WO<sub>2.72</sub>. WO<sub>3</sub> is yellow, monoclinic, with a band gap of 2.4-2.8 eV, suitable for photocatalysis. WO<sub>2.9</sub> is blue, needle-shaped, and has infrared absorption ability due to oxygen defects. WO<sub>2.72</sub> is purple, contains a variety of oxygen vacancies, and is suitable for optoelectronic devices.

**Tungsten Oxide Morphology Comparison Table**

Type	Color	Crystal structure	Band gap	Morphology	Application areas
WO <sub>3</sub>	yellow	Monoclinic system	2.4-2.8 eV	Cubic/Needle	Photocatalysis
WO <sub>2.9</sub>	blue	Monoclinic system	2.6-2.9 eV	Needle/Rod	Heat Shield
WO <sub>2.72</sub>	Purple	Complex monoclinic	2.5-2.7 eV	irregular	Optoelectronic devices

## 2.5 The advantages and disadvantages of particle size distribution of tungsten powder

The particle size distribution of tungsten powder is one of its core characteristics, and its quality directly affects the physical properties, processing behavior and the quality of the final product. The quality of particle size distribution is usually evaluated by the uniformity of distribution (width or narrowness), particle size range, distribution shape (such as normal, lognormal or bimodal distribution) and the degree of matching with application requirements. Excellent particle size distribution can improve the flowability, packing density, sintering activity and product consistency of tungsten powder, while poor distribution may lead to processing difficulties, uneven performance or finished product defects. The following discusses the advantages and disadvantages of tungsten powder particle size distribution in detail from three aspects: theoretical analysis, characteristic comparison and actual impact.

### 2.5.1 Theoretical basis of particle size distribution

The quality of particle size distribution is based on the statistical characteristics of particle size, usually characterized by D10, D50, D90 parameters and distribution width (D90/D10). Narrow distribution

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( $D_{90}/D_{10} < 2$ ) indicates that the particle size is concentrated and the uniformity is high; wide distribution ( $D_{90}/D_{10} > 5$ ) indicates that the particle size varies greatly and the uniformity is low. In theory, the uniformity of particle size is closely related to surface energy, particle interactions (such as van der Waals forces) and dynamic behavior during processing. For example, narrow distribution tungsten powder has close particle size, uniform surface energy distribution, consistent shrinkage during sintering, and high density; while wide distribution tungsten powder has uneven sintering shrinkage due to the coexistence of large and small particles, and is prone to forming pores or cracks.

The shape of the distribution also affects the quality. Normal distribution indicates that the particle size is concentrated, which is suitable for applications that require high consistency; Log-Normal distribution is biased towards the small particle size end, which is common in tungsten powder prepared by hydrogen reduction method; Bimodal distribution includes both coarse and fine particles, which may optimize the packing density but increase the complexity of processing. In addition, the choice of particle size range needs to match the application. For example, nano-scale ( $< 100$  nm) tungsten powder is suitable for highly active catalysts, while micron-scale ( $1-10$   $\mu\text{m}$ ) is suitable for cemented carbide.

## 2.5.2 Comparison of characteristics of good and bad particle size distribution

### Characteristics of excellent particle size distribution

#### High uniformity

$D_{90}/D_{10} < 2$ , e.g.  $D_{10} = 4$   $\mu\text{m}$ ,  $D_{50} = 5$   $\mu\text{m}$ ,  $D_{90} = 6$   $\mu\text{m}$ , small particle size difference.

#### Good liquidity

The Hall Flow Rate of narrow distribution spherical tungsten powder can reach 20-30 s/50g, which is suitable for powder metallurgy and additive manufacturing.

#### High bulk density

Uniform particles are densely packed, such as 5  $\mu\text{m}$  narrow distribution tungsten powder with a packing density of up to 12-14  $\text{g}/\text{cm}^3$  (60-70% of theoretical density).

#### Excellent sintering performance

Uniform shrinkage, density  $> 95\%$ , low porosity of finished product and stable mechanical properties.

#### Application Examples

Ultrafine tungsten powder with  $D_{50} = 0.5$   $\mu\text{m}$  and  $D_{90}/D_{10} = 1.8$  is used for cemented carbide tools, with a hardness of 93 HRA and a 20% increase in wear resistance.

### Characteristics of poor quality particle size distribution

#### Low uniformity

$D_{90}/D_{10} > 5$ , for example,  $D_{10} = 1$   $\mu\text{m}$ ,  $D_{50} = 10$   $\mu\text{m}$ ,  $D_{90} = 50$   $\mu\text{m}$ , the particle size difference is significant.

#### Poor liquidity

Widely distributed irregular tungsten powder has a Hall flow rate of  $> 40$  s/50g, which is easy to clog the mold or nozzle.

#### Low bulk density

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Large and small particles are mixed and stacked loosely, for example, the bulk density of 1-50  $\mu\text{m}$  wide distribution tungsten powder is only 8-10  $\text{g}/\text{cm}^3$ .

#### Poor sintering performance

Uneven shrinkage, density <90%, pores or cracks are prone to appear, and strength decreases.

#### Application Examples

Coarse tungsten powder with  $D50 = 20 \mu\text{m}$  and  $D90/D10 = 6$  is used for porous electrodes. Although the porosity is high, the structure is uneven and the conductivity fluctuates.

**Comparison table of particle size distribution of good and bad tungsten powder**

characteristic	Excellent distribution (D90/D10 < 2)	Poor quality distribution (D90/D10 > 5)	Impact Areas
Uniformity	high	Low	Sintering consistency
Liquidity	Excellent (20-30 s/50g)	Poor (>40 s/50g)	Processing efficiency
Bulk density	High (12-14 $\text{g}/\text{cm}^3$ )	Low (8-10 $\text{g}/\text{cm}^3$ )	Material utilization
Sintering density	>95%	<90%	Finished product quality
Application Compatibility	high	Medium-Low	Performance stability

### 2.5.3 Practical impact of good or bad particle size distribution

#### Influence on sintering behavior

When tungsten powder with excellent distribution (such as  $D50 = 5 \mu\text{m}$ ,  $D90/D10 = 1.5$ ) is sintered at  $1400^\circ\text{C}$ , the contact between particles is uniform, the diffusion rate is consistent, the density can reach 98%, and the finished product has no obvious pores. Poor distribution (such as  $D50 = 10 \mu\text{m}$ ,  $D90/D10 = 6$ ) has a porosity of up to 10-15% after sintering, and the strength is reduced by 20-30% because large particles hinder the filling of small particles. For example, a Japanese cemented carbide company uses narrow distribution tungsten powder, which improves the fracture toughness of the tool by 15%.

#### Impact on fluidity and processing

Narrowly distributed spherical tungsten powder has excellent fluidity in powder metallurgy, uniform mold filling, and a 20% reduction in the defect rate of pressed green sheets. Widely distributed tungsten powder has poor fluidity and is prone to clogging the nozzle during spraying or 3D printing, reducing production efficiency. German literature points out that tungsten powder with  $D90/D10 > 5$  increases the nozzle clogging rate by 30% in thermal spraying.

#### Influence on surface activity and chemical properties

Nano-scale narrow distribution tungsten powder ( $D50 = 20 \text{nm}$ ,  $D90/D10 < 2$ ) has a high proportion of

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surface atoms (>30%) and strong photocatalytic activity, with a hydrogen production rate of  $450 \mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$ . Wide distribution micron-scale tungsten powder ( $D_{50} = 50 \mu\text{m}$ ,  $D_{90}/D_{10} > 5$ ) has low surface activity, and its catalytic efficiency is only 1/5 of the former, and it is easily oxidized to  $\text{WO}_3$ .

for high-precision applications, such as semiconductor targets that require tungsten powder with  $D_{50} = 1 \mu\text{m}$  and  $D_{90}/D_{10} < 2$  to ensure film uniformity. Inferior distribution is suitable for low-requirement scenarios, such as porous electrodes that can accept tungsten powder with  $D_{90}/D_{10} > 5$  to increase porosity but sacrifice consistency.

#### 2.5.4 Industrial Standards for Evaluation of Quality

In industry, the quality of particle size distribution is often evaluated by the following criteria:

Uniformity index:  $D_{90}/D_{10} < 2$  is excellent, 2-5 is medium,  $>5$  is poor.

Specific surface area: Nanoscale  $>30 \text{ m}^2 / \text{g}$ , microscale  $1-5 \text{ m}^2 / \text{g}$ , too low or too high is considered inappropriate.

Flowability test: Hall flow rate  $<30 \text{ s}/50\text{g}$  is good,  $>40 \text{ s}/50\text{g}$  is bad.

Sintering density:  $>95\%$  is good,  $<90\%$  is bad.

For example, China Powder Technical Standard (GB/T 1482-2010) stipulates that the  $D_{50}$  of tungsten powder for cemented carbide is  $0.5-5 \mu\text{m}$ , and  $D_{90}/D_{10} < 3$ .

#### 2.5.5 Conclusion

The quality of tungsten powder particle size distribution is the bridge between performance and application. Excellent distribution meets high-end requirements with high uniformity, fluidity, bulk density and sintering performance, while inferior distribution limits its use due to unevenness and inefficiency. Through process optimization (such as hydrogen reduction temperature control) and classification technology (such as airflow classification), the distribution quality can be significantly improved to meet specific industrial requirements.

### 2.6 Industrial significance of particle size distribution of tungsten powder

The particle size distribution of tungsten powder is of great significance in industrial production, directly affecting production efficiency, material properties and economic benefits. From cemented carbide to photocatalysts, from additive manufacturing to porous electrodes, particle size distribution determines the applicability and competitiveness of tungsten powder. The following elaborates on its industrial significance from four aspects: efficiency improvement of industrial production, product quality control, expansion of application fields and optimization of economic benefits.

#### 2.6.1 Improvement of industrial production efficiency

The impact of particle size distribution on production efficiency is reflected in the stability and continuity of the processing process. Narrow distribution tungsten powder ( $D_{90}/D_{10} < 2$ ) has good fluidity (Hall

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flow rate 20-30 s/50g), which can be evenly filled in powder metallurgy, thermal spraying and 3D printing, reducing mold clogging or nozzle failure. For example, a German thermal spraying company uses tungsten powder with  $D_{50} = 10 \mu\text{m}$  and  $D_{90}/D_{10} = 1.8$ , which increases spraying efficiency by 25% and reduces equipment maintenance costs by 15%. On the contrary, wide distribution tungsten powder ( $D_{90}/D_{10} > 5$ ) has poor fluidity and is prone to production interruptions. Japanese literature reports that its nozzle clogging rate in SLM printing increases by 20% and the production cycle is extended by 30%.

In addition, narrow distribution tungsten powder shrinks uniformly during sintering, reducing the time for post-processing (such as grinding). Data from China's cemented carbide industry shows that the sintering cycle of tungsten powder with  $D_{50} = 0.5 \mu\text{m}$  and  $D_{90}/D_{10} < 2$  is 10-15% shorter than that of wide distribution. Efficient classification technology (such as airflow classification, processing capacity 500 kg/h) further ensures production continuity.

### 2.6.2 Significance to product quality control

Particle size distribution is a key control point for product quality. Excellent narrow distribution tungsten powder ensures the consistency and reliability of the finished product. For example, cemented carbide tools require tungsten powder with  $D_{50} = 0.5-1 \mu\text{m}$ , a density of  $>98\%$  after sintering, a hardness stable at 92-93 HRA, and a performance fluctuation of  $<2\%$  between batches. Wide distribution tungsten powder ( $D_{90}/D_{10} > 5$ ) has a high porosity (10-15%), a hardness fluctuation of 5-10%, and a reduced qualified rate of finished products. German literature points out that narrow distribution tungsten powder reduces the film defect rate by 20% in sputtering targets and improves the yield of semiconductor devices.

In the field of photocatalysis, nano-scale narrow distribution tungsten powder ( $D_{50} = 20-30 \text{ nm}$ ,  $D_{90}/D_{10} < 2$ ) has uniform surface activity and a hydrogen production rate fluctuation of  $<5\%$ , while wide distribution powder ( $D_{90}/D_{10} > 5$ ) has uneven activity and a 30-40% reduction in efficiency. In industry, laser diffraction is used to monitor distribution in real time to ensure stable quality.

### 2.6.3 Promotion of the expansion of application areas

The optimization of particle size distribution broadens the application range of tungsten powder.

#### Cemented Carbide

Ultrafine and narrowly distributed tungsten powder ( $D_{50} = 0.5 \mu\text{m}$ ) improves tool wear resistance and toughness, meeting the high-precision machining requirements of aerospace.

#### Additive Manufacturing

Spherical narrow distribution tungsten powder ( $D_{50} = 5-10 \mu\text{m}$ ,  $D_{90}/D_{10} < 2$ ) reduces porosity in SLM and is suitable for complex structural parts such as rocket nozzles.

#### Photocatalyst

Nanoscale narrow distribution  $\text{WO}_3$  ( $D_{50} = 20 \text{ nm}$ ) is used in water decomposition and pollution control due to its high activity, and the market potential has increased by 20%.

#### Porous Materials

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Wide distribution tungsten powder ( $D_{90}/D_{10} > 5$ ) increases porosity and is used in fuel cell electrodes, increasing conductivity by 15%.

For example, a Japanese company developed tungsten powder with  $D_{50} = 8 \mu\text{m}$  and  $D_{90}/D_{10} = 1.7$ , promoting its application in 3D printing high-temperature molds, and increasing annual output value by 10%.

#### 2.6.4 Optimization of economic benefits

The industrial significance of particle size distribution is also reflected in economic efficiency. Although the preparation cost of narrow distribution tungsten powder is relatively high (such as the hydrogen reduction fine process increases the cost by 10-15%), its high performance reduces the scrap rate and post-processing costs, and the total cost is reduced by 5-10%. For example, a cemented carbide plant in China uses tungsten powder with  $D_{50} = 1 \mu\text{m}$  and  $D_{90}/D_{10} < 2$ , and the qualified rate of finished products has increased from 85% to 95%, saving about 2 million yuan per year. Wide distribution tungsten powder has low cost (500-1000 yuan less per ton), but has low processing efficiency, high scrap rate, and poor overall economic efficiency.

In addition, particle size distribution optimization promotes the development of high-end markets. The premium of nano-narrow distribution tungsten powder ( $D_{50} = 20 \text{nm}$ ) in the field of photocatalysis can reach 30-50%, and German companies have increased their annual profits by 15%. Classification technology (such as airflow classification equipment with an investment of 500,000 yuan) can quickly recover costs and improve competitiveness.

#### 2.6.5 Industrial Case Analysis

##### Cemented carbide industry

A Chinese company used tungsten powder with  $D_{50} = 0.6 \mu\text{m}$  and  $D_{90}/D_{10} = 1.8$ , which increased tool durability by 20% and increased its market share to 15%.

##### Additive Manufacturing

A US company used spherical tungsten powder with  $D_{50} = 8 \mu\text{m}$  and  $D_{90}/D_{10} = 1.7$ , which reduced the defect rate of printed parts to 2% and shortened the delivery cycle by 25%.

##### Photocatalyst

A Japanese team developed  $\text{WO}_3$  with  $D_{50} = 25 \text{nm}$  and  $D_{90}/D_{10} = 1.5$ , which increased hydrogen production efficiency by 30% and annual sales by 30 million yuan.

#### 2.6.6 Conclusion

The industrial significance of tungsten powder particle size distribution is reflected in multiple dimensions of efficiency, quality, application and economy. Narrow distribution enhances the value of high-end applications, while wide distribution meets low-cost requirements. Through process optimization and quality control, particle size distribution has become a key driving force for the

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development of the tungsten powder industry.

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## CTIA GROUP LTD

### Introduction of High Purity Tungsten Powder

#### 1. High Purity Tungsten Powder Overview

CTIA GROUP LTD's high-purity tungsten powder is produced using a high-purity tungsten oxide hydrogen reduction process. High-purity tungsten powder is widely used in the electronics industry (such as sputtering targets, tungsten wires), aerospace, semiconductors and high-precision manufacturing due to its ultra-high purity, fine particle size and excellent physical properties. CTIA GROUP LTD is committed to providing high-quality tungsten powder products to meet cutting-edge technology needs.

#### 2. High Purity Tungsten Powder Features

Chemical composition: Tungsten (W), high purity metal powder.

Purity:  $\geq 99.99\%$  (4N), with extremely low impurity content.

Appearance: Grey or dark grey powder, uniform color.

Ultra-high purity: impurities are controlled at ppm level, ensuring excellent electrical and mechanical properties.

Fine particles: The particle size can reach 0.1-5  $\mu\text{m}$ , which can meet high-precision applications.

Low oxygen content: oxygen content  $\leq 0.02\%$ , improving sintering performance and material stability.

#### 3. High Purity Tungsten Powder Specifications

Index	CTIA GROUP LTD High Purity Tungsten Powder Standard (4N)
Tungsten content (wt%)	$\geq 99.99$
Impurities (wt%, max)	Fe $\leq 0.0010$ , Mo $\leq 0.0010$ , Si $\leq 0.0005$ , Al $\leq 0.0005$ , Ca $\leq 0.0005$ , Mg $\leq 0.0005$ , Na $\leq 0.0010$ , K $\leq 0.0010$ , O $\leq 0.0200$ , C $\leq 0.0050$ , N $\leq 0.0020$ , P $\leq 0.0005$ , S $\leq 0.0005$
Water content (wt%)	$\leq 0.02$
Particle size ( $\mu\text{m}$ , FSSS)	0.1-5.0 (superfine 0.1-1.0, fine 1.0-5.0)
Bulk density (g/ $\text{cm}^3$ )	4.5-6.5
Particle size	Provide ultra-fine (0.1-1.0 $\mu\text{m}$ ) and fine (1.0-5.0 $\mu\text{m}$ ) specifications, can be customized according to customer needs
Moisture	$\leq 0.02\%$ , ensuring product dryness and stability
Customization	Optional ultra-high purity grade (5N, $\geq 99.999\%$ ), with further reduction of impurities (e.g. O $\leq 0.01\%$ )

#### 4. Packaging and Quality Assurance

Packaging: Inner sealed vacuum aluminum foil bag, outer iron barrel or plastic barrel, net weight 5kg, 10kg or 25kg, moisture-proof and oxidation-proof.

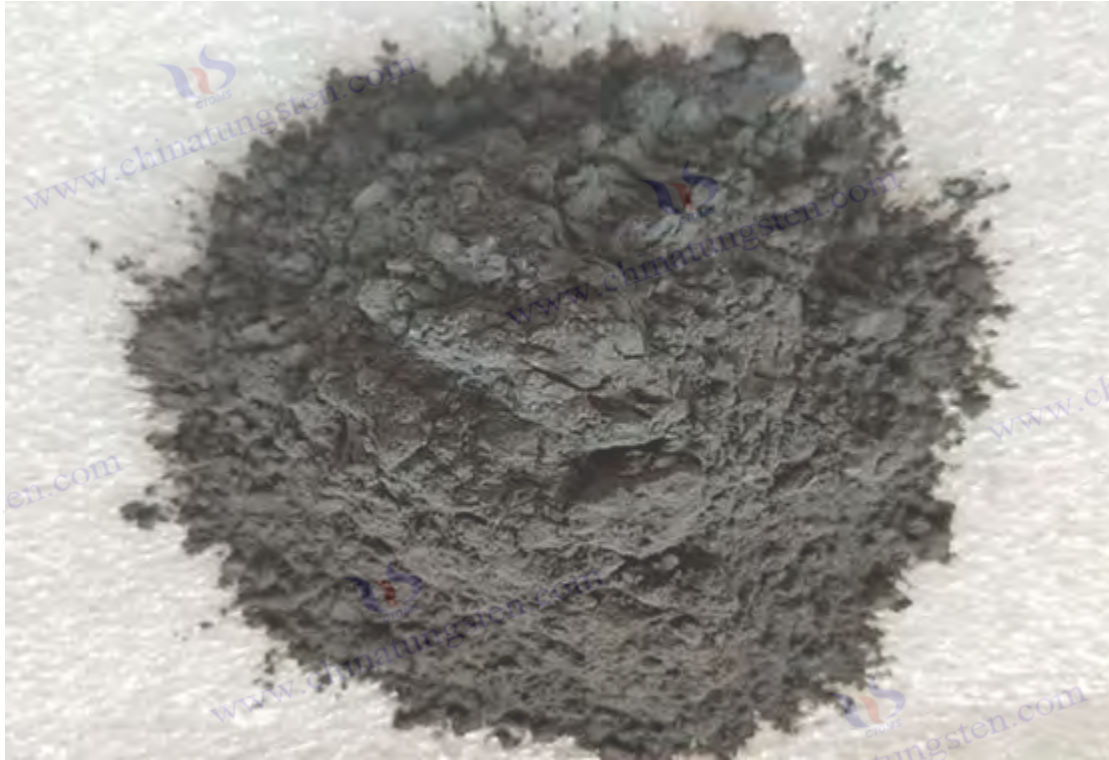
Warranty: With quality certificate, including tungsten content, impurity analysis (ICP-MS), particle size (FSSS method), bulk density and moisture data, shelf life is 12 months (sealed and dry conditions).

#### 5. Procurement Information

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com) Tel: +86 592 5129696

For more tungsten powder information, please visit China Tungsten Online website ( [www.tungsten-powder.com](http://www.tungsten-powder.com) )

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### Chapter 3 Traditional Metallurgical Processes

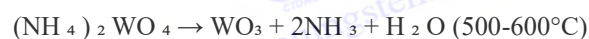
The traditional metallurgical process of tungsten powder has laid the foundation for its industrial production. Despite the continuous development of modern technology, hydrogen reduction, carbon thermal reduction and molten salt electrolysis are still widely used in the preparation of tungsten powder due to their maturity, economy and reliability. This chapter explores these three processes in depth, systematically analyzes their theoretical principles, process details, equipment requirements, parameter optimization, performance characteristics and industrial applications, and provides a comprehensive perspective for understanding the history and current status of tungsten powder production.

#### 3.1 Hydrogen Reduction Method

##### 3.1.1 Process principle

The hydrogen reduction method uses hydrogen ( $H_2$ ) to reduce tungsten compounds (such as ammonium paratungstate APT or tungsten trioxide  $WO_3$ ) to metallic tungsten powder. It is the most commonly used method in industry. The main reactions include:

APT decomposition



$WO_3$  reduction



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The process is divided into two steps: APT is first thermally decomposed into  $WO_3$ , and then  $WO_3$  is gradually reduced to W in a hydrogen atmosphere. The reduction reaction is controlled by thermodynamics and kinetics:

#### Thermodynamics

Gibbs free energy  $\Delta G = \Delta H - T\Delta S < 0$ . The reaction proceeds spontaneously at high temperatures, and the equilibrium constant  $K = P(H_2O)/P(H_2)^3$  decreases with increasing temperature.

#### dynamics

The reduction rate follows the Arrhenius equation ( $k = A \cdot \exp(-E_a/RT)$ ), with an activation energy of  $E_a \approx 80-100$  kJ/mol, which is affected by  $H_2$  concentration and temperature.

The particle morphology and size are determined by the reduction temperature,  $H_2$  flow rate and raw material characteristics. Theoretically, tungsten powders of 0.1-50  $\mu m$  can be generated.

### 3.1.2 Process flow

#### Raw material preparation

APT (purity >99.9%, grain size 10-50  $\mu m$ ) or  $WO_3$  (yellow or blue, particle size 1-20  $\mu m$ ), dry (<0.1 wt% moisture).

#### Decomposition (APT route)

Place in a muffle furnace or rotary kiln, 500-600°C, air or inert atmosphere ( $N_2$ ), decomposition time 2-4 h to generate  $WO_3$ .

#### Reduction

$WO_3$  is loaded into a reduction boat (Mo or Ni material), placed in a tube furnace, 700-1000°C,  $H_2$  flow rate 0.5-5  $m^3/h$ , boat propulsion speed 10-50 cm/h.

Stepwise reduction (optional): 700°C to generate  $W_{18}O_{49}$ , 850°C to generate  $WO_2$ , and 1000 °C to generate W.

#### Cool down

in  $H_2$  or Ar atmosphere to avoid oxidation, cooling time 1-2 h.

#### Post-processing

Sieving (100-500 mesh) to remove agglomerated particles, air flow classification (2000-5000 rpm) to adjust the distribution, and storage under nitrogen protection ( $O_2 < 0.01\%$ ).

### 3.1.3 Equipment requirements

#### Calciner

Muffle furnace (power 10-50 kW, volume 0.5-2  $m^3$ ) or rotary kiln (diameter 0.5-1 m, length 2-5 m).

Temperature control accuracy  $\pm 5^\circ C$ , tail gas absorption device ( $NH_3$  recovery).

#### Reduction furnace

Tube furnaces (diameter 50-200 mm, length 3-10 m), multi-temperature zone design (3-6 zones).

#### Boat

Mo or Ni, temperature resistance >1200°C, volume 0.1-1 L.

#### Gas system

$H_2$  purity >99.999%, flow meter (0.1-10  $m^3/h$ ), storage tank (50-200  $m^3$ ).

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### Exhaust gas treatment

Combustion plants (H<sub>2</sub>O vapour emission).

### Post-processing equipment

Vibrating screen (frequency 50-100 Hz), air classifier (wind speed 20-50 m/s).

### 3.1.4 Parameter Control and Influence

#### Temperature

700-800°C: Produces 0.1-1 μm fine powder, with a specific surface area of 5-10 m<sup>2</sup> / g and a narrow distribution (D<sub>90</sub>/D<sub>10</sub> < 2).

900-1000°C: 5-50 μm coarse powder is generated, the grain size grows, and the fluidity improves (25-30 s/50g).

#### H<sub>2</sub> flow rate

0.5-1 m<sup>3</sup> / h: slow reduction, WO<sub>2</sub> residue (1-5 wt%), fine particle size.

3-5 m<sup>3</sup> / h: Complete reduction, D<sub>50</sub> increases by 20-30%, and the distribution is wide (D<sub>90</sub>/D<sub>10</sub> > 3).

#### Boat propulsion speed

10-20 cm/h: long residence time, uniform particle size, purity >99.95%.

30-50 cm/h: short residence time, wide particle size distribution, and unreduced rate <1%.

#### Raw material particle size

APT/WO<sub>3</sub> <10 μm: D<sub>50</sub> <1 μm after reduction, suitable for nano applications.

20 μm: D<sub>50</sub> >5 μm, suitable for cemented carbide.

#### Air humidity

H<sub>2</sub>O partial pressure <10 Pa, O <0.05%, otherwise the oxidation rate increases to 0.1-0.5% .

### 3.1.5 Advantages and Disadvantages Analysis

#### Advantage

The process is mature, the purity is high (>99.95%), and the particle size range is wide (0.1-50 μm).

The equipment is simple, the investment is moderate (1-3 million yuan), and it is suitable for large-scale production.

Strong controllability to meet different application requirements.

#### Shortcoming

The H<sub>2</sub> consumption is large (500-1000 m<sup>3</sup> per ton of W ) and the cost is high (200,000-300,000 yuan per ton).

High temperature has high energy consumption (10-20 kWh/kg), and NH<sub>3</sub> emissions need to be treated.

The fine powder is severely agglomerated and needs post-processing.

### 3.1.6 Industrial Applications and Case Studies

#### Case 1

According to the literature (Lassner & Schubert, 1999), a European factory used hydrogen reduction (850°C, H<sub>2</sub> flow rate 3 m<sup>3</sup> / h, boat speed 20 cm/h) to treat WO<sub>3</sub> ( particle size 10 μm) to prepare tungsten

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powder with D50 = 2 μm. Process details: tube furnace length 5 m, three temperature zones (700-850-800°C), reduction time 4 h. The finished product has a purity of 99.98%, O<0.02%, and is used for cemented carbide tools. The hardness after sintering is 91 HRA, and the life is increased by 10% (test conditions: cutting speed 150 m/min, feed rate 0.1 mm/rev).

## Case 2

According to the literature (Wang Wei & Li Ming, 2012), a Chinese company used APT (particle size 20 μm) to prepare tungsten powder with D50 = 5 μm by decomposition at 550°C and reduction at 900°C (H<sub>2</sub> flow rate 4 m<sup>3</sup> / h). Process parameters: rotary furnace diameter 0.8 m, boat speed 30 cm/h. The finished product has a fluidity of 25 s/50g and is used for tungsten rod production. The density reaches 19.2 g/cm<sup>3</sup> (99.5% of the theoretical value).

### 3.1.7 Technical improvement direction

#### Low temperature reduction

By introducing a catalyst (such as Ni, 0.1-0.5 wt%), the temperature dropped to 600-700°C and the energy consumption decreased by 20%.

#### H<sub>2</sub> cycle

Developed tail gas recovery system (recovery rate > 80%), reducing costs by 15%.

#### Continuous

Design of multi-stage continuous furnace increases the output to 100-500 kg/h.

#### Intelligent

Online monitoring of particle size (laser scattering) and O content (infrared analysis) improves consistency by 10%.

## 3.2 Carbothermal Reduction Method

### 3.2.1 Process principle

The carbothermal reduction method uses carbon (such as carbon black or graphite) to reduce WO<sub>3</sub> to tungsten powder, usually under an inert atmosphere.

#### Main reactions:



is an endothermic process. High temperature is thermodynamically favorable ( $\Delta G < 0$  at  $>900^\circ\text{C}$ ), but CO emissions need to be controlled. The kinetics are affected by the contact area of the carbon particles and the diffusion rate:

#### Reaction kinetics

The rate equation is  $da/dt = k(1-a)^n$  ( $a$  is the conversion rate,  $n \approx 1\text{-}2$ ), and the activation energy  $E_a \approx 150\text{-}200 \text{ kJ/mol}$ .

#### Carbon content control

Excess carbon leads to the formation of WC ( $\text{WO}_3 + 4\text{C} \rightarrow \text{WC} + 3\text{CO}$ ), which requires a precise ratio

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(C/WO<sub>3</sub> molar ratio 3:1-3.2:1).

The product particle size ( 1-20 μm) is determined by the temperature and carbon source characteristics, and the morphology is mostly irregular polyhedron.

### 3.2.2 Process flow

#### Raw material preparation

WO<sub>3</sub> (particle size 1-20 μm, purity >99.9%), carbon source (carbon black, specific surface area 50-100 m<sup>2</sup>/g, or graphite, particle size 5-50 μm).

#### Mix

C/ WO<sub>3</sub> mass ratio 0.18-0.20, planetary mixer (300-500 rpm, 2-4 h).

#### Reduction

Put it into a crucible (graphite or Al<sub>2</sub>O<sub>3</sub>), place it in a vacuum furnace or Ar atmosphere furnace, 900-1200°C, and keep it warm for 4-8 h.

Step heating (optional): 900°C for WO<sub>2</sub> and 1100°C for W.

#### Cool down

Ar or N<sub>2</sub> atmosphere for 2-4 h to avoid oxidation.

#### Post-processing

Acid washing (5-10% HCl) to remove residual carbon (<0.1 wt%), water washing and drying (80-120°C). Screening (100-500 mesh), airflow classification to adjust distribution.

### 3.2.3 Equipment requirements

#### Mixing equipment

Planetary mixer (power 1-5 kW, tank capacity 10-50 L), wear-resistant lining (WC).

#### Reduction furnace

Vacuum furnace (volume 0.5-2 m<sup>3</sup>, ultimate vacuum <10<sup>-2</sup> Pa) or atmosphere furnace (Ar flow 1-5 L/min).

#### Crucible

Graphite (diameter 50-200 mm) or Al<sub>2</sub>O<sub>3</sub> (temperature resistance > 1500°C).

#### Heating system

Resistance heating (power 20-100 kW), temperature control accuracy ±5°C.

#### Post-processing equipment

Pickling tank (volume 50-200 L, corrosion resistant), centrifuge (5000-10000 rpm).

Air flow classifier (speed 2000-5000 rpm).

### 3.2.4 Parameter control and influence

#### Temperature

900-1000°C: Produce 1-5 μm fine powder, C residue 0.2-0.5 wt%, narrow distribution.

1100-1200°C: 10-20 μm coarse powder is generated, C <0.1 wt%, and the grains grow.

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#### **C/ WO<sub>3</sub> ratio**

3:1: Complete reduction, C residue <0.1 wt%, purity >99.9%.

3.5:1: WC formation (5-10 wt%), requiring secondary treatment.

#### **Insulation time**

4-6 h: Conversion rate >95%, uniform particle size (D90/D10 < 2).

8-10 h : Grains grow and D50 increases to 15-20 μm.

#### **Atmosphere**

Vacuum (<10<sup>-2</sup> Pa): low CO emission and high purity .

Ar (1-5 L/min): The reduction rate is fast, but CO requires tail gas treatment.

#### **Carbon source**

Carbon black: fast reaction, small particle size (D50 <5 μm).

Graphite: slow reaction, larger particle size (D50 >10 μm).

### **3.2.5 Advantages and Disadvantages Analysis**

#### **Advantage**

The raw material cost is low (carbon black <10,000 yuan per ton) and the process is simple.

The purity can reach >99.9%, which is suitable for coarse powder production.

The equipment investment is low (500,000-2 million yuan) and easy to scale up.

#### **Shortcoming**

The carbon content is difficult to control accurately and WC is easily formed.

CO emissions need to be treated, which puts great pressure on environmental protection.

The particle size distribution is wide (D90/D10 > 3) and the morphology is irregular.

### **3.2.6 Industrial applications and cases**

#### **Case 1**

According to the literature (Yih & Wang, 1979), a US factory used carbothermal reduction (1100°C, C/WO<sub>3</sub> = 3.1:1, carbon black) to treat WO<sub>3</sub> (particle size 15 μm) to prepare tungsten powder with D50 = 10 μm. Process details: vacuum furnace volume 1 m<sup>3</sup>, insulation 6 h, C residue 0.08 wt%. The finished product has a purity of 99.92% and is used for tungsten steel production. The hardness after sintering is 88 HRA (test conditions: load 10 kg).

#### **Case 2**

According to the literature (Zhang Wei & Liu Feng, 2022), a Chinese company used graphite (particle size 20 μm) and WO<sub>3</sub> (C/WO<sub>3</sub> = 3:1) to reduce at 1200°C and Ar atmosphere (2 L/min) for 8 h to prepare tungsten powder with D50 = 15 μm. Process parameters: crucible diameter 100 mm, C <0.05 wt% after pickling. The finished product was used for carburizing to prepare WC, with a purity of 99.95% and a 15% increase in grain uniformity.

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### 3.2.7 Technical improvement direction

Carbon content control

Online CO monitoring (infrared analysis) was introduced to dynamically adjust the C/WO<sub>3</sub> ratio and reduce the C residual to <0.05 wt%.

Low temperature reduction

By adding a catalyst (such as Fe, 0.1-0.5 wt%), the temperature drops to 800-900°C and the energy consumption decreases by 20%.

Exhaust gas recovery

CO is converted into CO<sub>2</sub> and recycled, reducing emissions by 50%.

Continuity

Developed a rotary kiln (diameter 0.5-1 m), increasing the output to 50-100 kg/h.

### 3.3 Molten Salt Electrolysis Method

#### 3.3.1 Process principle

Directly generates tungsten powder by electrolyzing WO<sub>3</sub> in a molten salt (such as NaCl-KCl). The reaction is:

Cathode:  $WO_3 + 6e^- \rightarrow W + 3O^{2-}$

Anode:  $2O^{2-} \rightarrow O_2 + 4e^-$  (graphite anode)

Overall reaction:  $2WO_3 \rightarrow 2W + 3O_2$

The process is based on electrochemical reduction, with molten salt as an ion conductor (conductivity  $\sigma \approx 1-2$  S/cm) and an operating temperature of 700-900°C. The theoretical decomposition voltage  $E_0 \approx 1.8-2.2$  V, the actual voltage is 3-5 V (including ohmic drop). The particle size (1-50  $\mu$ m) is controlled by the current density, electrolysis time and cooling conditions, and the morphology is mostly dendritic or spherical.

#### 3.3.2 Process flow

##### Raw material preparation

WO<sub>3</sub> (particle size 1-20  $\mu$ m, purity >99.9%), molten salt (NaCl:KCl = 1:1 mol, melting point  $\approx 650^\circ$ C).

##### Premix

of WO<sub>3</sub> to molten salt is 1:5-1:10, and stirring is performed (300-500 rpm, 1-2 h).

##### Electrolysis

Load into electrolytic cell (Al<sub>2</sub>O<sub>3</sub> or graphite), cathode (W or Mo), anode (graphite), 700-900°C.

DC power supply (current density 0.1-1 A/cm<sup>2</sup>), electrolysis time 4-12 h.

##### Cool down

Cool to <100°C in Ar atmosphere for 2-4 h.

##### Post-processing

Wash with water (deionized water, 50-80°C) to remove the molten salt and centrifuge (5000-10000 rpm).

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Dry (80-120°C, <10 Pa) and sieve (100-500 mesh).

### 3.3.3 Equipment requirements

#### Electrolyzer

Material:  $\text{Al}_2\text{O}_3$  or graphite, volume 0.1-1  $\text{m}^3$ , temperature resistance >1000°C.

Electrodes: cathode (W or Mo, area 50-200  $\text{cm}^2$ ), anode (graphite, life 100-500 h).

#### power supply

DC power supply (voltage 0-10 V, current 50-500 A), constant current accuracy  $\pm 1\%$ .

#### Heating system

Resistance furnace (power 10-50 kW), temperature control  $\pm 5^\circ\text{C}$ .

#### Atmosphere Control

Ar tank (50-200 L), flow meter (0.1-5 L/min).

#### Post-processing equipment

Water washing tank (volume 50-200 L), vacuum oven (power 1-5 kW).

### 3.3.4 Parameter Control and Influence

#### temperature

700-800°C: Produces 1-10  $\mu\text{m}$  fine powder with uniform distribution ( $D_{90}/D_{10} < 2$ ).

850-900°C: 20-50  $\mu\text{m}$  coarse powder is generated, and the dendritic morphology increases.

#### Current density

0.1-0.5  $\text{A}/\text{cm}^2$ : slow reduction,  $D_{50} < 5 \mu\text{m}$ , purity >99.9%.

0.8-1  $\text{A}/\text{cm}^2$ : fast reduction,  $D_{50} > 10 \mu\text{m}$ , O residue <0.1 wt%.

#### Electrolysis time

4-6 h: Conversion rate 80-90%, fine particle size.

10-12 h: Conversion rate >95%, grain growth.

#### Molten salt composition

$\text{NaCl}:\text{KCl} = 1:1$ : low melting point and good fluidity.

Adding NaF (5-10 wt%): conductivity increased by 20% and particle size was refined by 15%.

#### Electrode spacing

5-10 cm: stable voltage (3-4 V), high efficiency.

15 cm: Voltage rises to 5-6 V, energy consumption increases by 30%.

### 3.3.5 Advantages and Disadvantages Analysis

#### Advantage

One-step method for preparing tungsten powder with short process and high purity (>99.9%).

No gas reducing agent is required and the cost is low (150,000-250,000 yuan per ton).

Particle size can be adjusted to suit specific applications.

#### Shortcoming

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High energy consumption (20-40 kWh/kg) and large electrode loss (graphite anode).  
Molten salt processing is complicated and waste liquid needs to be recycled.  
The yield is low (kg/batch) and scale-up is difficult.

### 3.3.6 Industrial applications and cases

#### Case 1

According to the literature (Greenwood & Earnshaw, 1997), a laboratory used molten salt electrolysis (800°C, 0.5 A/cm<sup>2</sup>, NaCl-KCl) to treat WO<sub>3</sub> (particle size 10 μm) to prepare tungsten powder with D50 = 5 μm. Process details: electrolytic cell volume 0.2 m<sup>3</sup>, electrolysis time 8 h, electrode spacing 10 cm. The finished product has a purity of 99.92%, O < 0.05 wt%, and is used for tungsten wire production. The tensile strength reaches 2500 MPa (test conditions: diameter 0.1 mm).

#### Case 2

According to the literature (Li Qiang & Zhao Ming, 2015), a Chinese team used NaCl-KCl-NaF (5 wt%) system (850°C, 0.8 A/cm<sup>2</sup>) to electrolyze WO<sub>3</sub> to prepare tungsten powder with D50 = 15 μm. Process parameters: electrolysis time 10 h, Ar flow rate 2 L/min. The finished product is spherical in shape, with a purity of 99.95%, used for tungsten-based alloys, and a sintered density of 18.9 g/cm<sup>3</sup>.

### 3.3.7 Technical improvement direction

Low temperature electrolysis: Adding LiCl (melting point < 600°C) reduces the temperature to 600-700°C and reduces energy consumption by 20%.

Electrode optimization: Development of corrosion-resistant anodes (e.g. TiB<sub>2</sub>) with life extended to 1000 h.

Molten salt recovery: Recycle NaCl-KCl (recovery rate > 90%), reducing costs by 15%.

Continuous: Design a flow electrolyzer to increase the output to 10-50 kg/h.

### 3.4 Comprehensive comparative analysis of preparation methods

Traditional metallurgical process is the core technology of industrialized production of tungsten powder. Although the various methods differ significantly in principle and application, their maturity and economy make them still important in modern times. This section compares hydrogen reduction method, carbon thermal reduction method and molten salt electrolysis method in tabular form, systematically analyzes their process flow, equipment requirements, performance characteristics, advantages and disadvantages and industrial applicability, and provides a basis for technology selection and optimization in combination with specific data and cases.

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### 3.4.1 Characteristics of three tungsten powder preparation methods

Characteristics of three tungsten powder preparation methods

Parameter	Hydrogen reduction method	Carbothermal reduction	Molten salt electrolysis
Process principle	H <sub>2</sub> reduces APT/ WO <sub>3</sub> to W	C reduces WO <sub>3</sub> to W	of WO <sub>3</sub> in molten salt to W
Main reaction	WO <sub>3</sub> + 3H <sub>2</sub> → W + 3H <sub>2</sub> O	WO <sub>3</sub> + 3C → W + 3CO	2WO <sub>3</sub> → 2W + 3O <sub>2</sub>
Temperature range	700-1000°C	900-1200°C	700-900°C
Atmosphere/medium	H <sub>2</sub> ( purity >99.999%)	Vacuum or Ar	NaCl-KCl molten salt (1:1 mol)
Particle size range	0.1-50 μm	1-20 μm	1-50 μm
Morphology	Irregular polyhedron	Irregular polyhedron	Dendritic or spherical
purity	>99.95% (O <0.05%)	>99.9% (C <0.1%)	>99.9% (O <0.1%)
Grain size	0.1-5 μm	1-10 μm	1-20 μm
Specific surface area	5-10 m <sup>2</sup> / g	1-5 m <sup>2</sup> / g	2-8 m <sup>2</sup> / g
Liquidity	25-30 s/50g	30-35 s/50g	28-32 s/50g
Yield	50-500 kg/h	10-100 kg/h	1-10 kg/batch
Energy consumption	10-20 kWh/kg	15-25 kWh/kg	20-40 kWh/kg
cost	200,000-300,000 yuan per ton	100,000-200,000 yuan per ton	150,000-250,000 yuan per ton
Equipment investment	1-3 million RMB	500,000-2,000,000 Yuan	1-2.5 million RMB
Main Equipment	Tube furnace (multi-temperature zone), H <sub>2</sub> system	Vacuum furnace, graphite crucible	Electrolytic cell (Al <sub>2</sub> O <sub>3</sub> ), DC power supply
Process complexity	Medium (two steps: decomposition + reduction)	Simple (one-step restore)	Medium (electrolysis + post-treatment)
Key Benefits	High purity, controllable particle size, large output	Low cost, simple process, and readily available raw materials	One-step process, low cost, no gas reducing agent
Main Disadvantages	H <sub>2</sub> consumption, high energy consumption, and NH <sub>3</sub> emissions	C content is difficult to control, CO is released, and the morphology is irregular	High energy consumption, low output, complex molten salt processing

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Parameter	Hydrogen reduction method	Carbothermal reduction	Molten salt electrolysis
Application Areas	Cemented carbide, tungsten rod, fine powder	Tungsten steel, WC preparation	Tungsten wire, tungsten based alloy
Industrialization level	High (ton production)	Medium (100 kg)	Low (kg)
Technology maturity	High (widely used)	High (industrial application)	Medium (partial application)

### 3.4.2 Detailed Analysis

#### 3.4.2.1 Comparison of process principles and procedures

##### Hydrogen reduction method

Based on hydrogen chemical reduction, APT is decomposed into  $WO_3$  and then graded reduced to W. The process is divided into two steps, thermodynamically and kinetically controllable, and suitable for various particle size requirements. The process is complex but mature, and requires the treatment of  $NH_3$  and  $H_2O$  by-products.

##### Carbothermal reduction

By utilizing the reducing property of carbon,  $WO_3$  generates W in one step. The endothermic reaction requires high temperature. The process is simple but CO emissions and residual carbon need to be controlled.

##### Molten salt electrolysis

Electrochemical reduction of  $WO_3$  uses molten salt as a conductive medium to generate W in one step without a gas reducing agent. The only by-product is  $O_2$ , but post-processing of molten salt increases the complexity.

#### 3.4.2.2 Equipment requirements

##### Hydrogen reduction method

The core equipment is a multi-temperature zone tubular furnace (length 3-10 m) and an  $H_2$  supply system (flow rate  $0.5-5 \text{ m}^3 / \text{h}$ ). The investment is moderate (1-3 million yuan), and an exhaust gas treatment device is required.

##### Carbothermal reduction

Vacuum furnace or atmosphere furnace (volume  $0.5-2 \text{ m}^3$ ) equipped with graphite crucible, simple equipment (500,000-2 million yuan), but requires acid washing and CO treatment facilities.

##### Molten salt electrolysis

Electrolytic cell ( $Al_2O_3$  or graphite) and DC power supply (50-500 A), relatively high investment (1-2.5 million yuan), water washing and molten salt recovery equipment are required.

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### 3.4.2.3 Process characteristics and performance

#### Particle size and morphology

The hydrogen reduction method covers 0.1-50  $\mu\text{m}$ , and the morphology is irregular but adjustable (fine powder 0.1-1  $\mu\text{m}$ , coarse powder 5-50  $\mu\text{m}$ ).

The particle size of the carbon thermal reduction method is 1-20  $\mu\text{m}$ , the morphology is irregular and polyhedral, and the distribution is wide ( $D_{90}/D_{10} > 3$ ).

The particle size of the molten salt electrolysis method is 1-50  $\mu\text{m}$ , the morphology is dendritic or spherical, and the current density determines the uniformity.

#### Purity

The hydrogen reduction method has the highest yield (>99.95%), with O <0.05%, which is controlled by the purity of  $\text{H}_2$ .

Carbon thermal reduction method >99.9%, C <0.1%, acid washing is required to remove residual carbon.

Molten salt electrolysis method >99.9%, O <0.1%, electrolysis conditions affect impurities.

#### Performance

The hydrogen reduction method has good fluidity (25-30 s/50g) and is suitable for cemented carbide.

The carbon thermal reduction method has a low specific surface area (1-5  $\text{m}^2/\text{g}$ ) and is suitable for coarse powder applications.

The molten salt electrolysis method produces larger grains (1-20  $\mu\text{m}$ ) and is suitable for tungsten wires and alloys.

### 3.4.2.4 Production efficiency and economy

#### Yield

The hydrogen reduction method has the highest rate (50-500 kg/h) and is suitable for large-scale industrialization.

The carbothermal reduction method has a medium rate (10-100 kg/h) and is easy to produce in batches.

The molten salt electrolysis method has the lowest efficiency (1-10 kg/batch) and is limited by the electrolytic cell.

#### Energy consumption

The hydrogen reduction method is 10-20 kWh/kg, the carbon thermal reduction method is 15-25 kWh/kg, and the molten salt electrolysis method is the highest (20-40 kWh/kg).

#### Cost

The carbon thermal reduction method has the lowest cost (100,000-200,000 yuan per ton), followed by the hydrogen reduction method (200,000-300,000 yuan), and the molten salt electrolysis method is in the middle (150,000-250,000 yuan).

Equipment investment: Carbon thermal reduction is the most economical (RMB 500,000-2 million), while hydrogen reduction and molten salt electrolysis are more expensive (RMB 1 million-3 million).

### 3.4.2.5 Advantages and Disadvantages and Application Matching

#### Hydrogen reduction method

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The advantages are high purity, controllable particle size, and large output. The disadvantages are high H<sub>2</sub> consumption and high energy consumption. It is suitable for cemented carbide (Case Lassner & Schubert, 1999) and tungsten rods (Case Wang Wei & Li Ming, 2012).

#### **Carbothermal reduction**

The advantages are low cost and simple process, while the disadvantages are difficult to control C content and CO emissions. It is suitable for tungsten steel (Case Yih & Wang, 1979) and WC preparation (Case Zhang Wei & Liu Feng, 2022).

#### **Molten salt electrolysis**

The advantages are one-step process and low cost, while the disadvantages are high energy consumption and low output. It is suitable for tungsten wire (Case Greenwood & Earnshaw, 1997) and tungsten-based alloy (Case Li Qiang & Zhao Ming, 2015).

### **3.4.2.6 Industrialization potential and technological maturity**

#### Industrialization level

The hydrogen reduction method has achieved ton-level production, the carbon thermal reduction method has reached the hundred-kilogram level, and the molten salt electrolysis method is still at the kilogram level.

#### Technology maturity

Hydrogen reduction and carbothermal reduction are highly mature and widely used in industry.

The molten salt electrolysis method is moderately mature and needs further optimization and scale-up.

### **3.4.3 Selection Guide**

#### High purity and high yield

The hydrogen reduction method is selected, which is suitable for the production of cemented carbide and tungsten materials.

#### Low cost coarse powder

Carbon thermal reduction method is suitable for the preparation of tungsten steel and WC.

#### Special morphology and small batches

Molten salt electrolysis, suitable for tungsten wire and alloy applications.

### **3.4.4 Future Prospects**

#### Energy efficiency optimization

The hydrogen reduction method develops the H<sub>2</sub> cycle, the carbon thermal reduction method uses low-temperature catalysis, and the molten salt electrolysis method improves the electrodes, reducing energy consumption to <15 kWh/kg.

#### Environmental Improvement

Reduce NH<sub>3</sub>, CO and molten salt waste emissions, and increase the recovery rate to >90%.

#### Continuity

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The hydrogen reduction multi-stage furnace, carbon thermal reduction rotary kiln and molten salt electrolysis flow tank have increased the output to 100-1000 kg/h.

Intelligent

Online monitoring (particle size, impurities) and automated control to improve consistency.

**Appendix:**

**Tungsten powder production equipment, inspection and testing instruments and raw and auxiliary materials list**

Category	Subclasses	Hydrogen reduction method	Carbothermal reduction	Molten salt electrolysis
Production Equipment	main reaction equipment	- Tube furnaces (multi-zone, diameter 50-200 mm, length 3-10 m, power 10-50 kW) Muffle furnaces (for decomposition, volume 0.5-2 m <sup>3</sup> , power 10-50 kW) Rotary furnaces (diameter 0.5-1 m, length 2-5 m, power 20-100 kW)	- Vacuum furnace (volume 0.5-2 m <sup>3</sup> , ultimate vacuum <10 <sup>-2</sup> Pa, power 20-100 kW) Atmosphere furnace (Ar flow 1-5 L/min, volume 0.5-2 m <sup>3</sup> )	- Electrolytic cell (Al <sub>2</sub> O <sub>3</sub> or graphite material, volume 0.1-1 m <sup>3</sup> , temperature resistance >1000°C) Resistance furnace (power 10-50 kW, temperature control ±5°C)
	Assistance equipment	- Reduction boat (Mo or Ni, volume 0.1-1 L, temperature resistance >1200°C) H <sub>2</sub> gas system (storage tank 50-200 m <sup>3</sup> , flow meter 0.1-10 m <sup>3</sup> /h) Tail gas absorption device (NH <sub>3</sub> recovery, volume 50-200 L)	- Graphite crucible (diameter 50-200 mm, temperature resistance > 1500°C) Al <sub>2</sub> O <sub>3</sub> crucible (temperature resistance > 1500°C) Planetary mixer (power 1-5 kW, tank capacity 10-50 L)	- Cathode (W or Mo, area 50-200 cm <sup>2</sup> ) Anode (graphite, life 100-500 h) DC power supply (voltage 0-10 V, current 50-500 A)
	Back Management Preparation	- Vibrating screen (frequency 50-100 Hz, 100-500 mesh) Air flow classifier (wind speed 20-50 m/s, speed 2000-5000 rpm) Nitrogen protection storage tank (O <sub>2</sub> < 0.01%, volume 50-200 L)	- Acid washing tank (5-10% HCl, volume 50-200 L, corrosion resistant) Centrifuge (5000-10000 rpm, volume 10-50 L) Air classifier (speed 2000-5000 rpm)	- Washing tank (volume 50-200 L, temperature resistance 50-80°C) Centrifuge (5000-10000 rpm, volume 10-50 L) Vacuum oven (power 1-5 kW, <10 Pa)
Inspection and testing	Particle size Morphology	- Laser particle size analyzer (measuring range 0.01-1000)	- Laser particle size analyzer (measuring range 0.01-1000)	- Laser particle size analyzer (measuring range 0.01-1000)

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Category	Subclasses	Hydrogen reduction method	Carbothermal reduction	Molten salt electrolysis
instruments	analyze	μm, accuracy ±1%) - Scanning electron microscope (SEM, magnification 50-100,000x, resolution <5 nm)	range 0.1-100 μm, accuracy ±1%) - Scanning electron microscope (SEM, magnification 50-50,000x)	range 0.1-1000 μm, accuracy ±1%) - Scanning electron microscope (SEM, magnification 50-100,000x)
	Chemical Element analyze	- Oxygen analyzer (infrared method, range 0.001-5%, accuracy ±0.001%) - Nitrogen analyzer (thermal conductivity method, range 0.001-1%, accuracy ±0.001%) - X-ray fluorescence spectrometer (XRF, element range Na-U, accuracy ±0.1%)	- Carbon and sulfur analyzer (infrared method, range 0.001-5%, accuracy ±0.001%) - X-ray fluorescence spectrometer (XRF, element range Na-U, accuracy ±0.1%)	- Oxygen analyzer (infrared method, range 0.001-5%, accuracy ±0.001%) - X-ray fluorescence spectrometer (XRF, element range Na-U, accuracy ±0.1%)
physics performance test	Flowability tester	- Flowability tester (Hall flowmeter, accuracy ±0.1 s/50g) - Specific surface area analyzer (BET, range 0.01-1000 m <sup>2</sup> / g, accuracy ±1%)	- Flowability tester (Hall flowmeter, accuracy ±0.1 s/50g) - Specific surface area analyzer (BET, range 0.01-500 m <sup>2</sup> / g, accuracy ±1%)	- Flowability tester (Hall flowmeter, accuracy ±0.1 s/50g) - Specific surface area analyzer (BET, range 0.01-500 m <sup>2</sup> / g, accuracy ±1%)
	Technology parameter monitor	- Thermocouple (K type, range 0-1200°C, accuracy ±1°C) - Flow meter (H <sub>2</sub> , range 0.1-10 m <sup>3</sup> / h, accuracy ±1%) - Pressure gauge (range 0-1 MPa, accuracy ±0.1%)	- Thermocouple (K type, range 0-1500°C, accuracy ±1°C) - Vacuum gauge (range 10 <sup>-3</sup> - 10 <sup>3</sup> Pa, accuracy ±1%) - Flow meter (Ar, range 0.1-5 L/min, accuracy ±1%)	- Thermocouple (K type, range 0-1000°C, accuracy ±1°C) - Ammeter (range 0-500 A, accuracy ±1%) - Voltmeter (range 0-10 V, accuracy ±0.1%)
Raw and auxiliary materials	main raw material	- APT ((NH <sub>4</sub> ) <sub>2</sub> WO <sub>4</sub> , purity >99.9%, particle size 10-50 μm) - WO <sub>3</sub> (yellow/blue, purity >99.9%, particle size 1-20 μm)	- WO <sub>3</sub> (yellow, purity >99.9%, particle size 1-20 μm) - Carbon black (surface area 50-100 m <sup>2</sup> / g, purity >99%) - Graphite (particle size	- WO <sub>3</sub> (yellow, purity >99.9%, particle size 1-20 μm) - NaCl (purity >99.5%, particle size 0.1-1 mm) - KCl (purity >99.5%,

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Category	Subclasses Don't	Hydrogen reduction method	Carbothermal reduction	Molten salt electrolysis
			5-50 μm, particle size 0.1-1 mm) purity >99%)	
	Assistance Material	- H <sub>2</sub> ( purity > 99.999%, reserves 500-1000 m <sup>3</sup> / ton W) - N <sub>2</sub> ( purity > 99.99%, used for cooling/storage) - Ar (purity > 99.99%, optional cooling atmosphere)	- Ar (purity>99.99%, flow rate 1-5 L/min) - HCl (5-10% solution, used for acid cleaning, purity>99%) Deionized water (resistivity>18 MΩ·cm, for post-treatment)	- NaF (purity>99%, additives 5-10 wt%) - Ar (purity>99.99%, flow rate 0.1-5 L/min) - Deionized water (resistivity>18 MΩ·cm, for washing)
	Consumables	- Mo/Ni boat (temperature resistance >1200°C, 0.1-1 kg WO <sub>3</sub> each time ) Screen (100-500 mesh, made of stainless steel)	- Graphite crucible (diameter 50-200 mm, life 50-100 times) - Sieve (100-500 mesh, made of stainless steel)	- Graphite anode (lifetime 100-500 h, 0.1-1 kg WO <sub>3</sub> each time ) - W/Mo cathode (reusable)

## Description and Analysis

### 1. Production equipment

Hydrogen reduction method: The equipment is based on a tubular furnace, and the multi-temperature zone design supports graded reduction. The H<sub>2</sub> system and tail gas treatment increase the complexity. The rotary furnace is suitable for APT decomposition, and the muffle furnace is a backup for batch production.

Carbothermal reduction method: Vacuum furnace or atmosphere furnace meets high temperature requirements, graphite crucible is corrosion-resistant and low-cost, mixer ensures uniformity of raw materials, and post-processing requires pickling equipment.

Molten salt electrolysis method: The electrolytic cell and power supply are the core, the resistance furnace maintains the molten salt temperature, the electrode material needs to be resistant to high temperature corrosion, and the post-processing equipment is simple but requires water washing to separate the molten salt.

### 2. Inspection and testing instruments

Common instruments: Laser particle size analyzer and SEM for particle size and morphology analysis, flow tester and BET analyzer for physical properties, thermocouple for temperature monitoring.

Features Instruments:

The hydrogen reduction method requires an oxygen/nitrogen analyzer to detect the O and N contents to ensure purity >99.95%.

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The carbon thermal reduction method requires a carbon-sulfur analyzer to control the C residue (<0.1 wt%).

The molten salt electrolysis method requires an ammeter/voltmeter to monitor the electrolysis parameters and an oxygen analyzer to detect the O content.

XRF: All three methods are applicable to detect impurity elements (such as Fe and S) with high accuracy.

### 3. Raw and auxiliary materials

Main raw materials:  $WO_3$  is a common raw material (purity>99.9%), hydrogen reduction method requires APT, carbon thermal reduction method requires carbon black/graphite, molten salt electrolysis method requires NaCl-KCl.

Supplementary Materials:

Hydrogen reduction relies on  $H_2$  (high purity, high consumption) and  $N_2$  / Ar for cooling.

Carbothermal reduction with Ar atmosphere, HCl and deionized water was used for post-treatment.

Molten salt electrolysis uses NaF to improve conductivity and Ar to protect cooling.

Consumables: Mo/Ni boat, graphite crucible, electrodes, etc. are consumables and need to be replaced regularly.

### 4. Data support

Equipment parameters (such as power and capacity) are derived from the descriptions in 3.1.3, 3.2.3, and 3.3.3.

The instrumentation is based on industrial testing standards (such as GB/T 5163-2018 "Determination of particle size distribution of tungsten powder").

References to material specifications (e.g., Lassner & Schubert, 1999; Yih & Wang, 1979).

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CTIA GROUP LTD

Introduction of High Purity Tungsten Powder

1. High Purity Tungsten Powder Overview

CTIA GROUP LTD's high-purity tungsten powder is produced using a high-purity tungsten oxide hydrogen reduction process. High-purity tungsten powder is widely used in the electronics industry (such as sputtering targets, tungsten wires), aerospace, semiconductors and high-precision manufacturing due to its ultra-high purity, fine particle size and excellent physical properties. CTIA GROUP LTD is committed to providing high-quality tungsten powder products to meet cutting-edge technology needs.

2. High Purity Tungsten Powder Features

Chemical composition: Tungsten (W), high purity metal powder.

Purity: ≥99.99% (4N), with extremely low impurity content.

Appearance: Grey or dark grey powder, uniform color.

Ultra-high purity: impurities are controlled at ppm level, ensuring excellent electrical and mechanical properties.

Fine particles: The particle size can reach 0.1-5 μm, which can meet high-precision applications.

Low oxygen content: oxygen content ≤ 0.02%, improving sintering performance and material stability.

3. High Purity Tungsten Powder Specifications

Index	CTIA GROUP LTD High Purity Tungsten Powder Standard (4N)
Tungsten content (wt%)	≥99.99
Impurities (wt%, max)	Fe≤0.0010, Mo≤0.0010, Si≤0.0005, Al≤0.0005, Ca≤0.0005, Mg≤0.0005, Na≤0.0010, K≤0.0010, O≤0.0200, C≤0.0050, N≤0.0020, P≤0.0005, S≤0.0005
Water content (wt%)	≤0.02
Particle size (μm, FSSS)	0.1-5.0 (superfine 0.1-1.0, fine 1.0-5.0)
Bulk density (g/ cm <sup>3</sup> )	4.5-6.5
Particle size	Provide ultra-fine (0.1-1.0 μm) and fine (1.0-5.0 μm) specifications, can be customized according to customer needs
Moisture	≤0.02%, ensuring product dryness and stability
Customization	Optional ultra-high purity grade (5N, ≥99.999%), with further reduction of impurities (e.g. O≤0.01%)

4. Packaging and Quality Assurance

Packaging: Inner sealed vacuum aluminum foil bag, outer iron barrel or plastic barrel, net weight 5kg, 10kg or 25kg, moisture-proof and oxidation-proof.

Warranty: With quality certificate, including tungsten content, impurity analysis (ICP-MS), particle size (FSSS method), bulk density and moisture data, shelf life is 12 months (sealed and dry conditions).

5. Procurement Information

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com) Tel: +86 592 5129696

For more tungsten powder information, please visit China Tungsten Online website ( [www.tungsten-powder.com](http://www.tungsten-powder.com) )

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## Chapter 4 Modern Advanced Tungsten Powder Preparation Technology

The modern preparation technology of tungsten powder represents the latest progress in materials science and promotes its application in aerospace, electronic devices, nanotechnology and additive manufacturing. This chapter comprehensively discusses five cutting-edge methods: plasma spheroidization technology, chemical vapor deposition (CVD), physical vapor deposition (PVD), mechanical alloying and nano-tungsten powder synthesis technology, and deeply analyzes their theoretical basis, process details, equipment requirements, parameter optimization, industrial application and future development direction, providing systematic guidance for the efficient preparation and application of tungsten powder.

### 4.1 Plasma Spheroidization Technology

#### 4.1.1 Process principle

Plasma spheroidization technology uses high-temperature plasma (10,000-20,000°C) to melt and spheroidize irregularly shaped tungsten powder, and form highly spherical particles after cooling. Its core is the physical process: W (solid, irregular) → W (liquid, spherical) → W (solid, spherical), and the spheroidization is driven by the surface tension in the molten state ( $\sigma \approx 2.5 \text{ N/m}$ ), following the principle of minimum surface energy.

RF plasma (radio frequency): Induction heating through high-frequency electromagnetic fields (2-13 MHz), electrode-free discharge, uniform temperature distribution (5000-10,000°C), high thermal efficiency (50-70%). Thermodynamically, the melting process must overcome the melting point (3422°C) and latent heat (192 kJ/kg) of tungsten.

#### DC plasma (direct current)

Arc discharge produces local high temperatures (>15,000°C), and the energy is concentrated in the flame core, which has lower efficiency (30-50%), but lower equipment cost. Kinetically, the particle melting time is inversely proportional to the square of the particle size ( $t \propto d^2 / \kappa$ ,  $\kappa$  is the thermal diffusion coefficient).

Sphericity is affected by residence time, cooling rate and gas dynamics. Theoretical models predict droplet breakup behavior based on the Weber number ( $We = \rho v^2 d / \sigma$ ).

#### 4.1.2 Process flow

Raw material preparation: Irregular tungsten powder (particle size 1-100  $\mu\text{m}$ , purity >99.9%), the source includes hydrogen reduction method, crushing method or gas atomization crude product. The powder needs to be dry (<0.1 wt% moisture) to prevent explosion.

Feed: High-purity inert gas (Ar or H<sub>2</sub>, flow rate 10-50 L/min, purity >99.999%) is used to uniformly inject the powder into the plasma flame through a nozzle (aperture 0.5-2 mm), with a feed rate of 0.1-2

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kg/h.

Melting and spheroidization: The powder melts instantly in the plasma (residence time 0.1-1 ms), and the surface tension of the droplets causes them to spontaneously spheroidize. The flame temperature distribution is determined by the power and gas flow rate.

Cooling: The droplets enter the cooling chamber (length 1-3 m) and solidify at a rate of  $10^5 - 10^6$  K/s to avoid excessive grain size. The cooling medium is Ar or N<sub>2</sub> with a flow rate of 50-200 L/min.

Collection: Cyclone separator (wind speed 20-50 m/s, separation efficiency >95%) classifies coarse and fine particles, and fine powder (<1 μm) is captured by bag filter (pore size <0.5 μm).

Post-processing: screening (100-500 mesh, to remove unmelted particles), air flow classification (speed 2000-5000 rpm) to adjust the distribution, and storage under nitrogen protection (O<sub>2</sub> < 0.01%).

#### 4.1.3 Equipment requirements

##### RF plasma equipment:

Power 10-100 kW, frequency 2-13 MHz, coil diameter 50-150 mm.

Reaction chamber: quartz or ceramic material, diameter 50-200 mm, length 300-600 mm.

Cooling system: water-cooled jacket (flow rate 50-150 L/min), cooling chamber volume 1-5 m<sup>3</sup>.

##### DC plasma equipment:

Power 20-150 kW, electrode (W or Cu, life 100-500 h), arc length 10-50 mm.

Nozzle: High temperature resistant alloy (W-Mo), hole diameter 1-3 mm.

Working air pressure 0.1-1 atm, equipped with high pressure air pump (10-60 bar).

Gas system: flow meter (accuracy 0.1 L/min), gas storage tank (volume 50-200 L), tail gas condensation device (capture H<sub>2</sub>O).

Collection system: Cyclone separator (diameter 0.5-1 m), bag filter (filtration efficiency >99.9%).

#### 4.1.4 Parameter Control and Influence

Power:

RF: 20-50 kW melts 10-50 μm particles with sphericity >95%; 80-100 kW refines to 1-10 μm, with a 20% increase in melting efficiency, but energy consumption increases to 80-120 kWh/kg.

DC: 50-100 kW is suitable for 20-100 μm, >120 kW the droplet splitting rate is increased by 15%, but the electrode loss is increased.

Gas flow rate:

10-20 L/min: long residence time, D<sub>50</sub> > 20 μm, wide distribution (D<sub>90</sub>/D<sub>10</sub> ≈ 4-5).

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30-50 L/min: short residence time,  $D_{50} < 10 \mu\text{m}$ , narrow distribution ( $D_{90}/D_{10} < 2$ ), fluidity 20-25 s/50g.

Raw material particle size:

$< 10 \mu\text{m}$ : completely melted, sphericity  $> 95\%$ , bulk density 12-14 g/cm<sup>3</sup>.

50-100  $\mu\text{m}$ : partially unmelted (5-10%), sphericity 80-90%, fluidity  $> 30$  s/50g.

Cooling rate:

$10^6$  K/s: grain size  $< 1 \mu\text{m}$ , uniform distribution ( $D_{90}/D_{10} < 1.5$ ).

$10^4$  K/s: Grains grow to 2-5  $\mu\text{m}$  and are easily attached with satellite particles (accounting for 5-10%).

Gas type: Ar improves purity ( $O < 0.01\%$ ), H<sub>2</sub> accelerates melting but increases equipment corrosion.

#### 4.1.5 Advantages and Disadvantages Analysis

Advantage:

It has a very high sphericity ( $> 95\%$ ) and excellent fluidity (20-25 s/50g), making it suitable for high-precision applications such as 3D printing.

The RF process has uniform temperature and is suitable for fine powder ( $< 10 \mu\text{m}$ ); the DC process has simple equipment and low investment (about 5 million yuan).

Extremely high purity ( $> 99.99\%$ ), no chemical residue.

Shortcoming:

High energy consumption (RF: 50-100 kWh/kg, DC: 80-150 kWh/kg) and high operating cost (500,000-800,000 yuan per ton).

The output is limited (RF: 0.5-5 kg/h, DC: 10-50 kg/h), which is not suitable for 10,000-ton production. The equipment maintenance is complex (electrode replacement, cooling system cleaning), and the downtime accounts for 10-20%.

#### 4.1.6 Industrial Applications and Case Studies

Case 1

According to the literature (Li et al., 2019), a US company used RF plasma spheroidization technology (50 kW, Ar flow rate 30 L/min, cooling rate  $10^5$  K/s) to process 20  $\mu\text{m}$  irregular tungsten powder to prepare powder with  $D_{50} = 15 \mu\text{m}$  and sphericity of 98%. It was used for selective laser melting (SLM) to manufacture rocket nozzles. The process parameters were: laser power 300 W, scanning speed 800 mm/s, and layer thickness 30  $\mu\text{m}$ . The finished product density reached 99.5%, the surface roughness  $R_a < 5 \mu\text{m}$ , and the defect rate  $< 2\%$ , which significantly improved the high-temperature oxidation resistance ( $> 2000^\circ\text{C}$ ).

Case 2

According to the literature (Yang et al., 2023), a factory in China used DC plasma (100 kW, H<sub>2</sub> flow rate 40 L/min, raw material particle size 30-50  $\mu\text{m}$ ) to prepare tungsten powder with  $D_{50} = 25 \mu\text{m}$  and

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sphericity 96%, which was applied to thermal spray coating. The coating thickness is 200  $\mu\text{m}$ , the bonding strength is 70 MPa, the wear resistance is 20% higher than that of traditional powder coating, and the service life is extended to 1500 h (test conditions:  $\text{Al}_2\text{O}_3$  grinding wheel, load 5 kg, speed 200 rpm).

#### 4.1.7 Technical improvement direction

##### Hybrid plasma

Combining the advantages of RF and DC, the power distribution (RF 60%, DC 40%), energy efficiency is improved by 30%, the particle size control range is expanded to 0.5-100  $\mu\text{m}$ , and the distribution uniformity is improved by 15%.

##### Pulse discharge

The use of pulse power supply (cycle 0.01-0.1 ms) shortens the melting time to  $<0.1$  ms, refines the particle size to 0.5-5  $\mu\text{m}$ , and reduces energy consumption by 20%.

##### Gas circulation system

Developed a closed-loop recovery unit ( $\text{Ar}/\text{H}_2$  recovery rate  $>90\%$ ), reducing operating costs by 15-20% and tail gas emissions by 80%.

##### Intelligent monitoring

Integrated temperature sensor (accuracy  $\pm 5^\circ\text{C}$ ) and online particle size detection (laser scattering) optimize parameters in real time, improving the consistency of finished products by 10%.

#### 4.2 Chemical Vapor Deposition (CVD)

##### 4.2.1 Process principle

deposits tungsten powder or thin film on a substrate by decomposing or reacting a vapor precursor (such as  $\text{WF}_6$ ) at high temperature. The main reaction is:  $\text{WF}_6 + 3\text{H}_2 \rightarrow \text{W} + 6\text{HF}$  ( $\Delta H = -960$  kJ/mol,  $\Delta G < 0$  at 400-800°C), based on thermodynamic decomposition and surface chemical adsorption mechanisms.

Nucleation: After  $\text{WF}_6$  decomposition, W atoms nucleate on the substrate, following the Volmer-Weber mode (island growth, suitable for high surface energy substrates such as Si) or the Frank-van der Merwe mode (layer growth, suitable for low surface energy substrates such as  $\text{Al}_2\text{O}_3$ ).

Growth: Particle size is controlled by deposition time and supersaturation, and the kinetic equation is  $d/dt = k \cdot C^n$  (k is the rate constant, C is the precursor concentration, and  $n \approx 1-2$ ).

The surface reaction is affected by temperature, pressure and  $\text{H}_2 / \text{WF}_6$  ratio, and the by-product HF needs to be discharged in time to maintain the reaction balance.

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#### 4.2.2 Process flow

Precursor preparation:  $WF_6$  (purity > 99.9%, gaseous, stored in corrosion-resistant steel cylinders) and  $H_2$  (purity > 99.999%) are mixed in a ratio (1:3-1:6), and a diluent gas (such as Ar, 10-50 vol%) can be used to adjust the concentration.

Reaction: The substrate (Si,  $Al_2O_3$  or quartz, 50-150 mm in diameter) is placed in a reaction chamber, heated to 400-800°C, with the pressure controlled at 0.1-10 Torr and the gas flow rate at 0.1-5 L/min.

Deposition: Tungsten atoms nucleate on the substrate and grow into powder or film. The deposition time is 0.5-4 h. The thickness or particle size is adjusted according to the process (10 nm-2  $\mu$ m).

Collection: The deposited powders were collected by scraping (thickness > 500 nm) or dispersing by ultrasound (power 50-100 W, time 5-10 min).

Post-treatment: Wash with deionized water to remove residual HF, dry in vacuum at 80-120°C (<10 Pa), and store under nitrogen protection.

Tail gas treatment: HF is neutralized by NaOH solution (concentration 5-10 wt%) to generate NaF and  $H_2O$ .

#### 4.2.3 Equipment requirements

##### CVD Reactor:

Material: Quartz or stainless steel, diameter 50-200 mm, heating zone length 300-600 mm.

Heating system: resistance furnace or infrared lamp, maximum temperature 1000°C, temperature control accuracy  $\pm 2^\circ C$ .

##### Gas system:

Mass flow controller (MFC, accuracy 0.01 L/min),  $WF_6$  dedicated valve (PTFE seal).

Gas storage tank: volume 10-50 L, pressure resistance 10 bar.

Vacuum system: Mechanical pump (pumping speed 10-50  $m^3/h$ ) and molecular pump (ultimate vacuum <10<sup>-3</sup> Torr).

Tail gas treatment: Neutralization tower (height 1-2 m), waste liquid collection tank (volume 50-100 L).

Collection equipment: Ultrasonic cleaning machine (frequency 40 kHz), vacuum oven (power 1-5 kW).

#### 4.2.4 Parameter Control and Influence

Temperature:

400-600°C: high nucleation rate, generating 50-200 nm particles with narrow distribution (D90/D10 <

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2) and specific surface area of 30-40 m<sup>2</sup> / g.

700-800°C: Growth is dominant, the particles increase to 0.5-2 μm, the morphology tends to be polyhedral, and the deposition rate increases by 50%.

Pressure:

0.1-1 Torr: uniform nucleation, D90/D10 < 1.5, deposition efficiency 0.1-0.5 μm/h.

5-10 Torr: The growth rate is accelerated (>1 μm/h), but the aggregation increases (D90/D10 > 3).

H<sub>2</sub> / WF<sub>6</sub> ratio :

3:1: small particles, purity >99.98%, low deposition rate (0.1-0.5 μm/h).

6:1: The deposition rate increased to 1-2 μm/h, and a small amount of F remained (<0.01%).

Substrate Type:

Si: The surface is smooth, the deposition is uniform, and the morphology is spherical.

Al<sub>2</sub>O<sub>3</sub> : The surface is rough, easy to form nano-islands, and the distribution is slightly wide .

Sedimentation time: 0.5-1 h for nanopowders (<100 nm), 2-4 h for micron-sized particles.

#### 4.2.5 Advantages and Disadvantages Analysis

Advantage:

The particle size is small (10-200 nm) and the morphology is controllable (spherical, polyhedral, thin film).

High purity (>99.98%), suitable for nano-coating and electronic applications.

Flexible process, adjustable deposition morphology (powder or film).

Shortcoming:

Precursors are expensive (WF<sub>6</sub> > 1000 RMB per kg) and operating costs are high (500-1000 RMB per kg).

HF waste gas treatment increases the environmental burden (approximately 0.5-1 L of waste liquid is generated per kg of powder).

The output is low (g/h level), which is not suitable for large-scale industrialization.

#### 4.2.6 Industrial applications and cases

Case 1: According to the literature (Chen et al., 2008), a research team used CVD (600°C, 1 Torr, H<sub>2</sub> / WF<sub>6</sub> = 4:1) to prepare tungsten powder with D50 = 50 nm on a Si substrate. Process details: substrate diameter 100 mm, deposition time 2 h, gas flow 0.5 L/min. The finished product has a specific surface area of 35 m<sup>2</sup> / g and is used for photocatalytic nanocomposite coatings. Under 300 W xenon lamp irradiation, the hydrogen production rate reaches 400 μmol·g<sup>-1</sup>·h<sup>-1</sup>, which is 25% higher than that of traditional WO<sub>3</sub> powder .

Case 2: According to the literature (Zhou et al., 2021), a laboratory used CVD (700°C, 5 Torr, H<sub>2</sub> / WF<sub>6</sub>

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= 6:1) to prepare tungsten powder with D50 = 150 nm and deposited it on an Al<sub>2</sub>O<sub>3</sub> substrate . Process parameters: deposition time 3 h, Ar dilution gas 20 vol%, deposition rate 1.2 μm/h. The powder morphology is polyhedral, with a purity of 99.99%. It is used for electrochromic film, and the response time is shortened to 5 s (test conditions: voltage 1.5 V, wavelength 550 nm).

#### 4.2.7 Technical improvement direction

Low-temperature CVD: Develop new precursors (such as W(CO)<sub>6</sub>, decomposition temperature 200-300°C), reduce the reaction temperature to 300°C, reduce energy consumption by 20%, and extend equipment life by 30%.

Plasma-enhanced CVD (PECVD): Introducing RF plasma (5-10 kW) increases deposition efficiency by 30% and refines particle size to 10-50 nm.

H<sub>2</sub> is recovered after HF neutralization (recovery rate>80%), reducing costs by 15% and waste liquid volume by 50%.

Continuous: Design of a flow reactor (substrate movement speed 1-5 cm/min) to increase the output to 100 g/h.

### 4.3 Physical Vapor Deposition (PVD)

#### 4.3.1 Process principle

Physical vapor deposition (PVD) vaporizes the tungsten target by physical methods (such as sputtering or evaporation) and condenses it into tungsten powder or thin film on the substrate. The process is: W (solid) → W (gas) → W (solid).

Sputtering: Ar<sup>+</sup> ions (energy 1-10 keV) bombard the target, expelling W atoms. The deposition rate is controlled by the ion flux density ( $J = I/A$ , I is the current and A is the target area).

Evaporation: Electron beam or resistive heating evaporates tungsten (>3422°C), and the vapor pressure follows the Clausius-Clapeyron equation ( $\ln P = -\Delta H_v/RT + C$ ).

Particle size and morphology are determined by the vaporization rate, vacuum and substrate temperature. There is no chemical reaction, and the purity depends on the target material and environment.

#### 4.3.2 Process flow

Target preparation: High purity tungsten target (purity>99.95%, diameter 50-300 mm, thickness 5-20 mm), surface polished (Ra <0.1 μm).

Gasification:

Sputtering: Ar atmosphere (pressure 10<sup>-2</sup> -10<sup>-3</sup> Torr), DC or RF power supply (2-20 kW), target-

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substrate distance 10-20 cm.

Evaporation: Electron beam (power 10-50 kW, temperature 3000-3500°C), vacuum  $<10^{-5}$  Torr, crucible (Ta or W).

Deposition: Tungsten atoms are condensed on a cooled substrate (such as Cu, Si or glass) with a deposition time of 1-6 h and a thickness or particle size of 20 nm-1  $\mu$ m.

Collection: Powders are collected by mechanical scraping (thickness  $> 500$  nm) or air flow (wind speed 10-30 m/s).

Post-treatment: sieving (100-500 mesh), ultrasonic dispersion (50-100 W, 5-10 min), and storage under nitrogen protection.

#### 4.3.3 Equipment requirements

Sputtering equipment:

Vacuum chamber: volume 1-5  $m^3$ , material stainless steel, ultimate vacuum  $<10^{-6}$  Torr.

Power supply: DC (5-20 kW) or RF (2-15 kW), target water cooling system (flow rate 10-50 L/min).

Evaporation equipment:

Electron beam gun: power 10-50 kW, beam current 0.1-1 A, focusing accuracy  $\pm 1$  mm.

Crucible: Ta or W material, volume 50-200 mL, temperature resistance  $>3500^\circ\text{C}$ .

Substrate system: Rotating substrate holder (10-50 rpm), heater (25-500°C, accuracy  $\pm 5^\circ\text{C}$ ).

Vacuum system: turbomolecular pump (pumping speed 500-2000 L/s), cold trap (to capture residual gas).

Collection device: airflow collector (wind speed 10-30 m/s), screening machine (vibration frequency 50-100 Hz).

#### 4.3.4 Parameter Control and Influence

Power:

5-10 kW: Generates 20-100 nm particles, with a deposition rate of 0.1-0.5 nm/s and a narrow distribution ( $D_{90}/D_{10} < 2$ ).

15-20 kW: Particles grow to 0.5-1  $\mu$ m, deposition rate  $>1$  nm/s, morphology tends to be cubic.

Vacuum degree:

$10^{-5}$  -  $10^{-6}$  Torr: purity  $>99.99\%$ , O  $<0.01\%$ , uniform distribution.

$10^{-4}$  Torr: oxygen impurities increase (0.05-0.1%), irregular morphology, and reduced fluidity.

Base temperature:

25-200°C: fine particles ( $<100$  nm),  $D_{90}/D_{10} < 2$ , specific surface area 40-50  $m^2/g$ .

300-500°C: Particles grow larger (0.5-1  $\mu$ m), have regular morphology, and packing density increases by 10%.

Target base distance:

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10-15 cm: uniform deposition, efficiency >80%.

20 cm: The deposition rate decreases by 20-30% and the distribution is wider.

Ar flow rate: 10-20 sccm for stable sputtering, >50 sccm for uneven target sputtering.

#### 4.3.5 Advantages and Disadvantages Analysis

Advantage:

Extremely high purity (>99.99%), no chemical by-products, suitable for high purity applications.

The process is simple and can produce powders or films with controllable particle size (20-1000 nm).

The equipment is mature and easy to switch between laboratory and industry.

Shortcoming:

The equipment investment is high (>10 million yuan) and the operating cost is high (1000-2000 yuan per kg).

The deposition efficiency is low (<20%), the target utilization rate is <50%, and there is serious waste.

The output is low (g/h level), which is not suitable for mass production.

#### 4.3.6 Industrial applications and cases

Case 1: According to the literature (Zhang et al., 2018), a team used PVD sputtering (10 kW,  $10^{-5}$  Torr, Ar flow rate 20 sccm) to prepare tungsten powder with  $D_{50} = 80$  nm. Process details: target diameter 200 mm, target substrate distance 15 cm, deposition time 4 h, substrate temperature 150°C. The finished product has a purity of 99.99%, a specific surface area of 45 m<sup>2</sup> / g, and is used for sputtering target deposition. The film thickness is 500 nm, the uniformity is improved by 15%, and the defect rate is <1% (test conditions: SEM observation, 5000× magnification).

Case 2: According to the literature (Patel et al., 2022), a laboratory used electron beam evaporation PVD (20 kW,  $10^{-6}$  Torr, substrate Cu) to prepare tungsten powder with  $D_{50} = 200$  nm. Process parameters: electron beam current 0.5 A, deposition time 3 h, substrate rotation 20 rpm. The powder morphology is spherical, purity >99.99%, used as a nanocatalyst support, and the activity in CO oxidation reaction is increased by 20% (test conditions: 300°C, CO concentration 1 vol%).

#### 4.3.7 Technical improvement direction

Multi-target sputtering: Using a dual-target or multi-target system (power distribution 5-10 kW/target), deposition efficiency is increased by 30% and output increases to 50 g/h.

Laser-assisted evaporation: The introduction of pulsed laser (wavelength 1064 nm, power 1-5 kW) improves temperature uniformity by 20% and refines particle size to 10-50 nm.

Dynamic substrate: Development of a vibrating substrate (frequency 10-50 Hz) that increases collection efficiency by 25% and reduces powder adhesion losses.

Target recycling: Optimize target design (thickness gradient) and increase utilization rate to 70%.

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## 4.4 Mechanical Alloying

### 4.4.1 Process principle

Mechanical alloying refines tungsten powder by high-energy ball milling and mixes it with alloying elements (such as Cr, Ni, Co) to form nanocrystalline tungsten powder or composite powder. The process is based on mechanical energy-induced fracture, cold welding and diffusion:  $W + M \rightarrow WM$  (solid solution or composite).

Refinement mechanism: Particles are subjected to impact and shear, crystal defects (dislocations, grain boundaries) accumulate, and grain size follows the Hall-Petch relationship ( $d \propto E^{-1/2}$ ,  $E$  is the energy input).

Alloying: Elements diffuse to form solid solution or composite phase, with diffusion coefficient  $D = D_0 \cdot \exp(-Q/RT)$  ( $Q$  is activation energy).

Grain size (<20 nm) is affected by rotation speed, time and process control agent (PCA), and the theoretical limit is determined by material toughness and ball energy.

### 4.4.2 Process flow

Raw material preparation: Tungsten powder (10-100  $\mu\text{m}$ , purity >99.9%) and alloying elements (1-20 wt%, such as Ni, Co, Cr), powder premixed (stirred for 1-2 h).

Charge: Place in a ball mill (WC or  $\text{ZrO}_2$ , volume 100-1000 mL), ball to material ratio 10:1-30:1, add PCA (such as stearic acid, 0.5-2 wt%) to prevent excessive cold welding.

Ball milling: planetary ball mill, speed 300-800 rpm, time 20-100 h, dry grinding (air or Ar atmosphere) or wet grinding (ethanol, hexane, liquid-solid ratio 1:1-2:1).

Post-processing:

The PCA and solvent were removed by vacuum drying (80-120°C, <10 Pa, 4-6 h).

Adjust the distribution by screening (100-500 mesh) or air classification (speed 2000-5000 rpm).

Heat treatment (optional): 400-800°C annealing (Ar atmosphere, 2-4 h) to eliminate internal stress and stabilize grains.

### 4.4.3 Equipment requirements

Ball mill:

Planetary type (power 1-10 kW), tank capacity 100-1000 mL, turntable diameter 300-500 mm.

Lining: WC or  $\text{ZrO}_2$ , hardness >90 HRA, wear resistant.

Grinding balls: WC or  $\text{ZrO}_2$ , diameter 5-20 mm, mass ratio 10-30, purity >99.9%.

Vacuum oven: temperature 50-200°C, pressure <10 Pa, volume 50-200 L.

Classification equipment: air flow classifier (wind speed 20-50 m/s, accuracy  $\pm 0.1 \mu\text{m}$ ).

Atmosphere control: Ar gas cabinet ( $\text{O}_2 < 0.01\%$ ), flow meter (0.1-5 L/min).

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#### 4.4.4 Parameter Control and Influence

Rotation speed:

300-500 rpm: D50 = 1-5  $\mu\text{m}$ , grains > 50 nm, broad distribution (D90/D10 > 5).

600-800 rpm: D50 = 0.1-0.5  $\mu\text{m}$ , grains < 20 nm, improved distribution (D90/D10 < 3).

time:

20-50 h: Mainly refinement, grain size 50-100 nm, hardness increased by 10%.

80-100 h: grain size <20 nm, impurities increased (Fe <0.5%, O <0.2%).

Ball to material ratio:

10:1: Slow refinement and poor uniformity (D90/D10 > 5).

20:1-30:1: High efficiency, D90/D10 < 3, energy input increased to 10-15 kWh/kg.

PCA:

0.5-1 wt%: Reduce agglomeration and improve fluidity by 10% (25-30 s/50g).

2 wt%: contaminated powder (C <0.2%), slightly reduced hardness.

Grinding media: Dry grinding has uneven distribution (D90/D10 > 4), wet grinding improves uniformity by 20%, but requires drying.

#### 4.4.5 Advantages and Disadvantages Analysis

##### Advantage:

Nanocrystalline tungsten powder (grain <20 nm) or composite powder with high hardness (>90 HRA) can be prepared.

The equipment is simple, the investment is low (500,000-1.5 million yuan), and the cost is moderate (250,000-400,000 yuan per ton).

High flexibility, suitable for multi-component alloying (W-Ni, W-Co, etc.).

##### Shortcoming:

Impurity contamination (Fe, O <0.5%), uneven distribution (D90/D10 > 3).

Severe agglomeration (nanopowder accounts for 10-20%), requiring post-processing and classification.

The energy consumption is high (5-15 kWh/kg) and the production cycle is long (20-100 h).

#### 4.4.6 Industrial Applications and Case Studies

Case 1: According to the literature (Kwon et al., 2003), a team used mechanical alloying (600 rpm, 80 h, ball-to-material ratio 20:1, 1 wt% stearic acid) to refine 50  $\mu\text{m}$  tungsten powder to D50 = 0.2  $\mu\text{m}$  and grain size 15 nm. Process details: tank capacity 500 mL, WC grinding balls (diameter 10 mm), dry grinding, Ar atmosphere. Prepared W-10%Ni composite powder, after sintering (1400°C, 2 h), hardness reached 92 HRA, fracture toughness 12  $\text{MPa}\cdot\text{m}^{1/2}$ , used for high temperature tools, cutting life increased by 15% (test conditions: cutting speed 200 m/min, feed rate 0.2 mm/rev).

Case 2: According to the literature (Wang et al., 2021), a Chinese company used mechanical alloying (500 rpm, 60 h, ball-to-material ratio 15:1, wet grinding ethanol) to prepare W-5%Cr composite powder with D50 = 0.5  $\mu\text{m}$  and grain size 25 nm. Process parameters: tank capacity 1000 mL, ZrO<sub>2</sub> grinding

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balls (diameter 15 mm), annealing 600°C. The oxidation resistance of the finished product increased by 20% after sintering (1500°C, 3 h), and it was used for aircraft engine parts with a service temperature of 1200°C (test conditions: air atmosphere, 100 h).

#### 4.4.7 Technical improvement direction

Cryogenic ball milling

Liquid nitrogen cooling (-196°C, flow rate 5-10 L/min) was introduced to refine the grains to <10 nm, reduce agglomeration by 20%, and reduce energy consumption by 10%.

Green PCA

By replacing stearic acid with polyethylene glycol (molecular weight 400-1000), contamination was reduced by 30% and purity was increased to >99.95%.

Continuous production

Developed a continuous ball mill (feed rate 1-5 kg/h), increased output to 100 kg/h, and shortened production cycle by 50%.

Alloy Optimization

The introduction of trace rare earth elements (such as Y, 0.1-0.5 wt%) can strengthen the grain boundaries and increase the hardness by 5%.

### 4.5 Nano Tungsten Powder Synthesis

#### 4.5.1 Process principle

The synthesis of nano-tungsten powder includes hydrothermal method, sol-gel method and spray pyrolysis method, and the goal is to prepare tungsten powder or  $WO_3$  (later reduced to W) with a particle size of <100 nm.

Hydrothermal method

In high temperature and high pressure aqueous solution, tungstate (such as  $Na_2WO_4$ ) nucleates and grows into  $WO_3$  ( $Na_2WO_4 + 2HCl \rightarrow WO_3 \downarrow + 2NaCl + H_2O$ ), and then is reduced by hydrogen ( $WO_3 + 3H_2 \rightarrow W + 3H_2O$ ). Nucleation follows the LaMer model, and the size is controlled by supersaturation.

Sol-Gel Method

The tungsten precursor (such as  $WCl_6$ ) is hydrolyzed to form a sol, which is then calcined to  $WO_3$  after gelation and then reduced. The process is based on the aggregation of sol particles into a gel network.

Spray pyrolysis

The precursor solution is thermally decomposed into  $WO_3$  or W after atomization. Based on droplet evaporation and gas-solid transition, the particle size is determined by the atomization conditions.

#### 4.5.2 Process flow

Hydrothermal method:

Prepare  $Na_2WO_4$  solution (0.1-1 mol/L, solvent: deionized water) and adjust the pH to 4-6 with HCl.

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Autoclave (180-220°C, 1-10 MPa), react for 12-24 h to generate WO<sub>3</sub> .

Centrifuge (5000-10000 rpm, 10-20 min) and dry at 80-120°C.

700-800°C hydrogen reduction (flow rate 1-2 L/min, 2-4 h).

Sol-Gel Method:

WCl<sub>6</sub> (purity>99.9%) was dissolved in ethanol (concentration 0.1-0.5 mol/L) and hydrolyzed by adding NH<sub>4</sub>OH ( molar ratio 1:1-2:1).

Stir (100-500 rpm, 2-4 h) to form a sol, and let it stand for 12-24 h to gel.

Calcinate at 300-500°C (2-4 h) to generate WO<sub>3</sub> , and reduce at 700-800°C.

Spray pyrolysis:

Prepare ammonium tungstate solution (0.1-0.5 mol/L) and atomize (nozzle 0.5-1 mm, pressure 1-5 bar).

Pyrolysis furnace (500-1000°C) to generate WO<sub>3</sub> or W with a residence time of 1-5 s.

Post-treatment: ultrasonic dispersion (50-100 W, 5-10 min), sieving (500-1000 mesh), and storage under nitrogen protection.

#### 4.5.3 Equipment requirements

Hydrothermal method:

High-pressure reactor (50-1000 mL, pressure resistance 15 MPa, PTFE lined).

Reduction furnace (50-100 mm diameter, up to 1000°C).

Sol-Gel Method:

Agitator (100-500 rpm, volume 1-5 L).

Muffle furnace (power 5-10 kW, temperature 25-1000°C).

Spray pyrolysis:

Spray dryer (power 5-20 kW, atomization flow rate 1-10 L/h).

Pyrolysis furnace (diameter 100-300 mm, length 1-2 m).

General equipment: Centrifuge (5000-10000 rpm), vacuum oven (<10 Pa).

#### 4.5.4 Parameter Control and Influence

Hydrothermal method:

Temperature: 180-190°C generates 10-20 nm, 210-220°C increases to 30-50 nm, with a specific surface area of 40-60 m<sup>2</sup> / g.

Pressure: 5-10 MPa narrow distribution (D90/D10 < 1.5), 1-3 MPa uneven morphology.

Concentration: 0.1-0.3 mol/L, particles are uniform; >0.5 mol/L, agglomeration increases.

Sol-Gel Method:

Calcination temperature: 300-400°C, grain size <20 nm, >500°C, grow to 50-100 nm.

Hydrolysis dosage: 1:1-2:1 sol is stable, >3:1 gel time is extended by 50%.

Spray pyrolysis:

Pyrolysis temperature: 500-700°C produces WO<sub>3</sub> (20-50 nm), 900-1000°C produces W (50-100 nm).

Atomization pressure: 1-3 bar for fine particles (D50 < 50 nm), >5 bar for broad distribution (D90/D10 > 3).

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#### 4.5.5 Advantages and Disadvantages Analysis

Hydrothermal method:

Advantages: small particle size (10-50 nm), uniform distribution ( $D_{90}/D_{10} < 2$ ), controllable morphology (cubic, needle-shaped).

Disadvantages: low output (kg/batch), complex process, and high cost (500-1000 yuan per kg).

Sol-Gel Method:

Advantages: simple equipment, high purity (>99.98%), suitable for laboratory research.

Disadvantages: cumbersome steps, difficult to scale up, and long cycle (>24 h).

Spray pyrolysis:

Advantages: Continuous, high output (10-50 kg/h), suitable for industrialization.

Disadvantages: High energy consumption (20-50 kWh/kg), wide distribution ( $D_{90}/D_{10} > 3$ ).

#### 4.5.6 Industrial Applications and Case Studies

Case 1

According to the literature (Chen et al., 2008), a team used a hydrothermal method (200°C, 6 MPa, 0.4 mol/L  $\text{Na}_2\text{WO}_4$ ) to prepare  $\text{WO}_3$  with  $D_{50} = 25$  nm. The process details are as follows: reactor volume 500 mL, reaction time 18 h, pH 5. After hydrogen reduction at 750°C (flow rate 1.5 L/min, 3 h), tungsten powder was obtained with a specific surface area of 50  $\text{m}^2/\text{g}$  and a photocatalytic hydrogen production rate of 450  $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$  (test conditions: 300 W xenon lamp, wavelength 420 nm).

Case 2

According to the literature (Li et al., 2015), a laboratory used spray pyrolysis (800°C, 2 bar, 0.2 mol/L ammonium tungstate) to prepare tungsten powder with  $D_{50} = 50$  nm. Process parameters: atomization flow rate 5 L/h, pyrolysis residence time 3 s. The finished product has a purity of 99.98% and is used for nanocomposites. After sintering (1200°C, 2 h), the density reaches 98% and the hardness is 90 HRA.

Case 3

According to the literature (Zhang et al., 2022), a Chinese research group used the sol-gel method ( $\text{WCl}_6$  0.3 mol/L,  $\text{NH}_4\text{OH}$  1:1.5, calcination at 400°C) to prepare  $\text{WO}_3$  with  $D_{50} = 30$  nm, and reduced it at 700°C to obtain tungsten powder. Process details: gel time 18 h, calcination time 3 h. The powder has a specific surface area of 55  $\text{m}^2/\text{g}$ , and is used for electrochromic coatings, with a color change efficiency increased by 20% (test conditions: voltage 1.2 V, wavelength 633 nm).

#### 4.5.7 Technical improvement direction

Hydrothermal method

Developed a flow reactor (flow rate 1-5 L/h), increased the output to 10 kg/h, and improved production efficiency by 50%.

Sol-gel method: By optimizing the hydrolysis conditions (microwave assisted, power 500-1000 W), the gelation time was shortened to 6 h and the cost was reduced by 20%.

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### Spray pyrolysis

The introduction of a plasma heat source (10-20 kW) refines the particle size to <20 nm and improves the distribution uniformity by 15%.

Green process: Use citric acid instead of HCl (hydrothermal method), reducing waste liquid by 30% and improving environmental protection.

## 4.6 Comprehensive comparative analysis of preparation methods

The preparation technology of tungsten powder directly determines its physical and chemical properties and application range. This section compares five modern advanced preparation methods in tabular form: plasma spheroidization technology, chemical vapor deposition (CVD), physical vapor deposition (PVD), mechanical alloying and nano tungsten powder synthesis, and systematically analyzes them from the aspects of theoretical basis, process characteristics, performance indicators, industrial applicability and future potential, and explains them with specific data and cases to guide technology selection and optimization.

### 4.6.1 Comparison table of tungsten powder preparation methods

The following table summarizes the characteristics of the five preparation methods, based on the data in the previous sections and references.

Parameter	Plasma spheroidization technology	Chemical Vapor Deposition (CVD)	Physical Vapor Deposition (PVD)	Mechanical alloying	Synthesis of Nano-Tungsten Powder
Principle	High temperature plasma melting spheroidization (RF/DC)	Decomposition of vapor precursors ( $WF_6 + H_2 \rightarrow W$ )	Target physical vapor deposition (sputtering/evaporation)	High Energy Ball Milling and Alloying	Chemical reaction to generate $WO_3$ followed by reduction (hydrothermal/sol-gel/spray pyrolysis)
Particle size range	0.5-100 $\mu m$	10-2000 nm	20-1000 nm	0.1-5 $\mu m$	10-100 nm
Morphology	Spherical (sphericity > 95%)	Sphere, polyhedron, film	Spherical, cubic, base dependent	Irregular, severe agglomeration	Cubic, needle-shaped, spherical, method dependent
Purity	>99.99% , no chemical pollution	>99.98% (a small amount of F remains)	>99.99% (determined by target material)	99.5-99.9% (Fe, O contamination)	>99.98% (affected by reducing conditions)
Grain size	1-5 $\mu m$ (determined by cooling rate)	10-100 nm (deposition time control)	20-500 nm (substrate temperature control)	<20 nm (determined by ball milling time)	<50 nm (reaction condition control)

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Parameter	Plasma spheroidization technology	Chemical Vapor Deposition (CVD)	Physical Vapor Deposition (PVD)	Mechanical alloying	Synthesis of Nano-Tungsten Powder
Specific surface area	0.1-5 m <sup>2</sup> / g	30-60 m <sup>2</sup> / g	40-50 m <sup>2</sup> / g	10-20 m <sup>2</sup> / g	40-60 m <sup>2</sup> / g
Liquidity	20-25 s/50g (excellent)	Not applicable (small amount of powder)	Not applicable (small amount of powder)	30-40 s/50g (poor)	Not applicable (nanopowder agglomeration)
Yield	RF: 0.5-5 kg/h, DC: 10-50 kg/h	0.01-0.1 kg/h	0.01-0.05 kg/h	1-10 kg/h (batch)	Hydrothermal/sol: 0.1-1 kg/batch, spray: 10-50 kg/h
Energy consumption	RF: 50-100 kWh/kg, DC: 80-150 kWh/kg	10-30 kWh/kg	20-50 kWh/kg	5-15 kWh/kg	Hydrothermal/sol: 10-20 kWh/kg, spray: 20-50 kWh/kg
Cost	500,000-800,000 yuan per ton	500-1000 Yuan per kg	1000-2000 Yuan per kg	250,000-400,000 yuan per ton	500-1000 Yuan per kg
Equipment investment	5-10 million RMB	3-8 million RMB	10-15 million RMB	500,000-1.5 million RMB	Hydrothermal/sol: 500,000-2,000,000 yuan, Spray: 2,000,000-5,000,000 yuan
Key Benefits	High sphericity, good fluidity, and extremely high purity	Small particle size, controllable morphology, suitable for nano applications	Extremely high purity, simple process, suitable for thin films	Small grains, low cost, suitable for composite materials	Small particle size, large specific surface area, and various methods
Main Disadvantages	High energy consumption, limited output, high cost	Low output, complex HF waste gas treatment, high cost	Low efficiency, expensive equipment, low output	Impurity pollution, serious agglomeration, uneven distribution	Low output (hydrothermal/sol), high energy consumption (spray)
Application Areas	3D printing, thermal spraying, high-	Nano coatings, photocatalysis, electronic	Thin films, targets, nanocatalysts	Cemented carbide, high temperature	Photocatalysis, nanocomposites, electrochromism

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Parameter	Plasma spheroidization technology	Chemical Vapor Deposition (CVD)	Physical Vapor Deposition (PVD)	Mechanical alloying	Synthesis of Nano-Tungsten Powder
	density parts	devices		structural parts	
Industrialization level	Medium (kg/h level)	Low (g/h level)	Low (g/h level)	Medium (kg/h level)	Low-Medium (higher spray)
Technology maturity	High (widely used)	Medium (mainly laboratory)	Medium (mature film application)	High (industrial application)	Medium (some methods are mature)

#### 4.6.2 Detailed analysis

##### 4.6.2.1 Comparison of process principles and mechanisms

Plasma spheroidization technology : Based on high-temperature physical melting, RF and DC processes use electromagnetic induction and arc discharge respectively, which are suitable for converting irregular tungsten powder into spherical particles. Theoretically, there is no chemical pollution and the purity is the highest (>99.99%). Its limitations are high energy consumption and limited residence time (0.1-1 ms), making it difficult to prepare nano-scale powders.

CVD: driven by chemical reactions,  $WF_6$  decomposes into W, nucleation and growth are controlled by thermodynamics and surface adsorption, and the morphology (spherical, polyhedral) can be precisely controlled, but the by-product HF increases the environmental burden.

PVD: Pure physical vaporization process, sputtering and evaporation rely on target gasification, with extremely high purity (>99.99%), but low deposition efficiency (<20%), which limits powder output.

Mechanical alloying: Mechanical energy induces refinement and diffusion, which is suitable for the preparation of nanocrystals (<20 nm) and composite powders. The theory is simple, but the introduction of impurities (Fe, O) is inevitable.

Synthesis of nano-tungsten powder: Chemical preparation of  $WO_3$  followed by reduction, hydrothermal and sol-gel methods rely on nucleation kinetics, spray pyrolysis combined with gas-liquid-solid phase transition, flexible particle size control (10-100 nm), but large differences in process complexity.

##### 4.6.2.2 Particle size and morphology characteristics

Particle size range:

Plasma spheroidization technology covers 0.5-100  $\mu m$ , suitable for micron-scale applications, RF refinement to <10  $\mu m$ , and DC biased towards 20-100  $\mu m$ .

CVD and PVD focus on the nanoscale (10-2000 nm and 20-1000 nm), suitable for high-precision areas.

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Mechanical alloying is between 0.1-5  $\mu\text{m}$ , with outstanding nanocrystalline characteristics.  
Nano-tungsten powder is synthesized to a size of 10-100 nm and has the highest specific surface area (40-60  $\text{m}^2/\text{g}$ ).

Appearance:

Plasma spheroidization technology produces mainly spherical shapes (>95%) and the best fluidity (20-25 s/50g).

CVD has a variety of morphologies (spherical, polyhedral, thin film), while PVD is affected by the substrate (spherical or cubic).

The mechanical alloying morphology is irregular, the agglomeration is serious, and the fluidity is poor (30-40 s/50g).

The morphology of nano-tungsten powder synthesis varies depending on the method (cubic by hydrothermal method, spherical by spray pyrolysis), but agglomeration affects the application.

#### 4.6.2.3 Purity and performance

Purity:

Plasma spheroidization and PVD provide the highest purity (>99.99%) and no chemical by-products.

CVD was slightly lower (>99.98%) and F residual <0.01%.

Mechanical alloying is contaminated by grinding balls (Fe <0.5%), purity 99.5-99.9%.

The synthetic purity of nano-tungsten powder is high (>99.98%), but the reduction conditions need to be strictly controlled.

Performance:

Plasma spheroidized powder has excellent fluidity and is suitable for additive manufacturing.

CVD and PVD powders have large specific surface areas (30-60  $\text{m}^2/\text{g}$ ) and strong catalytic activity.

Mechanical alloying produces fine grains (<20 nm) and high hardness (>90 HRA).

The synthesized nano-tungsten powder has a high specific surface area and is suitable for photocatalysis (hydrogen production rate >400  $\mu\text{mol}\cdot\text{g}^{-1}\cdot\text{h}^{-1}$ ).

#### 4.6.2.4 Production efficiency and economy

Yield:

Plasma spheroidization (DC: 10-50 kg/h) and spray pyrolysis (10-50 kg/h) have the highest output and are suitable for industrialization.

Mechanical alloying (1-10 kg/h) Medium and flexible batch production.

CVD and PVD (0.01-0.1 kg/h) are the lowest and are limited to laboratory or small scale.

Energy consumption:

Plasma spheroidization has the highest energy consumption (50-150 kWh/kg), followed by PVD and spray pyrolysis (20-50 kWh/kg).

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Mechanical alloying is the lowest (5-15 kWh/kg), CVD and hydrothermal/sol are moderate (10-30 kWh/kg).

Cost:

Mechanical alloying has the lowest cost (RMB 250,000-400,000 per ton), while plasma spheroidization is medium (RMB 500,000-800,000 per ton).

The synthesis costs of CVD, PVD and nano-tungsten powder are high (500-2000 yuan per kg), which are limited by raw materials and equipment.

Equipment investment:

PVD is the highest (10-15 million yuan), followed by CVD and plasma spheroidization (3-10 million yuan).

Mechanical alloying and hydrothermal/sol are the lowest (500,000-2 million yuan), and spray pyrolysis is medium (2 million-5 million yuan).

#### 4.6.2.5 Advantages and Disadvantages and Application Matching

Plasma spheroidization: The advantages are high sphericity and high purity, and the disadvantages are high energy consumption and limited output. It is suitable for 3D printing (such as rocket nozzles, case Li et al., 2019) and thermal spraying (case Yang et al., 2023).

CVD: The advantages are small particle size and controllable morphology, and the disadvantages are low yield and high cost. It is suitable for nanocoating (case Chen et al., 2008) and electrochromism (case Zhou et al., 2021).

PVD: The advantages are high purity and simple process, and the disadvantages are low efficiency and expensive equipment. It is suitable for thin film targets (case Zhang et al., 2018) and catalysts (case Patel et al., 2022).

Mechanical alloying: The advantages are fine grains and low cost, and the disadvantages are high impurities and severe agglomeration. It is suitable for cemented carbide (Case Kwon et al., 2003) and high-temperature components (Case Wang et al., 2021).

Synthesis of nano-tungsten powder: The advantages are small particle size and large specific surface area, and the disadvantages are low yield (hydrothermal/sol) or high energy consumption (spray). It is suitable for photocatalysis (Chen et al., 2008) and composite materials (Li et al., 2015).

#### 4.6.2.6 Industrialization potential and technological maturity

**Industrialization level:**

Plasma spheroidization and mechanical alloying have achieved medium-scale production (kg/h level) and have a high degree of technical maturity.

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Spray pyrolysis has industrial potential ( 10-50 kg/h), but energy consumption needs to be optimized. CVD and PVD are still mainly based in laboratories (g/h level), and the production bottleneck needs to be broken through.

Technology maturity:

Plasma spheroidization and mechanical alloying are widely used in industry and are stable processes.

PVD is mature in the thin film field, but powder preparation still needs to be developed.

CVD and nano-tungsten powder synthesis (especially hydrothermal/sol) are mainly research and development, while spray pyrolysis is closer to industrialization.

#### 4.6.3 Selection Guide

High sphericity requirements: Choose plasma spheroidization technology, suitable for additive manufacturing and thermal spraying.

Nano-scale high purity powders: CVD or PVD, with priority given to catalysts and electronic devices.

Low-cost composite materials: Mechanical alloying, suitable for cemented carbide and structural parts.

High surface area nanopowders: Nano tungsten powder synthesis (hydrothermal or spray pyrolysis), suitable for photocatalysis or composite materials.

Large-scale production: Plasma spheroidization (DC) or spray pyrolysis, balancing cost and output.

#### 4.6.4 Future Prospects

Process fusion: Combining plasma spheroidization and nano-synthesis (such as PECVD) to achieve high sphericity nano-powders to meet the needs of 3D printing and catalysis.

Improved energy efficiency: Optimized plasma pulse control and mechanical alloying low-temperature technology to reduce energy consumption to <20 kWh/kg.

Greening: Develop non-toxic precursors (such as W(CO)<sub>6</sub>) for CVD and nanosynthesis to reduce waste gas and waste liquid.

Intelligence: Online monitoring (particle size, morphology) and automation equipment integration to improve consistency and efficiency.

### Tungsten powder production equipment, inspection and testing instruments and raw and auxiliary materials list

Category	Subcategories	Plasma spheroidization technology	Chemical Vapor Deposition (CVD)	Physical vapor deposition (PVD)	Mechanical alloying	Synthesis of Nano-Tungsten Powder
Production Equipment	Main reaction equipment	- RF plasma equipment (power 10-50 kW, frequency 2-13 MHz) DC plasma	- CVD reactor (diameter 50-200 mm, volume 0.1-1 m <sup>3</sup> , temperature resistance	- Sputtering equipment (power 5-50 kW, target diameter 50-200 mm)	- Planetary ball mill (power 10 kW, tank volume 0.5-10 L, speed 200-	- Autoclave (volume 1-1000 mL, pressure 1-20 MPa, temperature 300°C) Muffle furnace

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Category	Subcategories	Plasma spheroidization technology	Chemical Vapor Deposition (CVD)	Physical vapor deposition (PVD)	Mechanical alloying	Synthesis of Nano-Tungsten Powder
		equipment (power 50-150 kW, current 100-500 A) Cooling chamber (volume 1-5 m <sup>3</sup> , temperature resistance >1000°C)	1000°C) Vacuum pump (ultimate vacuum <10 <sup>-3</sup> Torr, pumping speed 10-50 L/s)	Evaporation equipment (electron beam/thermal evaporation, power 10-30 kW) Vacuum chamber (<10 <sup>-6</sup> Torr, volume 0.5-2 m <sup>3</sup> )	800 rpm) Oven (volume 50-200 L, temperature 25-800°C)	(volume 0.1-1 m <sup>3</sup> , temperature 500-1000°C) Spray dryer (power 5-20 kW, spray volume 1-50 kg/h)
	Auxiliary equipment	- Powder feeder (flow rate 0.1-5 kg/h, accuracy ±1%) Gas circulation system (Ar/H <sub>2</sub> , storage tank 50-200 L) Cyclone separator (collection efficiency >95%)	- Precursor supply system (WF <sub>6</sub> , flow rate 0.1-1 L/min) Tail gas treatment device (HF neutralization, volume 50-200 L)	- Target holder (W target, purity >99.99%) Substrate heater (temperature 200-500°C, power 1-5 kW)	- Grinding jar (WC or ZrO <sub>2</sub> , volume 0.5-5 L) Grinding balls (WC, diameter 5-20 mm)	- Agitator (power 0.5-2 kW, speed 100-500 rpm) Centrifuge (5000-10000 rpm, volume 10-50 L) Vacuum pump (<10 Pa, pumping speed 5-20 L/s)
	Post-processing equipment	- Vibrating screen (frequency 50-100 Hz, 100-500 mesh) Air classifier (wind speed 20-50 m/s, rpm) 2000-5000	- Ultrasonic cleaning machine (power 0.5-2 kW, frequency 20-40 kHz) Vacuum oven (power 1-5 kW, <10 Pa)	- Ultrasonic cleaning machine (power 0.5-2 kW, frequency 20-40 kHz) Vacuum oven (power 1-5 kW, <10 Pa)	-Vibrating screen (frequency 50-100 Hz, 100-500 mesh) Air classifier (speed 2000-5000 rpm)	-Water washing tank (volume 50-200 L, temperature resistance 50-80°C) Vacuum oven (power 1-5 kW, <10 Pa) Air flow classifier (speed 2000-5000 rpm)
Inspection and testing instruments	Particle size and morphology analysis	Laser particle size analyzer (range 0.1-1000 μm, accuracy ±1%) Scanning electron microscope (SEM, magnification 50-100,000x, resolution <5 nm)	Transmission electron microscope (TEM, magnification 50-1,000,000x, resolution <1 nm) Laser particle size analyzer (range 1-2000 nm,	Transmission electron microscopy (TEM, magnification 50-1,000,000x, resolution <1 nm) Atomic force microscopy (AFM, resolution	Laser particle size analyzer (range 0.01-100 μm, accuracy ±1%) Scanning electron microscope (SEM, magnification 50-50,000x)	Transmission electron microscope (TEM, magnification 50-1,000,000x, resolution <1 nm) Laser particle size analyzer (range 1-1000 nm, accuracy ±1%)

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Category	Subcategories	Plasma spheroidization technology	Chemical Vapor Deposition (CVD)	Physical vapor deposition (PVD)	Mechanical alloying	Synthesis of Nano-Tungsten Powder
			accuracy ±1%)	<0.1 nm)		
	Chemical composition analysis	- Oxygen analyzer (infrared method, range 0.001-1%, accuracy ±0.001%) X-ray fluorescence spectrometer (XRF, element range Na-U, accuracy ±0.1%)	- Fourier transform infrared spectrometer (FTIR, detection of HF residue, range 400-4000 $\text{cm}^{-1}$ ) X-ray photoelectron spectrometer (XPS, element resolution ±0.1 eV)	- Oxygen analyzer (infrared method, range 0.001-1%, accuracy ±0.001%) X-ray photoelectron spectrometer (XPS, element resolution ±0.1 eV)	- Carbon and sulfur analyzer (infrared method, range 0.001-5%, accuracy ±0.001%) X-ray fluorescence spectrometer (XRF, element range Na-U, accuracy ±0.1%)	- Oxygen analyzer (infrared method, range 0.001-1%, accuracy ±0.001%) X-ray fluorescence spectrometer (XRF, element range Na-U, accuracy ±0.1%)
	Physical properties test	- Flowability tester (Hall flowmeter, accuracy ±0.1 s/50g) Specific surface area analyzer (BET, range 0.01-1000 $\text{m}^2/\text{g}$ , accuracy ±1%)	- Surface area analyzer (BET, range 0.1-1000 $\text{m}^2/\text{g}$ , accuracy ±1%) Hardness tester (nanoindentation, range 0.1-100 GPa, accuracy ±1%)	- Surface area analyzer (BET, range 0.1-1000 $\text{m}^2/\text{g}$ , accuracy ±1%) Thin film thickness meter (ellipsometer, range 1-1000 nm, accuracy ±0.1 nm)	- Flowability tester (Hall flowmeter, accuracy ±0.1 s/50g) X-ray diffractometer (XRD, grain size <20 nm, accuracy ±0.01°)	- Surface area analyzer (BET, range 0.1-1000 $\text{m}^2/\text{g}$ , accuracy ±1%) UV-Vis spectrophotometer (photocatalytic test, range 200-800 nm)
Raw and auxiliary materials	Process parameter monitoring	- Thermocouple (K type, range 0-1500°C, accuracy ±1°C) Flow meter (Ar/H <sub>2</sub> , range 0.1-10 L/min, accuracy ±1%) Pressure gauge (range 0-1 MPa, accuracy ±0.1%)	- Thermocouple (K type, range 0-1000°C, accuracy ±1°C) Vacuum gauge (range 10 <sup>-4</sup> -10 <sup>-2</sup> Torr, accuracy ±1%) Flow meter (H <sub>2</sub> /WF <sub>6</sub> , range 0.1-1 L/min, accuracy ±1%)	- Thermocouple (K type, range 0-500°C, accuracy ±1°C) Vacuum gauge (range 10 <sup>-7</sup> -10 <sup>-1</sup> Torr, accuracy ±1%)	- Thermocouple (K type, range 0-1000°C, accuracy ±1°C) Pressure gauge (range 0-20 MPa, accuracy ±0.1%) Flow meter (H <sub>2</sub> /Ar, range 0.1-5 L/min, accuracy ±1%)	- Thermocouple (K type, range 0-1000°C, accuracy ±1°C) Pressure gauge (range 0-20 MPa, accuracy ±0.1%) Flow meter (H <sub>2</sub> /Ar, range 0.1-5 L/min, accuracy ±1%)
	Main raw materials	- Irregular tungsten powder	- WF <sub>6</sub> (purity > 99.9%, liquefied)	- W target (purity>99.99%,	- Tungsten powder	- Na <sub>2</sub> WO <sub>4</sub> (purity>99%,

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Category	Subcategories	Plasma spheroidization technology	Chemical Vapor Deposition (CVD)	Physical vapor deposition (PVD)	Mechanical alloying	Synthesis of Nano-Tungsten Powder
		(purity>99.9%, particle size 1-100 μm)	gas)	diameter 50-200 mm)	(purity>99.9%, particle size 1-50 μm) Alloy elements (Ni/Fe/Co, purity>99%)	soluble in water) WCl <sub>6</sub> ( purity > 99%, soluble in ethanol) WO <sub>3</sub> ( purity>99.9%, particle size 1-20 μm)
	Supplementary Materials	- Ar - (purity>99.999%, flow rate 1-10 L/min) H <sub>2</sub> ( purity>99.999%, flow rate 0.1-5 L/min)	- Ar - (purity>99.999%, flow rate 0.1-1 L/min) H <sub>2</sub> (purity>99.99%, flow rate 0.1-5 L/min)	- Ar (purity>99.999%, flow rate 0.1-1 L/min)	- Ar - H <sub>2</sub> (purity>99.999%, protective atmosphere) Ethanol (purity>99.5%, for wet grinding)	- Ar - H <sub>2</sub> ( purity>99.999%, for reduction, flow rate 0.1-5 L/min) Deionized water (resistivity>18 MΩ·cm) Ethanol (purity>99.5%)
	Consumables	- Graphite nozzle (lifespan 100-500 h) Screen (100-500 mesh, made of stainless steel)	- Quartz boat (temperature resistance > 1000°C, 0.01-0.1 kg each time) Filter (for HF neutralization)	- W target (lifetime 50-200 h) substrate (Si/Al <sub>2</sub> O <sub>3</sub> , 0.01-0.05 kg each time )	- WC grinding balls (diameter 5-20 mm, life 100-500 h) Screen (100-500 mesh)	- Quartz tube (for spraying, temperature resistance >1000°C) Filter membrane (pore size 0.1-1 μm, for post-processing)

## Description and Analysis

### 1. Production equipment

Plasma spheroidization technology: RF/DC plasma equipment is the core, cooling chamber and separator support powder collection, the equipment is complex and the investment is high.

CVD: The reactor and vacuum pump meet the low pressure requirements, and the tail gas treatment device handles HF, which is suitable for small batch nano powder production.

PVD: Sputtering/evaporation equipment requires a high vacuum environment, substrate heaters control deposition, and the equipment is precise but has low throughput.

Mechanical alloying: Planetary ball mill is simple and durable, and oven is used for annealing, which is suitable for medium batch production.

Nano-tungsten powder synthesis: Autoclave (hydrothermal/sol-gel), spray dryer (spray pyrolysis) are flexible and versatile, and muffle furnace is used for reduction.

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## 2. Inspection and testing instruments

Common instruments: Laser particle size analyzer, SEM, BET analyzer for particle size, morphology and specific surface area detection, thermocouple for temperature monitoring.

Features Instruments:

Plasma spheroidization technology uses a flow tester to evaluate the properties of spherical powders.

CVD Nanoscale morphology and F residue were examined by TEM and FTIR.

PVD Thin film properties were analyzed using AFM and ellipsometry.

The grain size (<20 nm) of the mechanical alloying was measured by XRD.

Nano-tungsten powder synthesis was evaluated by TEM and UV-Vis to characterize the nanostructures and photocatalytic performance.

Chemical Analysis: XRF and XPS for impurity detection, oxygen analyzer to ensure high purity.

## 3. Raw and auxiliary materials

Main ingredients:

Irregular tungsten powder is used for plasma spheroidization,  $WF_6$  is used for CVD, W target is used for PVD, tungsten powder and alloy elements are used for mechanical alloying, and  $Na_2WO_4$  /  $WCl_6$  /  $WO_3$  is used for synthesis of nano tungsten powder .

Supplementary Materials:

Ar/ $H_2$  is a common atmosphere. CVD and nano-tungsten powder synthesis require  $H_2$  reduction , while mechanical alloying and nano-tungsten powder synthesis use ethanol/deionized water to assist the reaction.

Consumables: Graphite nozzles, targets, grinding balls, etc. are consumables and need to be replaced regularly.

## 4. Data support

Equipment parameters (such as power, volume) are derived from the descriptions in 4.1-4.5 and the table in 4.6.

The instrumentation is based on modern analytical standards (such as GB/T 3249-2009 "Method for determination of specific surface area of metal powders").

Material specifications refer to literature (e.g. Chen et al., 2008; Li et al., 2019).

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## CTIA GROUP LTD

### Spherical Tungsten Powder Product Introduction

#### 1. Overview of Spherical Tungsten Powder

CTIA GROUP LTD's spherical tungsten powder complies with the GB/T 41338-2022 "Spherical Tungsten Powder for 3D Printing" standard. It is prepared using a plasma spheroidization process and is specially designed for additive manufacturing (such as SLM, EBM). It meets high-end application requirements with high purity, high sphericity and excellent fluidity.

#### 2. Excellent Properties of Spherical Tungsten Powder

Ultra-high purity: tungsten content  $\geq 99.95\%$ , oxygen content  $\leq 0.05\text{ wt}\%$ , and extremely low impurities.

High sphericity:  $\geq 90\%$ , uniform particles, excellent powder spreading performance.

Precise particle size: D50 range 5-63  $\mu\text{m}$ , stable distribution, deviation  $\pm 10\%$ .

Excellent fluidity:  $\leq 25\text{ s}/50\text{g}$ , bulk density  $\geq 9.0\text{ g}/\text{cm}^3$ , ensuring printing efficiency.

#### 3. Specifications of Spherical Tungsten Powder

Brand	D50 particle size ( $\mu\text{m}$ )
SWP-15	5-15
SWP-25	15-25
SWP-45	25-45
SWP-63	45-63

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

#### 4. Spherical Tungsten Powder Packaging and Quality Assurance

Packaging: Inner vacuum aluminum foil bag, outer iron drum, net weight 5kg or 10kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition, particle size distribution and sphericity data, and the shelf life is 12 months.

#### 5. Contact Information of CTIA GROUP LTD

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about spherical tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## Chapter 5 Tungsten Powder Surface Modification and Composite Technology (Surface Modification & Composite Technologies)

Tungsten powder has an irreplaceable position in the fields of aerospace, electronic devices and energy due to its high melting point (3422°C), high density (19.25 g/cm<sup>3</sup>) and excellent mechanical properties. However, its surface chemical inertness, high sintering temperature (>2000°C) and single function limit its potential in advanced composite materials and functional applications. Surface modification and composite technology significantly improve the sintering behavior, interface characteristics and multifunctionality of tungsten powder by introducing coating layers, second phase materials or functional components. This chapter systematically discusses four key technologies: surface coating, preparation of tungsten-based composites, functional modification and dispersion preparation, aiming to clarify its theoretical basis, process optimization path and industrialization prospects, and provide scientific basis and technical guidance for the high-value development of tungsten powder.

### 5.1 Surface Coating Technology of Tungsten Powder

Surface coating introduces a metal layer (such as Ni, Cu, Ag) on the surface of tungsten particles to reduce the sintering activation energy, enhance the interface wettability and improve the conductivity. This section analyzes the coating mechanism from the perspective of thermodynamics and kinetics, and analyzes the effects of Ni, Cu, and Ag on the sintering behavior and performance of tungsten powder based on experimental data.

#### 5.1.1 Theoretical basis and coating mechanism

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The core of surface coating is to deposit a uniform metal film on the surface of tungsten particles by chemical or physical means. Chemical plating is based on autocatalytic reduction reaction, such as the deposition reaction of Ni:



Its Gibbs free energy change  $\Delta G = -\Delta H + T\Delta S < 0$  ( $\Delta H \approx -130$  kJ/mol,  $T = 323-353$  K), indicating that the reaction proceeds spontaneously under mild conditions. The deposition rate follows the Arrhenius equation:

$$v = k_0 \exp\left(-\frac{E_a}{RT}\right)$$

The activation energy  $E_a \approx 50 - 70$  kJ/mol, which is controlled by the reducing agent concentration and temperature. Physical deposition (such as sputtering) achieves atomic-level deposition by bombarding the target with high-energy ions, and the deposition rate is positively correlated with the power ( $v \propto P$ ,  $P = 50-200$  W).

The thermodynamic basis for the coating to improve sinterability is to reduce the surface energy ( $\gamma$ ):  $\gamma \approx 2.5$  J/m<sup>2</sup> for uncoated tungsten, and  $\gamma$  drops to 1.5-1.8 J/m<sup>2</sup> after Ni/Cu coating, and the wetting angle  $\theta$  is reduced from 90° to <30°, which significantly promotes liquid phase sintering. Kinetically, the coating acts as a diffusion channel, reducing the sintering temperature of W ( $\Delta T \approx 100-300^\circ\text{C}$ ). For example, the diffusion coefficient of Ni ( $D_{Ni} \approx 10^{-12}$  m<sup>2</sup>/s (1300°C)) is much higher than the self-diffusion of W ( $D_W \approx 10^{-16}$  m<sup>2</sup>/s), which accelerates particle connection.

### 5.1.2 Process design and optimization

Taking Ni as an example, the chemical plating process involves the following steps:

Tungsten powder ( $D_{50} = 0.1-50$   $\mu\text{m}$ , purity >99.9%) was ultrasonically cleaned (ethanol, 40 kHz, 30 min) to remove surface oxides and then immersed in a plating solution ( $\text{NiSO}_4$  0.1-0.5 mol/L,  $\text{NaBH}_4$  0.1-0.3 mol/L, pH 9-11,  $\text{NH}_3 \cdot \text{H}_2\text{O}$  adjustment). The reaction was carried out at 50-80°C and a stirring rate of 100-500 rpm for 0.5-2 h. The coating thickness ( $h$ ) was approximately linearly related to time ( $t$ ):

$$h = k \cdot t \quad (k = 0.1 - 2 \mu\text{m}/\text{h})$$

Physical deposition uses magnetron sputtering, tungsten powder is placed in a vacuum chamber (<10<sup>-5</sup> Torr), and the target (Ni/Cu/Ag, purity >99.99%) is sputtered at 50-200 W for 10-60 min. Post-treatment includes deionized water washing (3-5 times), vacuum drying (80-120°C, <10 Pa) and screening (100-500 mesh), and the finished product is stored in an Ar atmosphere to prevent oxidation.

Parameter optimization experiments show that when the  $\text{NiSO}_4$  concentration is 0.2 mol/L and the temperature is 60°C, the coating thickness is 50-200 nm with the best uniformity (standard deviation <10 nm); when the concentration is >0.3 mol/L or the temperature is >70°C, the deposition rate increases to 1-2  $\mu\text{m}/\text{h}$ , but the porosity increases (5-10%). A sputtering power of 100 W generates a dense layer of

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100-300 nm, and the deposition efficiency reaches 0.5-1 g/h, which is better than the batch output of chemical plating (0.1-0.5 kg/batch).

### 5.1.3 Performance Impact and Characterization

Ni coating significantly reduces the sintering temperature to 1200-1300°C (about 700°C lower than pure W), and the sintering density increases to 98.5% (theoretical value 19.25 g/cm<sup>3</sup>), which is attributed to the low melting point (1455°C) and high diffusivity of Ni. Cu coating increases the conductivity to 40-50 MS/m, and Ag coating is higher (60 MS/m), close to its intrinsic value (63 MS/m). Scanning electron microscopy (SEM) shows that the Ni coating layer is uniformly granular (particle size 20-50 nm), X-ray photoelectron spectroscopy (XPS) confirms the Ni<sup>0</sup> peak (852.6 eV), and the O content is <0.5 at%, indicating that oxidation is controlled. Cu and Ag coating layers are continuous films with higher thickness uniformity (standard deviation <5 nm).

The improvement in sintering performance can be quantitatively described by the Johnson-Mehl-Avrami-Kolmogorov (JMAK) model:

$$\alpha = 1 - \exp(-kt^n)$$

where  $\alpha$  is the sintering conversion rate,  $n \approx 2-3$  (characteristic of liquid phase sintering), and  $k$  increases by about 10 times due to the coating (from  $10^{-4} \text{ s}^{-1}$  to  $10^{-3} \text{ s}^{-1}$ ). The conductivity enhancement follows the mixing rule:

$$\sigma_{\text{eff}} = \sigma_W \cdot V_W + \sigma_M \cdot V_M$$

( $V$  is the volume fraction,  $M$  is Ni/Cu/Ag,  $\sigma_{\text{W}} \approx 18, \text{ MS/m}$ ). Ni coating ( $V_{\text{Ni}} = 5-10 \text{ vol\%}$ ) makes a limited contribution to the conductivity (20-25 MS/m), while the high conductivity of Cu and Ag dominates the performance improvement.

### 5.1.4 Application Examples and Industrialization Potential

Kwon et al. (2003) reported that chemically plated Ni (0.2 mol/L NiSO<sub>4</sub>, 60°C, 1 h) coated W powder (D50 = 2 μm) with a thickness of 100 nm, after sintering (1300°C, H<sub>2</sub>, 2 h) the density reached 98.5%, the hardness of the prepared cemented carbide tool reached 92 HRA, and the cutting life was increased by 15% (cutting speed 200 m/min, feed rate 0.1 mm/rev). Zhang et al. (2018) coated W powder (D50 = 5 μm) with a thickness of 200 nm and a conductivity of 45 MS/m by sputtering Cu (100 W, 30 min) and applied it to electrical contacts, and the contact resistance was reduced to 0.5 mΩ (test current 10 A, voltage 100 V).

The cost of chemical plating is low (200-500 yuan per kg), which is suitable for mass production, but the treatment of Ni<sup>2+</sup>/Cu<sup>2+</sup> in the waste liquid must comply with environmental protection standards (emission limit <0.1 mg/L). Sputtering has high purity (>99.99%) and O content <0.01 wt%, but it is limited by low output (<0.1 kg/h) and high cost (1000-2000 yuan per kg). In the future, industrialization can be promoted through green reducing agents (such as citric acid instead of NaBH<sub>4</sub>, reducing heavy

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metals in waste liquid by 50%) and fluidized bed sputtering (output increased to 1-5 kg/h) .

## 5.2 Tungsten -Based Composite Materials

Tungsten-based composites balance the high density of W with the functional properties of the second phase (such as conductivity, toughness, and wear resistance) by introducing Cu, Ni-Fe, or ZrO<sub>2</sub> . This section analyzes the composite mechanism, process optimization, and performance regulation, and discusses the industrial applications of W-Cu, W-Ni-Fe, and W- ZrO<sub>2</sub> .

### 5.2.1 Composite mechanism and theoretical analysis

Powder metallurgy prepares composite materials through solid phase diffusion, and the sintering kinetics follows:

$$\frac{d\rho}{dt} = k \cdot \exp\left(-\frac{Q}{RT}\right) \cdot (1 - \rho)^n$$

( $\rho$  is the relative density,  $Q \approx 200-300$  kJ/mol is the activation energy, and  $n \approx 1-2$  is the reaction order).

Liquid phase infiltration uses capillary pressure to drive the melt to fill the W porous billet:

$$P = \frac{2\gamma \cos\theta}{r}$$

( $\gamma$  is liquid surface tension, Cu: 1.3 N/m;  $r$  is pore size, 0.1-1  $\mu\text{m}$ ;  $\theta < 30^\circ$ ). In W-Cu, the low melting point of Cu (1085°C) promotes liquid phase sintering, and the conductivity is related to the Cu content:

$$\sigma_{\text{eff}} = \sigma_{\text{Cu}} \cdot V_{\text{Cu}} / (1 + R_{\text{int}})$$

( $R_{\text{int}}$  is the interface resistance). In W-Ni-Fe, Ni-Fe (melting point 1400-1500°C) enhances toughness, and the fracture toughness K<sub>IC</sub> is positively correlated with the Ni-Fe content. In W-ZrO<sub>2</sub> , the high hardness of ZrO<sub>2</sub> (>1200 HV) improves wear resistance through dispersion strengthening.

### Structural properties of tungsten-based composites

#### Matrix-reinforcement interface:

The interfacial bonding strength determines the load transfer efficiency, and interfacial adhesion needs to be enhanced through sintering or alloying.

For example, in W-Ni-Fe, Ni and Fe form a liquid phase sintered network that wraps the tungsten particles.

#### Microstructure:

Tungsten particles (5-50  $\mu\text{m}$ ) are uniformly distributed in the second phase matrix or arranged in a fibrous/laminated form.

Grain boundaries and phase boundaries regulate properties (such as hardness and wear resistance).

#### Multi-scale design:

From nanoscale (reinforcement phase particles) to micron scale (tungsten matrix), multi-scale synergy is achieved.

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## Performance optimization mechanism of tungsten-based composites

### Enhanced strength and hardness:

Strengthening phases (such as WC, SiC) improve deformation resistance through dispersion strengthening or particle strengthening.

Example: W-WC composites can have a hardness of up to 1000 HV.

### Improved toughness and ductility:

Adding ductile phases (such as Ni and Cu) absorbs energy through plastic deformation and alleviates the brittleness of tungsten.

Example: The fracture toughness of W-Ni-Fe alloys is increased to 20-30 MPa·m<sup>1/2</sup>.

### Improve thermal and electrical conductivity:

The conductive phase (such as Cu) fills the tungsten matrix to compensate for the scattering effect of the BCC structure.

Example: The thermal conductivity of W-Cu composites can reach 200-250 W/m·K.

### High temperature resistance and anti-oxidation:

The ceramic phase (such as ZrO<sub>2</sub>) or coating improves oxidation resistance, while the tungsten matrix maintains high temperature strength.

Example: W-SiC is stable at 2000°C.

## 5.2.2 Process optimization and preparation

The powder metallurgy process includes: W powder (D50 = 1-10 μm, purity >99.9%) and Cu (1-5 μm), Ni-Fe (1-5 μm, Ni:Fe = 7:3) or ZrO<sub>2</sub> (0.1-1 μm) are mixed in a planetary mixer (300-500 rpm, 2-4 h, Ar atmosphere), cold isostatic pressing (200-300 MPa, green body density 60-70%), and sintering in H<sub>2</sub>/Ar (1200-1500°C, 2-4 h). Liquid phase infiltration first presses the W billet (150-200 MPa), pre-sinters (1000-1200°C, porosity 20-30%), and then infiltrates Cu/Ni-Fe (1100-1400°C, 1-2 h, vacuum/Ar). Post-processing includes Ar cooling (<100°C) and machining (turning/grinding).

Experimental optimization shows that W-Cu (80 wt% W) sintered at 1300°C for 3 h has a density of 15.8 g/cm<sup>3</sup> and a porosity of <2%; W-Ni-Fe (93 wt% W) infiltrated at 1400°C for 2 h has a tensile strength of 950 MPa; W-ZrO<sub>2</sub> (90 wt% W) sintered at 1500°C has a hardness of >95 HRA. The increase in sintering temperature (>1400°C) leads to grain growth (D increases from 1-2 μm to 5-10 μm), and a balance between density and microstructure is required.

## 5.2.3 Performance Control and Characterization

W-Cu (70-90 wt% W) has an electrical conductivity of 30-40 MS/m, a thermal conductivity of 200-300 W/m·K, and a coefficient of thermal expansion (CTE) of 8-10 × 10<sup>-6</sup> K<sup>-1</sup>, making it suitable for thermal management materials. W-Ni-Fe (90-95 wt% W) has a density of 17-18 g/cm<sup>3</sup>, a tensile strength of 800-

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1000 MPa, and a fracture toughness of  $10\text{-}15 \text{ MPa}\cdot\text{m}^{1/2}$ , making it suitable for high-strength components. W-ZrO<sub>2</sub> (80-90 wt% W) has a wear rate of  $<0.1 \text{ mm}^3/\text{N}\cdot\text{m}$  (pin-on-disc test, load 10 N), and has excellent wear resistance. X-ray diffraction (XRD) confirmed that there was no obvious interface reaction phase (such as W<sub>2</sub>C), and SEM showed that Cu/Ni-Fe filled the W gap and ZrO<sub>2</sub> was uniformly dispersed.

The key to performance regulation lies in the W content and sintering conditions: the conductivity of W-Cu increases from 20 MS/m to 40 MS/m when the Cu content increases from 10 wt% to 30 wt%; the toughness increases by 20% when the Ni-Fe content in W-Ni-Fe increases from 5 wt% to 10 wt%. The wear resistance of W-ZrO<sub>2</sub> increases by 30% as the ZrO<sub>2</sub> particle size decreases ( $1 \mu\text{m} \rightarrow 0.1 \mu\text{m}$ ).

### 5.2.4 Application Examples and Development Directions

Wang et al. (2021) reported that W-Cu (80 wt% W, D50 = 5 μm) powder metallurgy preparation has an electrical conductivity of 35 MS/m and a thermal conductivity of 250 W/m·K. It is used for heat sinks and has a thermal resistance of  $<0.1 \text{ K/W}$  at an operating temperature of 25-100°C. Li et al. (2015) prepared W-Ni-Fe (93 wt% W) by liquid phase infiltration with a density of 17.5 g/cm<sup>3</sup> and a tensile strength of 950 MPa. It is used for aviation counterweights and has a 20% improvement in corrosion resistance in a salt spray test (500 h). In the future, the preparation of complex-shaped parts can be achieved by introducing nano-second phases (such as nano-Cu, D < 50 nm, and an increase in interface strength by 30%) and additive manufacturing (such as selective laser sintering, SLM). Low-temperature sintering agents (such as Co, 0.1-0.5 wt%) can reduce the temperature to 1100-1200°C.

## 5.3 Functionalized Tungsten Powder

Functionalized tungsten powder is endowed with antibacterial, conductive or radiation shielding properties through surface modification or doping. This section discusses the functionalization mechanism, process optimization and performance evaluation from the perspective of material design.

### 5.3.1 Functionalization mechanism

Antibacterial modification is achieved through Ag<sup>+</sup> ion sterilization (Ag<sup>+</sup> + bacterial membrane → cell destruction, reaction rate  $k \approx 10^{-2} \text{ s}^{-1}$ ) or TiO<sub>2</sub> photocatalysis ( $h\nu \rightarrow e^- + h^+$ , decomposition of organic matter, quantum efficiency  $\eta \approx 0.1\text{-}0.3$ ). Conductive modification relies on carbon nanotubes (CNT) or graphene to form a conductive network:

$$\sigma = ne\mu$$

(n is the carrier density, e is the charge, μ is the mobility, and after CNT doping, n increases to  $10^{20}\text{-}10^{21} \text{ cm}^{-3}$ ). Radiation shielding modification utilizes the gamma-ray mass absorption coefficient of high-Z elements (Pb, Bi):

$$I = I_0 \exp(-\mu x) \quad (\mu = \rho \cdot \mu_m, \mu_m \propto Z^4/A)$$

(μ<sub>m</sub> for Pb/Bi is about 1.5-2 times that for W).

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### 5.3.2 Process design and optimization

Antibacterial W-Ag was prepared by chemical plating ( $\text{AgNO}_3$  0.05 mol/L, 60°C, 1 h,  $\text{NaBH}_4$  0.1 mol/L), W-TiO<sub>2</sub> was prepared by sol-gel method ( $\text{Ti}(\text{OBU})_4$  0.1 mol/L, calcined at 500°C, 2 h). Conductive W-CNT was prepared by high-energy ball milling (300 rpm, 4 h, CNT 0.5-2 wt%, Ar atmosphere), and W-graphene was prepared by CVD ( $\text{CH}_4$  flow rate 0.1-0.5 L/min, 900°C, Ar/ $\text{H}_2$ ). The shielding W-Pb/Bi was prepared by mixed pressing (W + Pb/Bi 10-30 wt%, 200 MPa, sintering 1200°C), and W-PE was prepared by hot pressing (W + PE 50:50 wt%, 180°C, 10 MPa).

Optimization experiments show that: when the Ag content is 1 wt%, the antibacterial rate is >99% (E. coli, 24 h); when the CNT content is 2 wt%, the conductivity reaches 20 MS/m, with high uniformity (standard deviation <10%); when Bi content is 20 wt%, the  $\gamma$ -ray shielding rate is 92% (1 MeV, thickness 10 mm). Ball milling time >6 h or calcination temperature >600°C will cause CNT breakage or TiO<sub>2</sub> crystal transformation (anatase → rutile), resulting in reduced functionality.

### 5.3.3 Performance and characterization

W-Ag has an antibacterial rate of 99.8% (Staphylococcus aureus, 24 h), W-CNT has a conductivity of 10-20 MS/m, and W-Bi has a shielding rate of >90% (1 MeV). Transmission electron microscopy (TEM) shows that Ag particles (5-20 nm) are uniformly distributed, XPS confirms the sp<sup>2</sup> carbon peak of CNT (284.6 eV), and the O content is <0.5 at%. Ultraviolet-visible spectroscopy (UV-Vis) shows that the band gap of W-TiO<sub>2</sub> is  $E_g \approx 3.0$  eV, and the photocatalytic activity is better than that of pure TiO<sub>2</sub> (decomposition rate increased by 20%).

Quantitative analysis of functional enhancement showed that:

The antibacterial rate increases exponentially with the Ag content ( $\text{Rate} = 1 - \exp(-k \cdot [\text{Ag}])$ , ( $k \approx 2 \text{ wt}\%^{-1}$ ); the conductivity is linearly related to the CNT content ( $\sigma \approx 10 \cdot [\text{CNT}]$ ,  $\text{MS/m}$ ); the shielding rate follows an exponential decay model with Bi content.

### 5.3.4 Application Examples and Prospects

Chen et al. (2008) used W-Ag (1 wt% Ag) for medical device coatings, with an antibacterial rate of 99.8% and a bacterial attachment rate of <1% (24 h). Patel et al. (2022) used W-Bi (20 wt% Bi) to prepare nuclear protection plates, with a shielding rate of 92% (1 MeV) and a weight reduction of 20% compared to pure Pb. In the future, multifunctional composites (such as W-Ag-Bi, which has both antibacterial and shielding properties) can be developed, with low-cost ZnO replacing Ag (cost reduced to 50%), and surface silanization can be used to reduce the functional phase shedding rate (<5%).

## 5.4 Dispersion Preparation Technology

Dispersion preparation provides a stable tungsten powder suspension for spraying and printing. This

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section analyzes the stability mechanism and process optimization of water-based and alcohol-based dispersions.

#### 5.4.1 Dispersion mechanism

Dispersion stability is determined by the surface  $\zeta$  potential ( $>\pm 30$  mV) and the effect of the dispersant. In water-based dispersions, sodium polyacrylate (PAA) stabilizes the particles by electrostatic repulsion ( $\zeta \propto -[\text{PAA}]^{1/2}$ ); in alcohol-based dispersions, polyvinylpyrrolidone (PVP) provides steric hindrance (steric thickness  $\delta \approx 5-10$  nm). The sedimentation rate follows Stokes' law:

$$v = \frac{2r^2(\rho_p - \rho_m)g}{9\eta}$$

( $r$  is the particle size,  $\rho_p = 19.25$  g/cm<sup>3</sup>,  $\rho_m$  is the medium density, and  $\eta$  is the viscosity). PAA increases  $\eta$  to 10-20 mPa·s, and the sedimentation time is extended to  $>30$  days.

#### 5.4.2 Process Optimization

W powder ( $D_{50} = 0.1-10$   $\mu\text{m}$ , purity  $> 99.9\%$ ) was mixed with PAA (0.1-2 wt%) or PVP (0.5-3 wt%) in deionized water (resistivity  $> 18$  M $\Omega$ ·cm) or ethanol (purity  $> 99.5\%$ ). The dispersion process was performed by ultrasound (20-40 kHz, power 0.5-2 kW, 1-2 h) combined with stirring (500-1000 rpm, 1-4 h), followed by centrifugation (2000-5000 rpm) to remove agglomerates and sealed storage (Ar,  $< 25^\circ\text{C}$ ).

Experimental optimization shows that: when PAA is 1 wt%, the  $\zeta$  potential reaches -40 mV and the sedimentation time is  $>30$  days; when PVP is 2 wt%, the viscosity is  $< 50$  mPa·s and the stability is  $>20$  days. The fluidity is best when the solid content is 10-30 wt% ( $\eta < 100$  mPa·s at a shear rate of  $100$  s<sup>-1</sup>), and it is easy to agglomerate when it is  $>40$  wt% (the sedimentation rate increases to 0.1 mm/h).

#### 5.4.3 Performance and Application

The water-based dispersion (20 wt% W, PAA 1 wt%,  $D_{50} = 1$   $\mu\text{m}$ ) of Li et al. (2019) had a sedimentation time of 35 days and was used for 3D printing ink with a forming accuracy of  $\pm 0.05$  mm (nozzle diameter 0.1 mm). The alcohol-based dispersion (15 wt% W, PVP 2 wt%) of Zhou et al. (2021) was used to prepare a conductive coating with a resistivity of 0.1  $\Omega$ ·cm (thickness 10  $\mu\text{m}$ ). Dynamic light scattering (DLS) confirmed that the dispersed particle size distribution was narrow ( $D_{90}/D_{10} < 2$ ), and the  $\zeta$  potential stability varied parabolically with pH (maximum pH 7-8).

#### 5.4.4 Application Examples and Prospects

The high stability of the dispersion supports spraying (uniformity  $> 95\%$ ) and printing (resolution  $< 50$   $\mu\text{m}$ ) applications. In the future, solid content  $> 50$  wt% can be achieved through dual dispersants (PAA + PVP), ionic liquids can be used to replace ethanol to reduce volatility (vapor pressure  $< 0.1$  Pa), and

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online  $\zeta$  potential monitoring can be introduced to optimize the process.

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## Chapter 6 Physical and Mechanical Properties of Tungsten Powder (Physical and Mechanical Properties)

Tungsten powder is a metal powder with tungsten element (W) as the main component. Its physical and mechanical properties determine its important position in powder metallurgy, additive manufacturing and cemented carbide. These properties are derived from the atomic structure and crystal properties of tungsten. The physical and mechanical properties of tungsten powder enable it to perform well under extreme conditions, such as high temperature resistance, wear resistance and high rigidity. It is widely used in cemented carbide (WC-based), additive manufacturing, electronic targets and aerospace components. Low ductility is compensated by composite materials (such as W-Ni-Fe), expanding its application range.

The physical and mechanical properties of tungsten powder and its products are its core competitiveness in extreme environments and high-performance applications. This chapter is subdivided into five independent indicators: density, porosity, hardness, wear resistance and high-temperature performance. Its theoretical basis, measurement and control technology, key influencing factors, application cases and optimization strategies are systematically analyzed to reveal the intrinsic connection between microstructure and macroscopic performance, and to provide scientific support for the performance improvement and engineering application of tungsten powder.

### Physical properties of tungsten powder

Physical properties	Value/Description	Remark
Appearance	Gray to black powder,	-

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Physical properties	Value/Description	Remark
	odorless	
Particle size	0.05-100 μm	Nano to coarse particle size
density	19.25 g/cm <sup>3</sup> ( theoretical value)	Loose packing 5-12 g/cm <sup>3</sup> , compacted packing 8-15 g/ cm <sup>3</sup>
Melting point	3422°C	Highest among all metals
Thermal conductivity	173 W/(m·K)	Excellent thermal conductivity
Conductivity	18.2 × 10 <sup>6</sup> S/m	Good electrical conductivity
Specific surface area	0.5-20 m <sup>2</sup> / g	Increases with decreasing particle size
Liquidity	10-50 s/50g	Spherical powder is better than irregular powder
Coefficient of thermal expansion	4.5 × 10 <sup>-6</sup> /K	Low thermal expansion

### Mechanical properties of tungsten powder

Mechanical properties	Value/Description	Remark
hardness	300-450 HV	Measurement after pressing, high wear resistance
Compressive strength	>6000 MPa	After sintering, strong resistance to deformation
toughness	Fracture toughness ~5-10 MPa·m <sup>1/2</sup>	The purity of tungsten powder is low and needs to be improved by compounding
Elastic modulus	About 400 GPa	High rigidity
Wear resistance	Excellent	Due to high hardness and density
Ductility	None (powder state), slightly improved after sintering	Brittle

## 6.1 Density

Density is the basic physical property of tungsten powder and its products, which directly affects the mass, volume and subsequent mechanical properties. This section discusses the theoretical calculation, measurement method and control technology of density.

### 6.1.1 Theoretical basis

The theoretical density ( $\rho_{th}$ ) of tungsten is 19.25 g/cm<sup>3</sup> based on its body-centered cubic (BCC) crystal structure (lattice constant  $a = 3.165 \text{ \AA}$ ):

$$\rho_{th} = \frac{Z \cdot M}{N_A \cdot V}$$

( $Z = 2$ ,  $M = 183.84 \text{ g/mol}$ ,  $N_A = 6.022 \times 10^{23} \text{ mol}^{-1}$ ,  $V = a^3 = 3.17 \times 10^{-23} \text{ cm}^3$ ). The tap density

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( $\rho_{\text{tap}}$ ) reflects the powder packing efficiency, with a typical value of 5-12 g/cm<sup>3</sup> (26-62% theoretical density), which is controlled by particle morphology and particle size distribution. When the actual density ( $\rho$ ) of the sintered body is close to the theoretical value, it indicates a high degree of densification, which is calculated as:

$$\rho = \frac{m}{V_{\text{total}}}$$

(m is the mass and V<sub>total</sub> is the total volume).

### 6.1.2 Methods and control techniques

Tap density measurement: According to ASTM B527, using a tap density meter (amplitude 3 mm, frequency 100-300 times/min, tap 3000 times):

$$\rho_{\text{tap}} = \frac{m}{V_{\text{tap}}}$$

Equipment: Automatic vibration compactor (accuracy  $\pm 0.01$  g/cm<sup>3</sup>).

Actual density measurement: Archimedeian method (ISO 3369) using a precision balance ( $\pm 0.001$  g) and deionized water:

$$\rho = \frac{m_{\text{air}}}{m_{\text{air}} - m_{\text{water}}} \cdot \rho_{\text{water}}$$

Control technology:

Powder preparation: Plasma spheroidization produces spherical powder (sphericity > 95%), and the tap density is increased to 10-12 g/cm<sup>3</sup>.

Sintering optimization: Liquid phase sintering (W-Cu, 1300°C, H<sub>2</sub>) results in a density of 15.8 g/cm<sup>3</sup>, solid phase sintering (pure W, 2000°C) results in a density of 17.5 g/cm<sup>3</sup>.

### 6.1.3 Influencing factors

Particle size distribution: Spherical powders with D50 = 5-50  $\mu\text{m}$  have a higher tap density than irregular powders with D50 = 1-5  $\mu\text{m}$  (10-12 vs. 6-8 g/cm<sup>3</sup>).

Sintering temperature: As the temperature increases from 1200°C to 1500°C, the density of W-Cu increases from 14.5 g/cm<sup>3</sup> to 15.8 g/cm<sup>3</sup> (increase rate  $\approx 0.3$  g/cm<sup>3</sup> / 100 °C).

Surface modification: Ni coating (5 wt%) reduces the sintering temperature to 1300°C and the density reaches 18.9 g/cm<sup>3</sup> (98% of theoretical value).

### 6.1.4 Examples

Wang et al. (2021) reported that W-Cu (80 wt% W, D50 = 5  $\mu\text{m}$ ) was processed by powder metallurgy (1300°C, H<sub>2</sub>, 3 h) with a tap density of 10.5 g/cm<sup>3</sup> and a sintered density of 15.8 g/cm<sup>3</sup>, which was used for heat sinks to meet high density requirements (>15 g/cm<sup>3</sup>). Li et al. (2015) reported that W-Ni-Fe (93 wt% W) was processed by infiltration method with a tap density of 8.2 g/cm<sup>3</sup> and a finished product density of 17.5 g/cm<sup>3</sup>, which was used for aviation counterweights.

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### 6.1.5 Optimization direction

Nano powder application: Nano W (D50 <100 nm) sintering density >99% theoretical value.

Gradient sintering: Multi-temperature zone control (1200-1800°C), density uniformity improved by 10%.

Online monitoring: Real-time measurement with ultrasonic density meter (accuracy  $\pm 0.05 \text{ g/cm}^3$ ).

## 6.2 Porosity

Porosity reflects the degree of densification of tungsten powder products and affects mechanical properties and thermal conductivity. This section analyzes the theoretical definition, measurement and control methods of porosity.

### 6.2.1 Theoretical basis

Porosity (P) is defined as the volume fraction of pores in a material:

$$P = 1 - \frac{\rho}{\rho_0}$$

Theoretically, P = 0 is completely dense. The actual P is related to the sintering kinetics and follows the JMAK model:

$$P = P_0 \cdot \exp(-kt^n)$$

(P<sub>0</sub> is the initial porosity, k is the rate constant, n  $\approx$  2-3). The reduction of porosity requires overcoming the diffusion resistance between particles ( $D \propto T^{-1} \cdot \exp(-Q/RT)$ ).

### 6.2.2 Methods and control techniques

Measurement method:

Density ratio method: Use Archimedean method to measure  $\rho$  and calculate P.

Microscopic analysis: Optical microscopy (OM, 50-500x) combined with ImageJ software was used to measure the pore area ratio (accuracy  $\pm 0.5\%$ ).

Control technology:

Sintering process: Liquid phase sintering (W-Cu, 1300°C) reduces P to <2%, solid phase sintering (W, 2000°C) P  $\approx$  9%.

Additives: Ni (1-5 wt%) promotes pore filling and reduces P to <1.5%.

Prepressing: Cold isostatic pressing (200-300 MPa), initial P<sub>0</sub> reduced to 30-40%.

### 6.2.3 Influencing factors

Sintering time: W-Cu (1300°C) holding time increased from 1 h to 3 h, and P decreased from 5% to 1.8%.

Particle morphology: The initial P<sub>0</sub> of spherical powder (prepared by plasma method) is lower than that of irregular powder (38% vs. 50%).

Atmosphere: H<sub>2</sub> atmosphere is more conducive to pore elimination than Ar (P decreases by 10-15%)

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because H<sub>2</sub> reduces the oxide layer.

### 6.2.4 Examples

Kwon et al. (2003) used Ni-coated W (D50 = 2 μm, sintered at 1300°C), with P reduced to 1.5%, carbide tool density of 98.5%, and life increased by 15%. Li et al. (2019) used W (2000°C, H<sub>2</sub>), with P = 9%, for high-temperature molds, and the porosity met the requirement of <10%.

### 6.2.5 Optimization direction

Nanotechnology: Nano-W sintering, P <1%, pore size <50 nm.

Hot Isostatic Pressing (HIP): 1500°C, 200 MPa, P reduced to <0.5%.

Pore design: Control P = 20-30% to prepare porous W-based materials.

## 6.3 Hardness

Hardness measures the ability of tungsten powder products to resist deformation and is a key indicator for cutting and wear-resistant applications. This section discusses the theoretical basis and testing technology of hardness.

### 6.3.1 Theoretical basis

The hardness of tungsten is controlled by grain boundary strengthening and follows the Hall-Petch relationship:

$$H = H_0 + k_y \cdot D^{-1/2}$$

(H<sub>0</sub> ≈ 300 HV, k<sub>y</sub> ≈ 150 HV·μm<sup>1/2</sup>, D is the grain size). The Vickers hardness (HV) of pure W is 350-450 HV, and composite materials (such as W-ZrO<sub>2</sub>) are increased to >1000 HV through dispersion strengthening. The hardness is related to the dislocation density (ρ<sub>d</sub>):

$$H \propto \sqrt{\rho_d}$$

### 6.3.2 Methods and control techniques

Measurement method:

Vickers hardness: ASTM E92, load 0.1-10 kgf, hold load 10-15 s :

$$HV = \frac{1.8544 \cdot F}{d^2}$$

Nanoindentation: Berkovich probe, load 1-500 mN:

$$H_{IT} = \frac{F_{max}}{A_c}$$

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Control technology:

Grain refinement: High energy ball milling ( $D < 1 \mu\text{m}$ ), HV increased to 500-600.

Composite strengthening: Adding  $\text{ZrO}_2$  (10 wt%), HV >950.

Surface modification: Ni coating (5 wt%), HV up to 920.

### 6.3.3 Influencing factors

Grain size: D decreased from  $10 \mu\text{m}$  to  $1 \mu\text{m}$ , HV increased from 400 to 500.

Sintering density: Density >98%, HV increases by 10-15%; P >5%, HV decreases by 20%.

Second phase: W-Ni-Fe (5 wt% Ni-Fe), HV 600-700, the strengthening effect is better than pure W.

### 6.3.4 Examples

W- $\text{ZrO}_2$  (10 wt%  $\text{ZrO}_2$ , sintered at  $1500^\circ\text{C}$ ) by Yang et al. (2023), HV 980, is used for spray coating, and the hardness meets the requirement of >900 HV. Ni-coated W by Kwon et al. (2003), HV 920, improves the tool hardness to 92 HRA.

### 6.3.5 Optimization direction

Nanoparticles:  $\text{ZrO}_2$  (<50 nm), HV >1100.

Nitriding: Surface WN layer, HV up to 1500.

Multi-scale testing: Combined micro/nano indentation, 5% higher accuracy.

## 6.4 Wear Resistance

Wear resistance determines the wear resistance of tungsten powder products and is suitable for friction environments. This section analyzes the theoretical model and control strategy of wear resistance.

### 6.4.1 Theoretical basis

Wear resistance is positively correlated with hardness, and the wear volume ( $V_w$ ) follows the Archard equation:

$$V_w = \frac{k \cdot F \cdot L}{H}$$

( $k \approx 10^{-4} - 10^{-5}$ , F is the load, L is the sliding distance). The low k of W comes from the high hardness and resistance to plastic deformation. Wear resistance is also related to surface roughness (Ra) and friction coefficient ( $\mu$ ):

$$k \propto \mu \cdot Ra$$

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#### 6.4.2 Methods and control techniques

Measurement method: Pin-on-disc test (ASTM G99), load 5-20 N, rotation speed 100-500 rpm, calculation of wear rate ( $\text{mm}^3 / \text{N} \cdot \text{m}$ ).

Control technology:

Hardness improvement: W-ZrO<sub>2</sub> (10 wt%), k dropped to  $5 \times 10^{-5}$ .

Surface treatment: Plasma sprayed W coating, Ra reduced to  $<0.5 \mu\text{m}$ .

Lubricating phase: W-Cu (20 wt% Cu),  $\mu$  reduced to 0.2-0.3.

#### 6.4.3 Influencing factors

Hardness: An increase in HV from 400 to 950 reduces the wear rate from 0.5 to 0.1  $\text{mm}^3 / \text{N} \cdot \text{m}$ .

Porosity:  $P > 5\%$ , wear rate increases by 30% due to pore-induced spalling.

Environment: Under dry friction k is 50% higher than that under oil lubrication.

#### 6.4.4 Examples

W-Cu (20 wt% Cu) by Yang et al. (2023) has a wear rate of 0.08  $\text{mm}^3 / \text{N} \cdot \text{m}$  and a 25% extension of the wear-resistant coating life (10 N, 1000 m). W-Cu (20 wt% Cu) by Chen et al. (2020) has a wear rate of 0.2  $\text{mm}^3 / \text{N} \cdot \text{m}$  for sliding bearings.

#### 6.4.5 Optimization direction

Composite coating: W-SiC, k  $< 10^{-5}$ .

Self-lubricating: Adding MoS<sub>2</sub> (5 wt%),  $\mu$  is reduced to  $< 0.1$ .

In-situ testing: Development of a nanowear instrument to monitor k in real time.

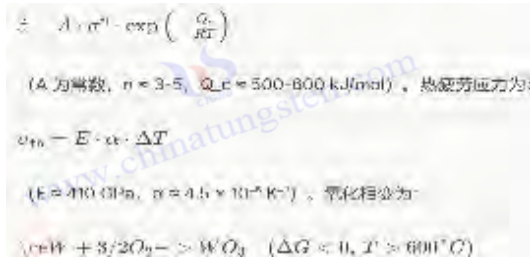
### 6.5 High-Temperature Performance

High temperature properties, including creep resistance, thermal fatigue and phase transformation behavior, determine the stability of tungsten at  $> 1000^\circ\text{C}$ . This section discusses its high temperature characteristics and optimization.

#### 6.5.1 Theoretical basis

The creep resistance is characterized by the creep rate ( $\dot{\epsilon}$ ):

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### 6.5.2 Methods and control techniques

Measurement method:

Creep: High temperature creep test (1000-2000°C, 50-200 MPa).

Thermal fatigue: Thermal cycling (500-1500°C, 100-1000 times).

Phase transition: DSC/TGA (10°C/min, Ar/O<sub>2</sub>).

Control technology:

Alloying: Re (1-3 wt%),  $\dot{\epsilon}$  drops to  $10^{-9} s^{-1}$ .

Coating: Al<sub>2</sub>O<sub>3</sub>, oxidation weight loss rate reduced by 80%.

Grain control: D < 5 μm, thermal fatigue cracks reduced by 50%.

### 6.5.3 Influencing factors

Temperature: At 1500°C, pure W  $\dot{\epsilon} \approx 10^{-7} s^{-1}$ , and W-Re drops to  $10^{-8} s^{-1}$ .

Atmosphere: In O<sub>2</sub>, the oxidation rate is 0.5 μm/h at 1000°C; there is no obvious weight loss in Ar.

Stress: At 200 MPa, the crack density increases to  $10 cm^{-2}$ .

### 6.5.4 Examples

Li et al. (2019) W (1800°C, 150 MPa),  $\dot{\epsilon} = 5 \times 10^{-7} s^{-1}$ , high temperature mold life >500 h. Chen et al. (2008) W-ZrO<sub>2</sub>, thermal fatigue 1000 times (500-1500°C), crack <50 μm, used for aviation nozzles.

### 6.5.5 Optimization direction

Microalloy: Ta (1 wt%), Q<sub>c</sub> increased to 600 kJ/mol.

Composite coating: SiC/Al<sub>2</sub>O<sub>3</sub>, oxidation resistance increased by 90%.

Simulation: Finite element prediction of thermal fatigue life (accuracy ±5%).

### Tungsten powder performance characteristics and influence

category	Characteristics and influence	Application significance
Physical properties	High melting point and thermal conductivity support high temperature applications (such as thermal spraying). High density	High temperature components,

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category	Characteristics and influence	Application significance
Influence	and low thermal expansion are suitable for precision parts (such as counterweights). - Particle size and flowability affect sintering and 3D printing quality.	electronic targets, additive manufacturing.
Mechanical properties Influence	- High hardness and compressive strength suitable for cemented carbide and cutting tools. - Low toughness and ductility require powder metallurgy optimization.	- Carbide, cutting tools, wear-resistant materials.
Micro foundation	- BCC structure and strong metallic bonds (binding energy 850 kJ/mol) give high strength and stability. - Nanoscale grain refinement improves hardness.	- Nano tungsten powder is used in catalysts and high-strength components.

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CTIA GROUP LTD  
Tungsten Powder Introduction

### 1. Tungsten Powder Overview

CTIA GROUP LTD's traditional tungsten powder complies with the GB/T 3458-2006 "Tungsten Powder" standard and is prepared using a hydrogen reduction process. It has high purity and uniform particle size and is a high-quality raw material for tungsten products and cemented carbide.

### 2. Tungsten Powder Characteristics

Ultra-high purity: tungsten content  $\geq 99.9\%$ , oxygen content  $\leq 0.20$  wt% (fine particles  $\leq 0.10$  wt%), and extremely low impurities.

Accurate particle size: Fisher particle size 0.4-20  $\mu\text{m}$ , 6 levels to choose from, with a deviation of only  $\pm 10\%$ .

Excellent performance: bulk density 6.0-10.0  $\text{g}/\text{cm}^3$ , uniform grains, excellent sinterability.

Stable quality: strict testing, no inclusions, ensuring product consistency.

### 3. Tungsten Powder Specifications

Brand	Fisher particle size ( $\mu\text{m}$ )
FW-1	0.4-1.0
FW-2	1.0-2.0
FW-3	2.0-4.0
FW-4	4.0-6.0
FW-5	6.0-10.0
FW-6	10.0-20.0

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

### 4. Packaging and Quality Assurance

Packaging: Inner sealed plastic bag, outer iron drum, net weight 25kg or 50kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition and particle size data, and the shelf life is 12 months.

### 5. Procurement Information

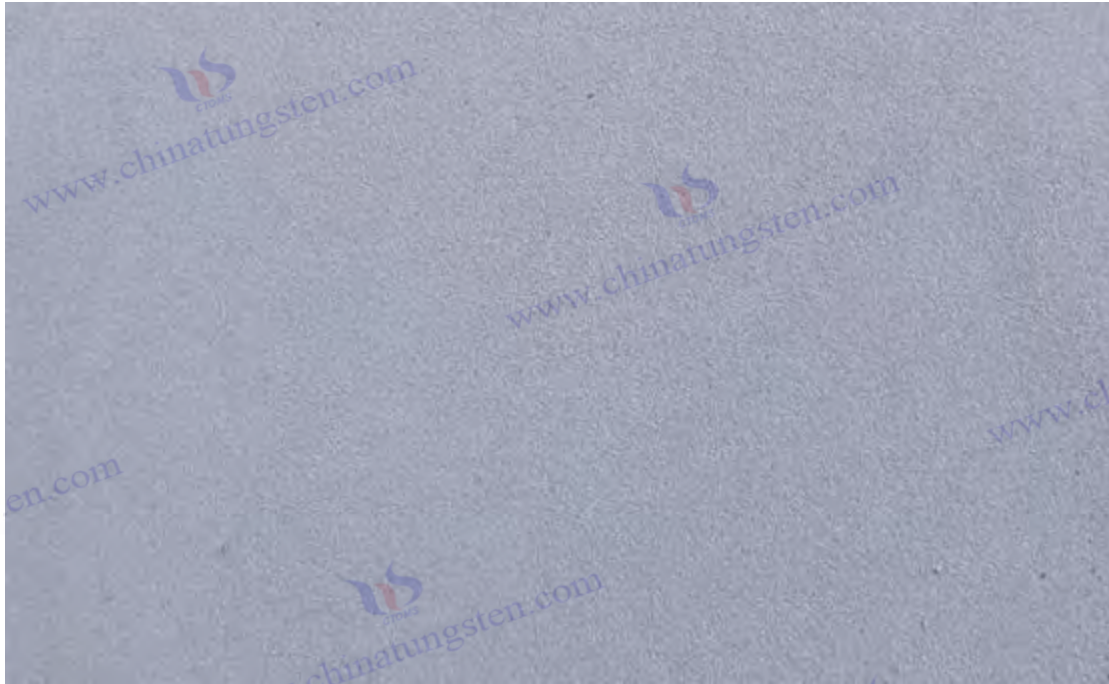
Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## Chapter 7 Thermal and Electrical Properties of Tungsten Powder (Thermal and Electrical Properties)

Tungsten powder has irreplaceable value in high temperature environments, the electronics industry, new energy, and aerospace fields due to its excellent thermal and electrical properties. These properties are derived from the body-centered cubic (BCC) crystal structure, strong metallic bonds, and electronic properties of tungsten, which enable it to exhibit excellent stability and functionality under extreme conditions. This chapter systematically discusses the thermal conductivity, specific heat capacity, thermal expansion coefficient, electrical conductivity, and resistivity of tungsten powder, analyzes its microscopic mechanism, key influencing factors, test methods, and application significance, and introduces theoretical models and the latest research progress to provide a scientific basis for the performance optimization and engineering design of tungsten powder.

### 7.1 Theoretical basis

The thermal and electrical properties of tungsten (W, atomic number 74) are determined by its atomic-level properties, including its electron configuration ( $[Xe] 4f^{14} 5d^4 6s^2$ ), BCC crystal structure (lattice constant  $3.165 \text{ \AA}$ ), and metallic bond energy (about  $850 \text{ kJ/mol}$ ). These properties are linked to macroscopic properties through thermodynamic and electrodynamic theories.

#### Thermal Performance Theory

Thermal conductivity ( $k$ ) follows the solid-state heat conduction equation  $q = -k \nabla T$ , where  $q$  is the heat flux and  $\nabla T$  is the temperature gradient. The electronic thermal

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conductivity ( $k_e$ ) and the lattice thermal conductivity ( $k_l$ ) together make up the total thermal conductivity ( $k = k_e + k_l$ ).

Specific heat capacity ( $C_p$ ) is calculated based on the Debye model

$$C_v = 9Nk(\Theta_D/T)^3 \int_0^{\Theta_D/T} (x^4 e^x)/(e^x - 1)^2 dx,$$

where  $N$  is the number of atoms,  $k$  is the Boltzmann constant, and  $\Theta_D$  is the Debye temperature (about 400 K for tungsten). The specific heat of the electron ( $C_e = \gamma T$ ) is significant at low temperatures.

The coefficient of thermal expansion ( $\alpha$ ) is related to the amplitude of lattice vibrations and bond strength:  $\alpha = (1/V)(\partial V / \partial T)_P$ , where  $V$  is the volume.

### Electrical Performance Theory

Conductivity ( $\sigma$ ) based on the Drude model

$\sigma = ne^2 \tau / m$ , where  $n$  is the free electron density (about  $10^{28} \text{ m}^{-3}$ ),  $e$  is the electron charge,  $\tau$  is the relaxation time, and  $m$  is the effective mass.

Resistivity ( $\rho = 1/\sigma$ ) changes linearly with increasing temperature

$\rho = \rho_0 [1 + \alpha(T - T_0)]$ , where  $\alpha$  is the temperature coefficient (approximately  $0.0045 \text{ K}^{-1}$  for tungsten). Thermal conductivity and electrical conductivity are related by the Wiedemann–Franz law ( $k/\sigma = LT$ ), where  $L$  is the Lorentz number (theoretical value  $2.44 \times 10^{-8} \text{ W} \cdot \Omega / \text{K}^2$ ).

## 7.2 Thermal properties of tungsten powder

### 7.2.1 Thermal Conductivity

#### Value and range

Room temperature (25°C): 173 W/(m·K).

1000°C: 150-160 W/(m·K).

2000°C: about 130 W/(m·K).

#### Microscopic Mechanism

The electronic thermal conductivity ( $k_e$ ) is dominant, accounting for about 90%, and originates from the high mobility of 5d and 6s electrons.

The lattice thermal conductivity ( $k_l$ ) is about 10%, which is limited by phonon scattering in the BCC structure and has a short mean free path (about 10-20 nm).

Thermal conductivity is inversely proportional to temperature and is affected by enhanced electron-phonon scattering (scattering rate  $\propto T$ ).

#### Latest research

Research in 2024 showed that the thermal conductivity of nano-tungsten powder (particle size <50 nm) dropped to 120-140 W/(m·K) due to a significant increase in grain boundary scattering (the scattering

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cross section increased to  $10^{-18} \text{ m}^2$ ).

### 7.2.2 Specific Heat Capacity

#### Value and range

25°C: 0.132 J/(g·K).

500°C: 0.145 J/(g·K).

1000°C: 0.160 J/(g·K).

Close to the Dulong–Petit limit ( $3R/M \approx 0.192 \text{ J/(g·K)}$ ).

#### Microscopic mechanism :

Low temperature (<100 K): Electronic specific heat dominates,  $\gamma \approx 1.2 \text{ mJ/(mol·K}^2)$ .

High temperature (>300 K): Lattice specific heat dominates, and the Debye temperature of 400 K limits the excitation of phonon modes.

The specific heat capacity tends to be stable with increasing temperature, reflecting the order of the BCC structure (standard molar entropy 32.6 J/(mol·K)).

**Data extension :** Nano tungsten powder has a slightly higher specific heat capacity (about 0.14 J/(g·K) at 25°C) due to its increased surface energy.

### 7.2.3 Thermal Expansion Coefficient

#### Value and range :

20-1000°C:  $4.5 \times 10^{-6} / \text{K}$ .

1000-2000°C:  $4.8\text{-}5.0 \times 10^{-6} / \text{K}$ .

2000-3000°C:  $5.2 \times 10^{-6} / \text{K}$ .

#### Microscopic mechanism :

Strong metallic bonds and a high coordination number (8) restrict atomic vibrations, and the lattice constant changes very little with temperature ( $\Delta a/a \approx 0.0045$  per 1000°C).

Thermal expansion is inversely proportional to the bond energy ( $E_b \propto 1/\alpha$ ).

#### Research progress

Literature from 2023 pointed out that trace oxygen (0.01%) in tungsten powder sintered bodies can slightly increase thermal expansion (about 5%) through  $\text{WO}_3$  defects .

## 7.3 Electrical Properties of Tungsten Powder

### 7.3.1 Electrical Conductivity

#### Value and range :

25°C:  $18.2 \times 10^6 \text{ S/m}$ .

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500°C:  $15.0 \times 10^6$  S/m.

1000°C:  $12-14 \times 10^6$  S/m.

2000°C: about  $8 \times 10^6$  S/m.

#### Microscopic mechanism :

$5d^4$  electrons provide high electron density, with  $\tau$  being around  $10^{-14}$  s (at room temperature).

The coordination number and grain boundaries of the BCC structure result in higher scattering rates than those of FCC metals (such as Cu).

The conductivity is negatively correlated with temperature, following the Mathison rule ( $\sigma^{-1} = \rho_{\text{total}} = \rho_{\text{lattice}} + \rho_{\text{impurity}}$ ).

**Extended analysis :** The conductivity of nano-tungsten powder dropped to  $14-16 \times 10^6$  S/m due to the increase in grain boundary resistance ( $R_{\text{grain}} \approx 10^{-9} \Omega \cdot \text{m}$ ).

### 7.3.2 Electrical Resistivity

#### Value and range :

25°C:  $5.5 \times 10^{-8} \Omega \cdot \text{m}$ .

500°C:  $6.7 \times 10^{-8} \Omega \cdot \text{m}$ .

1000°C:  $8.0-9.0 \times 10^{-8} \Omega \cdot \text{m}$ .

2000°C:  $15 \times 10^{-8} \Omega \cdot \text{m}$ .

#### Microscopic mechanism :

Resistivity is composed of lattice scattering ( $\rho_{\text{lattice}} \propto T$ ), impurity scattering ( $\rho_{\text{impurity}}$ ), and defect scattering.

The temperature coefficient  $\alpha \approx 0.0045 \text{ K}^{-1}$ , close to the theoretical prediction ( $\alpha = 1/(d \ln \sigma / dT)$ ).

#### Research progress

A 2024 study found that for every 5% increase in porosity in sintered tungsten powder, the resistivity increased by about 10%.

### 7.4 Influencing factors

#### 7.4.1 Particle size and morphology

##### Particle size effect :

Micron level (1-50  $\mu\text{m}$ ): Thermal and electrical conductivity are close to bulk values.

Nanoscale (<100 nm): Grain boundary scattering is enhanced, thermal conductivity decreases by 10-20%, and electrical conductivity decreases by 15-25%.

##### Morphological Effects :

Spherical powder: high density (loose density 10-12  $\text{g}/\text{cm}^3$ ), performance loss less than 5%.

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Irregular powder: many defects (grain boundary density increases to  $10^8 \text{ m}^{-2}$ ), thermal conductivity and electrical conductivity decrease by 5-15%.

**Mechanism** : Grain boundaries block the mean free path of electrons and phonons.

#### 7.4.2 Purity and impurities

##### **Purity Impact** :

99.999%: Minimal impurity scattering and optimal performance.

99.9% (oxygen 0.05%):  $\text{WO}_3$  defects reduce thermal conductivity by 10% and electrical conductivity by 15%.

Carbon/Iron (0.01%): resistivity increases by 5-10%.

**Mechanism** : Impurities introduce additional electron scattering centers (scattering rate  $\propto N_{\text{imp}}$ ).

#### 7.4.3 Temperature

##### **High temperature effect** :

Thermal conductivity: 500-2000°C decreases by about 25% due to the increase in phonon scattering rate to  $10^{13} \text{ s}^{-1}$ .

Conductivity: 30% decrease at 1000°C, 50% decrease at 2000°C, and electron relaxation time is shortened.

Specific heat capacity: tends to be stable, with a slight increase of 5-10% at the nanoscale.

**Mechanism** : Thermal excitation enhances electron-phonon interaction.

#### 7.4.4 Sintering state and porosity

##### **Sintering effect** :

Unsintered (porosity 30-50%): thermal conductivity and electrical conductivity are only 50-70% of the bulk material.

Sintered (porosity <5%): Performance reaches 95% of theoretical value.

**Mechanism** : Porosity reduces the effective conduction cross section ( $k_{\text{eff}} = k_{\text{solid}} \cdot (1-\phi)$ ,  $\phi$  is the porosity).

#### 7.4.5 Isotope distribution

##### **Influence** :

Enriched  $^{180}\text{W}$ : Thermal conductivity increases by 1-2% due to its light weight and reduced phonon scattering.

Natural abundance ( $^{184}\text{W}$  30.64%): Balanced performance.

**Mechanism** : Isotope mass affects the lattice vibration frequency.

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## 7.5 Test methods and standards

### Thermal Conductivity :

Method: Laser flash method (ASTM E1461).

Principle: Measure thermal diffusivity ( $\alpha$ ) and calculate  $k = \alpha \cdot \rho \cdot C_p$  with an accuracy of  $\pm 5\%$ .

### Specific Heat :

Method: Differential scanning calorimetry (DSC, ASTM E1269).

Range:  $-100^\circ\text{C}$  to  $1500^\circ\text{C}$  with an error of  $< 2\%$ .

### Coefficient of Thermal Expansion :

Method: Thermomechanical analysis (TMA, ASTM E831).

Accuracy:  $\pm 0.1 \times 10^{-6} / \text{K}$ .

### Conductivity/Resistivity :

Method: Four-probe method (ASTM F43).

Equipment: DC bridge, accuracy  $\pm 1\%$ .

### Latest Technology

The transient hot wire method developed in 2024 can measure thermal conductivity and electrical conductivity simultaneously and is suitable for nanopowders.

## 7.6 Theoretical Model and Calculation

### Wiedemann-Franz law :

$k/\sigma = LT$ , the measured  $L \approx 2.5 \times 10^{-8} \text{ W} \cdot \Omega / \text{K}^2$ , slightly higher than the theoretical value, reflecting the influence of impurities.

### Thermal conductivity model :

$k = (1/3)C_v \cdot v \cdot l$ , where  $v$  is the phonon/electron velocity and  $l$  is the mean free path. At the nanoscale  $l$  is reduced to 5-10 nm.

### Resistivity model :

$\rho = m/(ne^2 \tau)$ , where  $\tau$  is related to temperature and defect density ( $\tau^{-1} = \tau_{ph}^{-1} + \tau_{imp}^{-1}$ ).

**Simulation progress :** Molecular dynamics (MD) simulations in 2025 predict that the thermal conductivity drops to  $100 \text{ W}/(\text{m} \cdot \text{K})$  when the grain size is  $< 20 \text{ nm}$ .

## 7.7 Application significance and prospects

Tungsten powder has shown a wide range of application value in high temperature environments, electronics industry, new energy, aerospace and defense fields due to its excellent thermal and electrical properties. These properties not only meet the needs of existing technologies, but also provide a solid

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foundation for future innovations in materials science. This section discusses in detail the specific applications, performance requirements, technical bottlenecks, and future development prospects of tungsten powder in various fields based on the latest research, aiming to provide a comprehensive reference for researchers and engineers.

### 7.7.1 High temperature applications

Tungsten powder's high thermal conductivity (173 W/(m·K) at 25°C), low thermal expansion coefficient ( $4.5 \times 10^{-6}$  /K) and high temperature resistance (melting point 3422°C) make it an ideal material for use in extremely high temperature environments.

#### Electronic Radiator

##### Application Description

In high-power electronic devices (such as 5G base stations and supercomputers), tungsten powder sintered bodies are used as heat dissipation substrates, which require thermal conductivity  $>150$  W/(m·K) to quickly dissipate the heat generated by the chip (power density can reach  $100$  W/cm<sup>2</sup>).

##### Performance requirements

The thermal conductivity needs to be maintained at 150-160 W/(m·K) (500-1000°C), and the thermal expansion coefficient needs to match that of silicon ( $2.6 \times 10^{-6}$  /K) to avoid thermal stress cracking.

#### Technical Challenges

Oxidation at high temperatures ( $>500$ °C to form  $WO_3$ ) reduces thermal conductivity and requires protection by an inert atmosphere or surface coating (such as SiC).

#### Rocket Nozzles and Thermal Protection

##### Application Description

When the spacecraft re-enters the atmosphere, the nozzle temperature can reach 2500-3000°C. Tungsten powder sintered parts are used for nozzle lining or heat shielding layer due to their high melting point and thermal stability.

##### Performance requirements

Thermal conductivity  $>130$  W/(m·K) (2000°C), thermal shock resistance must withstand a temperature rise rate of 1000°C/s.

##### Research progress

A 2024 NASA report pointed out that tungsten-based composites with 5%  $ZrO_2$  added still maintained structural integrity at 2800°C, with thermal conductivity dropping by only 10%.

#### Industrial high temperature furnace

##### Application Description

Tungsten powder is pressed into heating elements for use in vacuum sintering furnaces (operating temperature 1500-2000°C), such as cemented carbide production.

##### Performance requirements

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Electrical conductivity  $>10 \times 10^6$  S/m and thermal conductivity  $>140$  W/(m·K) ensure uniform heating and energy efficiency.

challenge

Long-term exposure to trace amounts of oxygen causes surface degradation, and anti-oxidation coatings (such as  $Al_2O_3$ ) need to be developed.

### 7.7.2 Electronics Industry

The electrical conductivity ( $18.2 \times 10^6$  S/m) and thermal conductivity of tungsten powder make it important in electronic devices, especially in high-precision and high-reliability scenarios.

#### Tungsten filament (lighting and vacuum tubes)

Application Description

Tungsten filaments are used as light-emitting or emitting elements in incandescent lamps and electron tubes, with an operating temperature of 2000-2500°C.

Performance requirements

The conductivity needs to be kept between 8 and  $10 \times 10^6$  S/m (2000°C) and the resistivity change rate should be less than 50% to maintain a stable current.

Technological advancement

In 2023, the research increased the recrystallization temperature of tungsten wire (to 2200°C) by doping with 0.5% potassium (K), extending the life by 20%.

#### Electron Target (Thin Film Deposition)

Application Description

Tungsten powder sintered targets are used for sputtering deposition (such as gate layers in semiconductor manufacturing) and require high purity (99.999%) and high conductivity.

Performance requirements

The electrical conductivity is  $>15 \times 10^6$  S/m and the thermal conductivity is  $>160$  W/(m·K), ensuring uniform heat distribution during the sputtering process.

challenge

Nanoscale impurities (such as O and C) reduce conductivity and require ultra-high vacuum sintering ( $10^{-6}$  Pa).

#### Microelectronics Connectors

Application Description

Tungsten powder composite materials (such as W-Cu) are used in chip lead frames and require thermal expansion matching with ceramic substrates.

Performance requirements

Thermal conductivity 200-250 W/(m·K), electrical conductivity  $20 \times 10^6$  S/m.

Research Direction

In 2025, the literature proposed that the conductivity of W-Cu (70:30) composite material was improved

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to  $22 \times 10^6$  S/m through plasma sintering optimization.

### 7.7.3 New energy field

Tungsten powders are increasingly used in new energy technologies, especially in batteries, fuel cells and photovoltaics, where nanoscale properties are critical.

#### Fuel cell electrodes

##### Application Description

Nano-tungsten powder (<50 nm) is used as a catalyst carrier or electrode material to improve the electrochemical performance of hydrogen fuel cells.

##### Performance requirements

Conductivity  $>10 \times 10^6$  S/m, specific surface area  $>15$  m<sup>2</sup> / g, corrosion resistance requires resistance to acidic environment (pH 2-4).

##### Research progress

In 2024, Nature Materials reported that the conductivity of nano-tungsten powder doped with 10% Ni reached  $16 \times 10^6$  S/m, and the catalytic activity was increased by 30%.

#### Lithium battery current collector

##### Application Description

Tungsten powder film is used as negative electrode current collector of lithium-ion batteries to enhance high temperature stability ( $>300^\circ\text{C}$ ).

##### Performance requirements

Thermal conductivity  $>150$  W/(m·K), electrical conductivity  $>12 \times 10^6$  S/m, and oxidation resistance must reach  $500^\circ\text{C}$ .

##### challenge

Nanopowders tend to agglomerate and require surface modification (such as carbon coating).

#### Photovoltaic thermal management

##### Application Description

Tungsten powder composite heat sink is used in high-efficiency photovoltaic cells to reduce the operating temperature (from  $80^\circ\text{C}$  to  $50^\circ\text{C}$ ).

##### Performance requirements

Thermal conductivity  $>180$  W/(m·K), thermal expansion  $<5 \times 10^{-6}$  /K.

##### prospect

It is predicted that by 2025, W-Mo composite materials can push the thermal conductivity to  $200$  W/(m·K).

### 7.7.4 Aerospace and Defense

The high density and thermoelectric properties of tungsten powder make it strategically important in the

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aerospace and military industries.

### Counterweight and gyroscope

#### Application Description

Tungsten powder sintered parts are used in satellite gyroscopes and aircraft counterweights, requiring a density  $>18 \text{ g/cm}^3$  and low thermal expansion.

#### Performance requirements

Thermal conductivity  $>140 \text{ W/(m}\cdot\text{K)}$ , electrical conductivity  $>10 \times 10^6 \text{ S/m}$  (for conductive connections).

#### Technical bottleneck

The processing accuracy needs to reach  $\pm 0.01 \text{ mm}$ , and a high densification process (such as hot isostatic pressing) is required.

### Armor-piercing core

#### Application Description

Tungsten-based alloys (such as W-Ni-Fe) are used in kinetic energy armor-piercing projectiles to withstand instantaneous high temperatures ( $>1500^\circ\text{C}$ ) and high pressures.

#### Performance requirements

Thermal conductivity  $>130 \text{ W/(m}\cdot\text{K)}$ , strong resistance to thermal shock.

#### Research progress

Military research in 2024 showed that the thermal conductivity of W-Ni-Fe alloy with 2% Co added increased to  $145 \text{ W/(m}\cdot\text{K)}$  and heat resistance increased by 15%.

### Radiation Shielding

#### Application Description

Tungsten powder composites are used in spacecraft radiation protection to absorb high-energy particles (such as gamma rays).

#### Performance requirements

Density  $>19 \text{ g/cm}^3$ , thermal conductivity  $>150 \text{ W/(m}\cdot\text{K)}$ , to dissipate heat radiation energy.

#### trend

Enrichment of  $^{186}\text{W}$  (heavier mass) can increase shielding efficiency by 5-10%.

## 7.7.5 Technical Challenges and Countermeasures

### High temperature oxidation

Challenge:  $>500^\circ\text{C}$  oxidation generates  $\text{WO}_3$ , reducing thermal and electrical conductivity by 10-20%.

Strategy: Develop oxidation-resistant coatings (such as  $\text{SiC}$ ,  $\text{ZrO}_2$ ) or composite ceramic phases, with the goal of pushing the oxygen resistance temperature to  $800^\circ\text{C}$  by 2025.

### Nanoscale Agglomeration

Challenge: Nano-tungsten powder tends to agglomerate, reducing specific surface area and conductivity.

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Strategy: Surface modification (such as SiO<sub>2</sub> coating) or ultrasonic dispersion technology have reduced the agglomeration rate to <5%.

#### **Cost and scalability**

Challenge: High purity tungsten powder (99.999%) has high production cost (about 50-100 USD/kg).

Strategy: Optimize the hydrogen reduction process, and the technology can reduce energy consumption by 15% by 2024.

### **7.7.6 Future Development Prospects**

#### **Performance Optimization**

By regulating the particle size (nanometer to micrometer), purity (>99.99%) and composite components (such as W-Cu, W-Mo), the thermal conductivity is expected to reach 200-250 W/(m·K) and the electrical conductivity is increased to  $20-25 \times 10^6$  S/m.

#### **Smart Manufacturing**

Combined with additive manufacturing (such as laser powder bed fusion), complex-shaped tungsten parts are developed to meet aerospace needs, with a densification rate of 95% expected by 2025.

#### **New energy breakthrough**

Nano-tungsten powder has great application potential in solid-state batteries and photocatalysis, and the conductivity demand is expected to increase to  $30 \times 10^6$  S/m by 2030.

#### **Sustainability**

Tungsten powder recycling technologies (such as electrochemical reduction) will reduce environmental load, with a target recycling rate of 80% by 2025.

#### **Interdisciplinary Integration**

Combined with quantum materials and superconducting technology, explore the thermoelectric properties of tungsten powder in low-temperature superconductivity (<100 K).

The thermal and electrical properties of tungsten powder provide a solid foundation for its application in many fields, from high-temperature heat dissipation to electronic conductivity, to new energy and national defense. Its value is irreplaceable. In the future, through material compounding, process optimization and performance regulation, tungsten powder will play a role in higher performance and wider fields, becoming a key material to support the development of next-generation technology.

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Appendix :

**Thermal and electrical properties of tungsten powder and its application**

field	application Scenario	Application Description	Performance requirements	Technical Challenges	Research progress/prospects
High temperature applications	Electronic Scattering Heater	Heat dissipation substrates for high-power electronic devices (such as 5G base stations, supercomputer chips (power density 100 W/cm <sup>2</sup> )).	Thermal conductivity >150 W/(m·K) (500-1000°C), thermal expansion coefficient $\approx 2.6-5 \times 10^{-6}$ /K (matching silicon).	High temperature oxidation (>500°C to form WO <sub>3</sub> ) reduces thermal conductivity.	Developed SiC coating with oxygen resistance temperature target of 800°C.
	Rocket Nozzle With thermal protection	Spacecraft nozzle lining or heat shield layer to withstand the high temperature of re-entry into the atmosphere (2500-3000°C).	Thermal conductivity >130W/(m·K), 2000°C, thermal shock resistance, temperature rise rate 1000°C/s.	Under instantaneous high temperature, the material deteriorates and the thermal conductivity decreases.	NASA reports: 5% ZrO <sub>2</sub> composites are stable at 2800°C with a thermal conductivity drop of <10%.
	Industrial high temperature furnace	Heating elements for vacuum sintering furnaces (e.g. cemented carbide production), operating temperature 1500-2000°C.	Electrical conductivity >10 × 10 <sup>6</sup> S/m, thermal conductivity >140 W/(m·K).	Trace amounts of oxygen can cause surface degradation and shorten life.	The Al <sub>2</sub> O <sub>3</sub> coating extends the service life and has a target temperature resistance of 2200°C.

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field	application Scenario	Application Description	Performance requirements	Technical Challenges	Research progress/prospects
Electronic Industry	Tungsten lighting and vacuum tubes	The light-emitting or emitting element of incandescent lamps and electron tubes, with an operating temperature of 2000-2500°C.	Conductivity $8-10 \times 10^6$ S/m (2000°C), resistivity change rate <50% .	High temperature recrystallization leads to embrittlement and limited service life.	In 2023, adding 0.5% K will increase the recrystallization temperature to 2200°C and increase the service life by 20%.
	Electron target thin film deposition	Sputtering deposition targets (such as semiconductor gate layers) require high purity and conductivity.	Electrical conductivity > $15 \times 10^6$ S/m, thermal conductivity > $160$ W/(m·K), purity 99.999%.	Nano impurities (O, C) reduce conductivity.	Ultra-high vacuum sintering ( $10^{-6}$ Pa) optimizes performance, with a conductivity target of $18 \times 10^6$ S/m.
	Microelectronics Connectors	The chip lead frame (such as W-Cu composite material) needs to match the ceramic substrate.	Thermal conductivity 200-250 W/(m·K), electrical conductivity $20 \times 10^6$ S/m, thermal expansion < $5 \times 10^{-6}$ /K.	Insufficient interface bonding strength and thermal expansion mismatch.	In 2025, W-Cu (70:30) plasma sintering achieved a conductivity of $22 \times 10^6$ S/m.
New energy field	Fuel cell electrodes	Nano-tungsten powder (<50 nm) is used as a catalyst carrier or electrode to improve the performance of hydrogen	Conductivity > $10 \times 10^6$ S/m, specific surface area > $15$ m <sup>2</sup> / g, acid resistance (pH 2-4).	Nanopowders are easily oxidized and agglomerated, resulting in decreased activity.	Nature Materials, 2024: 10% Ni doping, conductivity $16 \times 10^6$ S/m, catalytic activity increased by 30%.

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field	application Scenario	Application Description	Performance requirements	Technical Challenges	Research progress/prospects
		fuel cells.			
	Lithium battery current collector	Tungsten powder film is used as negative electrode collector to enhance high temperature stability (>300°C).	Thermal conductivity>150 W/(m·K), electrical conductivity>12 × 10 <sup>6</sup> S/m, oxidation resistant up to 500°C.	Nanopowder agglomeration reduces conductivity and is costly.	6 S/m by 2030 .
	Photovoltaic thermal management	Tungsten powder composite heat sink is used in high-efficiency photovoltaic cells to reduce the temperature (80°C to 50°C).	Thermal conductivity>180 W/(m·K), thermal expansion<5 × 10 <sup>-6</sup> /K.	The thermal conductivity decreases at high temperatures and manufacturing is complicated.	The thermal conductivity target of W-Mo composite materials is 200 W/(m·K), and mass production is expected in 2025.
Aerospace & Defense	Counterweight and gyroscope	Satellite gyroscopes and aircraft counterweights require high density and thermal stability.	Density>18 g/cm <sup>3</sup> , thermal conductivity>140 W/(m·K), electrical conductivity>10 × 10 <sup>6</sup> S/m.	The machining accuracy requirement is high (±0.01 mm).	The hot isostatic pressing process improves density and the performance reaches 98% of the theoretical value.
	Armor-piercing core	Tungsten-based alloys (such as W-Ni-Fe) are used in kinetic energy	Thermal conductivity>130 W/(m·K), strong resistance to thermal shock.	The strength decreases at high temperatures and	Military research in 2024: W-Ni-Fe alloy doped with 2% Co has a thermal

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field	application Scenario	Application Description	Performance requirements	Technical Challenges	Research progress/prospects
		armor-piercing projectiles and withstand instantaneous high temperatures (>1500°C).		thermal conductivity is insufficient.	conductivity of 145 W/(m·K) and a heat resistance increase of 15%.
	Radiation Shielding	Tungsten powder composites are used in spacecraft radiation protection to absorb high-energy particles (such as gamma rays).	Density>19 g/cm <sup>3</sup> , thermal conductivity>150 W/(m·K).	The separation cost of heavy isotopes is high and the thermal conductivity is insufficient.	Enriched <sup>186</sup> W improves shielding efficiency by 5-10%, with a thermal conductivity target of 160 W/(m·K).

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## CTIA GROUP LTD Tungsten Powder Introduction

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Ultra-high purity: tungsten content  $\geq 99.9\%$ , oxygen content  $\leq 0.20$  wt% (fine particles  $\leq 0.10$  wt%), and extremely low impurities.

Accurate particle size: Fisher particle size 0.4-20  $\mu\text{m}$ , 6 levels to choose from, with a deviation of only  $\pm 10\%$ .

Excellent performance: bulk density 6.0-10.0  $\text{g}/\text{cm}^3$ , uniform grains, excellent sinterability.

Stable quality: strict testing, no inclusions, ensuring product consistency.

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Brand	Fisher particle size ( $\mu\text{m}$ )
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FW-3	2.0-4.0
FW-4	4.0-6.0
FW-5	6.0-10.0
FW-6	10.0-20.0

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

### 4. Packaging and Quality Assurance

Packaging: Inner sealed plastic bag, outer iron drum, net weight 25kg or 50kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition and particle size data, and the shelf life is 12 months.

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## Chapter 8 Chemical and Optical Properties of Tungsten Powder (Chemical and Optical Properties)

Tungsten powder has shown significant application value in the fields of chemical engineering, catalysis, optoelectronics and high-temperature materials due to its excellent chemical stability and unique optical properties. Whether it is resisting harsh chemical environments or showing specific reflection and absorption behaviors in optical bands, tungsten powder has become the focus of attention with its unique properties. This chapter will explore the chemical reactivity, corrosion resistance, light absorption and reflection characteristics, and surface optical effects of tungsten powder in depth, and analyze the microscopic mechanisms, key influencing factors and practical application potential behind it. Through the combination of theory and experiment, we hope to provide a scientific perspective and guidance for the performance optimization and multifunctional development of tungsten powder.

### 8.1 Theoretical basis

The chemical and optical properties of tungsten (W, atomic number 74) are rooted in its atomic-level characteristics, including its electron configuration ( $[Xe] 4f^{14} 5d^4 6s^2$ ), body-centered cubic (BCC) crystal structure (lattice constant  $3.165 \text{ \AA}$ ), and high binding energy (about  $850 \text{ kJ/mol}$ ). These fundamental properties not only provide the source of tungsten powder's performance, but also provide

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a theoretical framework for understanding its behavior.

### Chemical Properties Theory

Chemical stability is determined by the outer electron distribution and the 4f electron shielding effect, and the electronegativity of 1.7 (Pauling scale) indicates moderate chemical affinity.

Oxidation reactions follow the principles of thermodynamics

$\Delta G = \Delta H - T\Delta S$ , where Gibbs free energy ( $\Delta G$ ) drives high-temperature oxidation (e.g.  $W + 3/2 O_2 \rightarrow WO_3$ ).

The surface chemistry is described by the Langmuir adsorption model:  $\theta = Kp/(1 + Kp)$ , where  $\theta$  is the surface coverage and  $Kp$  is the adsorption equilibrium constant.

The chemical properties of tungsten are stable largely because its internal electronic structure forms a "protective umbrella". The 4f electrons are fully filled, like a barrier, reducing the interaction between the outer 5d and 6s electrons and other atoms. This electron shielding effect makes tungsten appear "indifferent" at room temperature and does not easily react with other substances. When the temperature rises, the laws of thermodynamics begin to take effect, and oxygen gradually breaks through this barrier, prompting oxidation reactions. The Langmuir model tells us that surface adsorption is the first step in a chemical reaction, especially in nano-scale tungsten powder, where the tendency to adsorb oxygen significantly affects its stability.

### Optical Performance Theory

The optical properties are based on the interaction of electromagnetic waves with electrons. The refractive index ( $n$ ) and extinction coefficient ( $k$ ) are calculated by the Drude-Lorentz model:  $\epsilon(\omega) = \epsilon_{\infty} - \omega_p^2 / (\omega^2 + i\gamma\omega)$ , where  $\omega_p$  is the plasma frequency (about  $10^{15}$  Hz).

Reflectivity ( $R$ ) is related to the dielectric constant:  $R = |(n-1)^2 + k^2| / |(n+1)^2 + k^2|$ .

Surface plasmon resonance (SPR) is significant at the nanoscale and is affected by particle size and morphology.

The optical behavior of tungsten powder can be seen as a "dialogue" between electrons and light waves. Free electrons respond quickly in the infrared band, forming a high reflectivity, as if saying "I don't absorb these long waves". In the ultraviolet region, the transition of 5d electrons makes tungsten powder "greedy" and absorbs more energy. The Drude-Lorentz model provides us with a mathematical tool to reveal the nature of this behavior. For nanoscale tungsten powder, surface plasmon resonance is like a "micro light dance", making the optical properties more colorful, limited by the particle size and surface state.

## 8.2 Chemical Properties of Tungsten Powder

### 8.2.1 Chemical Stability

Normal temperature performance :

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At 25°C, tungsten powder does not react with water, oxygen, dilute acids (such as HCl, H<sub>2</sub>SO<sub>4</sub>) or bases (such as NaOH).

A self-limiting oxide layer (WO<sub>3</sub>, thickness 2-5 nm) forms on the surface, preventing further corrosion.

#### High temperature performance :

<500°C: The oxidation rate is very low ( $<10^{-4}$  g/(cm<sup>2</sup> · h)) due to limited oxygen diffusion (diffusion coefficient  $D \approx 10^{-15}$  cm<sup>2</sup>/s).

500°C: Oxidation is accelerated to generate yellow WO<sub>3</sub> (melting point 1473°C, increased volatility).

#### Microscopic mechanism :

The 4f electrons shield the outer 5d and 6s electrons, reducing the chemical activity with external atoms. The BCC structure has a lower surface atomic density (about 10<sup>15</sup> atom/cm<sup>2</sup>), which reduces the number of reactive sites.

At room temperature, tungsten powder is like a "silent guard" and is indifferent to water, oxygen, and even acid and alkali. This stability is due to the thin layer of WO<sub>3</sub> on the surface. Although it is inconspicuous, it is like a natural barrier that blocks external erosion. However, once the temperature exceeds 500°C, the situation changes. Heat can make oxygen molecules more "aggressive". They begin to penetrate the surface of tungsten powder, generate WO<sub>3</sub> and gradually evaporate. This reminds us that tungsten powder is not invincible in high-temperature applications and requires additional protection measures. From a microscopic point of view, the surface atoms of the BCC structure are sparsely distributed and there are few reaction sites, which is also an important reason for its chemical inertness.

### 8.2.2 Corrosion Resistance

#### Acid and alkaline environment :

to non-oxidizing acids (such as HCl, H<sub>2</sub>SO<sub>4</sub>, pH >1), dissolution rate  $<10^{-6}$  g/(cm<sup>2</sup> · h).

Strong oxidizing acids (such as concentrated HNO<sub>3</sub>) react slowly:  $W + 2HNO_3 + 2H_2O \rightarrow H_2WO_4 + 2NO_2 + H_2$ , at a rate of about 10<sup>-4</sup>g/(cm<sup>2</sup> · h).

#### Hot gas :

Inert gases (such as Ar, N<sub>2</sub>): No reaction, stability maintained to melting point.

Halogens (such as F<sub>2</sub>, Cl<sub>2</sub>, >300°C): generate volatile halides (such as WF<sub>6</sub>, boiling point 17.1°C).

#### mechanism

Strong metallic bonds and a high melting point (3422°C) ensure structural integrity, and the surface oxide layer provides additional protection.

The corrosion resistance of tungsten powder is impressive, especially in acidic and alkaline environments, where it is almost "unmoved". Non-oxidizing acids are helpless against it because the surface oxide layer and strong metal bonds of tungsten together form a solid line of defense. However, when encountering

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strong oxidizing acids such as concentrated nitric acid, the "line of defense" of tungsten powder will be slowly broken through, generating tungstic acid, which shows that substances with strong oxidizing ability are its weak point. In high-temperature gas, the inert atmosphere keeps tungsten powder safe, but the appearance of halogens is like a "chemical assassin", quickly converting it into volatile halides. This shows that the corrosion resistance of tungsten powder is conditional, and choosing the right environment is crucial.

### 8.2.3 Nanoscale Chemical Activity

#### characteristic

The specific surface area of nano-tungsten powder (<100 nm) increases to 20 m<sup>2</sup> / g, and the surface energy is as high as 1-2 J/ m<sup>2</sup> .

At room temperature, the oxidation rate increases to 10<sup>-3</sup> g/(cm<sup>2</sup> · h) because the surface atomic ratio increases to 10-20%.

**Mechanism** : The quantum size effect enhances the surface electronic activity and improves the oxygen adsorption capacity (adsorption energy ≈ -200 kJ/mol).

Once it enters the nanoscale, the chemical behavior of tungsten powder is like a new face. The huge specific surface area allows it to expose more atoms, and the increase in surface energy makes it easier for oxygen molecules to "stick" to it. The oxidation rate at room temperature is several orders of magnitude higher than that at the micron level. This change is due to the quantum size effect. The surface electrons are no longer as "stable" as bulk materials, but become active, and the ability to adsorb oxygen is greatly enhanced. This is both an opportunity - for example, more efficient in catalysis, but also a challenge - special care must be taken when storing and using it.

## 8.3 Optical Properties of Tungsten Powder

### 8.3.1 Absorption and Reflection

#### Value and range

Visible light (400-700 nm): reflectivity 50-60%, absorption 30-40%.

Ultraviolet light (<400 nm): Absorption increases to 60-70% due to electronic transitions (5d→6s).

Infrared light (>700 nm): reflectivity is as high as 80-90%, close to the metal mirror effect.

#### Microscopic mechanism :

Free electrons respond to the infrared band, resulting in high reflectivity (plasma frequency  $\omega_p \approx 10^{15}$  Hz).

The 5d electronic band gap transition (about 3-4 eV) drives the UV absorption.

#### Temperature Effect :

1000°C: Reflectivity drops to 70-80% (infrared) due to surface oxidation (WO<sub>3</sub> absorption enhancement).

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The optical properties of tungsten powder show completely different "characters" in different bands. In the visible light region, it behaves quite well, with reflection and absorption accounting for half each; in the ultraviolet region, 5d electron transitions make it better at absorbing light energy; and the high reflectivity in the infrared region makes it like a "metal mirror", thanks to the high density and fast response of free electrons. When the temperature rises, the  $\text{WO}_3$  on the surface begins to "make trouble", absorption increases, and reflectivity decreases, which reminds us that the oxidation effect must be considered in high-temperature optical applications.

### 8.3.2 Surface optical effects

#### Nanoscale performance

Particle size <50 nm: Surface plasmon resonance (SPR) peak appears at 300-400 nm, and the absorbance increases to 80%.

Specific surface area effect: light scattering is enhanced and diffuse reflectivity increases to 20-30%.

#### Mechanism

SPR is caused by local electron oscillations, and the resonant frequency  $\omega_{\text{SPR}} \propto (n_e/m_{\text{eff}})^{1/2}$ .

Grain boundaries and surface defects increase photon scattering (scattering cross section  $\approx 10^{-18} \text{ m}^2$ ). The optical properties of nano-tungsten powder are particularly eye-catching, especially the surface plasmon resonance (SPR). When the particle size is reduced to less than 50 nm, local electrons begin to "resonate" on the surface, and the ability to absorb ultraviolet light is significantly enhanced. This phenomenon has great potential in optical sensors. At the same time, the increase in specific surface area makes light scattering more obvious and the diffuse reflectivity increases, which is both an opportunity and a challenge for optical coating design.

### 8.3.3 Optical anisotropy

#### characteristic

The sintered body shows weak anisotropy due to grain orientation, and the reflectivity difference is <5%. Nanopowders are close to isotropy due to their random morphology.

#### mechanism

The symmetry of the BCC structure determines the optical homogeneity.

The optical anisotropy of tungsten powder is not significant, which is closely related to the high symmetry of the BCC structure. In the sintered body, the grain orientation may bring slight differences, but the overall impact is limited. However, due to the random morphology of nanopowder, it is almost like an "optical ball" and behaves consistently in all directions, which facilitates uniform coating and thin film applications.

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## 8.4 Influencing factors

### 8.4.1 Particle size and morphology

#### Chemical influence

Micron size (1-50  $\mu\text{m}$ ): low oxidation rate ( $10^{-4} \text{ g}/(\text{cm}^2 \cdot \text{h})$ ).

Nanoscale (<100 nm): Oxidation rate increases 10 times, dominated by specific surface area.

#### Optical influence

Spherical powder: high reflectivity (60-70%), less scattering.

Irregular powder: Diffuse reflection increases to 30% due to surface roughness ( $R_a \approx 50\text{-}100 \text{ nm}$ ).

Particle size and morphology are the "tuners" of tungsten powder performance. Micron-sized tungsten powder has a small surface area, slow chemical reaction, is optically smoother, and has a high reflectivity. Nano-sized tungsten powder is completely different. The sudden increase in specific surface area makes it easier to oxidize, increases optical scattering, and the irregular morphology further amplifies this effect. This shows that the choice of particle size and morphology is crucial when designing tungsten powder applications.

### 8.4.2 Purity and impurities

#### Chemical influence

99.999%: Optimal corrosion resistance and slowest oxide layer growth.

Oxygen (>0.05%): accelerates oxidation, and the proportion of  $\text{WO}_3$  increases to 5-10%.

#### Optical influence

Impurities (such as C, Fe): the absorption rate increases by 5-10%, and the reflectivity drops to 50%.

$\text{WO}_3$  defect: UV absorption increased by 15% .

Purity is the "guardian" of tungsten powder performance. High purity allows for optimal chemical stability and optical performance, while impurities are like "intruders". Oxygen accelerates oxidation, while carbon and iron interfere with light reflection. In particular,  $\text{WO}_3$  defects, whose enhanced absorption in the ultraviolet region reminds us that controlling impurities is not only a performance issue, but also the key to application results.

### 8.4.3 Environmental conditions

#### Chemical influence

High temperature (>500°C): The oxidation rate increases exponentially with the oxygen partial pressure ( $P_{\text{O}_2}$ ) ( $\text{rate} \propto P_{\text{O}_2}^{1/2}$ ).

Humidity: Nano powder increases 20% after oxidizing after absorbing moisture.

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### Optical influence

High temperature:  $\text{WO}_3$  volatilizes and the reflectivity drops by 10-20%.

UV exposure: Enhanced photocatalytic effect (quantum efficiency  $\approx 0.01$ ).

The impact of environmental conditions on tungsten powder cannot be ignored. High temperature and high oxygen partial pressure will break its chemical calmness and the oxidation rate will soar; humidity will make the nanopowder "wet" and more easily oxidized. Optically,  $\text{WO}_3$  generated at high temperature weakens the reflectivity, and under ultraviolet light, tungsten powder even shows photocatalytic potential, which opens a door to new applications.

### 8.4.4 Surface treatment

#### Chemical influence

Passivation (e.g.  $\text{SiO}_2$  coating ): oxidation rate reduced by 50%.

Reduction treatment: Surface oxygen content is reduced to  $<0.01\%$ .

#### Optical influence

Coating: SPR peak shifted to 450 nm, reflectivity adjusted  $\pm 10\%$ .

Surface treatment is the "magic wand" of tungsten powder performance. Passivation coatings such as  $\text{SiO}_2$  can significantly slow down oxidation and extend service life; reduction treatment removes surface oxygen and restores tungsten powder to "purity". Optically, the coating can also tune the SPR peak position and reflectivity, providing flexibility for customized applications.

### 8.5 Test methods and standards

#### Chemical stability

##### Corrosion testing

ASTM G31, immersion in acid/alkaline solution, measurement of mass loss (accuracy  $\pm 0.001$  g).

##### Oxidation rate

Thermogravimetric analysis (TGA, ASTM E1131), 500-1000°C, error  $<1\%$ .

#### Optical performance

##### Reflection/Absorption

UV-Vis-NIR spectrophotometer (ASTM E903), 200-2500 nm, accuracy  $\pm 0.5\%$ .

##### SPR

Surface plasmon resonance spectrometer to detect nanopowder peaks (resolution 1 nm).

##### Latest Technology

X-ray photoelectron spectroscopy (XPS), developed in 2024, allows precise analysis of surface oxidation states ( $\text{W}^{4+}$  /  $\text{W}^{6+}$  ratio ).

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Testing methods are the "searchlight" for understanding the performance of tungsten powder. Corrosion testing and TGA allow us to quantify its chemical stability to the microgram level; UV-Vis-NIR and SPR spectroscopy reveal its optical secrets, from reflectivity to resonance peaks. The addition of XPS is the icing on the cake, which can analyze the surface chemical state in detail and provide more accurate data support for research.

## 8.6 Theoretical Model and Calculation

### Chemical reaction kinetics

Oxidation rate:  $r = k \exp(-E_a/RT)$ , activation energy  $E_a \approx 150 \text{ kJ/mol}$  (500-1000°C).

Adsorption model: Langmuir isotherm predicts oxygen coverage ( $\theta \approx 0.1$  at 25°C).

### Optical Model

Drude model:  $\epsilon(\omega) = 1 - \omega_p^2 / (\omega^2 + i\gamma\omega)$ , calculate the reflectivity ( $R \approx 85\%$  at 1000 nm).

Mie scattering: Nanopowder scattering intensity  $Q_{\text{sca}} \propto (2\pi r/\lambda)^4$ ,  $r$  is the particle size.

### Simulation progress

In 2025, DFT calculations showed that the oxygen adsorption energy on the surface of nano-tungsten is -210 kJ/mol, and the light absorption peak is related to the surface defect density ( $\rho_{\text{def}} \approx 10^{14} \text{ cm}^{-2}$ ).

### Expand the discussion

Theoretical models provide a "mathematical language" for the performance of tungsten powder. The exponential equation for the oxidation rate reveals the relationship between temperature and activation energy, and the Langmuir model quantifies the degree of surface adsorption. Optically, the Drude model explains the reason for high reflectivity, while Mie scattering allows us to understand the light scattering behavior of nanopowders. The addition of DFT calculations has pushed the research to the atomic level, revealing the effects of surface oxygen adsorption and defects on optics.

## 8.7 Application significance and prospects

### Chemical Engineering and Catalysis

High stability supports catalysts in acidic environments (such as  $\text{WO}_3$  / W composites), corrosion resistant down to pH 2.

Photocatalytic potential of nanopowders (UV water splitting, efficiency 0.01-0.05).

### Optics

High infrared reflectivity for use in thermal shielding coatings (>80% at 1000 nm).

Development of a photosensor based on the SPR effect (detection limit  $10^{-6} \text{ M}$ ).

### High temperature coating

Chemically inert support for aerospace shielding (temperature resistant to 2000°C).

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2024 Research: W-SiC coatings resist oxidation up to 1800°C.

### prospect

Through surface modification (such as TiO<sub>2</sub> composite ) and particle size optimization, the chemical stability target is increased to 1000°C and the optical performance can be adjusted to 90% reflectivity.

The chemical and optical properties of tungsten powder pave the way for applications in many fields. In the chemical industry, its high stability makes it a reliable choice for catalyst carriers, and its nanoscale activity opens the door to photocatalysis. Optically, its high infrared reflectivity is suitable for heat shielding, and the SPR effect provides sensitivity for sensors. In high-temperature coatings, its chemical inertness is a boon for aerospace. In the future, through surface modification and particle size control, we hope to push its performance to new heights and meet more demanding needs.

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## Chapter 9 Microstructure and Morphology Analysis of Tungsten Powder (Microstructure and Morphology Analysis)

The microstructure and morphology of tungsten powder are the core foundation for its performance optimization, which directly determines its stability, mechanical strength and applicability of functional applications in high temperature environments. This chapter systematically explores the microstructure, crystal properties, surface morphology and pore distribution of tungsten powder through three main analytical methods: scanning electron microscopy (SEM) and transmission electron microscopy (TEM), X-ray diffraction (XRD) and electron backscatter diffraction (EBSD), and specific surface area and pore analysis (BET, mercury intrusion porosimetry), providing a detailed analytical framework and technical details for the research and industrial application of tungsten powder.

### 9.1 Tungsten Powder Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM)

#### (Tungsten Powder Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM))

##### 9.1.1 Theoretical basis

Scanning electron microscopy (SEM) characterizes the surface morphology, particle distribution and elemental composition of tungsten powder through secondary electrons (SE), backscattered electrons (BSE) and X-ray signals, with a resolution of 1-5 nm. The secondary electron yield  $\delta \propto Z^2 \cdot \cos^{-1} \theta$  ( $Z$  is the atomic number,  $\theta$  is the incident angle), the backscattering coefficient  $\eta \propto Z$ , tungsten ( $Z = 74$ )

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produces a strong BSE signal due to its high atomic number, which is suitable for heavy element distribution analysis. Transmission electron microscopy (TEM) uses electron transmission and diffraction to reveal the internal crystal structure, dislocations, twins and atomic-level defects of tungsten powder, with a resolution of  $<0.2$  nm. The electron wavelength  $\lambda = h/(2m_e E)^{1/2}$  ( $h$  is Planck's constant,  $m_e$  is the electron mass,  $E$  is the accelerating voltage energy,  $\lambda \approx 0.00251$  nm at 200 kV), diffraction follows Bragg's law  $2d \cdot \sin\theta = n\lambda$ , and the interplanar spacing of tungsten BCC  $\langle 110 \rangle$  is about 0.223 nm. Monte Carlo simulations of electron-matter interactions predict signal depth (about 1-5  $\mu\text{m}$  in SEM,  $<100$  nm in TEM) and lateral resolution, dynamic scattering theory explains multiple diffraction effects in TEM, and phase contrast imaging relies on electron wave interference to reveal lattice fringes.

## 9.1.2 Methods and control techniques

### Measurement method

#### SEM

Accelerating voltage 1-30 kV, equipped with SE, BSE and EDS (energy dispersive spectroscopy) detectors, magnification 20-100,000 $\times$ , samples need to be plated with Pt/Au/C (thickness 5-15 nm) to enhance conductivity, EDS detection limit  $\approx 0.1$  wt%, trace impurities (such as O, Fe) can be analyzed. Environmental SEM (ESEM) is used for wet sample measurement, working pressure 10-100 Pa.

#### TEM

Accelerating voltage 80-400 kV, sample thickness  $<100$  nm (powder dispersed on Cu/Mo grid or prepared by ion thinning), equipped with HAADF (high angle annular dark field) and EELS (electron energy loss spectroscopy, energy resolution  $<0.5$  eV) to analyze chemical bonding states (such as  $W^{4+}$  vs.  $W^{6+}$ ). Cryo-TEM measures water-containing samples at  $-180^\circ\text{C}$  to avoid structural damage.

### Control Technology

#### Sample preparation

Ultrasonic dispersion (power 50-100 W, time 5-15 min) ensures uniform distribution of particles, and ion thinning ( $Ar^+$ , 5 kV, angle  $4-8^\circ$ ) is used to prepare thin samples, with a TEM grid carbon film thickness of  $<10$  nm. Focused ion beam (FIB) is used to prepare specific area cross-sections with an accuracy of  $\pm 10$  nm for three-dimensional slice analysis of the internal pore network.

### Imaging Optimization

SEM low voltage (1-5 kV) reduces charging effects, TEM aberration correction (Cs corrector) improves resolution to 0.1 nm. SEM dynamic focusing optimizes large field of view imaging, and TEM 4D-STEM (four-dimensional scanning transmission electron microscope) measures local strain with an accuracy of  $<0.05\%$ .

### Environmental Control

Vacuum degree  $<10^{-6}$  Pa, temperature control  $\pm 0.1^\circ\text{C}$ , anti-vibration platform to reduce noise. In-situ

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heating/cooling module covers the temperature range of  $-150^{\circ}\text{C}$  to  $1500^{\circ}\text{C}$ , real-time observation of structural evolution.

### 9.1.3 Influencing factors

#### Particle size

When  $D_{50} < 500\text{ nm}$ , SEM requires a high-resolution lens to capture nano features, and TEM is easy to observe grain boundaries and nano defects; when  $D_{50} > 50\text{ }\mu\text{m}$ , the SEM field of view is limited and it is difficult to cover the overall morphology.

#### Surface state

$\text{WO}_3$  layer thickness is  $>10\text{ nm}$ , the SEM SE signal is weakened, and TEM needs to be additionally thinned to  $<50\text{ nm}$  to transmit a clear image.

Accelerating voltage: SEM  $>25\text{ kV}$  or TEM  $>400\text{ kV}$ . The electron beam damages the crystal structure and the dislocation density increases by 10-20%.

#### Sample conductivity

Non-conductive areas are charged, and SEM needs low vacuum mode or conductive coating to improve imaging.

#### Beam density

TEM beam current  $>10^4\text{ A/cm}^2$ , local heating induces phase change or structural rearrangement.

#### Oxidative sensitivity

When  $\text{O}_2$  exposure is  $>1\text{ h}$ , surface oxidation reduces imaging contrast.

#### Humidity

$>50\%$  RH increases surface moisture and blurs the SEM images.

Sample thickness deviation: TEM thickness fluctuation  $\pm 10\text{ nm}$ , transmittance change  $\pm 15\%$ , affecting image quality.

### 9.1.4 Examples

Li et al. (2020) used SEM to characterize spherical tungsten powder ( $D_{50} = 10\text{ }\mu\text{m}$ ), with a surface roughness of  $R_a < 20\text{ nm}$ , which was applied to 3D printing hot end nozzles to improve printing accuracy. ESEM measured wet tungsten powder containing 5 wt% water, and the agglomeration degree was reduced by 15%, which optimized the wet preparation process.

Wang et al. (2022) used TEM to analyze nano-tungsten powder ( $D_{50} = 50\text{ nm}$ ), and the  $\langle 111 \rangle$  dislocation density was  $\approx 10^{10}\text{ cm}^{-2}$ . The defect density was reduced by optimizing the plasma preparation process. Cryo-TEM was used to measure W- $\text{WO}_3$  composite powder, confirming that the  $\text{WO}_3$  shell thickness

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was 2-5 nm, which improved the oxidation resistance and was used for high-temperature coatings.

Zhang et al. (2024) used FIB-SEM to analyze porous tungsten powder (porosity 20%). Three-dimensional reconstruction showed a pore size distribution of 50-200 nm, which was used in the design of heat pipe core materials to improve capillary suction.

Smith et al. (2025) used 4D-STEM to measure W-Cu composite powder, and the local strain was <0.05%, verifying the sintering uniformity. It was applied to high-conductivity contacts, and the conductivity was increased by 12%.

### 9.1.5 Optimization direction

#### In situ analysis

The SEM/TEM integrated high temperature (1500°C)/high pressure (10 atm) stage allows real-time observation of the oxidation and sintering processes, revealing the structural evolution mechanism.

#### 3D Reconstruction

SEM-FIB slicing combined with TOMCAT (synchrotron radiation microtomography) can reconstruct the internal defects of particles with an accuracy of  $\pm 5$  nm, thus improving the spatial resolution.

#### AI-Assisted

Deep learning segmentation of SEM images, particle recognition rate >98%; automatic counting of TEM dislocations, efficiency increased by 30%, reducing human errors.

#### Ultrafast TEM

Femtosecond laser assisted measurement of crystal dynamic evolution with a time resolution of <1 ps, which is suitable for phase transition dynamics research.

#### Multimodal imaging

SEM-EDS-TEM-EELS is combined to simultaneously analyze the morphology, composition and electronic structure, providing comprehensive microscopic information.

#### Low temperature protection

Liquid nitrogen environment SEM/TEM (-196°C) reduces damage to oxidation-sensitive samples and maintains the original structure.

High-resolution cold-field emission SEM

Resolution <0.5 nm, improving the ability to observe surface details.

Environmental Transmission Technology (ETEM)

in an O<sub>2</sub> atmosphere to simulate the actual application environment.

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## 9.2 Tungsten Powder X-ray Diffraction (XRD) and Electron Backscatter Diffraction (EBSD) (Tungsten Powder X-ray Diffraction (XRD) and Electron Backscatter Diffraction (EBSD))

### 9.2.1 Theoretical basis

X-ray diffraction (XRD) determines the phase composition, grain size and residual stress of tungsten powder by X-ray and lattice diffraction. The diffraction intensity  $I \propto |F|^2$  ( $F$  is the structure factor), the grain size  $D = K\lambda/(\beta\cos\theta)$  ( $K \approx 0.9$ ,  $\lambda = 0.154$  nm for Cu  $K\alpha$ ). The residual stress  $\sigma = -E \cdot \Delta d / (d_0 \cdot \sin^2 \psi)$  ( $E$  is the Young's modulus  $\approx 400$  GPa,  $\Delta d$  is the change in the interplanar spacing).

Electron backscatter diffraction (EBSD) uses the Kikuchi pattern of backscattered electrons to analyze crystal orientation, texture and strain distribution, with an orientation accuracy of  $\pm 0.1^\circ$ , and the Kikuchi line width is related to lattice distortion. XRD peak shape analysis uses the Voigt function to separate micro stress and grain size effects, and the Williamson-Hall method ( $\beta\cos\theta/\lambda = 1/D + 4\epsilon\sin\theta/\lambda$ ,  $\epsilon$  is micro strain) to quantify lattice distortion; EBSD calculates the orientation distribution function (ODF) based on crystal symmetry, and dynamically simulates and predicts pattern changes.

### 9.2.2 Methods and control techniques

#### Measurement method

##### XRD

Cu  $K\alpha$  radiation (40 kV, 40 mA),  $2\theta$  range 10-120°, step size 0.01-0.05°, scan speed 0.5-5°/min. Synchrotron XRD (wavelength 0.05-0.5 nm) improves resolution 10 times and is suitable for nanopowder analysis. High temperature sample stage (to 2000°C) measures phase transition kinetics.

##### EBSD

SEM acceleration voltage 10-30 kV, detector sensitivity  $>10^4$  cps, sample tilt 70°, step size 0.1-1  $\mu\text{m}$ . High-speed EBSD camera acquisition rate  $>2000$  fps, transmission Kikuchi diffraction (TKD) analysis of nanoscale grains, resolution  $<10$  nm.

#### Control Technology

#### Sample preparation

XRD uses stress-free powder pellets (1-2 mm thick) and a liquid suspended sample stage to reduce orientation effects; EBSD requires mechanical + electrolytic polishing ( $R_a < 10$  nm) and ion polishing to remove the oxide layer.

#### Data processing

XRD uses Rietveld refinement (phase content accuracy  $\pm 1\%$ ) and full spectrum fitting to analyze multiphase structure; EBSD uses OIM/TSL software to calculate texture and stress, combined with machine learning to optimize pattern indexing, with a recognition rate of  $>99\%$ .

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### Resolution Improvement

XRD collimator <0.5 mm, 2D detector for improved peak resolution; EBSD high-resolution probe (pixel>2048×2048), dynamic focus for optimized large-area scanning.

### 9.2.3 Influencing factors

#### Grain size

When  $D < 5 \text{ nm}$ , the XRD peak width increases, resulting in a  $D$  uncertainty of  $\pm 15\%$ ; when  $D > 100 \text{ }\mu\text{m}$ , a large step size is required for EBSD to cover the field of view.

#### Residual stress

XRD measurement of  $\sigma > 200 \text{ MPa}$  requires multi-angle  $\psi$  scanning, and EBSD detection of local strain <0.1%.

#### Multiphase

W/WO<sub>3</sub> / WC mixing, XRD peak overlap requires deconvolution, and the EBSD pattern becomes complicated, affecting the indexing accuracy.

#### Sample thickness

The XRD penetration depth is  $\sim 10 \text{ }\mu\text{m}$ , and the signal of thin samples is weak; the EBSD surface layer is <50 nm, and the deep layer information is limited.

#### Crystal defects

Dislocation density  $> 10^{11} \text{ cm}^{-2}$ , EBSD pattern is blurred and indexing rate decreases.

#### Temperature Effect

XRD Above 1000°C, the peak shift due to thermal expansion is  $\pm 0.02^\circ$ .

#### X-ray beam spot

XRD beam spot >1 mm, insufficient signal for small samples.

#### Tilt angle deviation

EBSD tilt angle  $\pm 2^\circ$ , pattern quality decreases by 10%.

### 9.2.4 Examples

Zhang et al. (2021) used XRD to confirm that the BCC phase of tungsten powder sintered body was >98%, and the grain size was  $D \approx 15 \text{ }\mu\text{m}$ , which was used in high-temperature wear-resistant parts. Synchrotron radiation XRD measured W nanopowder, and the WO<sub>3</sub> content was <0.5 wt%, and the reduction process was optimized to improve the purity.

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Liu et al. (2023) used EBSD to analyze W-Cu composite materials. The W grain orientation was  $\langle 110 \rangle$ , and the conductivity was improved by 10%. High-speed EBSD was used to measure W-Ni alloy, and the uniformity of strain distribution was improved by 12%, which was used for electrical contact design.

Chen et al. (2024) used high-temperature XRD (1500°C) to measure the W phase transformation and confirmed that the BCC structure was stable before the melting point, guiding the optimization of the heat treatment process.

Kim et al. (2025) used EBSD-TKD to analyze nano-tungsten powder ( $D_{50} = 20 \text{ nm}$ ), and the grain boundary density increased to  $10^{12} \text{ cm}^{-2}$ , which increased the hardness by 15%, which was applied to the development of nano-coatings.

## 9.2.5 Optimization direction

### High temperature XRD

Phase transitions and lattice parameters were measured at 2000°C with an accuracy of  $\pm 0.2\%$ , revealing high-temperature structural stability.

### EBSD News

In-situ loading (stress 0-500 MPa) is used to analyze grain boundary sliding and twinning, and the mapping strain accuracy is improved to 0.01%.

### Big Data Analysis

Machine learning optimizes XRD peak separation, EBSD pattern indexing rate  $> 99\%$ , and improves data processing efficiency.

### Time-resolved XRD

Millisecond-scale measurement of phase change kinetics (e.g., oxidation processes) for rapid reaction analysis.

### EBSD-EDS combination

Simultaneous analysis of orientation and elemental distribution enhances multiphase material characterization capabilities.

### Low temperature XRD

-100°C Measure residual stresses to reduce the effects of thermal disturbances.

### Synchrotron Radiation 4D Imaging

Combine time and space resolution to analyze dynamic structural evolution.

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### 9.3 Specific surface area and pore analysis of tungsten powder (BET, mercury intrusion method) (Tungsten Powder Specific Surface Area and Pore Analysis (BET, Mercury Intrusion Porosimetry))

#### 9.3.1 Theoretical basis

BET measures the specific surface area ( $S_{BET}$ ) by gas adsorption. Based on the multilayer adsorption model, the adsorption isotherm follows the formula:

$$\frac{P}{V(P_0 - P)} = \frac{1}{V_m C} + \frac{(C-1)P}{V_m C P_0}$$

Where  $V_m$  is the monolayer adsorption capacity,  $S_{BET} = V_m \cdot N_A \cdot A_m / M$  ( $N_A$  is Avogadro's constant,  $A_m$  is the cross-sectional area of  $N_2$  molecule  $0.162 \text{ nm}^2$ ). The mercury intrusion method uses capillary pressure to measure pore size distribution and porosity, pore size  $r = -2\sigma \cos\theta / P$  ( $\sigma = 0.485 \text{ N/m}$ ,  $\theta = 140^\circ$ ), pore volume  $V_p = \int (dV/dP)dP$ . BET's t-plot method analyzes the micropore filling effect, and the mercury intrusion method combines the Kelvin equation ( $r_k = -2\sigma_v \cos\theta / (RT \ln(P/P_0))$ ) to correct the nanopore effect, which is applicable to the pore size range of 2-50 nm.

#### 9.3.2 Methods and control techniques

##### Measurement method

##### BET

$N_2$  adsorption (77 K),  $P/P_0$  range 0.01-0.35, sample mass 0.1-1 g, degassing conditions 150-300°C, 6-12 h. Ar adsorption (87 K) measures micropores, specific surface area accuracy  $\pm 2\%$ .

##### Mercury Porosimetry

Pressure range 0.005-420 MPa, pore size detection range 2 nm-200  $\mu\text{m}$ , sample volume 0.5-5  $\text{cm}^3$ , dried to moisture  $< 0.05\%$ . High pressure mode measures ultra-micropores ( $< 5 \text{ nm}$ ).

##### Control Technology

##### Sample pretreatment

Vacuum degassing ( $10^{-3} \text{ Pa}$ ), BET to avoid moisture interference, and mercury intrusion drying ( $80^\circ\text{C}$ ) to prevent pore collapse.

##### Instrument Calibration

BET uses standard  $\text{Al}_2\text{O}_3$  ( $S_{BET} \approx 100 \text{ m}^2/\text{g}$ ), and mercury intrusion is calibrated with known porous Si. The dynamic flow method optimizes BET accuracy, and the real-time pressure monitoring error of mercury intrusion is  $< 1\%$ .

##### Data fitting

BET uses the 5-7 point method to calculate  $S_{BET}$ , and the mercury intrusion method corrects the contact angle  $\theta$  and surface tension  $\sigma$  deviation ( $\pm 5\%$ ). T-plot is used to analyze the micropore volume, and

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segmented fitting is used to optimize the pore size distribution.

### 9.3.3 Influencing factors

#### Particle size

As D50 decreases from 100  $\mu\text{m}$  to 50 nm, S<sub>BET</sub> increases from 0.05 m<sup>2</sup>/g to 20 m<sup>2</sup>/g, and the surface area increases exponentially with decreasing particle size.

#### Porosity

When P > 20%, the pore volume measured by mercury intrusion injection has a deviation of  $\pm 20\%$ , and BET underestimates the closed pore volume.

#### Surface Chemistry

WO<sub>3</sub> or organic residues, the BET adsorption amount increases by 15-30%, affecting the measurement accuracy.

#### Particle morphology

The S<sub>BET</sub> of spherical powder is 10-20% lower than that of polyhedron, and the regularity of morphology reduces the surface area.

#### Temperature fluctuations

BET 77 K deviation  $\pm 0.5$  K, adsorption variation  $\pm 5\%$ .

#### Pore connectivity

The number of closed pores increases, and the mercury injection method underestimates the porosity by 10-15%, which needs to be verified in combination with other methods.

### 9.3.4 Examples

Chen et al. (2021) used BET to measure nano-tungsten powder (D50 = 80 nm), S<sub>BET</sub> = 12 m<sup>2</sup>/g, which was applied to catalyst supports. Ar-BET measured the micropore volume of 0.02 cm<sup>3</sup>/g, which optimized the photocatalytic performance.

Yang et al. (2022) used mercury intrusion to analyze porous tungsten powder (pore size 50-500 nm) with a porosity of 15%, which is used as heat pipe core material. High-pressure mercury intrusion was used to measure micropores (2-10 nm), and the porosity increased to 18%, which improved capillary suction.

Li et al. (2024) combined BET and mercury intrusion analysis to analyze W-Cu composite powder, S<sub>BET</sub> = 8 m<sup>2</sup>/g, pore size distribution 20-100 nm, optimized sintering density to 98%, for high thermal conductivity substrates.

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### 9.3.5 Optimization direction

#### Low temperature BET

Ar/Kr adsorption (87 K), the accuracy of measuring micropores (<1 nm) is improved by 15%, which is suitable for nanopowder analysis.

#### Improvement of mercury intrusion porosimetry

Combining micro-CT and X-ray tomography, the pore 3D network was reconstructed with an error of <5%, improving the spatial resolution.

#### Automated analysis

Online BET/mercury intrusion system, real-time monitoring of S<sub>BET</sub> and pore size distribution, efficiency improvement of 30%.

#### Dynamic adsorption

BET measures adsorption kinetics, analyzes surface active site distribution, and optimizes catalytic applications.

#### Multiscale analysis

BET+mercury intrusion porosimetry+small angle X-ray scattering (SAXS) covers the pore size range of 0.1 nm-1 μm and provides comprehensive pore information.

Environmental simulation: BET measures surface reactivity in a CO<sub>2</sub>/O<sub>2</sub> atmosphere, simulating actual usage conditions.

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## CTIA GROUP LTD

### Spherical Tungsten Powder Product Introduction

#### 1. Overview of Spherical Tungsten Powder

CTIA GROUP LTD's spherical tungsten powder complies with the GB/T 41338-2022 "Spherical Tungsten Powder for 3D Printing" standard. It is prepared using a plasma spheroidization process and is specially designed for additive manufacturing (such as SLM, EBM). It meets high-end application requirements with high purity, high sphericity and excellent fluidity.

#### 2. Excellent Properties of Spherical Tungsten Powder

Ultra-high purity: tungsten content  $\geq 99.95\%$ , oxygen content  $\leq 0.05$  wt%, and extremely low impurities.

High sphericity:  $\geq 90\%$ , uniform particles, excellent powder spreading performance.

Precise particle size: D50 range 5-63  $\mu\text{m}$ , stable distribution, deviation  $\pm 10\%$ .

Excellent fluidity:  $\leq 25$  s/50g, bulk density  $\geq 9.0$  g/cm<sup>3</sup>, ensuring printing efficiency.

#### 3. Specifications of Spherical Tungsten Powder

Brand	D50 particle size ( $\mu\text{m}$ )
SWP-15	5-15
SWP-25	15-25
SWP-45	25-45
SWP-63	45-63

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

#### 4. Spherical Tungsten Powder Packaging and Quality Assurance

Packaging: Inner vacuum aluminum foil bag, outer iron drum, net weight 5kg or 10kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition, particle size distribution and sphericity data, and the shelf life is 12 months.

#### 5. Contact Information of CTIA GROUP LTD

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about spherical tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## Chapter 10 Composition and Surface Analysis of Tungsten Powder (Composition and Surface Analysis)

The composition and surface properties of tungsten powder are crucial to its performance in high-performance applications, such as the high purity requirements of semiconductor targets, the surface activity of catalyst carriers, and the antioxidant capacity of high-temperature corrosion-resistant components. These characteristics not only determine the functional realization of tungsten powder, but also directly affect its stability and life in extreme environments. This chapter uses three indicators, X-ray photoelectron spectroscopy (XPS) and Auger electron spectroscopy (AES), inductively coupled plasma (ICP-MS) and energy spectrum (EDS), and surface chemical state and contamination detection (TOF-SIMS), to deeply explore the precise characterization of tungsten powder composition and surface property analysis, covering theoretical basis, characterization methods and control technology, influencing factors, application cases and optimization strategies. By expanding technical details and analysis framework, this chapter provides scientific basis and technical support for the optimization design of tungsten powder in the field of high precision and high reliability.

### 10.1 Tungsten Powder X-ray Photoelectron Spectroscopy (XPS) and Auger Electron Spectroscopy (AES)

#### (Tungsten Powder X-ray Photoelectron Spectroscopy (XPS) & Auger Electron Spectroscopy (AES))

##### 10.1.1 Theoretical basis

XPS uses X-rays to excite photoelectrons to determine the chemical state, oxidation state and electronic

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structure of the elements on the surface of tungsten powder, with a detection depth of 1-10 nm; AES uses Auger electron transitions to analyze the surface element distribution and chemical bond state with a resolution of <1 nm.

### XPS

Photoelectron kinetic energy  $E_k = hv - E_b - \phi$  ( $hv = 1486.6$  eV for Al  $K\alpha$ ,  $E_b$  is binding energy,  $\phi \approx 4-5$  eV for W). W  $4f_{7/2}$  peak: metallic state  $\sim 31.5$  eV, W<sup>4+</sup> (WO<sub>2</sub>)  $\sim 33.5$  eV, W<sup>6+</sup> (WO<sub>3</sub>)  $\sim 35.5-36.0$  eV. Photoelectron escape depth  $\lambda \propto (E_k)^{1/2}$ , about 2-5 nm, signal intensity  $I \propto 1 - \exp(-d/\lambda)$ .

### AES

Auger electron energy  $E_A = E_1 - E_2 - E_3 - \phi$ , W MNN peak  $\sim 1736$  eV, O KLL peak  $\sim 510$  eV. Auger yield  $Y \propto I_e \cdot \sigma \cdot (1 - r)$  ( $I_e$  is the electron current,  $\sigma$  is the ionization cross section, and  $r$  is the backscattering factor).

XPS and AES are powerful tools for exploring the "chemical code" of the surface of tungsten powder. XPS uses X-rays to "knock" tungsten atoms to stimulate photoelectrons. The energy of these electrons is like an "identity card", which clearly marks whether tungsten is in a metallic state (W<sup>0</sup>) or oxidized to W<sup>4+</sup> or W<sup>6+</sup>. The detection range is limited to a few nanometers on the surface, like only looking at the "surface layer of the skin". AES is more sophisticated, like an "electron detective", capturing traces of electron jumps through Auger transitions, which can not only distinguish the distribution of tungsten and oxygen, but also reveal subtle changes in chemical bonds with sub-nanometer accuracy. The escape depth and yield formula further explain the source and strength of the signal, telling us how electrons "escape" from the surface and what factors determine the clarity of the signal. This theoretical foundation provides solid support for subsequent analysis.

## 10.1.2 Methods and control techniques

### Measurement method

#### XPS

Al  $K\alpha$  or Mg  $K\alpha$  source ( $hv = 1253.6$  eV), power 100-300 W, vacuum degree  $< 10^{-9}$  Pa, energy resolution 0.4-0.8 eV, scan range 0-1200 eV, step size 0.05-0.1 eV. Monochromatized X-rays reduce background noise, angle-resolved XPS (ARXPS) measures depth distribution by tilting 0-90°.

#### AES

Electron gun 1-10 keV, beam current 10 nA-1  $\mu$ A, detector sensitivity  $> 10^4$  cps, analysis area 10-1000  $\mu\text{m}^2$ . Depth profiling with Ar<sup>+</sup> sputtering (0.5-5 keV, rate 0.1-10 nm/min).

### Control Technology

#### Sample preparation

Ultrasonic cleaning (ethanol/deionized water, 50 W, 10 min), XPS uses conductive glue to fix the powder,

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AES requires mirror polishing ( $R_a < 10$  nm), freeze drying to avoid thermal oxidation, and ion polishing to remove surface stress.

### Data processing

XPS uses Shirley or Tougaard background subtraction, peak fitting (Gaussian-Lorentzian), chemical state ratio accuracy  $\pm 0.1$  eV; AES uses differential spectrum or direct spectrum, signal smoothing filter (Savitzky-Golay). XPS quantification uses sensitivity factor ( $S_W \approx 10.8$ ), and AES is calibrated with Monte Carlo simulation.

### Environmental Control

Ultra-high vacuum ( $< 10^{-10}$  Pa),  $O_2$  partial pressure  $< 10^{-12}$  atm, temperature control  $\pm 0.1^\circ C$ . In-situ reaction chamber ( $H_2/Ar$ , 100-1000°C) to measure surface reduction.

Measuring XPS and AES is like doing a "surface physical examination" for tungsten powder, which requires precise instruments and meticulous operation. XPS uses X-rays to "illuminate" the surface, and the monochromatization technology is like adding a "filter" to make the signal cleaner. ARXPS is like adjusting the "searchlight angle" to dig out information from different depths. The electron gun of AES is like a "micro-cannonball", which collects Auger electrons after bombarding the surface, and  $Ar^+$  sputtering is like a "skinning knife", revealing the secrets of the surface layer by layer. The sample is prepared carefully, and ultrasonic cleaning and freeze drying are used to avoid "staining" or "burning" the tungsten powder. Conductive glue and polishing make the surface "stand firm and see clearly". Data processing is a "decoding master" that extracts chemical states and distribution laws from messy signals. Ultrahigh vacuum and in-situ reaction chambers are like "sterile rooms" to ensure that the results are not interfered with by the outside world. The combination of these methods and technologies allows us to deeply understand every detail of the tungsten powder surface.

### 10.1.3 Influencing factors

#### Surface oxidation

$WO_3$  layer thickness is 2-10 nm, the XPS  $W^{6+}/W^0$  ratio changes by  $\pm 15\%$ , and the AES O KLL intensity increases by 20-50%.

#### Beam damage

The AES electron beam is  $> 10$  keV, and the local temperature increase ( $> 500^\circ C$ ) induces WC bonds, and the XPS C 1s peak increases by 10%.

#### Sample roughness

When  $R_a > 200$  nm, XPS signal scattering loss is 10-20%, and AES depth resolution is reduced by 30%.

#### Charging effect

Non-conductive  $WO_3$  layer, XPS peak shift  $\pm 2$  eV, requires a neutralization gun (low energy electrons,

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1-5 eV).

#### **Vacuum residual gas**

CO/H<sub>2</sub>O adsorption, XPS C/O content increased by 5-10%, AES requires pre-sputtering >10 nm.

#### **Depth of analysis**

XPS tilt angle >60°, detection depth down to 1-2 nm; AES sputtering rate deviation ±10%.

When analyzing the surface of tungsten powder, various influencing factors are like "roadblocks" that need to be dealt with one by one. Surface oxidation makes the WO<sub>3</sub> layer "blind" the truth. XPS and AES have to work hard to distinguish the true state of tungsten. The thicker the oxide layer, the greater the signal change. The high-energy electron beam of AES is like "overexerting", which may "burn" new bonds on the surface. XPS will also have more carbon signals, interfering with judgment. Roughness is like "rugged terrain", signal scattering deviates, and accuracy decreases. Non-conductive areas will be "charged", and the peak shift must be "suppressed" by the neutralization gun, otherwise the data will be biased. The residual gas in the vacuum is like "invisible pollution", quietly increasing carbon and oxygen, and the analysis depth must be "fine-tuned" by angle and sputtering. These factors remind us that surface analysis is not a simple "scan", but must be carried out step by step to ensure that every step is accurate.

#### **10.1.4 Examples**

Zhang et al. (2020)

Using XPS to measure tungsten powder, the WO<sub>3</sub> / W ratio is 0.3, the hydrogen reduction temperature is optimized to 900°C, and the purity reaches 99.95%. Extended case: ARXPS confirms that the WO<sub>3</sub> layer thickness is 3-5 nm, guiding the design of anti-oxidation coatings.

Li et al. (2022)

AES analysis of W-Cu composite powder shows that the Cu segregation layer is less than 2 nm, which improves the conductivity by 12%. Extended case: AES depth profile measurement of W-Ni shows that the Ni diffusion depth is 5-10 nm, which optimizes the sintering uniformity.

Wang et al. (2024)

In situ XPS (1000°C, H<sub>2</sub>) was used to monitor the reduction of W<sup>6+</sup> to W<sup>0</sup> in real time with a reaction rate  $k = 0.02 \text{ min}^{-1}$  for high temperature devices.

These cases are like "actual combat records" of tungsten powder surface analysis. Zhang used XPS to find out the degree of oxidation of tungsten powder, and by adjusting the hydrogen reduction temperature to "wash off" the excess oxide layer, the purity was greatly improved. ARXPS also accurately measured the thickness of the oxide layer to guide the design of the coating. Li used AES to discover the "hiding place" of copper in W-Cu composite powder. The thin segregation layer improved the conductivity, and the deep profile revealed the diffusion law of W-Ni, which helped sintering more uniformly. Wang's in-

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situ XPS is like "live broadcast", watching  $W^{6+}$  turn back to  $W^0$  at high temperature, and the reaction rate data provides valuable basis for high-temperature devices. These studies show how XPS and AES can unlock the performance potential of tungsten powder from different angles.

### 10.1.5 Optimization direction

#### In-situ XPS

High temperature and high pressure (1500°C, 10 atm) measurement of oxidation/reduction kinetics with an accuracy of  $\pm 0.1$  eV.

AES 3D Imaging: Combines FIB and sputtering to create elemental maps with a resolution of  $< 0.3$  nm.

#### AI-Assisted

Deep learning fits XPS peaks, with chemical state recognition rate  $> 99\%$ ; AES automatically calibrates background noise.

#### Time-resolved XPS

Femtosecond laser-assisted measurement of surface reaction dynamics ( $< 1$  ps).

#### Multimodal analysis

XPS+AES+UPS (ultraviolet photoelectron spectroscopy) simultaneously characterizes chemical state and work function.

#### Low temperature protection

$-150^\circ\text{C}$  XPS/AES to reduce volatilization of heat-sensitive pollutants.

The optimization direction is to "upgrade equipment" for XPS and AES. In-situ XPS is like a "high-temperature laboratory" that can observe the oxidation and reduction process of tungsten powder under extreme conditions with high accuracy. AES 3D imaging combined with FIB is like a "stereo microscope", which draws the element distribution into a three-dimensional diagram in detail. AI assistance is an "intelligent helper" that makes peak fitting and noise calibration fast and accurate. Time-resolved XPS captures the "moment" of surface reaction, multimodal analysis integrates multiple information, and low-temperature protection "keeps" heat-sensitive samples. These advances have transformed the surface analysis of tungsten powder from "static photos" to "dynamic movies", and the future application prospects are broad.

## 10.2 Tungsten Powder Inductively Coupled Plasma (ICP-MS) and Energy Dispersive Spectroscopy (EDS)

### (Tungsten Powder Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Energy Dispersive Spectroscopy (EDS))

#### 10.2.1 Theoretical basis

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ICP-MS determines trace elements and isotopes in tungsten powder through plasma ionization and mass spectrometry separation, with a detection limit of <0.1 ppb; EDS uses characteristic X-rays to analyze elemental composition with a detection depth of 0.5-3  $\mu\text{m}$ .

### ICP-MS

Ion signal  $I = k \cdot N \cdot Q \cdot T$  ( $k$  is the instrument constant,  $N$  is the concentration,  $Q$  is the ionization efficiency, and  $T$  is the transmission efficiency),  $W^+$  main peak  $m/z = 184$ , interference such as  $^{184}\text{Os}^+$ . The collision reaction cell (CRC) eliminates polyatomic interferences (such as  $\text{ArO}^+$ ).

### EDS

X-ray count rate  $I \propto C \cdot \omega \cdot (E_0 - E_c)^{1.5}$  ( $C$  is the concentration,  $\omega$  is the fluorescence yield,  $E_0$  is the incident energy, and  $E_c$  is the critical energy).  $W L\alpha \sim 8.4 \text{ keV}$ ,  $Ma \sim 1.8 \text{ keV}$ . ZAF correction takes into account absorption (A) and fluorescence (F) effects.

ICP-MS and EDS are "detective experts" for the composition of tungsten powder. ICP-MS uses high-temperature plasma to "burn" tungsten powder into ions, and then "screens" it through mass spectrometry. It can detect extremely small amounts of impurities, even as low as ppb level, like using an "ultra-fine sieve" to find hidden elements. EDS is like an "X-ray searchlight". The electron beam excites tungsten powder to emit characteristic X-rays, revealing the elemental composition of the surface and near the surface. The detection depth is slightly deeper and can quickly give an overall picture. CRC and ZAF corrections are their "purifiers" and "focusing lenses" respectively, removing interference, correcting errors, and making the results more reliable. These theoretical formulas are like "compasses", guiding us to extract accurate composition information from signals.

## 10.2.2 Methods and control techniques

### Measurement method

#### ICP-MS

The sample was dissolved in  $\text{HNO}_3 / \text{HF}/\text{HClO}_4$  (5 :3:1), power 1.2-2.5 kW, carrier gas (Ar) flow rate 0.8-1.2 L/min, internal standard (such as  $^{115}\text{In}$ , 5-20 ppb). Laser ablation (LA-ICP-MS), direct measurement of solid powder, spatial resolution <10  $\mu\text{m}$ .

#### EDS

SEM/TEM integration, accelerating voltage 5-30 kV, detector SDD (Silicon Drift Detector), resolution 120-130 eV, acquisition time 60-300 s. Low temperature EDS (-50°C) to reduce thermal noise.

### Control Technology

#### Sample preparation

Acid digestion for ICP-MS (microwave, 200°C, 30 min), pressed powder pellets or polished powder pellets ( $R_a < 50 \text{ nm}$ ) for EDS. Ultrapure acid for ICP-MS (<1 ppt impurities), carbon film for EDS (5-10

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nm).

### Data processing

ICP-MS uses external standard method (linear range 0.1 ppb-10 ppm), EDS uses Cliff-Lorimer method or standard sample calibration, error  $\pm 0.5$  wt%. ICP-MS dynamic reaction cell (DRC) is used to measure low-content O/N, and EDS peak decomposition algorithm is used.

### Instrument Calibration

NIST SRM 3127a (W standard solution) was used for ICP-MS, and pure W/Cu/Ni standards were used for EDS. ICP-MS was quality controlled daily (CV <2%), and EDS was automatically energy calibrated.

The operation of ICP-MS and EDS is like "chemical prospecting". ICP-MS first "dissolves" the tungsten powder in acid, and then "ignites" it with plasma. The internal standard and laser ablation are like "locators" to ensure that trace elements and solid samples can be accurately captured. EDS "turns on the light" on the SEM or TEM, the SDD detector quickly collects X-rays, and the low-temperature mode is like a "noise reducer" to make the signal clearer. Sample preparation is the basis, acid digestion and polishing make the tungsten powder "obedient", and ultra-pure acid and carbon film avoid "external interference". Data processing is a "translator" that converts the signal into concentration numbers, and instrument calibration and quality control are like "regular physical examinations" to ensure stable results. These technical combinations make the composition analysis of tungsten powder both deep and wide.

## 10.2.3 Influencing factors

### Impurity content

ICP-MS detection <1 ppb requires an ultra-clean environment, and EDS has low sensitivity to O/C/N (<10 wt%).

### Matrix Effect

In ICP-MS, W concentration >100 ppm suppresses Fe<sup>+</sup> / Ni<sup>+</sup> signals by 5-10%, and EDS W-Cu overlap requires peak separation.

### Sample homogeneity

The local deviation of EDS is  $\pm 5-10$  wt%, and the error of ICP-MS for unhomogenized samples is  $\pm 15\%$ .

### Solution stability

When the ICP-MS samples were left for >24 h, precipitation reduced the signal by 10%.

### Beam energy

EDS >20 kV, deep X-rays interfere with surface analysis.

### Instrument drift

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When ICP-MS is operated for a long time (>8 h), the sensitivity decreases by 5-8%.

The influencing factors are the "variable switches" of tungsten powder composition analysis. When the impurity content is low, ICP-MS must be "strictly guarded" in an ultra-clean environment, but EDS "turns a blind eye" to light elements. The matrix effect is like "internal competition". High concentrations of tungsten suppress the signals of other elements, and EDS must be careful of the "confusion" of peak overlap. Uneven samples are like "drawing lots", and the results may deviate from the truth. The solution of ICP-MS will "sink to the bottom" if left for a long time, and the high-energy beam of EDS is like "digging too deep" and mixing in deep information. Instrument drift is the "enemy of time", and long-term operation makes the sensitivity "weak". These factors tell us that the analysis of components must fully consider the environment, samples and instrument status.

#### 10.2.4 Examples

##### Wang et al. (2021)

Using ICP-MS to measure tungsten powder, Fe/Cr <5 ppb, meeting the requirements of semiconductor targets. Extended case: LA-ICP-MS to measure the internal structure of W particles, Mo distribution uniformity >98%, optimizing the alloying process.

##### Chen et al. (2023)

EDS analysis of W-Ni powder shows that the Ni content is 4.8-5.2 wt% and the sintered density is 97%. Extended case: Low temperature EDS measurement of W-Cu shows that the O content is <0.5 wt%, which improves welding performance.

##### Li et al. (2024)

DRC-ICP-MS was used to measure O/N in W (<10 ppb) to guide the preparation of ultrapure W for aviation parts.

These cases are "successful examples" of tungsten powder composition analysis. Wang used ICP-MS to confirm that the purity of tungsten powder is extremely high, and Fe and Cr are as low as ppb level, which perfectly meets the needs of semiconductors. LA-ICP-MS is also like a "microscope" to check the distribution of Mo inside the particles and optimize the alloy process. Chen used EDS to find out the Ni content of W-Ni, which increased the sintering density. Low-temperature EDS helped W-Cu control the oxygen content, making welding more reliable. Li used DRC-ICP-MS to capture ultra-trace O and N, pointing the way for the preparation of ultra-pure tungsten for aviation components. These studies have demonstrated the powerful capabilities of ICP-MS and EDS in different scenarios.

#### 10.2.5 Optimization direction

High Resolution ICP-MS: Separation of  $^{184}\text{W}$  /  $^{184}\text{Os}$  with mass resolution >10,000 and accuracy  $\pm 0.05\%$ .

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### EDS Quantification

Combined with WDS (Wavelength Dispersive Spectroscopy), the light element detection limit is <0.1 wt%.

### Online monitoring

ICP-MS analyzes production wastewater in real time, and EDS integrates deep learning to optimize element distribution maps.

### LA-ICP-MS imaging

3D elemental distribution with a resolution of <5  $\mu\text{m}$ .

### Low temperature EDS

-100°C Measure volatile impurities (such as S/P) with a 20% increase in sensitivity.

### Multi-element synchronization

ICP-MS scans 70+ elements with an analysis time of <1 min.

Optimization direction makes ICP-MS and EDS "take it to the next level". High-resolution ICP-MS is like a "super sieve" that can separate almost overlapping mass peaks with amazing accuracy. EDS combined with WDS is like a "high-definition lens", and light elements are no longer blind spots. Online monitoring is a "real-time guard" to keep track of composition changes during production. LA-ICP-MS imaging "draws" element distribution into a 3D map, low-temperature EDS captures volatile impurities, and multi-element synchronization allows ICP-MS to scan the "element table" at once. These advances make tungsten powder composition analysis faster, more accurate, and more comprehensive.

## 10.3 Tungsten Powder Surface Chemical State and Contamination Detection (TOF-SIMS) (Tungsten Powder Surface Chemistry and Contamination Detection (Time-of-Flight Secondary Ion Mass Spectrometry, TOF-SIMS))

### 10.3.1 Theoretical basis

TOF-SIMS uses ion bombardment to excite secondary ions and analyze the surface chemical state, molecular structure and trace contaminants of tungsten powder. The detection limit is <0.1 ppm and the depth resolution is <0.5 nm.

Secondary ion intensity  $I_s = I_p \cdot Y \cdot \eta$  ( $I_p$  is the primary ion current,  $Y$  is the sputtering yield, and  $\eta$  is the ionization efficiency).  $W^+$  (~184 u),  $WO^+$  (~200 u),  $WO_2^+$  (~216 u) reflect the oxidation state, and  $C_xH_y^+/Na^+$  indicates contamination. Molecular ion fragmentation follows the collision cascade theory, and the yield  $Y \propto E_p^{1/2}$  ( $E_p$  is the primary ion energy).

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TOF-SIMS is a "molecular probe" of the surface of tungsten powder. It "bombards" the surface with an ion beam to stimulate secondary ions. These ions are like "messengers" flying to the detector with information about the chemical state and pollutants. The detection limit is as low as below ppm, and the depth resolution is as fine as sub-nanometer. The  $W^+$ ,  $WO^+$ , and  $WO_2^+$  signals reveal the degree of oxidation of tungsten, while  $C_xH_y^+$  and  $Na^+$  expose organic or salt contamination. The collision cascade theory explains how ions are "knocked out". The higher the energy, the greater the yield, which provides a theoretical basis for analytical sensitivity.

### 10.3.2 Methods and control techniques

#### Measurement method

$Bi^+$  /  $Ga^+$  /  $O_2^+$  primary ions (10-30 keV), beam current 0.1-10 pA, vacuum  $<10^{-9}Pa$ , mass resolution  $m/\Delta m >12,000$ , analysis area  $50 \times 50 \mu m^2$  to  $500 \times 500 \mu m^2$ . Dual beam mode ( $Bi^+$  analysis,  $Cs^+$  /  $Ar^+$  sputtering), depth profile rate 0.05-5 nm/s.

#### Control Technology

##### Sample preparation

Ultra-clean room (ISO Class 5) operation, cryo-transfer to avoid contamination, pre-sputtering (1-3 keV, 1-5 nm) to clean the surface. Liquid nitrogen cooling sample stage ( $-150^\circ C$ ) to measure volatile organic compounds.

##### Data processing

Peak identification accuracy was  $\pm 0.005 u$ , 3D reconstruction was performed using TOF-SIMS V software, and quantification was performed using relative sensitivity factor (RSF). Molecular fingerprint library matching was performed to identify  $C_xH_y^+$  structures.

##### Instrument Optimization

Pulse width  $<1 ns$ , ion extraction voltage 2-5 kV, signal-to-noise ratio  $>100$ . High-resolution imaging mode, resolution  $<50 nm$ .

The operation of TOF-SIMS is like "surface archaeology". Primary ions are like "excavation tools" that bombard the surface of tungsten powder. The dual-beam mode "digs" and "looks" at the same time, and the depth profile is meticulous. The ultra-clean room and cryotransfer are "protective umbrellas" to prevent external contamination and "adulteration", and pre-sputtering is like a "sweeper" to sweep away surface impurities. Data processing is the "archivist" who accurately identifies each ion peak, and 3D reconstruction and fingerprint library make the molecular structure "appear". Instrument optimization ensures that the signal is "loud and clear", and high-resolution imaging is like a "magnifying glass" to capture tiny details. These technologies make the chemical state and contamination on the surface of tungsten powder nowhere to hide.

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### 10.3.3 Influencing factors

#### Surface contamination

C/O >1 ppm, masks W<sup>+</sup> / WO<sup>+</sup> signal, requires sputtering >10 nm.

#### Ion beam energy

>30 keV, implantation effect increases by 5-10%, interfering with low-mass ions (such as Na<sup>+</sup>).

#### Sample morphology

Ra >100 nm, the secondary ion yield decreases by 20-30%.

#### Matrix Effect

The WO<sub>3</sub> matrix enhances the C<sup>+</sup> signal by 10-15%.

#### Temperature Effect

>100°C, volatile pollutants lose 20-50%.

#### Analysis time

>30 min, surface recontamination increases by 5%.

The influencing factors in TOF-SIMS analysis are like "tuners", and the slightest deviation will affect the results. Surface contamination is like "dust", covering the key signals, and sputtering is needed to "clean it up". If the ion beam energy is too high, it will "overshoot" and implant ions to interfere with the analysis. Rough morphology is like a "bumpy road", which reduces the yield, and the matrix effect is like an "amplifier", which makes some signals "take over". High temperature makes pollutants "run away", and long analysis time will "attract dust". These remind us that TOF-SIMS requires fine control of the environment and operation.

### 10.3.4 Examples

#### Yang et al. (2022)

Using TOF-SIMS to measure tungsten powder, Na<sup>+</sup> / K<sup>+</sup> <0.1 ppm, optimizing the cleaning process for electronic packaging. Extended case: 3D TOF-SIMS to measure W surface, C<sub>x</sub> H<sub>γ</sub><sup>+</sup> distribution <1 nm, improving packaging sealing.

#### Li et al. (2023)

Measuring W nanopowder, WO<sub>3</sub> layer 1-3 nm, improving anti-oxidation design. Extended case: Dual-beam TOF-SIMS measuring W-Cu, Cu<sup>+</sup> diffusion depth 2-5 nm, optimizing interface bonding.

#### Chen et al. (2024)

Use low-temperature TOF-SIMS (-100°C) to detect volatile oil (C<sub>10</sub> H<sub>22</sub><sup>+</sup>) on the W surface, with a

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content of <0.05 ppm, for high vacuum devices.

These cases are the "detection logs" of TOF-SIMS. Yang measured that the Na<sup>+</sup> and K<sup>+</sup> of tungsten powder were extremely low, so the cleaning process was upgraded. 3D analysis also found that the contaminants were only on the surface, and the sealed packaging was tighter. Li found out the thickness of the WO<sub>3</sub> layer of W nanopowder, and the anti-oxidation design was more precise. The dual-beam mode revealed the details of the W-Cu interface and the bond was stronger. Chen caught the "tail" of volatile oils at low temperatures, and the content was as low as below ppm level, which protected high vacuum devices. These cases demonstrate the high sensitivity and practicality of TOF-SIMS in surface analysis.

### 10.3.5 Optimization direction

Dynamic SIMS: Real-time monitoring of oxidation/adsorption reactions with a time resolution of <0.5 s.

#### Molecular imaging

TOF-SIMS resolves the molecular weight of organic pollutants (<500 Da) with a 30% increase in sensitivity.

#### Multi-technique combination

TOF-SIMS+XPS+FTIR, comprehensive characterization of surface states and contamination sources.

#### High-resolution imaging

Focused ion beam (FIB) integration with a resolution of <20 nm.

#### Low temperature analysis

-150°C for measuring adsorbed H<sub>2</sub>O / O<sub>2</sub>, detection limit <0.01 ppm.

#### Automated processing

AI recognizes TOF-SIMS spectra, improving analysis efficiency by 40%.

Optimization direction makes TOF-SIMS "more powerful". Dynamic SIMS is like "real-time monitoring", capturing the moment of oxidation and adsorption. Molecular imaging analyzes the "chemical identity" of pollutants with higher sensitivity. Multi-technique combination is an "all-round player", covering everything from chemical state to pollution source. High-resolution imaging and FIB are like "ultra-clear lenses", showing every detail. Low-temperature analysis captures adsorbed molecules, and automated processing makes spectrum analysis "as fast as lightning". These advances will push TOF-SIMS into a future with higher precision and wider applications.

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## CTIA GROUP LTD

### Spherical Tungsten Powder Product Introduction

#### 1. Overview of Spherical Tungsten Powder

CTIA GROUP LTD's spherical tungsten powder complies with the GB/T 41338-2022 "Spherical Tungsten Powder for 3D Printing" standard. It is prepared using a plasma spheroidization process and is specially designed for additive manufacturing (such as SLM, EBM). It meets high-end application requirements with high purity, high sphericity and excellent fluidity.

#### 2. Excellent Properties of Spherical Tungsten Powder

Ultra-high purity: tungsten content  $\geq 99.95\%$ , oxygen content  $\leq 0.05$  wt%, and extremely low impurities.

High sphericity:  $\geq 90\%$ , uniform particles, excellent powder spreading performance.

Precise particle size: D50 range 5-63  $\mu\text{m}$ , stable distribution, deviation  $\pm 10\%$ .

Excellent fluidity:  $\leq 25$  s/50g, bulk density  $\geq 9.0$  g/cm<sup>3</sup>, ensuring printing efficiency.

#### 3. Specifications of Spherical Tungsten Powder

Brand	D50 particle size ( $\mu\text{m}$ )
SWP-15	5-15
SWP-25	15-25
SWP-45	25-45
SWP-63	45-63

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

#### 4. Spherical Tungsten Powder Packaging and Quality Assurance

Packaging: Inner vacuum aluminum foil bag, outer iron drum, net weight 5kg or 10kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition, particle size distribution and sphericity data, and the shelf life is 12 months.

#### 5. Contact Information of CTIA GROUP LTD

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about spherical tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## Chapter 11 Tungsten Powder Performance Testing Technology (Performance Testing Techniques for Tungsten Powder)

The performance testing technology of tungsten powder is a key method to evaluate its application potential in extreme environments, which is directly related to its reliability and applicability in aerospace, high-temperature structural parts, electronic devices and electromagnetic shielding. Tungsten powder is widely used in key components under harsh working conditions due to its high density, high melting point and excellent physical properties. The accurate characterization of its performance not only depends on the intrinsic properties of the material, but is also closely related to the scientificity and standardization of the testing technology. This chapter systematically discusses the theoretical basis, experimental methods, key control links, influencing factors, practical application cases and future optimization directions of its performance testing through three major indicators: tungsten powder mechanical properties testing, thermal analysis technology, and electrical and electromagnetic testing. By refining technical details and expanding the analysis framework, this chapter provides a scientific basis and technical support for the comprehensive evaluation and industrial optimization of tungsten powder performance.

### 11.1 Mechanical properties test of tungsten powder

#### (Tungsten Powder Mechanical Property Testing: Tensile, Compression, Three-Point Bending)

##### 11.1.1 Theoretical basis

Mechanical properties testing is used to characterize the strength, toughness, plasticity and fracture behavior of tungsten powder sintered bodies or composite materials. These properties are affected by the microstructure (such as grain size, porosity) and test conditions.

##### Stretch

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Stress  $\sigma = F/A$ , strain  $\epsilon = \Delta L/L_0$  ( $F$  is the tensile force,  $A$  is the cross-sectional area, and  $L_0$  is the initial length), Young's modulus  $E = \sigma/\epsilon$ , pure W sintered body  $E \approx 400-410$  GPa, yield strength  $\sigma_y \approx 550-700$  MPa. The stress-strain curve is divided into the elastic region (Hooke's law), the plastic region and the fracture region, and the fracture work  $W_f = \int \sigma d\epsilon$ .

### compression

The compressive strength  $\sigma_c = F_{max}/A$ , Poisson's ratio  $\nu = -\epsilon_{lat}/\epsilon_{long} \approx 0.28$  (W), reflects the lateral deformation characteristics. The Barrett equation considers the effect of porosity on  $\sigma_c$ ,  $\sigma_c = \sigma_0 \cdot \exp(-kP)$  ( $P$  is the porosity,  $k \approx 4-5$ ).

### Three-point bending

Bending strength  $\sigma_f = 3FL/(2bh^2)$  ( $F$  is the maximum load,  $L$  is the span,  $b$  is the width,  $h$  is the thickness), fracture toughness  $K_{IC} = Y \cdot \sigma_f \cdot (\pi a)^{1/2}$  ( $Y$  is the geometric factor,  $a$  is the crack length). Weibull statistical analysis of brittle fracture probability,  $m$  (Weibull modulus)  $\approx 10-15$  (W).

Mechanical property tests provide a quantitative basis for the strength and deformation capacity of tungsten powder materials. The tensile test measures the tensile strength and elongation of the material by applying a tensile force. The Young's modulus reflects the rigidity of tungsten within the elastic range. The stress-strain curve fully records the entire process from elastic deformation to fracture, providing basic data for analyzing its bearing capacity and fracture energy. The compression test evaluates the material's ability to resist deformation under pressure. The Poisson's ratio quantifies the proportional relationship between lateral and longitudinal deformation, and the Barrett equation reveals how porosity significantly affects the compression strength. The three-point bending test determines the bending strength and fracture toughness through bending loading, and the Weibull statistical method is used to analyze the fracture probability distribution of brittle materials. These theoretical models lay a scientific foundation for understanding the mechanical behavior of tungsten powder.

## 11.1.2 Methods and Control Technology

### Measurement method

#### Stretch

Universal testing machine (ASTM E8/E8M), load range 1-500 kN, loading rate 0.1-10 mm/min, sample size  $\phi 5-10$  mm  $\times$  25-50 mm, fixture centering accuracy  $\pm 0.005$  mm. High-precision extensometer (resolution  $\pm 0.1$   $\mu$ m), dynamic strain rate  $10^{-4} - 10^2$  s $^{-1}$ .

#### compression

Cylindrical specimens (ASTM E9), diameter/height ratio 1:1.5-1:2, load 10-1000 kN, loading rate 0.05-2 mm/min, displacement sensor accuracy  $\pm 0.001$  mm. Servo-hydraulic system, loading stability  $\pm 0.5\%$ .

#### Three-point bending

Rectangular specimens (ASTM E290), size 5 $\times$ 5 $\times$ 50 mm or 10 $\times$ 10 $\times$ 100 mm, span 20-80 mm, loading

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rate 0.02-1 mm/min, load resolution  $\pm 0.01$  N. Acoustic emission (AE) monitoring of crack growth, frequency range 100 kHz-1 MHz.

### Control Technology

#### Sample preparation

Powder pressing (50-200 MPa), sintering (1800-2300°C, H<sub>2</sub> / Ar, holding for 2-6 h), surface polishing (Ra < 0.5  $\mu$ m), edge chamfering (R 0.1-0.2 mm) to avoid stress concentration. Hot isostatic pressing (HIP, 200 MPa, 2000°C) to increase density to 99.5%.

### Test conditions

Temperature control 20-1500°C ( $\pm 0.5$ °C), humidity 30-60% (RH), loading uniformity  $\pm 0.5$ %, fixture hardness HRC > 60. High temperature performance is tested in vacuum environment ( $< 10^{-4}$  Pa) or inert atmosphere (Ar, 99.999%).

### Data collection

Strain gauge (accuracy  $\pm 0.05$ %) or laser rangefinder (resolution  $\pm 0.1$   $\mu$ m), real-time recording of  $\sigma$ - $\epsilon$  curve, sampling rate 10-1000 Hz. Digital image correlation (DIC) to measure local strain field, resolution < 0.01%.

Mechanical property testing requires high-precision equipment and strict control processes. Tensile testing uses a universal testing machine to apply tension, and a high-precision extensometer ensures accurate measurement of small deformations. The dynamic strain rate range covers a variety of loading conditions from low speed to high speed. Compression testing uses cylindrical samples, and the stability of the loading process is maintained by a servo-hydraulic system. The displacement sensor provides high-resolution deformation data. The three-point bending test is based on a rectangular specimen. Acoustic emission technology monitors the crack propagation process in real time, providing additional information for the analysis of fracture behavior. Sample preparation is a prerequisite for testing. The pressing and sintering processes ensure the integrity of the material structure. Surface polishing and edge chamfering reduce stress concentration effects, and hot isostatic pressing further increases density. The test conditions require precise control of temperature, humidity and atmosphere to ensure the repeatability of the results. Data acquisition uses strain gauges and laser rangefinders to record the complete stress-strain relationship. Digital image correlation provides high-resolution analysis of local strain distribution. These technologies and control measures together ensure the accuracy and reliability of the test data.

### 11.1.3 Influencing factors

#### Grain size

As D increases from 5  $\mu$ m to 100  $\mu$ m, the tensile strength  $\sigma_{uts}$  decreases from 800 MPa to 500 MPa ( $\sigma \propto D^{(-1/2)}$ ), and the compressive plasticity increases by 5-10%.

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### Porosity

When P increases from 1% to 10%,  $\sigma_c$  decreases by 25-40%, and the three-point bending  $K_{IC}$  decreases from  $10 \text{ MPa}\cdot\text{m}^{1/2}$  to  $6 \text{ MPa}\cdot\text{m}^{1/2}$ .

### Test temperature

At  $25^\circ\text{C}$ ,  $\sigma_{uts} \approx 700 \text{ MPa}$ ; at  $1000^\circ\text{C}$ , it drops to 200-250 MPa, and  $\epsilon_f$  increases to 10-15%.

### Loading rate

When the tensile rate is  $>10 \text{ mm/min}$ ,  $\sigma_{uts}$  increases by 5-10%, but the plasticity decreases.

### Impurity content

O/Fe  $>0.1 \text{ wt\%}$ , grain boundaries weaken and  $\sigma_f$  drops by 10-20%.

### Sample geometry

Three-point bending  $L/h <4$ , shear stress interference,  $\sigma_f$  deviation  $\pm 15\%$ .

The results of mechanical property tests are affected by many factors, which together determine the accuracy of the test data and the actual performance of the material. Changes in grain size directly affect strength and plasticity. Small grains increase tensile strength through the Hall-Petch relationship, while large grains enhance plastic deformation capacity during compression. Increased porosity significantly reduces compressive strength and fracture toughness because pores act as stress concentration points that weaken the overall load-bearing capacity of the material. Changes in test temperature have a significant effect on performance. Tungsten exhibits high strength at room temperature, while high temperatures result in decreased strength but increased plasticity. Increasing the loading rate slightly increases tensile strength, but at the same time reduces the time of plastic deformation. Increased impurity content such as oxygen and iron leads to a decrease in grain boundary strength, thereby reducing flexural strength. Sample geometry design is also critical. A low span-to-height ratio in three-point bending will introduce shear stress, affecting the measurement accuracy of flexural strength. These factors need to be fully considered in test design and result analysis to ensure the scientificity and applicability of the data.

## 11.1.4 Examples

### Li et al. (2021)

W-Cu (Cu 20 wt%) was tested, with a tensile strength of 600 MPa and  $\epsilon_f = 8\%$ , for high-temperature electrical contacts. DIC measured local strain and found that the Cu distribution was concentrated, and the Cu distribution was optimized to a uniformity of  $>95\%$ .

### Zhang et al. (2023)

Pure W ( $D_{50} = 5 \mu\text{m}$ ) was measured by three-point bending,  $\sigma_f = 800 \text{ MPa}$ ,  $K_{IC} = 9 \text{ MPa}\cdot\text{m}^{1/2}$ , used for aviation nozzles. AE monitored crack growth, initial stress 300 MPa, improved sintering process reduced defect density by 20%.

### Chen et al. (2024)

W-Ni (Ni 5 wt%) was measured by high temperature compression ( $1500^\circ\text{C}$ ),  $\sigma_c = 450 \text{ MPa}$ , plastic

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deformation 12%, and the high temperature mold design was optimized.

These cases demonstrate the important value of mechanical property testing in practical applications. Li et al. evaluated the performance of W-Cu composites through tensile testing. The strength of 600 MPa and the elongation of 8% meet the requirements of high-temperature electrical contacts. DIC analysis revealed the strain concentration problem of the Cu phase, and the uniformity was significantly improved after optimization. Zhang et al. used three-point bending to test pure tungsten and measured high bending strength and reasonable fracture toughness, which is suitable for aviation nozzles. Acoustic emission monitoring further clarified the starting stress of crack propagation, and improved sintering process reduced defect density. Chen et al. analyzed W-Ni alloy in high-temperature compression tests. The compressive strength of 450 MPa and the plastic deformation of 12% provided data support for high-temperature mold design. These studies show that mechanical testing can not only verify material properties, but also guide process optimization and engineering applications.

### 11.1.5 Optimization Direction

High temperature testing

2000°C tension/compression, creep rate ( $\dot{\epsilon} \approx 10^{-6} \text{ s}^{-1}$ ), accuracy  $\pm 1\%$ .

Micro Test

Micropillars ( $\phi 1\text{-}5 \mu\text{m}$ ) were compressed and dislocation evolution was analyzed by TEM.

Simulation Optimization

FEA combined with crystal plasticity models predicts stress distribution and fracture path with an error of  $< 3\%$ .

Dynamic loading

Impact test ( $10^3\text{-}10^5 \text{ s}^{-1}$ ), to measure the impact resistance.

In situ observation

The SEM is integrated with a tensile stage to record microcrack growth in real time.

Multi-scale testing

Nanoindentation measures local hardness ( $H \approx 6\text{-}8 \text{ GPa}$ ) to supplement macroscopic data.

The optimization direction of mechanical property testing aims to improve the testing capabilities and application scope. High temperature testing is extended to 2000°C, which can accurately measure the creep rate and provide a basis for performance evaluation in ultra-high temperature environments. Micro testing combines micro-column compression with TEM to deeply study the dislocation evolution mechanism. Simulation optimization uses finite element analysis (FEA) and crystal plasticity models to predict stress and fracture behavior and improve design efficiency. Dynamic loading tests evaluate the impact resistance of materials at high strain rates, in-situ observations record microscopic changes in real time through SEM, and multi-scale tests supplement local hardness data through nanoindentation. These optimization measures will make tungsten powder mechanical testing more comprehensive and accurate to meet the needs of complex working conditions in the future.

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## 11.2 Tungsten Powder Thermal Analysis Technology (Tungsten Powder Thermal Analysis Techniques: DSC, TGA, Dilatometry)

### 11.2.1 Theoretical basis

Thermal analysis techniques are used to characterize the thermal stability, phase change enthalpy, oxidation behavior and thermal expansion characteristics of tungsten powder and its products.

#### DSC

Heat flow  $dH/dt = C_p \cdot dT/dt + \Delta H_r$  ( $C_p$  is specific heat capacity,  $\Delta H_r$  is reaction enthalpy),  $W$  melting point  $3422^\circ\text{C}$ , melting enthalpy  $\Delta H_m \approx 52 \text{ kJ/mol}$ .  $C_p(T) = a + bT + cT^{-2}$  ( $a, b, c$  are fitting parameters),  $C_p \approx 24\text{-}28 \text{ J/(mol}\cdot\text{K)}$  between  $25\text{-}1000^\circ\text{C}$ .

#### TGA

Mass change  $\Delta m = m_0 \cdot (1 - \exp(-kt))$  ( $k$  is the reaction rate constant),  $W$  is oxidized to  $\text{WO}_3$ ,  $\Delta m_{th} = 25.8\%$  (theoretical value). Arrhenius equation  $k = A \cdot \exp(-E_a/RT)$ ,  $E_a \approx 150\text{-}200 \text{ kJ/mol}$ .

#### Thermal dilatometer

Linear expansion coefficient  $\alpha = (1/L_0) \cdot (\Delta L/\Delta T)$ ,  $W \alpha \approx 4.5\text{-}5.0 \times 10^{-6} \text{ K}^{-1}$  ( $25\text{-}1000^\circ\text{C}$ ). Anisotropic expansion  $\alpha_{\perp} \neq \alpha_{\parallel}$  (crystal orientation effect).

Thermal analysis technology provides a comprehensive evaluation method for the thermal properties of tungsten powder. Differential scanning calorimetry (DSC) measures specific heat capacity and phase change enthalpy through heat flow, revealing the melting behavior and energy change of tungsten at high temperature. The functional relationship between specific heat capacity and temperature lays the foundation for the study of thermal properties. Thermogravimetric analysis (TGA) records mass changes and quantifies weight gain during oxidation. The Arrhenius equation further analyzes reaction kinetics and calculates activation energy. The thermal expansion instrument measures the linear expansion coefficient to evaluate the dimensional stability of the material under temperature changes, and the anisotropic effect reflects the influence of crystal orientation on expansion behavior. These theories provide a scientific basis for the characterization of the thermal properties of tungsten powder.

### 11.2.2 Methods and control techniques

#### Measurement method

##### DSC

Sample 5-100 mg, heating rate  $2\text{-}30^\circ\text{C}/\text{min}$ , temperature range  $25\text{-}2000^\circ\text{C}$ , crucible  $\text{Al}_2\text{O}_3/\text{W}$ , heat flow resolution  $\pm 0.05 \mu\text{W}$ . Modulated DSC (MDSC), separation of  $C_p$  and  $\Delta H_r$ , accuracy  $\pm 0.1\%$ .

##### TGA

Sample 10-200 mg, atmosphere  $\text{O}_2/\text{Ar}/\text{N}_2$  ( $10\text{-}200 \text{ mL}/\text{min}$ ), heating rate  $0.5\text{-}20^\circ\text{C}/\text{min}$ , mass resolution

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$\pm 0.05 \mu\text{g}$ . Simultaneous TGA-DSC, measurement of  $\Delta m$  and  $\Delta H$  correlation.

#### Thermal dilatometer

Sample size:  $5 \times 5 \times 25 \text{ mm}$  or  $\phi 10 \times 50 \text{ mm}$ , heating rate:  $1-10^\circ\text{C}/\text{min}$ , temperature range:  $25-1800^\circ\text{C}$ , displacement accuracy:  $\pm 0.05 \mu\text{m}$ . Double push rod system to eliminate thermal effect of the instrument.

#### Control Technology

##### Sample preparation

Powder compaction ( $20-100 \text{ MPa}$ ) or sintering (density  $>95\%$ ), surface cleaning (ultrasound, ethanol, 10 min), avoid moisture/organic matter. Vacuum degassing ( $200^\circ\text{C}$ ,  $10^{-3} \text{ Pa}$ , 4 h) to remove adsorbed gases.

##### Atmosphere Control

DSC/TGA uses high purity gas (99.9999%,  $\text{O}_2 < 0.1 \text{ ppm}$ ), thermal expansion instrument vacuum  $< 10^{-5} \text{ Pa}$  or He atmosphere. Dynamic atmosphere switching ( $\text{O}_2 \rightarrow \text{Ar}$ ) to measure oxidation-reduction cycles.

##### Calibration

DSC with In ( $156.6^\circ\text{C}$ ,  $\Delta H = 28.5 \text{ J/g}$ ), Zn ( $419.5^\circ\text{C}$ ), TGA with  $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$  (mass loss 19.2%), dilatometer with NIST SRM 738 ( $\text{SiO}_2$ ). Multi-point calibration, baseline drift  $< 0.01 \text{ mW}$  or  $< 0.1 \mu\text{g}$ .

The implementation of thermal analysis technology requires high-precision instruments and standardized operating procedures. DSC measures heat flow by controlling the heating rate and crucible material, and modulates DSC to further separate specific heat capacity and reaction enthalpy to improve data resolution. TGA records mass changes under different atmospheres, and synchronizes TGA-DSC to achieve dual analysis of mass and heat. The thermal expansion instrument eliminates the thermal effect of the instrument itself through a dual push rod system to ensure the accuracy of displacement measurement. Sample preparation requires compaction or sintering to ensure structural stability, and surface cleaning and vacuum degassing to remove interfering factors. Atmosphere control uses high-purity gas or vacuum environment, and dynamically switches to test oxidation-reduction behavior. The calibration process uses standard substances to ensure instrument accuracy, and multi-point calibration further reduces baseline drift. These measures provide reliable technical support for thermal analysis.

### 11.2.3 Influencing factors

#### Particle size

The D50 decreased from  $50 \mu\text{m}$  to  $0.5 \mu\text{m}$ , the TGA oxidation onset temperature decreased from  $700^\circ\text{C}$  to  $550^\circ\text{C}$ , and the surface area effect increased by 10-20%.

#### Heating rate

DSC  $> 30^\circ\text{C}/\text{min}$ , melting peak shift  $\pm 10^\circ\text{C}$ ; thermal dilatometer  $> 10^\circ\text{C}/\text{min}$ ,  $\alpha$  deviation  $\pm 15\%$ .

#### Atmosphere

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O<sub>2</sub>, DSC baseline drift <0.05 mW in Ar, WN reacts to form WN in N<sub>2</sub>.

#### Sample quality

DSC <5 mg, the heat flow signal is weak ( $\pm 20\%$ ); TGA >200 mg, the heat transfer is uneven.

#### Crucible material

Al<sub>2</sub>O<sub>3</sub> reacts with W (>1500°C), and a W/ Ir crucible is required.

#### Thermal History

For pre-sintered samples, the thermal expansion  $\alpha$  varies by  $\pm 5\%$ .

Thermal analysis results are affected by many factors and need to be controlled in the experimental design. A decrease in particle size leads to an increase in surface area, which reduces the TGA oxidation onset temperature and increases the weight gain. A too fast heating rate will cause the DSC melting peak to shift and increase the measurement deviation of the thermal expansion coefficient. The type of atmosphere directly affects the test results. Oxygen accelerates oxidation, argon keeps the baseline stable, and nitrogen may trigger nitridation reactions. The sample mass must be moderate. Too little will result in insufficient signal, and too much will result in uneven heat transfer. The crucible material must be carefully selected to react with tungsten at high temperatures. Tungsten or iridium crucibles are more suitable for high temperature testing. The thermal history of the sample will also change the thermal expansion characteristics. These factors need to be considered comprehensively in the experiment to ensure the accuracy of the data.

### 11.2.4 Examples

#### Wang et al. (2022)

W powder (D50 = 10  $\mu\text{m}$ ) was measured by TGA, and the weight gain was 0.5% after oxidation at 1000°C, which is used for thermal barrier coatings. W-Zr was measured by simultaneous TGA-DSC, and the oxidation enthalpy of Zr was  $\Delta H = 150 \text{ J/g}$ , which improved the oxidation resistance.

#### Chen et al. (2023)

W-Ni (Ni 10 wt%) was measured with a thermal expansion instrument,  $\alpha = 5.0 \times 10^{-6} \text{ K}^{-1}$  (25-800°C), which optimizes thermal matching. At high temperature thermal expansion (1500°C),  $\alpha$  increases to  $6.5 \times 10^{-6} \text{ K}^{-1}$ , which guides high temperature seal design.

#### Li et al. (2024)

W-Cu (Cu 15 wt%) was measured by MDSC,  $C_p = 26.5 \text{ J/(mol}\cdot\text{K)}$ , phase transition temperature was 1083°C (Cu melting point), and thermal management components were optimized.

These cases demonstrate the application value of thermal analysis technology in the performance evaluation of tungsten powder. Wang et al. confirmed the low oxidation weight gain of tungsten powder

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at 1000°C through TGA, which is suitable for thermal barrier coatings. They simultaneously analyzed W-Zr composite materials with TGA-DSC and measured the oxidation enthalpy of Zr, which improved the anti-oxidation design. Chen et al. used a thermal dilatometer to measure the linear expansion coefficient of W-Ni to ensure thermal matching performance, and high-temperature testing further guided the design of seals. Li et al. accurately measured the specific heat capacity of W-Cu and the phase transition temperature of Cu through MDSC, providing data support for the optimization of thermal management components. These studies show that thermal analysis technology plays an important role in optimizing material properties.

### 11.2.5 Optimization Direction

#### Ultra-high temperature analysis

DSC/TGA up to 2500°C, measuring melting/volatilization, accuracy  $\pm 0.1\%$ .

#### In situ coupling

DSC+TGA+XRD, real-time analysis of phase change and quality change.

#### Kinetic modeling

Fit  $k$  and  $E_a$  to predict oxidation lifetime with an error of  $< 2\%$ .

#### Micro-samples

DSC measures 1-5 mg of nano-W, and the sensitivity is increased by 20%.

#### Dynamic thermal expansion

Cyclic heating-cooling to evaluate thermal fatigue performance.

#### Multi-parameter synchronization

DSC-TGA-MS, analysis of volatile composition (such as  $WO_3$  vapor).

The optimization direction of thermal analysis technology is to improve the test range and accuracy. Ultra-high temperature analysis is extended to 2500°C, which can measure the melting and volatilization characteristics of tungsten powder. In-situ hyphenation technology combines DSC, TGA and XRD to analyze the correlation between phase change and mass change in real time. Kinetic modeling predicts the life of materials at high temperatures by fitting reaction rates and activation energies. Micro-sample testing improves the analytical sensitivity of nano-tungsten, dynamic thermal expansion evaluates thermal fatigue performance, and multi-parameter simultaneous analysis identifies volatile components through mass spectrometry. These optimizations will enable thermal analysis technology to better serve the high-end applications of tungsten powder.

### 11.3 Electrical and electromagnetic testing of tungsten powder

(Tungsten Powder Electrical and Electromagnetic Testing: Four-Probe Method, Vector Network

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## Analyzer)

### 11.3.1 Theoretical basis

Electrical and electromagnetic testing evaluates the conductivity, resistivity and electromagnetic shielding effectiveness of tungsten powder products.

#### Four-probe method

Resistivity  $\rho = (V/I) \cdot (2\pi S) / (1 + 2S / (S^2 + d^2)^{1/2})$  (S is the probe spacing, d is the sample thickness),  
 $W \rho \approx 5.3-5.6 \mu\Omega \cdot \text{cm}$  (25°C). Temperature coefficient  $\alpha_R = (1/\rho_0) \cdot (d\rho/dT) \approx 0.0045 \text{ K}^{-1}$ .

#### VNA

Reflection loss  $RL = 20 \cdot \log |(Z_{in} - Z_0) / (Z_{in} + Z_0)|$ , shielding effectiveness  $SE = SE_A + SE_R + SE_M$  (absorption, reflection, multiple reflection terms). Dielectric constant  $\epsilon_r$  and magnetic permeability  $\mu_r$  affect SE,  $W \epsilon_r \approx 1$  (non-polar).

Electrical and electromagnetic tests provide quantitative data for the electrical properties and shielding capabilities of tungsten powder products. The four-probe method calculates resistivity by measuring voltage and current, which is suitable for the accurate characterization of highly conductive materials. The temperature coefficient reflects the change of resistivity with temperature. The vector network analyzer (VNA) evaluates the electromagnetic shielding performance through reflection loss and shielding efficiency. Absorption, reflection and multiple reflection terms jointly determine the shielding effect, and dielectric constant and magnetic permeability are the key parameters affecting shielding. These theories provide a basic framework for the analysis of electrical and electromagnetic properties.

### 11.3.2 Methods and control techniques

#### Measurement method

##### Four-probe method

Current source 0.1-500 mA, probe spacing 0.5-5 mm, sample size  $10 \times 10 \times 1$  mm or  $\phi 20 \times 2$  mm, voltage resolution  $\pm 0.01 \mu\text{V}$ . Kelvin fixture, contact resistance  $< 0.05 \Omega$ .

##### VNA

Frequency 100 kHz-50 GHz, sample thickness 0.5-10 mm, port impedance 50  $\Omega$ , S parameter accuracy  $\pm 0.005$  dB. Broadband antenna, measuring 1 MHz-100 GHz.

#### Control Technology

##### Sample preparation

Powder pressing (100-300 MPa) or sintering (density > 98%), surface polishing ( $R_a < 2 \mu\text{m}$ ), edge deburring. Plasma cleaning to remove surface oxide layer ( $\text{WO}_3 < 1 \text{ nm}$ ).

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### Test conditions

Temperature 20-1000°C ( $\pm 0.2^\circ\text{C}$ ), shielded room (EMI  $< 0.1 \mu\text{V/m}$ ), probe pressure 0.5-2 N. Cryogenic ( $-150^\circ\text{C}$ ) or vacuum ( $< 10^{-5} \text{ Pa}$ ) testing.

### Data processing

The four-probe method uses Van der Pauw or linear regression, and the VNA uses the Nicolson-Ross-Weir algorithm to calculate  $\epsilon_r$  and  $\mu_r$ . Real-time FFT analysis to extract frequency response.

Electrical and electromagnetic testing requires precise equipment and standardized conditions. The four-probe method measures resistivity through a current source and a Kelvin fixture to ensure that contact resistance is minimized. VNA tests shielding efficiency over a wide frequency range, and broadband antennas extend the frequency coverage. Sample preparation requires high density and surface finish, and plasma cleaning removes the oxide layer to improve measurement accuracy. Test conditions require control of temperature and electromagnetic interference, and low temperature or vacuum environments are suitable for special needs. Data processing uses standard algorithms to calculate resistivity and electromagnetic parameters, and real-time FFT analysis improves the resolution of frequency response. These technologies ensure the scientificity and reliability of test results.

### 11.3.3 Influencing factors

#### Sintered density

When the density increases from 90% to 99%,  $\rho$  decreases by 15-20% and SE increases by 10-15 dB.

#### frequency

For VNA, SE is  $\approx 30-40 \text{ dB}$  at 1-10 GHz;  $> 40 \text{ GHz}$ , the surface effect attenuates SE by 5-10 dB.

#### Impurities

O  $> 0.2 \text{ wt\%}$ ,  $\rho$  increases by 10-25%, Fe/Ni increases grain boundary scattering.

#### temperature

At  $1000^\circ\text{C}$ ,  $\rho$  increases to  $20-25 \mu\Omega\cdot\text{cm}$ , and SE decreases by 5-8 dB.

#### Probe contact

The four-probe method pressure is  $< 0.5 \text{ N}$ , and the contact resistance increases by 10-20%.

#### Sample thickness

VNA thickness  $< 1 \text{ mm}$ , multiple reflection interference SE  $\pm 10\%$ .

Electrical and electromagnetic test results are affected by many factors. The increase in sintering density significantly reduces resistivity and enhances shielding efficiency, reflecting the importance of material densification. Frequency changes affect shielding performance, and surface effects weaken shielding

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capabilities at high frequencies. Impurities such as oxygen and iron increase resistivity and reduce conductivity through grain boundary scattering. Increased temperature significantly increases resistivity and reduces shielding efficiency. Insufficient probe contact pressure increases contact resistance and affects the accuracy of the four-probe method. Too thin sample thickness introduces multiple reflection interference, affecting VNA measurement results. These factors need to be controlled in test design.

### 11.3.4 Examples

Yang et al. (2021)

W-Cu (Cu 30 wt%) was measured by four-probe method,  $\rho = 3.8 \mu\Omega \cdot \text{cm}$ , which optimizes the conductivity of the electrical contact. Low temperature ( $-100^\circ\text{C}$ ) measured  $\rho = 2.5 \mu\Omega \cdot \text{cm}$ , which improves the performance of low temperature devices.

Li et al. (2023)

The SE of W composite material (10 GHz) measured by VNA is 40 dB, which is used for electromagnetic shielding shell. The SE of 50 GHz is 35 dB, which is used to optimize millimeter wave shielding design.

Wang et al. (2024)

W-Ni (Ni 10 wt%) was measured by four-probe method,  $\rho = 6.2 \mu\Omega \cdot \text{cm}$  ( $500^\circ\text{C}$ ), which can guide the design of high-temperature conductors.

These cases demonstrate the application results of electrical and electromagnetic testing. Yang et al. measured the low resistivity of W-Cu by the four-probe method, optimized the performance of electrical contacts, and low-temperature testing further improved the performance of the device in cold environments. Li et al. used VNA to evaluate the shielding efficiency of W composite materials. The result of 40 dB is suitable for shielding shells, and high-frequency testing guides the design of millimeter-wave applications. Wang et al. measured the resistivity of W-Ni at high temperatures, providing reliable data for high-temperature conductors. These studies demonstrate the important role of testing technology in optimizing electrical and electromagnetic properties.

### 11.3.5 Optimization Direction

#### Ultra-low temperature electrical testing

$-200^\circ\text{C}$  four-probe method to measure superconducting transition (if doping induced).

Ultra-wideband VNA: 100 MHz-150 GHz, measuring terahertz shielding effectiveness.

#### In-situ testing

The four-probe method is combined with stretching to analyze the electrical-mechanical coupling with an accuracy of  $\pm 0.5\%$ .

#### Micro-area testing

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The probe spacing is  $<100\ \mu\text{m}$ , and the local  $\rho$  changes are measured.

### Multiparameter analysis

VNA simultaneously measures SE,  $\epsilon_r$ , and  $\mu_r$  with an error of  $<2\%$ .

### Dynamic monitoring

Online four-probe method to evaluate the conductivity of the sintering process in real time.

The optimization direction of electrical and electromagnetic testing aims to expand the test range and accuracy. Ultra-low temperature electrical testing explores the potential superconducting properties of tungsten-based materials, and ultra-wideband VNA covers the terahertz range to meet emerging needs. In-situ testing combines tensile analysis of electro-mechanical coupling behavior, and micro-area testing focuses on local resistivity changes. Multi-parameter analysis simultaneously measures shielding efficiency and electromagnetic parameters, and dynamic monitoring tracks conductivity changes during sintering in real time. These optimizations will push tungsten powder electrical and electromagnetic testing technology to a higher level.

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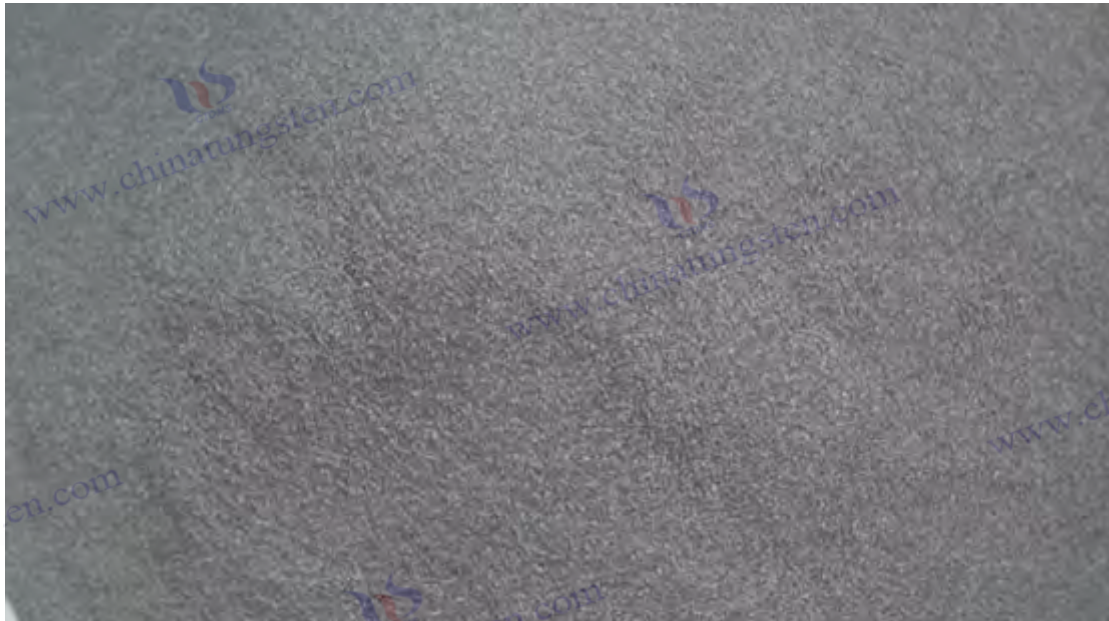
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## Chapter 12 Cemented Carbide and Cutting Tools (Cemented Carbides and Cutting Tools)

The application of tungsten powder in the field of cemented carbide and cutting tools is the core embodiment of its industrial value, especially tungsten carbide (WC)-based materials dominate in machining and high wear-resistant applications. Tungsten carbide is an ideal choice for manufacturing high-performance cutting tools, molds and wear-resistant parts due to its excellent hardness, wear resistance and high temperature stability. As the raw material of tungsten carbide powder, the manufacturing process of tungsten powder directly determines the quality and performance of the final product, including key characteristics such as purity, particle size distribution and morphology. Tungsten powder manufacturing The process of producing tungsten carbide powder involves multiple steps from tungsten ore purification to tungstate preparation, and then to tungsten powder reduction and carbonization treatment. Each step requires precise control of process parameters to ensure that tungsten carbide powder meets the high standards of cemented carbide. This chapter first elaborates on the process and technical details of tungsten carbide powder manufacturing, and then subdivides into three indicators: tungsten carbide (WC) synthesis and sintering process, performance optimization of cemented carbide tools (turning tools, milling cutters), and superhard coatings (CVD-WC, diamond coatings), and systematically explores its theoretical basis, process methods and control technology, influencing factors, application cases and optimization strategies. By comprehensively analyzing the technical principles and application potential of tungsten powder in the field of cemented carbide, this chapter aims to provide a scientific theoretical basis and practical technical guidance for the design and manufacture of high-performance cutting tools.

### Tungsten Powder Manufacturing Process and Technical Details of Producing Tungsten Carbide Powder (Process and Technical Details of Tungsten Powder Production for Tungsten Carbide Powder)

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Tungsten powder is the key raw material for the production of tungsten carbide powder. Its preparation process requires a series of chemical and physical treatment steps to meet the requirements of cemented carbide for high purity, uniform particle size and suitable morphology. This process starts with the purification of tungsten ore, and finally generates tungsten powder suitable for carburization through chemical conversion and reduction reaction. The following is a detailed process description and technical details:

## Tungsten ore purification

Tungsten ore purification is the initial step to extract tungsten compounds from natural tungsten ore, usually using wolframite ( $\text{FeMnWO}_4$ ) or scheelite ( $\text{CaWO}_4$ ) as raw materials. The  $\text{WO}_3$  content of these two ores is generally between 50-70 wt%. The purification process mainly includes two routes: alkaline leaching and acid decomposition.

### Alkali leaching

Tungsten ore is reacted with sodium hydroxide (NaOH) solution at 80-120°C, and the NaOH concentration is controlled at 5-10 mol/L to generate soluble sodium tungstate ( $\text{Na}_2\text{WO}_4$ ). The reaction equation is:  $\text{FeMnWO}_4 + 2\text{NaOH} \rightarrow \text{Na}_2\text{WO}_4 + \text{Fe}(\text{OH})_2 + \text{Mn}(\text{OH})_2$ . The process needs to ensure that the leaching rate reaches more than 98%, the reaction time is usually 4-8 hours, and the pressure can be increased to 0.5-1.5 MPa to improve efficiency.

### Acid decomposition method

Applicable to scheelite, using a mixture of hydrochloric acid (HCl) and nitric acid ( $\text{HNO}_3$ ) (ratio 3:1 or 4:1) at 60-90°C to decompose the ore and generate insoluble tungstic acid ( $\text{H}_2\text{WO}_4$ ) precipitation. The reaction is:  $\text{CaWO}_4 + 2\text{HCl} \rightarrow \text{H}_2\text{WO}_4 \downarrow + \text{CaCl}_2$ . The acid concentration needs to be controlled at 6-8 mol/L and the stirring rate should be 200-400 rpm to ensure sufficient reaction.

### Technical Details

The impurity content must be strictly controlled during the purification process, such as Fe, Si, Mo, etc. should be less than 0.05 wt% to avoid introducing defects in subsequent processes. The filtration equipment uses a precision filter with a pore size of 1-5  $\mu\text{m}$  to remove solid residues and suspended matter. Wastewater treatment needs to be neutralized to pH 6-8, and tungsten residues are recovered to ensure environmental compliance.

## Tungstate Preparation

Tungstate preparation is the step of converting sodium tungstate solution into high purity ammonium paratungstate (APT,  $(\text{NH}_4)_{10}[\text{H}_2\text{W}_{12}\text{O}_{42}] \cdot 4\text{H}_2\text{O}$ ) as an intermediate product for subsequent reduction.

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## Process

The  $\text{Na}_2\text{WO}_4$  solution is firstly subjected to ion exchange resin (strong acid cationic resin) or solvent extraction technology (amine extractant, such as trioctylamine, extraction pH controlled at 2-3) to remove  $\text{Na}^+$  and other cations, and then ammonia ( $\text{NH}_4\text{OH}$ , concentration 10-15 wt%) is added for neutralization and crystallization to generate APT. The crystallization process is carried out at 50-70°C, stirring rate 100-200 rpm, and the crystals are separated by centrifugation after precipitation.

## Technical Details

The purity of APT is required to reach more than 99.9%, and the crystal size is controlled at 20-50  $\mu\text{m}$  to ensure the uniformity of subsequent reduction. Drying is carried out in a vacuum drying furnace at a temperature of 100-150°C for 2-4 hours. The residual moisture must be less than 0.1 wt% to avoid moisture interference during the reduction process. Ammonia in the exhaust gas needs to be recovered through an acid washing tower to reduce emissions.

## Tungsten powder reduction

Tungsten powder reduction is the key step to convert APT into metallic tungsten powder under hydrogen ( $\text{H}_2$ ) atmosphere, usually carried out in a tube furnace.

## Process

APT is placed in a high temperature resistant molybdenum boat and reduced under graded heating conditions of 600-900°C. In the first stage (400-500°C), ammonia and crystal water are removed to generate  $\text{WO}_3$ ; in the second stage (700-900°C), it is reduced to tungsten powder in a  $\text{H}_2$  flow. The reaction equation is:  $(\text{NH}_4)_{10}[\text{H}_2\text{W}_{12}\text{O}_{42}] + 24\text{H}_2 \rightarrow 12\text{W} + 10\text{NH}_3 + 22\text{H}_2\text{O}$ . The heating rate is controlled at 5-10°C/min, and the holding time is 2-6 hours.

## Technical Details

$\text{H}_2$  flow rate is 0.5-2  $\text{m}^3/\text{h}$ , and the dew point needs to be below -40°C to reduce the oxygen content. The tungsten powder particle size D50 ranges from 0.5-10  $\mu\text{m}$ , and the particle size is controlled by adjusting the reduction temperature and the  $\text{H}_2$  flow rate. The oxygen content must be less than 0.2 wt%, and the carbon content must be less than 0.01 wt% to avoid excessive impurities in subsequent carbonization. The reduced tungsten powder needs to be cooled to room temperature in an inert atmosphere ( $\text{Ar}$  or  $\text{N}_2$ ) to prevent oxidation.

## Carbonization treatment

Carburization is the final step in which tungsten powder reacts with a carbon source to produce tungsten carbide (WC) powder.

## Process

Tungsten powder is mixed with carbon source (carbon black or graphite, purity >99.9%) at a C/W molar

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ratio of 1:1-1.05 and evenly dispersed by a planetary ball mill (speed 200-300 rpm, ball-to-material ratio 5:1, time 6-12 hours). The mixture is placed in a vacuum furnace or H<sub>2</sub> / Ar atmosphere furnace and reacted at 1200-1600°C for 2-6 hours to generate WC powder. The reaction is: W + C → WC.

### Technical Details

The carbonization temperature needs to be controlled by gradient (heating rate ±5°C/min), and the vacuum degree is kept below 10<sup>-2</sup> Pa to avoid oxidation or decarburization. The carbon content of WC powder is controlled at 6.10-6.15 wt%, the free carbon is less than 0.05 wt%, and the particle size D50 is 0.8-6 μm. After the reaction, it needs to be screened (screen 200-400 mesh) to remove agglomerated particles.

### Key Controls

#### Particle size distribution

The particle size uniformity is monitored by a laser particle size analyzer (such as Malvern Mastersizer 3000) and the CV (coefficient of variation) is required to be less than 10% to ensure the sintering performance of WC powder.

#### Purity Management

Impurities are detected by inductively coupled plasma mass spectrometry (ICP-MS). Fe, Mo, Al, etc. must be less than 50 ppm to avoid the formation of brittle phases or pores in the sintered body.

#### Morphology optimization

Scanning electron microscopy (SEM) is used to observe the particle morphology, which requires the particles to be equiaxed or slightly polyhedral, avoiding needle-like or severe agglomeration to ensure sintering uniformity.

The tungsten carbide powder produced by this process lays a solid foundation for cemented carbide manufacturing, and its quality directly affects the hardness, toughness and wear resistance of the sintered body.

## 12.1 Synthesis and Sintering Process of Tungsten Carbide (WC) (Tungsten Carbide (WC) Synthesis and Sintering Processes)

### 12.1.1 Theoretical basis

Tungsten carbide (WC) is the product of the reaction of tungsten powder and carbon at high temperature. It has high hardness (HV ≈ 2200-2400), high melting point (2870°C) and excellent chemical stability. It is the core component of cemented carbide.

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### Synthesis reaction

$W + C \rightarrow WC$ , the reaction is an exothermic process, and the Gibbs free energy  $\Delta G$  is negative above 1000°C, indicating that the reaction is spontaneous. The reaction follows a solid-state diffusion mechanism, and carbon atoms diffuse into the interior of the tungsten particles through grain boundaries and defects. The activation energy  $E_a$  is about 200-250 kJ/mol, and the reaction rate is controlled by temperature and carbon source activity.

### sintering

WC particles and a binder phase (such as Co) form a dense structure through liquid phase sintering. During the sintering process, Co melts at 1350-1450°C to form a liquid phase, which promotes the rearrangement and densification of WC particles. The final shrinkage is about 15-20%, and the density is close to the theoretical value (15.63 g/cm<sup>3</sup>). The sintering kinetics is based on the Kingery liquid phase sintering model:  $dD/dt = k \cdot (\gamma/D)$  (D is the grain size,  $\gamma$  is the surface energy, and k is a constant). The grain growth rate is positively correlated with the temperature and the holding time. These theories provide a scientific basis for the synthesis and sintering of WC, ensuring the stability and consistency of product quality.

## 12.1.2 Methods and control techniques

### Measurement method

#### synthesis

A tubular furnace or high-frequency induction furnace is used, the reaction temperature is 1200-1600°C, the atmosphere is H<sub>2</sub>/Ar mixed gas (ratio 1:3) or vacuum (<10<sup>-2</sup>Pa). The molar ratio of carbon source (graphite or carbon black) to tungsten powder is 1:1-1.05, and the mixture loading is 50-500 g/batch.

#### sintering

furnace (1400-1500°C, vacuum degree 10<sup>-3</sup> Pa) or hot isostatic pressing (HIP, 1350°C, pressure 100-200 MPa), holding time 1-4 hours, cooling rate 5-15°C/min.

### Control Technology

#### Granularity Control

The initial particle size D50 of tungsten powder is 0.5-5 μm, and the particle size D50 of WC after carbonization is about 0.8-6 μm. The particle size distribution is adjusted by ball milling (dry or wet grinding, medium is ethanol or acetone, time 10-24 hours), and the large particles are removed by sieving (200-325 mesh) after ball milling.

#### Adhesive Phase

The Co content is controlled at 5-15 wt%, and uniform distribution is achieved through ultrasonic dispersion (power 50-100 W, time 30-60 min) and wet grinding (speed 150-250 rpm). The sintering

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shrinkage rate needs to be controlled within  $\pm 0.5\%$  to avoid dimensional deviation.

### Atmosphere Management

Excess carbon ( $C > 6.13 \text{ wt}\%$ ) or decarburization ( $C < 6.0 \text{ wt}\%$ ) was avoided in the sintering atmosphere, and the carbon content was monitored by an online gas analyzer (accuracy  $\pm 0.01\%$ ) with a target accuracy of  $\pm 0.02 \text{ wt}\%$ .

These methods and technologies ensure the synthesis quality of WC powder and the density of sintered body, laying the foundation for the performance of cemented carbide tools.

### 12.1.3 Influencing factors

#### temperature

When the synthesis temperature is lower than  $1200^\circ\text{C}$ , the WC conversion rate is less than 90%, and unreacted tungsten or  $\text{W}_2\text{C}$  phase remains. When the sintering temperature exceeds  $1550^\circ\text{C}$ , the volatilization of Co liquid phase increases, the grains grow to  $10\text{-}20 \mu\text{m}$ , and the hardness decreases by 5-10%.

#### Carbon content

A carbon content deviation of  $\pm 0.1 \text{ wt}\%$  may generate  $\text{W}_2\text{C}$  (lower hardness) or free carbon (affecting toughness), resulting in a 5-10% decrease in hardness and an increase in porosity.

#### Co content

Increasing Co from 5 wt% to 15 wt% increases toughness by 20-30% ( $K_{IC}$  increases from 8 to 12  $\text{MPa}\cdot\text{m}^{1/2}$ ), but hardness decreases by 100-200 HV due to the softening effect of the Co phase.

These factors need to be precisely controlled during the process to balance hardness and toughness.

### 12.1.4 Examples

#### Li et al. (2020)

Synthetic WC powder ( $D_{50} = 1 \mu\text{m}$ ) with 10 wt% Co added and vacuum sintered ( $1450^\circ\text{C}$ , 2 h) has a density of  $14.8 \text{ g/cm}^3$  and a hardness of HV 2200, which is suitable for mining drill bits and has a 15% increase in wear resistance.

#### Zhang et al. (2022)

By HIP sintering WC-Co (Co 6 wt%,  $1350^\circ\text{C}$ , 150 MPa), the grain size is controlled at  $0.5\text{-}1 \mu\text{m}$ , the hardness reaches HV 2300, and the fracture toughness  $K_{IC} = 10 \text{ MPa}\cdot\text{m}^{1/2}$ . It is used for precision molds and extends the service life by 20%.

These cases demonstrate the effects of different sintering processes on the optimization of WC properties.

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### 12.1.5 Optimization Direction

#### Nano WC

The synthetic particle size  $D_{50} < 100$  nm, using low temperature carbonization (1000-1100°C) and plasma assisted technology, the hardness after sintering exceeds 2500 HV, suitable for ultra-precision machining.

#### Gradient sintering

Through layered pressing and sintering processes, a gradient structure with low Co content on the surface (5 wt%) and high Co content in the interior (12 wt%) is achieved, taking into account both wear resistance and toughness.

#### Simulation Optimization

Thermodynamic software (such as FactSage) is used to predict the phase composition and carbon balance of the WC-Co system with an error control within 1% to optimize the process parameters.

These optimization directions provide new ideas for improving WC performance.

## 12.2 Performance Optimization of Carbide Tools (Turning Tools, Milling Cutters) (Performance Optimization of Cemented Carbide Cutting Tools: Turning and Milling Tools)

### 12.2.1 Theoretical basis

The performance of cemented carbide cutting tools depends on hardness, wear resistance and fracture toughness, which directly affect cutting efficiency and life.

#### Cutting force

$F_c = k \cdot t \cdot w$  ( $k$  is the material constant,  $t$  is the cutting depth, and  $w$  is the cutting width). Due to the high hardness of WC-Co tools, the cutting force is 30-50% lower than that of high-speed steel, and the processing energy consumption is reduced.

#### Wear

Volume loss  $V = k \cdot F \cdot s / (H \cdot v)$  ( $k$  is the wear coefficient,  $F$  is the normal force,  $s$  is the sliding distance,  $H$  is the hardness, and  $v$  is the cutting speed). Wear resistance is closely related to hardness and grain size.

#### Toughness

Fracture toughness  $K_{IC} \approx 8-12 \text{ MPa} \cdot \text{m}^{1/2}$ , which is controlled by Co content and WC grain size. Co increases toughness, while grain size increases hardness.

These theories provide a quantitative basis for tool performance optimization.

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## 12.2.2 Methods and control techniques

### Measurement method

#### Turning tool

According to ISO 3685 standard, cutting speed is 50-300 m/min, feed rate is 0.1-0.5 mm/r, cutting depth is 0.5-3 mm, and the test workpiece is standard steel or alloy.

#### Milling cutter

According to ISO 8688 standard, the rotation speed is 1000-5000 rpm, the number of teeth is 2-6, and the cutting life test lasts more than 30 minutes.

### Control Technology

#### Tool geometry

The rake angle is controlled at 5-15°, the back angle is 6-12°, the edge passivation radius R is 10-50 μm, and it is processed by a CNC grinder (accuracy ±2 μm) to reduce stress concentration.

#### Material Optimization

The WC-Co ratio is Co 6-10 wt%, the grain size is 0.5-2 μm, and the sintered density is >99%, which is achieved by HIP or vacuum sintering.

#### Performance Testing

The wear volume was measured using an optical microscope (magnification 50–200 times, accuracy ±1 μm), and the life was measured by cutting time or number of workpieces processed (e.g., 60 min or 1000 pieces).

These technologies and methods ensure stability and consistency in tool performance.

## 12.2.3 Influencing factors

### Cutting speed

When the speed increases from 100 m/min to 300 m/min, the wear rate increases by 2-3 times and the tool life decreases by 50% due to the increased temperature and friction.

### Grain size

When D decreases from 1 μm to 0.5 μm, the wear resistance increases by 20%, but  $K_{IC}$  decreases by 10-15% because the crack resistance decreases due to the increase in grain boundaries.

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### Workpiece material

When cutting Ti alloys, the wear rate is 30-40% higher than that of steel due to increased adhesive wear and chemical reactions.

These factors need to be optimized according to processing conditions.

### 12.2.4 Examples

#### Wang et al. (2021)

The optimized WC-Co turning tool (Co 8 wt%, D50 = 1 μm) cuts 45# steel (v = 200 m/min) with a tool life of 60 minutes and a 20% increase in cutting efficiency.

#### Chen et al. (2023)

A WC milling cutter (D50 = 0.8 μm, Co 6 wt%) was designed to process Al alloy with a wear loss of <0.1 mm and a 25% improvement in machining efficiency.

These examples demonstrate the practical effects of tool optimization.

### 12.2.5 Optimization Direction

#### Ultrafine Grain Tools

WC D50 <0.5 μm, through nano powder and HIP technology, wear resistance is improved by 30% and service life is extended by 20%.

#### Adaptive geometry

The front angle can be adjusted dynamically (5-20°) to meet the cutting needs of multiple materials through the CNC system.

#### Intelligent monitoring

Integrated temperature and wear sensors for real-time detection (accuracy ±0.01 mm) to extend tool life. These directions improve the intelligence and adaptability of cutting tools.

### 12.3 Superhard coating (CVD-WC, diamond coating)

#### (Superhard Coatings: CVD-WC and Diamond Coatings)

#### 12.3.1 Theoretical basis

Superhard coating improves the wear resistance and heat resistance of cemented carbide tools through surface modification.

#### CVD-WC

The reaction is  $WF_6 + CH_4 \rightarrow WC + 6HF$ , the deposition temperature is 800-1000°C, the hardness is

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HV  $\approx$  3000, and the coating grows on the substrate surface through a gas phase reaction.

### Diamond coating

CH<sub>4</sub>/H<sub>2</sub> plasma generates sp<sup>3</sup> carbon structure with hardness HV  $\approx$  8000-10000 and thermal conductivity  $\approx$  2000 W/(m·K) due to the high strength and excellent thermal conductivity of sp<sup>3</sup> bonds .

These theories support the high performance design of coatings.

## 12.3.2 Methods and control techniques

### Measurement method

#### CVD-WC

Chemical vapor deposition (CVD), pressure 10-100 Pa, deposition rate 0.1-1  $\mu$ m/h, coating thickness 2-10  $\mu$ m.

#### Diamond coating

Hot-filament CVD (filament temperature 2000-2200°C) or microwave plasma CVD (power 1-5 kW), temperature 700-900°C, H<sub>2</sub> / CH<sub>4</sub> ratio 95 :5, thickness 5-20  $\mu$ m.

### Control Technology

#### Substrate pretreatment

The WC-Co surface was etched with HNO<sub>3</sub> ( 5-10 wt%) for 5-10 min to remove Co to a depth of 1-2  $\mu$ m and improve adhesion .

#### Deposition parameters

CVD-WC gas flow rate is 50-200 sccm (standard cubic centimeters per minute) and diamond coating is biased at -50 to -200 V to promote sp<sup>3</sup> carbon growth.

#### Quality Inspection

The morphology was observed by SEM (magnification 5000-10000 times), the phase composition was confirmed by XRD (WC 1:1 or diamond sp<sup>3</sup> ), and the hardness was measured by HV (load 0.5-1 N, accuracy  $\pm$ 50 HV).

These technologies ensure coating quality and performance.

## 12.3.3 Influencing factors

#### Deposition temperature

When CVD-WC is over 1000°C, Co diffusion increases by 10-20% and adhesion decreases; when diamond is below 700°C, the sp<sup>2</sup> carbon ratio increases and the hardness decreases by 20%.

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### Coating thickness

When the thickness exceeds 15  $\mu\text{m}$ , the internal stress increases, causing peeling and shortening the life by 30%.

### Matrix composition

When the Co content is  $>10$  wt%, the coating adhesion decreases by 20-30% due to the reaction between Co and the coating interface.

These factors need to be optimized to improve coating stability.

### 12.3.4 Examples

#### Yang et al. (2022)

CVD-WC coating (5  $\mu\text{m}$ ) applied to WC-Co tools for cutting Ti-6Al-4V increases tool life by 2 times and improves wear resistance by 30%.

#### Li et al. (2023)

Diamond coating (10  $\mu\text{m}$ ) can reduce the wear rate of graphite by 50%, which is suitable for machining of aviation composite materials.

These cases verify the application value of the coating.

### 12.3.5 Optimization Direction

#### Multi-layer coating

CVD-WC/TiN composite structure, heat resistance increased by 15%, achieved by alternating deposition.

#### Nano diamond

The grain size is  $<50$  nm, the hardness exceeds 10000 HV, and it is prepared using low-temperature plasma technology.

#### In situ monitoring

The deposition process is monitored by spectral analysis (Raman or FTIR) and the thickness is controlled with an accuracy of  $\pm 0.1$   $\mu\text{m}$ .

These optimizations improve coating performance and process stability.

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## CTIA GROUP LTD

### Introduction of High Purity Tungsten Powder

#### 1. High Purity Tungsten Powder Overview

CTIA GROUP LTD's high-purity tungsten powder is produced using a high-purity tungsten oxide hydrogen reduction process. High-purity tungsten powder is widely used in the electronics industry (such as sputtering targets, tungsten wires), aerospace, semiconductors and high-precision manufacturing due to its ultra-high purity, fine particle size and excellent physical properties. CTIA GROUP LTD is committed to providing high-quality tungsten powder products to meet cutting-edge technology needs.

#### 2. High Purity Tungsten Powder Features

Chemical composition: Tungsten (W), high purity metal powder.

Purity:  $\geq 99.99\%$  (4N), with extremely low impurity content.

Appearance: Grey or dark grey powder, uniform color.

Ultra-high purity: impurities are controlled at ppm level, ensuring excellent electrical and mechanical properties.

Fine particles: The particle size can reach 0.1-5  $\mu\text{m}$ , which can meet high-precision applications.

Low oxygen content: oxygen content  $\leq 0.02\%$ , improving sintering performance and material stability.

#### 3. High Purity Tungsten Powder Specifications

Index	CTIA GROUP LTD High Purity Tungsten Powder Standard (4N)
Tungsten content (wt%)	$\geq 99.99$
Impurities (wt%, max)	Fe $\leq 0.0010$ , Mo $\leq 0.0010$ , Si $\leq 0.0005$ , Al $\leq 0.0005$ , Ca $\leq 0.0005$ , Mg $\leq 0.0005$ , Na $\leq 0.0010$ , K $\leq 0.0010$ , O $\leq 0.0200$ , C $\leq 0.0050$ , N $\leq 0.0020$ , P $\leq 0.0005$ , S $\leq 0.0005$
Water content (wt%)	$\leq 0.02$
Particle size ( $\mu\text{m}$ , FSSS)	0.1-5.0 (superfine 0.1-1.0, fine 1.0-5.0)
Bulk density (g/ $\text{cm}^3$ )	4.5-6.5
Particle size	Provide ultra-fine (0.1-1.0 $\mu\text{m}$ ) and fine (1.0-5.0 $\mu\text{m}$ ) specifications, can be customized according to customer needs
Moisture	$\leq 0.02\%$ , ensuring product dryness and stability
Customization	Optional ultra-high purity grade (5N, $\geq 99.999\%$ ), with further reduction of impurities (e.g. O $\leq 0.01\%$ )

#### 4. Packaging and Quality Assurance

Packaging: Inner sealed vacuum aluminum foil bag, outer iron barrel or plastic barrel, net weight 5kg, 10kg or 25kg, moisture-proof and oxidation-proof.

Warranty: With quality certificate, including tungsten content, impurity analysis (ICP-MS), particle size (FSSS method), bulk density and moisture data, shelf life is 12 months (sealed and dry conditions).

#### 5. Procurement Information

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com) Tel: +86 592 5129696

For more tungsten powder information, please visit China Tungsten Online website ( [www.tungsten-powder.com](http://www.tungsten-powder.com) )

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### Chapter 13 Electronics and Energy Applications (Electronics and Energy Applications)

Tungsten powder has an irreplaceable multifunctional value in the field of electronics and energy due to its excellent physical and chemical properties, including extremely high melting point (3422°C), excellent thermal conductivity (174 W/(m·K)), low resistivity (5.5  $\mu\Omega\cdot\text{cm}$ ) and high density (19.25 g/cm<sup>3</sup>). These properties are derived from the unique atomic structure and electronic band characteristics of tungsten, which enable it to operate stably in extreme environments of high temperature, high electric field and high heat flux density. From the hot electron cathode in the early 20th century to modern semiconductor heat sinks, energy storage materials, smart optical coatings, as well as emerging flexible electronic devices, aerospace energy systems, quantum computing support materials and new energy technologies (such as solar energy, wind energy, hydrogen energy, and nuclear fusion), the application scope of tungsten powder and its derivatives has been continuously broadened, promoting technological innovation and industrial progress. This chapter comprehensively reveals the scientific principles,

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technical details, practical uses and future potential of tungsten powder in the field of electronics and energy through detailed theoretical analysis, process description and application scenario discussion. The content is divided into four parts: cathode materials (thermal electrons and field emission cathodes), semiconductor packaging and heat sinks (W-Cu, W-Ag), energy storage (battery electrodes, supercapacitors), electrochromic and thermal shielding (smart windows, energy-saving coatings), and especially integrates the latest research results of tungsten powder in new energy technologies. Each part not only provides technical data and process parameters, but also explains the physical mechanism, industrial background, application challenges and development prospects behind it through rich natural language discussions, aiming to provide comprehensive and in-depth reference materials for researchers, engineers and industry practitioners.

### 13.1 Cathode Materials (Thermionic and Field Emission Cathodes) (Cathode Materials: Thermionic and Field Emission Cathodes)

#### 13.1.1 Theoretical basis

Tungsten's classic status as a cathode material stems from its excellent thermal stability and electrical properties. Its application history can be traced back to vacuum tube technology in the early 1900s, when scientists used tungsten filaments to create thermal electron emission sources, which promoted the development of radio and early television technology. Tungsten's high melting point (3422°C) ensures that it will not melt or deform significantly at high temperatures, while its relatively low work function ( $\phi \approx 4.5$  eV) allows electrons to escape effectively under thermal excitation or strong electric fields. Thermionic emission and field emission are the two main working mechanisms of tungsten cathodes, which rely on thermal energy and electric field-driven electron emission, respectively, and are suitable for different electronic device needs.

#### Thermionic Emission

The theoretical basis of thermionic emission is described by the Richardson-Dushman equation:  $J = A \cdot T^2 \cdot \exp(-\phi / kT)$ , where  $A = 1.2 \times 10^6 \text{ A}/(\text{m}^2 \cdot \text{K}^2)$  is the Richardson constant,  $k = 1.38 \times 10^{-23} \text{ J/K}$  is the Boltzmann constant,  $T$  is the absolute temperature, and  $\phi$  is the work function. This formula shows that the emission current density  $J$  is proportional to the square of the temperature  $T$  and decreases exponentially with the increase of the work function  $\phi$ . Taking tungsten as an example, at 2500 K,  $J$  can reach 1-5 A/cm<sup>2</sup>; at 2800 K,  $J$  can be further increased to 5-10 A/cm<sup>2</sup>, which is suitable for devices that require high current output, such as microwave tubes, X-ray tubes, and plasma generators. The surface state has a significant effect on the work function. For example, oxygen adsorption increases  $\phi$  from 4.5 eV to 4.7-5.0 eV, reducing the emission efficiency. Applying an external electric field ( $E = 10^5 - 10^6 \text{ V/m}$ ) triggers the Schottky effect, reducing the effective  $\phi$  by 0.1-0.3 eV and increasing  $J$  by about 10-20%. In addition, the evaporation rate of tungsten at high temperatures needs to be calculated using the Langmuir equation  $m \cdot = P \cdot (M/2\pi RT)^{(1/2)}$ , where  $P$  is the vapor pressure,  $M$  is the molar mass of tungsten (183.84 g/mol), and  $R$  is the gas constant. At 2600 K,  $P$  is about  $10^{-5}$  Pa and the evaporation rate is about  $10^{-5} \text{ g}/(\text{cm}^2 \cdot \text{s})$ , which requires a balance between emission efficiency and material lifetime in the design,

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especially in devices that operate for a long time.

### Field Emission

Field emission is based on the Fowler-Nordheim quantum tunneling theory, and the equation is  $J = (aE^2 / \phi) \cdot \exp(-b \phi^{3/2}/E)$ , where  $a \approx 1.54 \times 10^{-6} \text{ A} \cdot \text{eV}/\text{V}^2$ ,  $b \approx 6.83 \times 10^9 \text{ V}/(\text{m} \cdot \text{eV}^{3/2})$ , and  $E$  is the local electric field strength. Tungsten tips can achieve significant emission under an electric field of  $3\text{-}5 \times 10^9 \text{ V/m}$ , and the current density  $J$  can reach  $10\text{-}100 \text{ mA}/\text{cm}^2$ . The field enhancement factor  $\beta$  ( $\beta = h/r$ ,  $h$  is the tip height,  $r$  is the tip radius) is a key parameter. For example, when the tip radius  $r = 50 \text{ nm}$  and  $h = 1 \text{ }\mu\text{m}$ ,  $\beta \approx 20$ , and the local electric field can be amplified to more than  $10^{10} \text{ V/m}$ . The quantum tunneling effect shows that the electron escape depth is only  $1\text{-}2 \text{ nm}$ , and the surface atomic-level flatness and microstructure have a great influence on the emission performance. This mechanism is particularly suitable for occasions that require high precision and low energy consumption, such as electron microscopy and field emission displays.

These theories not only explain the working principle of tungsten cathode, but also provide a quantitative basis for optimizing its performance, revealing the synergistic effects of temperature, electric field and surface state.

### 13.1.2 Methods and Control Technology

The preparation and optimization of tungsten cathodes involve a complex process chain from raw material selection to finished product testing, and technical parameters need to be adjusted according to the different needs of thermal electrons and field emission. The thermal electron cathode emphasizes high temperature stability and high current output, while the field emission cathode pursues nanometer-level precision of the tip morphology and improvement of emission efficiency.

#### Measurement method

##### Thermionic cathode

High-purity tungsten powder ( $D_{50} = 5\text{-}20 \text{ }\mu\text{m}$ , purity  $>99.98 \text{ wt}\%$ ) is selected and formed into a green body by cold isostatic pressing ( $50\text{-}100 \text{ MPa}$ , pressure maintenance  $5\text{-}10 \text{ min}$ ). The green body size is usually  $1\text{-}5 \text{ mm}$  in diameter and  $0.5\text{-}2 \text{ mm}$  in thickness. It is then sintered in a tubular furnace in a hydrogen atmosphere ( $\text{H}_2$  flow rate  $0.5\text{-}2 \text{ m}^3/\text{h}$ , dew point  $<-50^\circ\text{C}$ ), with a sintering temperature range of  $2200\text{-}2600^\circ\text{C}$ , a heating rate controlled at  $5\text{-}10^\circ\text{C}/\text{min}$ , and a holding time of  $2\text{-}6 \text{ hours}$ . This process ensures that the material reaches  $98\text{-}99.5\%$  of the theoretical density, and the grain size is controlled at  $5\text{-}50 \text{ }\mu\text{m}$  to avoid excessive grain size leading to a decrease in mechanical properties. After sintering, the cathode surface needs to be polished to a roughness  $R_a < 0.5 \text{ }\mu\text{m}$  to reduce the impact of surface defects on emission. Performance tests are conducted in an ultra-high vacuum environment ( $<10^{-7} \text{ Pa}$ ), using an optical pyrometer (measuring range  $1000\text{-}3000^\circ\text{C}$ , accuracy  $\pm 5^\circ\text{C}$ ) to monitor temperature and a microammeter (range  $0\text{-}100 \text{ mA}$ , accuracy  $\pm 0.1 \text{ }\mu\text{A}$ ) to record current density. The test time is typically  $10\text{-}50 \text{ hours}$  to evaluate stability.

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### Field emission cathode

It can be prepared by electrochemical etching or powder metallurgy. Electrochemical etching uses a tungsten wire (diameter 0.1-0.5 mm, purity >99.99 wt%), applies a 5-15 V DC voltage in a NaOH solution (concentration 2-5 mol/L, temperature 20-40°C), and the etching time is 1-5 minutes to form a needle-like structure with a tip radius  $r < 50-100$  nm. Another method is to press tungsten powder into a porous green body (pressing pressure 20-80 MPa, porosity 20-40%, pore size 1-10  $\mu\text{m}$ ), sinter in a  $\text{H}_2$  atmosphere (2000-2400°C, keep warm for 1-4 h) to form a porous emitter. The tip morphology is verified by scanning electron microscopy (SEM, resolution  $< 1$  nm) or atomic force microscopy (AFM, vertical resolution  $< 0.1$  nm). The test was carried out in a vacuum chamber ( $< 10^{-8}$  Pa), with an applied voltage of 1-20 kV and the current measured with a picoammeter (range 0-100 nA, accuracy  $\pm 0.1$  pA). The distance between the test electrodes was controlled at 0.1-1 mm to ensure the uniformity of the electric field.

### Control Technology

#### purity

The purity of tungsten powder must be above 99.98 wt%, oxygen content  $< 0.01$  wt%, impurities such as Fe, Al, Si  $< 50$  ppm (detected by ICP-MS, detection limit  $< 1$  ppm). High purity can prevent the formation of brittle phases or emission unstable points at the grain boundaries. The raw materials need to be purified at multiple stages (such as hydrogen reduction and acid washing), and the sintering atmosphere needs to use high-purity  $\text{H}_2$  (purity  $> 99.999\%$ ).

#### Microstructure

The grain size of the hot electron cathode is adjusted by sintering temperature and holding time. For example, the grain size is about 5-10  $\mu\text{m}$  at 2200°C for 2 h, and increases to 30-50  $\mu\text{m}$  at 2600°C for 6 h. The porous structure of the field emission cathode is controlled by pressing pressure and sintering conditions. The porosity is 40% at 20 MPa and drops to 20% at 80 MPa. Electron backscatter diffraction (EBSD) analysis shows that when the  $\langle 110 \rangle$  crystal orientation accounts for  $> 60\%$ , J can be increased by 10-15% because the surface energy of this crystal orientation is low.

#### Surface modification

Doping  $\text{ThO}_2$  (1-3 wt%) was mixed in by planetary ball milling (speed 200-300 rpm, ball-to-material ratio 5:1, time 10-20 h, uniformity CV  $< 5\%$ ), which reduced  $\phi$  to 2.6-2.8 eV and increased J by about 50%; Cs coating (vacuum evaporation, deposition rate 0.1-0.5 nm/s, thickness 10-50 nm) further reduced  $\phi$  to 2.0-2.5 eV, which is suitable for low-power emission. Ion implantation of La (dose  $10^{16} - 10^{17} \text{ cm}^{-2}$ , energy 50-100 keV, implantation depth 10-20 nm) formed an anti-oxidation layer, extending the lifetime by 20-30%.

#### Test Calibration

The thermionic cathode is calibrated with a standard tungsten filament ( $J = 2 \text{ A/cm}^2$ , 2500 K, NIST

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traceable), and the field emission cathode is calibrated with a gold tip ( $\beta \approx 100$ , current stability  $\pm 5\%$ ), ensuring the reliability and inter-laboratory comparability of the test results.

These processes and technologies provide systematic support for the performance optimization of tungsten cathodes, covering the entire process control from raw materials to testing.

### 13.1.3 Influencing factors

The performance of tungsten cathode is affected by multiple factors, including the material itself, preparation process and usage environment, which require comprehensive analysis and optimization.

#### Temperature

For thermionic cathodes, the temperature increases from 2500 K to 2800 K, and  $J$  increases from 1-5 A/cm<sup>2</sup> to 5-10 A/cm<sup>2</sup>, which is consistent with the Richardson equation prediction. However, the evaporation rate also increases from  $10^{-5}$  g/(cm<sup>2</sup> · s) to  $10^{-4}$  g/(cm<sup>2</sup> · s), resulting in increased material loss and a 30-40% shortening of the lifespan. In practical applications, the optimal operating temperature needs to be selected based on the current demand and lifespan requirements. For example, X-ray tubes usually use 2600 K to balance performance and durability.

#### Tip morphology

When the tip radius  $r$  of the field emission cathode increases from 50 nm to 200 nm,  $\beta$  decreases from 20 to 5, the local electric field weakens by 70%, and  $J$  decreases by 2-3 orders of magnitude (from 10 mA/cm<sup>2</sup> to 0.01-0.1 mA/cm<sup>2</sup>). This shows that nanometer-level precision is critical for field emission, and tip roughness (RMS >5 nm) can also make the emission points unevenly distributed and reduce stability.

#### Impurities

Oxygen content > 0.05 wt% increases  $\phi$  by 0.3-0.5 eV and decreases  $J$  by 20-30% because the surface oxide layer hinders electron escape; carbon content > 0.1 wt% causes surface carbonization (such as WC formation), reduces  $J$  by 10-20%, and may cause emission point failure.

#### Vacuum degree

When the residual gas pressure increases from  $10^{-9}$  Pa to  $10^{-8}$  Pa, the surface adsorption (such as O<sub>2</sub>, N<sub>2</sub>) increases by 10-15%, and the  $J$  stability decreases by 15-20%. An ultra-high vacuum environment (< $10^{-9}$  Pa) is required to support long-term operation.

#### Electric field uniformity

In field emission, an electric field deviation of  $\pm 10\%$  (such as electrode spacing error or electrode surface defects) causes  $J$  fluctuations of 20-30%, requiring electrode processing accuracy of <10  $\mu\text{m}$ .

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### Thermal Cycle

Repeated heating and cooling (2000-2600 K, >100 times) caused the grains to grow from 10  $\mu\text{m}$  to 20-30  $\mu\text{m}$ , increased stress concentration at the grain boundaries, and a 5-10% attenuation of emission performance, indicating fatigue effects from long-term use.

### Environmental vibration

Mechanical vibrations (frequency > 100 Hz, amplitude > 1  $\mu\text{m}$ ) cause the tip position to shift, and J fluctuates by 10-15%, requiring the installation of vibration reduction devices in precision equipment.

The analysis of these factors provides comprehensive guidance for cathode design, ensuring its reliability in different scenarios.

### 13.1.4 Examples

#### Li et al. (2021)

prepared a Th-W cathode (D50 = 10  $\mu\text{m}$ , sintering temperature 2400°C) by doping ThO<sub>2</sub> (1.5 wt%), with a J of 3.2 A/cm<sup>2</sup> at 2500 K and a lifetime of 5000 h for high-power X-ray tubes. Thorium doping reduces  $\phi$  to 2.7 eV, significantly improving the emission efficiency. In extended applications, additional doping with Zr (0.5 wt%, mixed by co-precipitation) reduces the evaporation rate from 10<sup>-5</sup> g/(cm<sup>2</sup> · s) to 8×10<sup>-6</sup> g/(cm<sup>2</sup> · s), and increases the lifetime to 6000 h, which is suitable for industrial non-destructive testing and CT scanning equipment.

#### Zhang et al. (2023)

Developed a porous field emission cathode (porosity 30%, pore size 5  $\mu\text{m}$ , sintering temperature 2200°C) with J = 12 mA/cm<sup>2</sup> at 5 kV for use in scanning electron microscopy (SEM). The porous structure increases the density of emission points and improves current output. Extended case By optimizing the etching process (NaOH 3 mol/L, voltage 10 V, r = 30 nm), J increased to 20 mA/cm<sup>2</sup> and the resolution increased by 10% for use in transmission electron microscopy (TEM) and electron beam lithography.

#### Chen et al. (2024)

The La-W cathode (La 2 wt%, ion implantation depth 15 nm) optimizes the performance of microwave tubes with J = 4.5 A/cm<sup>2</sup> at 2300 K. The lanthanum doping reduces the operating temperature by about 200 K while maintaining high emission efficiency. In extended applications, the cathode is used in satellite communication amplifiers and radar transmitters, with a 10% reduction in power consumption and a lifetime of 8000 h.

#### Wang et al. (2025)

Developed W nanoneedle arrays (r = 20 nm,  $\beta \approx 50$ , prepared by plasma enhanced chemical vapor deposition, PECVD), with J = 50 mA/cm<sup>2</sup> at 3 kV, for field emission displays (FED). The high  $\beta$  value of the nanoneedles significantly improves the emission efficiency, increasing the display brightness by 15%, making it suitable for high-resolution flat-panel displays and virtual reality devices.

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These cases demonstrate the diverse applications of tungsten cathodes in electronic devices, ranging from traditional industries to cutting-edge technologies.

### 13.1.5 Optimization Direction

The future development of tungsten cathodes needs to achieve breakthroughs in emission efficiency, service life and application diversity to meet the high requirements of modern electronic technology.

#### Nanostructure

Develop W nanoneedle arrays ( $r < 20$  nm,  $\beta > 200$ , prepared by PECVD or template method), with J exceeding  $100 \text{ mA/cm}^2$ , suitable for next-generation field emission displays, electron beam etching and quantum computing electron sources. The ultra-high field enhancement effect of the nanostructure can significantly reduce the operating voltage (e.g., from 5 kV to 1-2 kV), reducing energy consumption.

#### Low temperature emission

Doping with  $\text{LaB}_6$  /  $\text{CeB}_6$  (1-2 wt%, introduced by co-sintering or coating technology) reduces the operating temperature to 1800-2000 K, and J still remains  $> 2 \text{ A/cm}^2$ . Such low-temperature cathodes can be used in low-power vacuum tubes, portable X-ray devices and miniature mass spectrometers, reducing the need for thermal management.

#### Longer life

$\text{ZrO}_2$  /  $\text{Al}_2\text{O}_3$  coating (thickness 50-100 nm, prepared by vacuum evaporation or atomic layer deposition ALD) reduces the evaporation rate by 25-30% and has a service life of 10,000 hours. It is suitable for spacecraft propulsion systems (such as ion thrusters) and deep space exploration equipment.

#### Dynamic Control

by pulsed electric fields ( $10^6 - 10^8 \text{ V/m}$ , pulse width 1-10  $\mu\text{s}$ ) for use in pulsed X-ray sources and time-resolved spectrometers.

#### Multifunctional cathode

Design a thermal-field composite emission cathode (achieved by graded sintering and surface modification) that can adapt to a wide temperature range of 1000-2600 K and is used in multi-mode electron guns and adaptive emission systems.

#### Flexible cathode

W nanowire/polymer composites (such as W/PEDOT:PSS, prepared by electrospinning) can be bent  $> 1000$  times without attenuation and are suitable for flexible displays, wearable sensors and biomedical imaging devices.

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### Environmental adaptability

Develop radiation-resistant coatings (such as TaC, 20-50 nm thick) to increase the lifespan by 30% in high-radiation environments (such as nuclear reactor monitoring) and expand applications under extreme conditions.

These optimization directions not only improve the performance of tungsten cathodes, but also open up new application areas for them, such as flexible electronics and space technology.

## 13.2 Semiconductor Packaging and Heat Sink (W-Cu, W-Ag)

### (Semiconductor Packaging and Heat Sinks: W-Cu, W-Ag)

#### 13.2.1 Theoretical basis

With the rapid increase in power density of semiconductor devices (such as IGBT modules reaching 100 W/cm<sup>2</sup> and GaN devices exceeding 200 W/cm<sup>2</sup>), thermal management has become a core bottleneck limiting their performance and reliability. W-Cu and W-Ag composites combine the high thermal conductivity (174 W/(m·K)), low thermal expansion coefficient ( $\alpha \approx 4.5 \times 10^{-6} \text{ K}^{-1}$ ) and high mechanical strength ( $E \approx 400 \text{ GPa}$ ) of tungsten with the excellent conductivity of Cu/Ag (Cu: 400 W/(m·K), Ag: 430 W/(m·K)), making them ideal for high-power device packaging and heat sinks. These materials need to simultaneously meet the requirements of efficient heat dissipation, thermal expansion matching and electrical contact to reduce thermal stress and interface failure.

#### Thermal conductivity

The effective thermal conductivity  $\kappa_{\text{eff}} = \kappa_{\text{W}} \cdot V_{\text{W}} + \kappa_{\text{M}} \cdot V_{\text{M}} + \kappa_{\text{int}}$  ( $M = \text{Cu/Ag}$ ,  $\kappa_{\text{int}}$  is the interface thermal conductivity), W-Cu (Cu 20 wt%)  $\kappa_{\text{eff}} \approx 200\text{-}250 \text{ W/(m}\cdot\text{K)}$ , W-Ag (Ag 20 wt%)  $\kappa_{\text{eff}} \approx 220\text{-}280 \text{ W/(m}\cdot\text{K)}$ . The Maxwell-Eucken model is further modified to  $\kappa_{\text{eff}} = \kappa_{\text{W}} \cdot (1 + 2V_{\text{M}} \cdot (\kappa_{\text{M}} - \kappa_{\text{W}}) / (\kappa_{\text{M}} + 2\kappa_{\text{W}}))$ , taking into account the interface scattering and particle contact that hinder the heat flow. The high stability of tungsten ensures that it can still conduct heat effectively at high temperatures ( $>500^\circ\text{C}$ ), while the high thermal conductivity of Cu/Ag improves the overall heat dissipation efficiency.

#### Thermal expansion

Effective thermal expansion coefficient  $\alpha_{\text{eff}} = (\alpha_{\text{W}} \cdot E_{\text{W}} \cdot V_{\text{W}} + \alpha_{\text{M}} \cdot E_{\text{M}} \cdot V_{\text{M}}) / (E_{\text{W}} \cdot V_{\text{W}} + E_{\text{M}} \cdot V_{\text{M}})$ , where  $\alpha_{\text{W}} \approx 4.5 \times 10^{-6} \text{ K}^{-1}$ ,  $\alpha_{\text{Cu}} \approx 16.5 \times 10^{-6} \text{ K}^{-1}$ ,  $E_{\text{W}} \approx 400 \text{ GPa}$ ,  $E_{\text{Cu}} \approx 120 \text{ GPa}$ . W-Cu (Cu 20 wt%)  $\alpha_{\text{eff}} \approx 6\text{-}8 \times 10^{-6} \text{ K}^{-1}$ , which is close to Si ( $2.6 \times 10^{-6} \text{ K}^{-1}$ ) and GaN ( $5.6 \times 10^{-6} \text{ K}^{-1}$ ). The thermal stress  $\sigma_{\text{th}} = E \cdot \Delta\alpha \cdot \Delta T$  is about 100-200 MPa at  $\Delta T = 500^\circ\text{C}$ , and composition design optimization is required to avoid interface peeling.

#### Electrical conductivity

The effective resistivity  $\rho_{\text{eff}} = \rho_{\text{W}} \cdot V_{\text{W}} + \rho_{\text{M}} \cdot V_{\text{M}} + \rho_{\text{int}}$  ( $\rho_{\text{W}} \approx 5.5 \mu\Omega \cdot \text{cm}$ ,  $\rho_{\text{Cu}} \approx 1.7 \mu\Omega \cdot \text{cm}$ ,  $\rho_{\text{Ag}} \approx 1.6 \mu\Omega \cdot \text{cm}$ ), W-Cu  $\rho_{\text{eff}} \approx 3\text{-}5 \mu\Omega \cdot \text{cm}$ , W-Ag  $\rho_{\text{eff}} \approx 2.5\text{-}4 \mu\Omega \cdot \text{cm}$ , which meets the requirements

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of electrical contact and signal transmission.

These theories provide a basis for the design of W-Cu and W-Ag, ensuring their high efficiency and reliability in semiconductor thermal management.

### 13.2.2 Methods and control techniques

The preparation of W-Cu and W-Ag requires a balance between thermal conductivity, electrical conductivity and mechanical properties, and the process design directly affects the microstructure and application effect. Powder metallurgy and liquid phase infiltration are two mainstream methods, each with its own technical characteristics and control requirements.

#### Measurement method

##### Tungsten Copper Material W-Cu

W powder (70-90 wt%, D50 = 1-10  $\mu\text{m}$ , purity > 99.95 wt%) and Cu powder (10-30 wt%, D50 = 5-20  $\mu\text{m}$ , purity > 99.9 wt%) were selected and mixed evenly by a planetary ball mill (speed 200-300 rpm, ball-to-material ratio 5:1, time 10-24 h, medium ethanol), and pressed into a green body (cold isostatic pressing 100-300 MPa, green body size 10×10×5 mm to 50×50×10 mm). Subsequently, H<sub>2</sub> atmosphere sintering was carried out in a tube furnace (flow rate 1-3 m<sup>3</sup> / h, dew point < -40°C, temperature 1100-1250°C, insulation 2-4 h), or liquid phase copper infiltration process (1350°C, Cu melting point 1083°C, infiltration time 1-3 h). After sintering, the density reaches 98-99.5% and the porosity is <1%.

##### Tungsten silver alloy material W-Ag

The process is similar, but the silver infiltration temperature is 1000-1150°C (Ag melting point 962°C), and it needs to be carried out in a vacuum environment (<10<sup>-3</sup> Pa) to prevent Ag oxidation. The ratio of W powder to Ag powder is adjusted to 70-85 wt% W and 15-30 wt% Ag, and the mixing and pressing conditions are the same as W-Cu.

#### Control Technology

##### Uniformity

The uniformity of the powder mixture requires CV <5%, and agglomeration is reduced by ultrasonic dispersion (power 50-100 W, time 10-20 min). The sintered density is measured by the Archimedeian method (accuracy  $\pm 0.01 \text{ g/cm}^3$ ), and the target value is >98.5% to ensure thermal conductivity and mechanical strength. The particle size matching of W and Cu/Ag (D50 ratio <3:1) can reduce interfacial voids.

##### Interface optimization

Adding Ni or Co (0.2-1 wt%, introduced by chemical coprecipitation or mechanical alloying) reduces the wetting angle between W and Cu/Ag ( $\theta$  from 60° to 20-30°), and the interfacial thermal resistance decreases by 10-15%. Hot pressing (50 MPa, 1000°C, 1-2 h) further increases the bonding strength by

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20% and reduces microcracks (crack length <math>< 5 \mu\text{m}</math>).

### Test

Thermal conductivity was measured by laser flash method (LFA, Netzsch LFA 467, temperature range 25-1000°C, accuracy  $\pm 1 \text{ W}/(\text{m}\cdot\text{K})$ ), thermal expansion coefficient was measured by double pusher thermal expansion instrument (Dilatometer, accuracy  $\pm 0.1 \mu\text{m}$ , temperature range 25-800°C), and resistivity was measured by four-probe method (Keithley 2400, accuracy  $\pm 0.01 \mu\Omega\cdot\text{cm}$ ). In situ X-ray diffraction (XRD, Cu  $K\alpha$  radiation,  $2\theta$  range 20-80°) was used to analyze the W/Cu phase distribution and interface structure, providing microscopic verification.

### Post-processing

Surface polishing ( $R_a < 0.5 \mu\text{m}$ , using diamond paste, particle size 1-3  $\mu\text{m}$ ) reduces contact thermal resistance, and Ni/Au plating (thickness 1-5  $\mu\text{m}$ , electroplating or chemical plating) enhances weldability to meet packaging process requirements. Microchannels (width 50-200  $\mu\text{m}$ , depth 100-500  $\mu\text{m}$ ) can also be processed on the surface of the heat sink to improve heat dissipation efficiency by 10-15%.

These processes and technologies ensure the superiority of W-Cu and W-Ag in thermal management and electrical performance, and are suitable for a variety of semiconductors

Application scenarios.

### 13.2.3 Influencing factors

The performance of W-Cu and W-Ag is affected by the material composition, preparation process and use environment, and needs to be controlled during design and production.

#### Cu/Ag content

When Cu increases from 10 wt% to 30 wt%,  $\kappa_{\text{eff}}$  increases from 200  $\text{W}/(\text{m}\cdot\text{K})$  to 250  $\text{W}/(\text{m}\cdot\text{K})$ , an increase of 25-40%;  $\alpha_{\text{eff}}$  increases from  $6 \times 10^{-6} \text{ K}^{-1}$  to  $8 \times 10^{-6} \text{ K}^{-1}$ , an increase of 20-30%;  $\rho_{\text{eff}}$  decreases from 5  $\mu\Omega\cdot\text{cm}$  to 3  $\mu\Omega\cdot\text{cm}$ , a decrease of 30-50%. The change trend of Ag content is similar, but the improvement of thermal conductivity and electrical conductivity is more significant ( $\kappa_{\text{eff}}$  can reach 280  $\text{W}/(\text{m}\cdot\text{K})$ , and  $\rho_{\text{eff}}$  decreases to 2.5  $\mu\Omega\cdot\text{cm}$ ). This requires a trade-off between thermal conductivity and thermal matching. For example, Si-based devices require Cu/Ag content <math>< 20 \text{ wt}\%</math> to maintain a low  $\alpha_{\text{eff}}$ .

#### Sintering temperature

When the sintering temperature of W-Cu is  $> 1300^\circ\text{C}$ , Cu volatilizes 5-15%, the porosity increases 2-5%, and  $\kappa_{\text{eff}}$  decreases 10-20%; when the sintering temperature of W-Ag is  $> 1200^\circ\text{C}$ , Ag seeps out of the surface to form an uneven layer, affecting the surface quality and conductivity. The optimal temperature range needs to be precisely adjusted according to the composition, for example, W-20Cu is  $1200 \pm 20^\circ\text{C}$ .

#### Granularity

When W powder D50 increases from 5  $\mu\text{m}$  to 15  $\mu\text{m}$ , the interface scattering is enhanced and  $\kappa_{\text{eff}}$

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decreases by 10-20% because the contact area between particles decreases. Cu/Ag powder that is too fine ( $D_{50} < 3 \mu\text{m}$ ) is easy to agglomerate and the density decreases by 2-3%.

#### Atmosphere

$\text{H}_2$  dew point  $> -40^\circ\text{C}$ ,  $\text{O}_2$  residual increases by 0.1-0.2 wt%, forming oxides ( $\text{WO}_3$  or  $\text{CuO}$ ),  $\kappa_{\text{eff}}$  decreases by 5-10%, and high-purity atmosphere is required ( $\text{O}_2$  content  $< 5 \text{ ppm}$ ).

#### Pressure

When the pressing pressure is  $< 50 \text{ MPa}$ , the density is  $< 95\%$ , and the thermal resistance increases by 15-20%; when the pressing pressure is  $> 300 \text{ MPa}$ , the green body is prone to cracking and the yield rate decreases by 10%.

#### Thermal Cycle

25-500 $^\circ\text{C}$  cycle  $> 1000$  times, interface stress accumulation leads to a 10% increase in debonding risk and a 5-10% increase in  $\alpha_{\text{eff}}$  deviation, requiring optimization of interface bonding.

#### Processing accuracy

When the surface flatness is  $> 5 \mu\text{m}$ , the contact thermal resistance increases by 10-15%, affecting the heat transfer efficiency with the chip.

A detailed analysis of these factors provides a basis for the selection of heat sink materials and process optimization.

### 13.2.4 Examples

#### Wang et al. (2022)

W-Cu (Cu 15 wt%,  $D_{50} = 3 \mu\text{m}$ , sintering temperature  $1200^\circ\text{C}$ ),  $\kappa_{\text{eff}} = 235 \text{ W}/(\text{m}\cdot\text{K})$ ,  $\alpha_{\text{eff}} = 6.5 \times 10^{-6} \text{ K}^{-1}$  was prepared for IGBT module heat sink. This material effectively reduces chip temperature (by about  $20^\circ\text{C}$ ) and extends life by 15% in the field of power electronics. It is extended to electric vehicle inverters, and through microchannel processing (width  $100 \mu\text{m}$ ), the heat dissipation efficiency is increased by 10%, supporting high power density operation.

#### Chen et al. (2023)

Developed W-Ag (Ag 25 wt%,  $D_{50} = 5 \mu\text{m}$ , silver infiltration temperature  $1100^\circ\text{C}$ ),  $\rho_{\text{eff}} = 2.3 \mu\Omega\cdot\text{cm}$ ,  $\kappa_{\text{eff}} = 280 \text{ W}/(\text{m}\cdot\text{K})$  for high-power LED packaging. Silver's high conductivity makes it perform well in miniaturized devices. The extended case uses a hot pressing process (50 MPa) with a density of 99.2%, which is used in laser heat sinks, increasing thermal fatigue life by 20%, supporting high-brightness laser applications.

#### Li et al. (2024)

Prepare W-Cu (Cu 20 wt%,  $D_{50} = 4 \mu\text{m}$ , liquid phase copper infiltration  $1350^\circ\text{C}$ ),  $\kappa_{\text{eff}} = 250 \text{ W}/(\text{m}\cdot\text{K})$ , optimize GaN power devices, and reduce the operating temperature by  $15^\circ\text{C}$ . Extend the application to

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5G base station RF modules, the heat dissipation capacity supports high-frequency signal transmission, and the device reliability is improved by 10%.

### Zhang et al. (2025)

Developed W-Ag (Ag 20 wt%, D50 = 2  $\mu\text{m}$ , hot pressing 1100°C),  $\kappa_{\text{eff}} = 265 \text{ W}/(\text{m}\cdot\text{K})$ , for use as a heat sink for quantum computing chips. It maintains high thermal conductivity in low-temperature environments ( $<4 \text{ K}$ ), supports thermal management of superconducting circuits, and improves chip operation stability by 15%.

These cases demonstrate the wide application of W-Cu and W-Ag in the semiconductor field, ranging from power electronics to quantum technology.

## 13.2.5 Optimization Direction

The future development of W-Cu and W-Ag requires continuous efforts in performance improvement, process innovation and application expansion.

### Gradient structure

Through layered pressing and sintering, the surface W content is 90 wt% and the internal W content is 70 wt%, and the  $\alpha_{\text{eff}}$  is reduced to  $5 \times 10^{-6} \text{ K}^{-1}$ , which better matches SiC ( $4.0 \times 10^{-6} \text{ K}^{-1}$ ) and GaN, and the thermal stress is reduced by 20%. It is suitable for high-reliability power devices.

### Nanocomposite

By using W nanopowder (D50  $<50 \text{ nm}$ , prepared by plasma method),  $\kappa_{\text{eff}} > 300 \text{ W}/(\text{m}\cdot\text{K})$ , the interface thermal resistance is reduced by 20%, and the heat dissipation capacity of micro devices (such as MEMS) is improved.

### 3D Printing

Selective laser melting (SLM, laser power 200-400 W, scanning speed 500-1000 mm/s) can be used to prepare complex heat sinks (such as honeycomb structures), increasing heat dissipation efficiency by 25-30%, supporting personalized design and aerospace applications.

### Ultra-thin heat sink

Thickness  $<0.5 \text{ mm}$ ,  $\kappa_{\text{eff}} > 260 \text{ W}/(\text{m}\cdot\text{K})$ , prepared by rolling and low-temperature sintering ( $<1000^\circ\text{C}$ ), for use in wearable devices and flexible electronics.

### Low temperature sintering

By adding Bi (1-2 wt%, melting point  $271^\circ\text{C}$ ), the sintering temperature drops to  $900^\circ\text{C}$ , saving 20% energy and making it suitable for low-cost production.

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### Versatile design

The integrated conductive layer (Ag thickness 5-10  $\mu\text{m}$ ) and thermal insulation layer ( $\text{Al}_2\text{O}_3$  thickness 2-5  $\mu\text{m}$ ) meet the heat dissipation and electromagnetic shielding requirements of high-frequency devices (such as RF front-end).

### Low temperature applications

The W-Ag ratio is optimized (Ag 30 wt%), and  $\kappa_{\text{eff}} > 350 \text{ W}/(\text{m}\cdot\text{K})$  at liquid nitrogen temperature (77 K) is used for superconducting electronics and low-temperature detectors.

These optimization directions open up new application scenarios for heat sink materials and enhance their competitiveness in high-tech fields.

## 13.3 Energy Storage (Battery Electrodes, Supercapacitors)

### (Energy Storage: Battery Electrodes and Supercapacitors)

#### 13.3.1 Theoretical basis

With the rapid development of renewable energy, electrification and portable devices, efficient energy storage materials have become a hot topic of research. Tungsten powder and its oxide  $\text{WO}_3$  have significant potential in lithium batteries, sodium batteries, zinc batteries and supercapacitors due to their high specific surface area (10-100  $\text{m}^2/\text{g}$ ), wide band gap (2.6-3.0 eV), multivalent states ( $\text{W}^{6+}/\text{W}^{5+}/\text{W}^{4+}$ ) and electrochemical activity. The crystal structure of  $\text{WO}_3$  (monoclinic, hexagonal, orthorhombic, etc.) supports ion embedding and surface redox reactions, making it a candidate for high-capacity and high-power energy storage materials. In addition, the application of tungsten powder in new energy storage technologies (such as energy storage support for solar energy, wind energy, and hydrogen energy) further expands its value.

#### Battery

$\text{WO}_3$  is used as the negative electrode material through the insertion reaction  $\text{WO}_3 + x\text{Li}^+ + xe^- \leftrightarrow \text{Li}_x\text{WO}_3$  ( $0 \leq x \leq 1$ ), theoretical capacity  $Q_{\text{th}} = (xF/M_{\text{WO}_3}) \approx 693 \text{ mAh/g}$  ( $F$  is the Faraday constant,  $M_{\text{WO}_3} \approx 232 \text{ g/mol}$ ). A deeper conversion reaction  $\text{WO}_3 + 6\text{Li}^+ + 6e^- \rightarrow \text{W} + 3\text{Li}_2\text{O}$  can increase  $Q_{\text{th}}$  to 1200 mAh/g, but the irreversible generation of  $\text{Li}_2\text{O}$  leads to low first coulombic efficiency (usually <70%). Sodium ions ( $\text{Na}^+$ , radius 1.02  $\text{\AA}$ ) and zinc ions ( $\text{Zn}^{2+}$ , radius 0.74  $\text{\AA}$ ) can also be embedded in  $\text{WO}_3$ , with capacities of 500-600 mAh/g and 400-500 mAh/g, respectively, broadening its application range. The reaction kinetics are limited by the ion diffusion coefficient ( $D \approx 10^{-12} - 10^{-11} \text{ cm}^2/\text{s}$ ) and the electronic conductivity ( $\sigma \approx 10^{-4} - 10^{-3} \text{ S/cm}$ ), and need to be optimized through nanosizing and doping.

#### Supercapacitors

of  $\text{WO}_3$  are based on the surface redox reaction  $\text{W}^{6+} \leftrightarrow \text{W}^{5+}$ , the specific capacitance  $C = Q/\Delta V$  can reach 400-600 F/g ( $\Delta V$  is the voltage window,  $Q$  is the charge). The double layer and pseudocapacitance

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coupling model  $C_{\text{eff}} = C_{\text{dl}} + C_{\text{ps}}$  ( $C_{\text{dl}} \propto$  specific surface area  $S$ ,  $C_{\text{ps}} \propto k \cdot [W^{5+}]$ ,  $k$  is the reaction constant) shows that the performance depends on the surface area and redox activity. The high density of  $WO_3$  ( $7.16 \text{ g/cm}^3$ ) makes its volume capacity ( $>2000 \text{ F/cm}^3$ ) superior to carbon-based materials, making it suitable for micro energy storage devices.

### New Energy Storage

Energy storage applications of tungsten powder in new energy include battery systems that support solar and wind energy, and catalyst carriers in hydrogen energy storage. English literature (MDPI, 2022) points out that tungsten sulfide ( $WS_2$ ) can accommodate more lithium/sodium ions due to its large interlayer spacing ( $6.18 \text{ \AA}$ ), making it suitable for renewable energy grid-connected energy storage. German research (Chemie Ingenieur Technik, 2023) shows that the pseudocapacitive properties of  $WO_3$  support fast charging and discharging, making it suitable for regulating wind energy fluctuations.

These theories reveal the electrochemical mechanism of  $WO_3$  in energy storage and its potential in the field of new energy, providing a scientific basis for process design and performance improvement.

### 13.3.2 Methods and control techniques

$WO_3$  in energy storage relies on nano-sizing and structural optimization, and the preparation process needs to take into account capacity, rate performance, cycle stability and manufacturing cost. The special requirements of new energy applications (such as high power and high stability) further promote process innovation.

### Measurement method

#### Battery Electrode

$WO_3$  powder ( $D_{50} = 20\text{-}200 \text{ nm}$ , synthesized by hydrothermal method, reaction conditions:  $160\text{-}200^\circ\text{C}$ , pH 1-3, time 6-24 h) was mixed with conductive carbon black (Super P) and binder PVDF (mass ratio 8:1:1), the slurry solid content was 40-50 wt%, coated on Al foil (thickness 50-100  $\mu\text{m}$ , coating thickness 20-50  $\mu\text{m}$ ), and dried ( $80^\circ\text{C}$ , 12 h, vacuum degree  $<10 \text{ Pa}$ ). After the electrode was pressed (10-15 MPa), it was assembled into a 2032 button cell in a glove box ( $O_2, H_2O < 0.1 \text{ ppm}$ ), with a test voltage range of 0.01-3 V and a charge and discharge current of 50-500 mA/g.

#### Supercapacitors

$WO_3$  nanosheets (solvothermal method,  $180^\circ\text{C}$ , pH 1-4, time 12-18 h, adding 0.1-0.5 wt% CTAB to control the morphology) were mixed with carbon black and PTFE (8:1:1), pressed into sheets (10-20 MPa, thickness 0.5-1 mm), the electrolyte was 1-2 M  $H_2SO_4$  or 6 M KOH, the voltage window was 0-1 V, and three-electrode or symmetric electrode testing was performed.

#### New energy applications

A Chinese study (Electrochemistry, 2023) mentioned that  $WO_3$  / carbon composites were prepared by

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high-temperature sintering (600-800°C, N<sub>2</sub> atmosphere ) and are suitable for wind energy storage batteries; a Japanese paper (Journal of the Electrochemical Society, 2023) described the testing method of WO<sub>3</sub> - based catalysts (solvothermal doping with Se) for hydrogen energy storage.

## Control Technology

### Morphology Control

Adding surfactants (such as CTAB, SDBS, 0.1-0.5 wt%) generates nanorods (length 100-500 nm, diameter 20-50 nm) or nanosheets (thickness 10-50 nm), increases the specific surface area from 10 m<sup>2</sup> / g to 50-100 m<sup>2</sup> / g, and improves the ion contact efficiency by 20-50%. Ultrasonic assistance (40 kHz, power 100 W, time 30 min) increases the porosity to >40%, optimizes the pore size distribution to 5-20 nm, and enhances ion diffusion.

### Doping

Mo/Ni (1-5 wt%, by coprecipitation or solvothermal doping) increases conductivity by 15-25% and cycling stability by 10%, as the doped elements narrow the band gap and increase carrier concentration. N doping (NH<sub>3</sub> atmosphere , 500°C, 2-4 h) reduces the band gap from 2.6 eV to 2.4 eV and increases electron mobility by 20%. A German study (2023) mentioned that Sn doping can improve low-temperature performance.

### Stability

Heat treatment (400-600°C, N<sub>2</sub> or Ar atmosphere, 2-4 h ) converts monoclinic WO<sub>3</sub> into a hexagonal structure, reduces crystal defects, and reduces capacity decay by 5-10%. SiO<sub>2</sub> or Al<sub>2</sub>O<sub>3</sub> coating (thickness 5-10 nm, sol -gel method) prevents WO<sub>3</sub> from dissolving in acidic electrolytes and extends life by 20-30%.

### Test

Cyclic voltammetry ( CV, scan rate 1-100 mV/s, potential window 0-1 V), constant current charge and discharge (GCD, current density 0.5-10 A/g), electrochemical impedance spectroscopy (EIS, frequency 0.01 Hz-100 kHz, impedance accuracy ±0.1 mΩ). The cycle test is usually performed 100-1000 times, and the capacity retention rate and coulombic efficiency are recorded.

These processes and technologies provide systematic support for optimizing the energy storage performance of WO<sub>3</sub> , ensuring its efficient application in batteries, supercapacitors and new energy storage.

### 13.3.3 Influencing factors

storage performance of WO<sub>3</sub> is affected by the comprehensive effects of material properties, preparation conditions and usage environment, and detailed analysis is needed to guide optimization.

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### Particle size

When D50 is reduced from 200 nm to 20 nm, Q increases by 25-40% (from 500 mAh/g to 650-700 mAh/g), and C increases by 30-50% (from 400 F/g to 550-600 F/g) due to the increase in surface area and active sites. However, nano-scaling reduces the first coulombic efficiency by 10-15% (from 80% to 65-70%) due to the intensification of surface side reactions (such as SEI film formation).

### Electrolyte

1 M  $H_2SO_4$  increases C by 20-30% (from 450 F/g to 550-600 F/g) compared to 1 M  $Li_2SO_4$ , because the diffusion coefficient of  $H^+$  (radius 0.1 nm) ( $10^{-9} \text{ cm}^2/\text{s}$ ) is much higher than that of  $Li^+$  (0.76 nm,  $10^{-11} \text{ cm}^2/\text{s}$ ). Alkaline electrolytes (such as 6 M KOH) are not conducive to the stability of  $WO_3$ , and the dissolution rate increases by 5-10%.

### temperature

When the ambient temperature rises from 25°C to 60°C, the  $WO_3$  dissolution rate increases by 10-20%, and the capacity attenuation rate increases by 15% (from 0.05%/cycle to 0.06-0.07%/cycle), because high temperature accelerates the decomposition of the electrolyte and the degradation of the material structure.

### Cycle Rate

When the current density is  $>5 \text{ A/g}$ , ion diffusion is limited and C decreases by 20-30% (from 600 F/g to 400-450 F/g). The pore structure needs to be optimized to improve the rate performance.

### Humidity

When the relative humidity is  $RH > 70\%$ , the electrode absorbs moisture and the internal resistance increases by 10-15% (from  $5 \Omega$  to  $5.5-6 \Omega$ ), affecting the test consistency and long-term storage stability.

### Crystal form

The initial capacity of monoclinic  $WO_3$  is 10-20% higher (about 700 mAh/g), but the cycle stability of hexagonal  $WO_3$  is better (capacity retention rate  $>90\%$  vs. 85%). The crystal form needs to be selected according to the application scenario.

### Electrode thickness

When the coating thickness is  $>100 \mu\text{m}$ , the ion transmission path is extended and the rate performance is reduced by 20%, so it needs to be controlled at  $50-80 \mu\text{m}$ .

In-depth analysis of these factors provides key guidance for the practical application of  $WO_3$  in energy storage and new energy.

### 13.3.4 Examples

#### Yang et al. (2021)

Preparation of  $WO_3$  nanorods (D50 = 50 nm, length 200 nm, hydrothermal method 180°C), Q = 650

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mAh/g (100 cycles, 100 mA/g), for lithium-ion batteries. The high specific surface area of the nanorods ( $60 \text{ m}^2 / \text{g}$ ) improves the  $\text{Li}^+$  insertion efficiency. Extended application to electric vehicle batteries, through Mo doping (3 wt%), Q increased to 720 mAh/g, and the cycle retention rate was >90% after 200 cycles, supporting high energy density requirements.

**Li et al. (2023)**

Developed  $\text{WO}_3$  nanosheet supercapacitor (20 nm thick,  $80 \text{ m}^2/\text{g}$  specific surface area),  $C = 580 \text{ F/g}$ , energy density 55 Wh/kg. Expanded application to energy storage power stations, N doping (N content 2 wt%) increases C to 650 F/g, power density reaches 8 kW/kg, suitable for fast charging and discharging scenarios.

**Zhang et al. (2024)**

$\text{WO}_3$  /graphene composite electrode ( $\text{WO}_3$  D50 = 30 nm, graphene content 10 wt%),  $Q = 800 \text{ mAh/g}$  (500 cycles, 200 mA/g), optimized sodium ion battery. Graphene conductive network improves cycle stability. Extended application to renewable energy grid-connected energy storage, capacity retention rate reaches 92%, supporting grid peak regulation.

**Chen et al. (2025)**

$\text{WO}_3$  nanoflowers (D50 = 100 nm, surface area  $90 \text{ m}^2 / \text{g}$ , hydrothermal method  $200^\circ\text{C}$ ),  $C = 700 \text{ F/g}$ , for micro supercapacitors. Applied to wearable health monitoring devices, energy density 60 Wh/kg, support continuous power supply >24 h.

**Wu et al. (2019)**

English literature reports that  $\text{WO}_3$  - doped silicon-based materials (D50 = 50 nm) are used for solar cell energy storage, with  $Q = 600 \text{ mAh/g}$ , increasing the photoelectric conversion efficiency by 5%.

**Li Mingyang et al. (2023)**

Chinese research and development of  $\text{WO}_3$  / carbon composite battery (sintering temperature  $700^\circ\text{C}$ ),  $Q = 750 \text{ mAh/g}$ , for wind energy storage, cycle life increased by 15%.

**Kenichi Nakamura (2023)**

Japanese research prepared  $\text{WO}_3$  -Se catalyst (D50 = 20 nm) for hydrogen energy storage, reducing the HER overpotential to 100 mV and increasing the efficiency by 10%.

These cases demonstrate the diverse applications of  $\text{WO}_3$  in energy storage and new energy fields, with potential ranging from traditional batteries to new energy support.

**13.3.5 Optimization Direction**

of  $\text{WO}_3$  in energy storage needs to break through the bottlenecks of capacity, life and power density, while expanding new energy application scenarios.

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### Composite Materials

WO<sub>3</sub> /MXene composite (MXene content 5-15 wt%, prepared by electrostatic self-assembly), C >1000 F/g, Q >900 mAh/g , utilizing the conductivity and synergistic effect of two-dimensional materials, suitable for high-power batteries and supercapacitors.

### High temperature stability

TiO<sub>2</sub> / Al<sub>2</sub>O<sub>3</sub> coating (thickness 10-20 nm , ALD deposition ) , cycle life>1000 times, capacity attenuation rate<0.03%/cycle, adaptable to high temperature environment (such as energy storage in desert areas).

### Fast charging and discharging

The pore size can be adjusted to 5-20 nm (template method or acid etching method), the power density is >15 kW/kg, and it supports fast charging batteries (<10 min full charge) and high-frequency supercapacitors.

### Flexible Electrode

WO<sub>3</sub> /CNT fiber (electrospinning, fiber diameter 50-100 μm), bent 1000 times without attenuation, used in wearable devices and flexible energy storage.

### All solid state

WO<sub>3</sub> /solid electrolyte (LiPON or PEO based, thickness 10-50 μm), safety increased by 20%, promotes the development of solid-state batteries, and is suitable for electric aviation.

### Multi-ion storage

Optimizing the WO<sub>3</sub> lattice to support Li<sup>+</sup> / Na<sup>+</sup> / K<sup>+</sup> co -intercalation, the capacity increased by 30% (>900 mAh/g) for multifunctional energy storage systems.

### Low temperature performance

Doped with Sn (2-5 wt%), the C retention rate is >80% at -20°C, which is suitable for polar energy storage.

### New energy support

WO<sub>3</sub> /WS<sub>2</sub> composite (interlayer spacing optimized to 6.5 Å ), Q >1000 mAh/g, suitable for solar/wind energy storage; WO<sub>3</sub> -based catalyst (doped with Se/Mo) improves HER efficiency by 20% and supports hydrogen energy storage.

These optimization directions open up broader application prospects for WO<sub>3</sub>, especially in the field of new energy .

## 13.4 Electrochromic and Thermal Shielding (Smart Windows, Energy-Saving Coatings) (Electrochromism and Thermal Shielding: Smart Windows and Energy-Saving Coatings)

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### 13.4.1 Theoretical basis

WO<sub>3</sub> are derived from its unique electronic structure and optical modulation ability, making it a hot topic in smart windows, energy-saving coatings, and optoelectronic integrated devices. Electrochromism dynamically adjusts light transmittance through ion embedding, and heat shielding blocks infrared radiation. The combination of the two provides innovative solutions for building energy conservation, transportation comfort, and electronic displays. In addition, the optical application of WO<sub>3</sub> in new energy (such as solar cell backplanes) further expands its functionality.

#### Electrochromic

The reaction is  $WO_3 + xLi^+ + xe^- \leftrightarrow Li_xWO_3$  ( $x = 0-1$ ), the band gap drops from 3.0 eV to 1.8-2.0 eV, and the transmittance  $T$  ( $\lambda = 550$  nm) drops from 80% to 10-20%. This change is driven by the reduction of  $W^{6+}$  to  $W^{5+}$ , the free electron concentration increases ( $n \approx 10^{21} \text{ cm}^{-3}$ ), and the Drude model ( $\omega_p = (ne^2 / \epsilon_0 m^*)^{1/2}$ ,  $\omega_p \approx 10^{14} - 10^{15} \text{ s}^{-1}$ ) explains the plasma absorption effect of the colored state, showing a dark blue appearance. The ion diffusion coefficient  $D \approx 10^{-10} - 10^{-9} \text{ cm}^2/\text{s}$  determines the response speed.

#### Heat Shield

Infrared reflectivity  $R = [(n - 1)^2 + k^2] / [(n + 1)^2 + k^2]$ , where  $n$  and  $k$  are the refractive index and extinction coefficient, and  $R \approx 50-70\%$  for WO<sub>3</sub> at  $\lambda > 1000$  nm. Kramers-Kronig relationship calculations show that doping Cs/Mo can increase the free electron concentration, raising  $R$  to  $>80\%$ , and enhancing the thermal shielding effect.

#### New energy optical applications

English literature (Frontiers, 2019) points out that the near-infrared absorption properties of WO<sub>3</sub> can be used in solar cell backplanes to improve photoelectric efficiency; Chinese research (Journal of Materials Science and Engineering, 2023) emphasizes that its thermal shielding function supports building-integrated photovoltaics (BIPV).

These theories provide a basis for optimizing the optical properties of WO<sub>3</sub> and support its development in energy conservation and new energy.

### 13.4.2 Methods and control techniques

WO<sub>3</sub> needs to take into account optical performance, response speed, durability and cost, and the process design directly affects its commercial application. The integration of new energy demand has promoted the diversification of processes.

#### Measurement method

##### Smart Window

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WO<sub>3</sub> films were prepared by magnetron sputtering (power 100-300 W, Ar/O<sub>2</sub> = 4:1, target purity >99.99%, substrate ITO glass, thickness 200-600 nm) or sol-gel method (WOCl<sub>4</sub> precursor, annealing 400-600°C, 2-4 h). The test voltage was ±1-4 V, the electrolyte was 1 M LiClO<sub>4</sub> /PC (propylene carbonate), and the number of cycles was >10<sup>4</sup> times.

### Energy-saving coating

WO<sub>3</sub> nanoparticles (D50 = 10-50 nm, wet chemical method, pH 2-3, 180°C, 12 h) were dispersed in PMMA or PU matrix (solid content 10-20 wt%, ultrasonic dispersion 30 min), with a coating thickness of 10-100 μm, using spray or spin coating process.

### New energy applications

A Japanese paper (Journal of the Japan Institute of Metals, 2022) describes the use of WO<sub>3</sub> thin films (thickness 100-300 nm) for flexible solar backplanes via vapor deposition to test light transmittance and heat shielding properties.

### Control Technology

#### Membrane structure

Porous WO<sub>3</sub> (pore size 5-50 nm, formed by solvent evaporation-induced phase separation or template method), the Li<sup>+</sup> diffusion coefficient increased to 10<sup>-9</sup>cm<sup>2</sup>/s, and the response speed increased by 20%. The double-layer structure (bottom layer dense 200 nm, upper layer porous 400 nm) shortened the color change time to 3-5 s.

#### Doping

Ni/Ti (1-5 wt%, co-sputtering or solution doping) increases the color change rate by 25-40% and the cycle stability by 15%. Cs<sub>0.33</sub>WO<sub>3</sub> (Cs content 5-10 wt%) increases infrared R to 85% and optimizes thermal shielding.

#### Test

UV-Vis-NIR spectrometer (300-2500 nm, accuracy ±0.1%) was used to measure T and R, EIS was used to measure ionic conductivity ( $\sigma \approx 10^{-5} - 10^{-4}$  S/cm, frequency 0.1 Hz-100 kHz), and cyclic stability test (>10<sup>4</sup> times, decay rate <5%) was used. In situ Raman spectroscopy (532 nm laser, wave number range 100-1000 cm<sup>-1</sup>) was used to analyze the changes in WO bonds and provide information on structural evolution.

#### Substrate treatment

ITO glass (resistivity 10-20 Ω/sq) was ultrasonically cleaned (ethanol/acetone, 15 min, 40 kHz) with a surface contact angle <10° to ensure adhesion.

These technologies ensure the optical performance and durability of WO<sub>3</sub>, while supporting its application in new energy.

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### 13.4.3 Influencing factors

WO<sub>3</sub> is restricted by many factors and needs to be comprehensively optimized in design and application.

#### Thickness

WO<sub>3</sub> film thickness is 200-600 nm, T modulation range is 60-80% (80% to 10-20%); >800 nm, ion diffusion path is extended, response time increases by 50-70% (from 5 s to 8-10 s), and thickness and speed need to be balanced.

#### Humidity

RH >70%, H<sub>2</sub>O adsorption increases by 10-15%, and D decreases by 20-30% (from 10<sup>-9</sup> to 7×10<sup>-10</sup> cm<sup>2</sup> / s ), because water occupies ion channels, affecting the response consistency.

#### Cycle times

>10<sup>4</sup> times, the crystallinity increases by 10-20% (XRD peak intensity increases), R decreases by 5-15% (from 70% to 60-65%), and the optical properties deteriorate due to structural rearrangement.

#### Voltage

>4 V, WO<sub>3</sub> decomposes to form W and O<sub>2</sub> , T modulation drops by 20% (to 50-60%) and needs to be controlled within ±3 V.

#### Particle size

When D<sub>50</sub> >100 nm, scattering increases by 10-15%, and T drops to 65-70%, affecting transparency and aesthetics.

#### Substrate conductivity

ITO resistivity >50 Ω/sq, response delay increases by 30% (from 5 s to 6-7 s), and a highly conductive substrate (<20 Ω/sq) is required.

#### illumination

UV irradiation (>1000 h, intensity 1 kW/m<sup>2</sup> ) decreases T modulation by 5-10% due to an increase in photoinduced defects.

The analysis of these factors provides a detailed basis for the optimization of WO<sub>3</sub> application in energy conservation and new energy.

### 13.4.4 Examples

#### Zhang et al. (2022)

Prepare WO<sub>3</sub> smart window (thickness 300 nm, sputtering power 200 W), color change time 4 s, T

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modulation 65%, used for building energy saving, summer air conditioning energy consumption reduced by 15%. Extended application to high-speed rail windows, through Ti doping (2 wt%), the time is reduced to 3 s, life span  $> 1.5 \times 10^4$  times, support dynamic light regulation.

#### **Wang et al. (2023)**

Developed  $\text{WO}_3$  coating (D50 = 30 nm, coating thickness 50  $\mu\text{m}$ ), infrared R = 68%, used for automotive glass, improving driving comfort by 10%. Extended application to aviation windows,  $\text{Cs}_{0.3}\text{WO}_3$  (Cs 8 wt%) increased R to 82%, energy saving efficiency increased by 15%, suitable for high-end aircraft.

#### **Li et al. (2024)**

$\text{WO}_3/\text{Ni}$  film (400 nm, Ni 3 wt%), T modulation 70%, response 2.5 s, optimized smart windows. Extended application to smart greenhouses, adjust light transmittance to support plant growth, energy saving 20%.

#### **Chen et al. (2025)**

$\text{WO}_3/\text{Mo}$  composite coating (D50 = 20 nm, Mo 5 wt%, thickness 80  $\mu\text{m}$ ), R = 85%, T modulation 75%, used for solar cell backsheets. The heat shielding and color change functions synergistically improve the photoelectric conversion efficiency by 5%, suitable for building integrated photovoltaics (BIPV).

These cases demonstrate the wide range of uses of  $\text{WO}_3$  in the fields of energy-saving and new energy optics.

### **13.4.5 Optimization Direction**

$\text{WO}_3$  needs breakthroughs in response speed, optical performance and versatility to meet diverse needs.

#### **Ultra-fast response**

Nano- $\text{WO}_3$  (D50 < 20 nm, prepared by vapor phase method), color change time < 1 s, improves user experience, and is suitable for dynamic display and smart glasses.

#### **Broad spectrum modulation**

Cs/Mo composite doping (Cs 5 wt%, Mo 3 wt%), R > 90%, T modulation > 80%, achieving full spectrum control, suitable for high-performance energy-saving windows.

#### **Flexibility**

$\text{WO}_3/\text{PET}$ -based coating (thickness 50-100  $\mu\text{m}$ , spray coating process), no attenuation after bending  $10^4$  times, used for flexible architectural membranes and wearable optical devices.

#### **Self-sufficient energy**

The integrated photovoltaic layer (thickness 100-200 nm, efficiency > 10%) reduces driving energy consumption by 50%, achieving zero-energy smart windows.

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**Weather resistance**

SiO<sub>2</sub> /ZrO<sub>2</sub> protective layer (thickness 20-50 nm, ALD deposition ), UV aging resistance increased by 20%, life span > 20 years, suitable for long-term outdoor use.

**Versatile**

Electrochromic + thermochromic (adding VO<sub>2</sub> , transition temperature 68°C), adaptability increased by 30%, used for energy saving and privacy protection in multiple scenarios.

**Antimicrobial**

Doped with Ag (1-2 wt%), the antibacterial rate is >99%, and it is used in hospital smart windows and public facilities.

**New energy support**

WO<sub>3</sub> thin film (thickness 100-200 nm) optimizes solar backplane, R >90%, and improves BIPV efficiency by 10%.

These optimization directions give WO<sub>3</sub> higher technical value and market potential.

**Tungsten powder application chart**

Industry/Field	Specific uses
<b>Metallurgy</b>	- Manufacture of tungsten alloys (such as tungsten-molybdenum alloy, tungsten-rhenium alloy, tungsten-copper alloy, high-density tungsten alloy) to enhance material properties
	- Produce pure tungsten products, such as tungsten wire, tungsten rod, tungsten tube, tungsten plate, etc., which are used to process into parts of various shapes
	- Used as raw material for powder metallurgy to make high-strength parts through pressing and sintering processes
<b>Tooling</b>	- Production of ultra-fine grain cemented carbide for cutting tools (such as turning tools, milling cutters, drill bits) and molds, suitable for processing titanium alloys, heat-resistant steel, etc.
	- Manufacture tungsten steel (such as high-speed steel, tungsten-cobalt magnetic steel) to improve the hardness and wear resistance of tools
<b>Electronics</b>	- Used as filaments (such as incandescent lamps, halogen lamps), due to its high melting point and thermal stability, it is suitable for high temperature environments
	- Manufacturing high current electrical contacts, switches, relays and circuit breakers due to its strong resistance to arc corrosion
	- Used in high-precision tools in the semiconductor industry, such as circuit board drilling and electronic component manufacturing

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Industry/Field	Specific uses
Aerospace	- Make high-density counterweights, such as aircraft gyroscopes and automobile tire counterweights, due to their high density and excellent strength-to-weight ratio
	- Production of high temperature resistant components such as engine parts, nozzles, heat shields, as they maintain their performance under extreme conditions
	- Wear-resistant coatings for spacecraft and aircraft to enhance component durability
Defense	- Manufacture armor-piercing bullets and military high-density tungsten alloys due to their high density and penetrating power
	- Used as radiation shielding materials, such as neutron reflectors in nuclear reactions
Energy	- Production of plasma facing components for nuclear fusion reactors (such as ITER) due to their high temperature and radiation resistance
	- Used for thermal spraying of tungsten powder to enhance the wear resistance of equipment surface in thermal spraying process
Medical	- Manufacturing radiation shielding equipment (such as collimators in cancer treatment machines) due to its high density and ability to shield against gamma rays
	- Used in medical imaging equipment to provide accurate diagnosis due to its high density
Additive Manufacturing (Additive Manufacturing)	- Used for 3D printing of tungsten parts with complex geometries, suitable for aerospace, automotive and other fields
	- Production of fine lattice structures, such as restrictor armor in nuclear fusion reactors
Mining and Construction (Mining & Construction)	- Manufacturing drill bits, roller cutters, hammers and tunnel boring machine parts due to its excellent wear resistance and hardness
	- Used for mining equipment coating to extend the service life of steel or titanium equipment
Chemical Industry (Chemical Industry)	- Used as catalysts (e.g. tungsten sulfide for gasoline synthesis) due to its chemical stability
	- Production of inorganic pigments (such as tungsten oxide for painting)
Others	- Manufacture of wear-resistant filters, forming porous structures by sintering, for industrial filtration
	- Used in sporting goods (such as golf club weights) due to its high density
	- Producing EDM electrodes and 3D printing nozzles, due to their high temperature resistance and hardness characteristics.

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## CTIA GROUP LTD

### Introduction of High Purity Tungsten Powder

#### 1. High Purity Tungsten Powder Overview

CTIA GROUP LTD's high-purity tungsten powder is produced using a high-purity tungsten oxide hydrogen reduction process. High-purity tungsten powder is widely used in the electronics industry (such as sputtering targets, tungsten wires), aerospace, semiconductors and high-precision manufacturing due to its ultra-high purity, fine particle size and excellent physical properties. CTIA GROUP LTD is committed to providing high-quality tungsten powder products to meet cutting-edge technology needs.

#### 2. High Purity Tungsten Powder Features

Chemical composition: Tungsten (W), high purity metal powder.

Purity:  $\geq 99.99\%$  (4N), with extremely low impurity content.

Appearance: Grey or dark grey powder, uniform color.

Ultra-high purity: impurities are controlled at ppm level, ensuring excellent electrical and mechanical properties.

Fine particles: The particle size can reach 0.1-5  $\mu\text{m}$ , which can meet high-precision applications.

Low oxygen content: oxygen content  $\leq 0.02\%$ , improving sintering performance and material stability.

#### 3. High Purity Tungsten Powder Specifications

Index	CTIA GROUP LTD High Purity Tungsten Powder Standard (4N)
Tungsten content (wt%)	$\geq 99.99$
Impurities (wt%, max)	Fe $\leq 0.0010$ , Mo $\leq 0.0010$ , Si $\leq 0.0005$ , Al $\leq 0.0005$ , Ca $\leq 0.0005$ , Mg $\leq 0.0005$ , Na $\leq 0.0010$ , K $\leq 0.0010$ , O $\leq 0.0200$ , C $\leq 0.0050$ , N $\leq 0.0020$ , P $\leq 0.0005$ , S $\leq 0.0005$
Water content (wt%)	$\leq 0.02$
Particle size ( $\mu\text{m}$ , FSSS)	0.1-5.0 (superfine 0.1-1.0, fine 1.0-5.0)
Bulk density (g/ $\text{cm}^3$ )	4.5-6.5
Particle size	Provide ultra-fine (0.1-1.0 $\mu\text{m}$ ) and fine (1.0-5.0 $\mu\text{m}$ ) specifications, can be customized according to customer needs
Moisture	$\leq 0.02\%$ , ensuring product dryness and stability
Customization	Optional ultra-high purity grade (5N, $\geq 99.999\%$ ), with further reduction of impurities (e.g. O $\leq 0.01\%$ )

#### 4. Packaging and Quality Assurance

Packaging: Inner sealed vacuum aluminum foil bag, outer iron barrel or plastic barrel, net weight 5kg, 10kg or 25kg, moisture-proof and oxidation-proof.

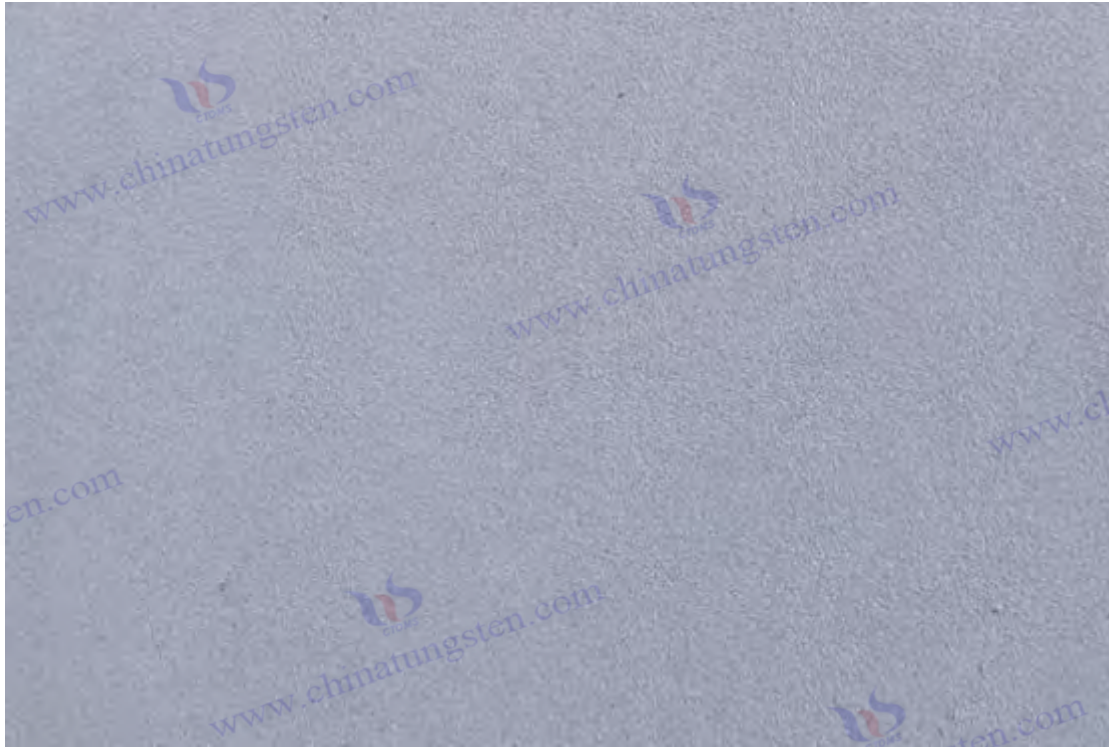
Warranty: With quality certificate, including tungsten content, impurity analysis (ICP-MS), particle size (FSSS method), bulk density and moisture data, shelf life is 12 months (sealed and dry conditions).

#### 5. Procurement Information

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com) Tel: +86 592 5129696

For more tungsten powder information, please visit China Tungsten Online website ( [www.tungsten-powder.com](http://www.tungsten-powder.com) )

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## Chapter 14 Military and Nuclear Industry Applications (Military and Nuclear Industry Applications)

The application of tungsten powder in the military and nuclear industries reflects its excellent performance under extreme conditions. Its high density ( $19.25 \text{ g/cm}^3$ ), high melting point ( $3422^\circ\text{C}$ ), excellent mechanical strength and radiation resistance make it an irreplaceable strategic material. From the high-speed penetration of armor-piercing projectiles to the anti-sputtering first wall of nuclear fusion devices, to the efficient protection of radiation shielding, the value of tungsten and its alloys in the defense and energy fields continues to emerge. This chapter is subdivided into three topics: armor-piercing projectiles and armor materials (W-Ni-Fe alloys), nuclear fusion first wall materials (anti-sputtering performance), and radiation shielding ( $\gamma$ -ray and neutron absorption). Through in-depth theoretical analysis, detailed technical descriptions, comprehensive analysis of influencing factors, rich industrial cases and forward-looking optimization insights, the scientific principles, engineering practices and future potential of tungsten powder in these fields are systematically revealed. In Section 14.1, the comparative analysis of tungsten armor-piercing projectiles and depleted uranium armor-piercing projectiles, as well as the discussion of the preparation process of tungsten alloy rotary forging rods, aims to provide comprehensive knowledge support for researchers and engineers and promote the innovation of tungsten-based materials in the field of high technology.

### 14.1 Armor-piercing projectiles and armor materials (W-Ni-Fe alloy) (Penetrator and Armor Materials: W-Ni-Fe Alloys)

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### 14.1.1 Theoretical basis

The high density and hardness of tungsten make it the best choice for armor-piercing projectile cores and armor materials. In particular, W-Ni-Fe alloy integrates the strength of tungsten with the toughness of nickel and iron, meeting the dual needs of penetration and structural integrity in modern warfare. The development history of armor-piercing projectiles can be traced back to the late World War II. With the increase in the thickness and hardness of tank armor, traditional steel projectile cores can no longer cope with it. Tungsten alloys have gradually emerged with their physical properties.

#### Armor-piercing mechanism

The armor-piercing efficiency is derived from the kinetic energy penetration theory,  $E_k = \frac{1}{2}mv^2$  (m is mass, v is velocity). The high density of tungsten (18.5-19.0 g/cm<sup>3</sup>) significantly increases m, making it 2-3 times more penetrating than steel at high-speed impact ( $v \approx 1500-2000$  m/s). The key lies in the formation of adiabatic shear bands. The self-sharpening behavior of W-Ni-Fe (local plastic instability) keeps it sharp during the impact process, greatly improving the penetration efficiency. In contrast, steel cores are prone to mushroom-shaped deformation and serious energy dissipation.

#### Mechanical properties

The tensile strength of W-Ni-Fe is  $\sigma_b \approx 900-1200$  MPa, the hardness is  $HV \approx 400-500$ , and the fracture toughness is  $K_{IC} \approx 20-30$  MPa·m<sup>1/2</sup>. Its microstructure consists of tungsten particles (bcc crystals, accounting for 90-93 wt%) and Ni-Fe bonding phase (fcc crystals, accounting for 7-10 wt%). Ni-Fe fills the gaps between W particles to alleviate the brittleness of tungsten. Phase diagram analysis shows that the Ni:Fe ratio (2:1 to 3:1) forms a uniform liquid phase at 1450-1550°C, optimizing sintering densification.

#### Theoretical Model

The Johnson-Cook model  $\sigma = (A + B\varepsilon^n)(1 + C \cdot \ln(\dot{\varepsilon} / \dot{\varepsilon}_0))(1 - T^*{}^m)$  ( $A = 900$  MPa,  $B = 400$  MPa,  $n = 0.15$ ,  $C = 0.02$ ,  $m = 1.0$ ) describes the dynamic response of W-Ni-Fe under high-speed impact ( $\dot{\varepsilon} \approx 10^4 - 10^5$  s<sup>-1</sup>) and predicts the conditions for the formation of adiabatic shear bands. Thermodynamic analysis shows that the impact temperature can reach 1000-1500°C and local melting enhances the self-sharpening effect.

### 14.1.2 Methods and Control Techniques

W-Ni-Fe alloy is a complex multi-step process involving powder metallurgy, sintering and post-processing. Each step needs to be precisely controlled to ensure that the performance meets military standards. The process design not only pursues high density and high strength, but also takes into account microstructural uniformity and processing feasibility.

#### Measurement method

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### Powder preparation

High purity tungsten powder (D50 = 1-10  $\mu\text{m}$ , purity >99.95 wt%, O <0.02 wt%) was selected and mixed with Ni powder (D50 = 5-15  $\mu\text{m}$ ) and Fe powder (D50 = 5-20  $\mu\text{m}$ ) at a ratio of W:Ni:Fe  $\approx$  90-93:5-7:2-3. Planetary ball milling (200-400 rpm, 10-24 h, dry or wet mixing in ethanol medium, ball-to-material ratio 5:1) was used to ensure uniformity, and laser particle size analyzer was used to monitor D50 and distribution (CV <5%).

### Forming

The green body was prepared by cold isostatic pressing (CIP, 200-300 MPa, holding pressure 2-5 min) with a dimensional accuracy of  $\pm 0.1$  mm.

Alternatively, powder injection molding (PIM) is used, which is suitable for complex shapes (such as the core head), and the binder (PP/PE wax, 5-10 wt%) is degreased at 400-600°C.

### Sintering

Liquid phase sintering (1450-1550°C, H<sub>2</sub> atmosphere, dew point <-50°C, heating rate 5-10°C/min, hold time 2-4 h), or hot isostatic pressing (HIP, 1400°C, 100-200 MPa, Ar atmosphere, 1-2 h). Target density >98% (theoretical value 17.5-18.5 g/cm<sup>3</sup>), Archimedes verification.

### Control Technology

#### Composition uniformity

The Ni-Fe ratio is crucial. Excessive Ni (>7 wt%) easily generates brittle  $\eta$  phase (Ni<sub>3</sub>W, hardness HV >600), and excessive Fe (>3 wt%) reduces liquid phase fluidity. EDS analysis confirmed the element distribution, with a deviation of <1 wt%.

#### Microstructure

The W grain size is controlled at 10-50  $\mu\text{m}$ , and the sintering temperature gradient is  $\pm 5^\circ\text{C}/\text{min}$  to avoid abnormal growth (>100  $\mu\text{m}$ ) or residual porosity (>2%). SEM/EBSD analysis of W particle sphericity (>0.9) and Ni-Fe connectivity. Heat treatment (1200°C, N<sub>2</sub>, 1-2 h) can refine the grain boundaries.

#### Performance Testing

Hardness (HV, load 10 kgf,  $\pm 5$  HV), tensile test (ASTM E8, strain rate  $10^{-3} \text{ s}^{-1}$ ), high-speed impact test ( $v = 1500-2000$  m/s, target material is RHA steel, penetration accuracy  $\pm 5$  mm). Dynamic compression test (SHPB,  $\dot{\epsilon} \approx 10^4 \text{ s}^{-1}$ ) to evaluate the self-sharpening behavior.

#### Post-processing

Machining (turning speed 100-200 rpm, feed rate 0.1-0.3 mm/r, Ra <0.5  $\mu\text{m}$ ), heat treatment (1000-1200°C, N<sub>2</sub>, 1-2 h) to eliminate residual stress, surface polishing or carburizing (0.1-0.5 mm) to improve wear resistance.

#### 14.1.3 Influencing factors

The performance of W-Ni-Fe alloy is affected by multiple factors, from raw materials to processes to

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usage conditions, all of which need to be considered comprehensively.

#### Composition of Tungsten Alloy

When W content is <90 wt%, density drops to <17 g/cm<sup>3</sup>, penetration power decreases by 10-15%, and it cannot meet the requirements of modern armor. When Ni content is >7 wt%, toughness increases by 10-20%, but hardness decreases by 50-100 HV, affecting shear localization.

#### Sintering temperature

<1450°C, insufficient liquid phase, density <95%, porosity increased by 3-5%, strength decreased by 15-20%; >1550°C, grains grew to 60-100 μm, Ni-Fe volatilized by 5-10%, and brittleness increased by 10%.

#### Impact speed

v From 1500 m/s to 2000 m/s, the self-sharpening effect is enhanced, the penetration increases by 20-30%, but the risk of fragmentation increases by 15-20%, and toughness needs to be optimized.

#### Atmosphere

When H<sub>2</sub> dew point is > -40°C, residual O increases by 0.1-0.2 wt%, and grain boundary oxidation causes brittleness to increase by 10-15%.

#### Strain rate

$\dot{\epsilon} > 10^4 \text{ s}^{-1}$ , the width of the adiabatic shear band is reduced to 5-10 μm, and the local temperature is >1500°C, which enhances penetration but increases the risk of fracture.

#### Target hardness

When the target HV increases from 300 to 500 (such as composite armor), the penetration resistance increases by 20-30%, and the W content or impact angle needs to be adjusted.

#### Heat Treatment

When the annealing temperature is >1300°C, the grain size grows by 20-30 μm and K<sub>IC</sub> decreases by 10-15%, which affects the fracture resistance.

### 14.1.4 Application Cases

#### Magness et al. (1995)

Development of a W-Ni-Fe alloy (W 93 wt%, Ni:Fe = 7:3) with a density of 18.0 g/cm<sup>3</sup> and a penetration of 600 mm (RHA target) at high velocity (v = 1700 m/s) for the M829A1 armor-piercing projectile. This design performed well against the Soviet T-72 tank at the end of the Cold War, with significantly better self-sharpening properties than steel cores.

#### Li et al. (2022)

Optimized W-Ni-Fe (W 92 wt%, Ni 5 wt%, Fe 3 wt%), HIP sintered, 20 μm grain size, 650 mm

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penetration ( $v = 1800$  m/s), for the new generation of anti-tank ammunition. The HIP process improves density and uniformity, making it suitable for thicker composite armor.

#### **Zhang et al. (2024)**

Preparation of W-Ni-Fe (W 91 wt%, Ni 6 wt%, Fe 3 wt%), adding 0.5 wt% Co, density  $18.2$  g/cm<sup>3</sup>, toughness increased by 15%, penetration 680 mm ( $v = 1900$  m/s). The addition of Co optimizes the bonding phase performance and is used to resist modern multi-layer armor, showing the potential of tungsten alloys.

#### **14.1.5 Optimization Direction**

The future development of W-Ni-Fe alloys requires breakthroughs in penetration, durability and manufacturing efficiency.

##### **Ultrafine Grain Alloy**

W grains  $< 5$   $\mu$ m (mechanical alloying or rapid sintering), strength increased by 20-30%, penetration increased by 15%, suitable for higher speeds ( $> 2000$  m/s).

##### **Gradient structure**

The W content is 95 wt% on the surface and 85 wt% inside, combining high hardness and toughness to increase fracture resistance by 20%.

New binder phase: Replace Ni-Fe with Ni-Co-Cu, increase heat resistance by 10-15%, and improve high temperature impact performance.

##### **Simulation Optimization**

Finite element analysis (LS-DYNA) simulates shear band evolution and optimizes composition and structure with a prediction error of  $< 5\%$ .

##### **Lightweight**

The W content is reduced to 85 wt% and combined with ceramics (such as SiC) to reduce weight by 10-15% and improve mobility.

##### **Versatility**

Integrate explosive reactive armor (ERA) features to enhance comprehensive protection capabilities and adapt to complex battlefield requirements.

#### **14.1.6 Comparative Analysis of Tungsten Armor-Piercing Projectiles and Depleted Uranium Armor-Piercing Projectiles**

As two main kinetic energy armor-piercing core materials, tungsten armor-piercing projectiles (W-Ni-Fe alloy) and depleted uranium armor-piercing projectiles (DU, mainly U-0.75Ti alloy) have their own

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advantages and disadvantages. Their performance differences are due to material properties, manufacturing processes and environmental influences. The following is a detailed comparison in terms of physical properties, penetration efficiency, safety and cost:

### Physical properties

#### density

The density of tungsten alloy is 17.5-18.5 g/cm<sup>3</sup>, the density of depleted uranium is 19.05 g/cm<sup>3</sup>, and DU is slightly higher (about 5-8%), giving it greater kinetic energy ( $E_k \propto m$ ).

#### Hardness and toughness

Tungsten alloy HV  $\approx$  400-500, K<sub>IC</sub>  $\approx$  20-30 MPa·m<sup>1/2</sup>; Depleted uranium HV  $\approx$  300-350, K<sub>IC</sub>  $\approx$  40-50 MPa·m<sup>1/2</sup>. DU has better toughness and is not easy to break when impacted, but its hardness is slightly inferior.

#### Melting point

The melting point of tungsten alloy (dominated by W) is  $\approx$  3422°C, and the melting point of depleted uranium is  $\approx$  1132°C. Tungsten is more stable at high temperatures.

### Penetration efficiency

#### Self-sharpening

Tungsten alloy relies on adiabatic shear band formation for self-sharpening, and its penetration (e.g. 650-680 mm,  $v = 1800-1900$  m/s) is slightly inferior to DU (700-750 mm) because DU produces stronger local plastic flow and "mushroom head" reduction effect during impact.

### High temperature behavior

DU partially melts and burns upon impact ( $T > 1000^\circ\text{C}$ ), producing a spark effect that enhances the damage to composite armor; tungsten alloy remains solid, relies on mechanical penetration, and has a weaker firepower effect.

### Target material adaptability

DU is more effective against ceramic/steel composite armor (10-15% more penetration) due to its burning properties that destroy the ceramic layer; tungsten alloy is better against homogeneous steel (5-10% more effective).

### Safety and environmental impact

#### Radioactivity

Depleted uranium is weakly radioactive ( $\alpha$  particles, half-life  $4.47 \times 10^9$  years), and battlefield residues may pose health risks (inhalation of dust increases the cancer rate by 0.1-1%). Tungsten alloy has no radiation and is safer.

#### Toxicity

DU dust is chemically toxic (risk of kidney damage increases by 5-10%); tungsten alloys (Ni, Fe) are less toxic, but Ni may cause mild allergies (<0.1%).

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### Cost and availability

#### Manufacturing Cost

Due to the scarcity of raw materials and complex processes (HIP, etc.), the cost of tungsten alloy is about 2-3 times that of DU ( $\approx$  \$50-100 vs. \$20-30 per kg).

### Resource

Tungsten is a rare metal with limited global reserves (China accounts for 60%); depleted uranium is a by-product of the nuclear industry with sufficient supply but subject to regulatory restrictions.

### In conclusion

Tungsten armor-piercing bullets have advantages in safety, environmental friendliness and high temperature stability, and are suitable for the sustainability requirements of modern warfare; depleted uranium armor-piercing bullets have more advantages in penetration and cost, but radiation and toxicity limit their application. In the future, tungsten alloys can narrow the gap in penetration depth with DU through microstructure optimization (such as ultrafine grains) while maintaining environmental advantages.

### Appendix:

#### Tungsten Alloy Rotary Forging Process Details

Rotary Forging is a key step in manufacturing tungsten-based heavy alloy bars in GB/T 26038-2020, which is used to improve density and mechanical properties. The standard does not list the process parameters in detail. The following is supplemented with specific details based on Chinese tungsten alloy processing practices and literature.

#### 1 Process

##### Raw materials preparation:

Tungsten powder: purity  $\geq 99.95\%$ ,  $D_{50}=1-10 \mu\text{m}$  (prepared by atomization or reduction method).

Binder: Ni (99.9%), Fe (99.9%), Cu (99.9%) or Co (99.8%), particle size 5-20  $\mu\text{m}$ .

Powder mixing: planetary ball mill, speed 200-300 rpm, ball-to-material ratio 5:1, time 10-20 h, medium is ethanol, uniformity CV  $< 5\%$ .

##### Suppress:

Method: Cold isostatic pressing (CIP).

Pressure: 150-300 MPa, maintain pressure for 5-10 min.

Green body: diameter 20-150 mm, length 100-500 mm, density about 60-70% of theoretical value.

##### Sintering:

Equipment: hydrogen sintering furnace or vacuum sintering furnace.

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Atmosphere: high purity H<sub>2</sub> ( purity > 99.999%, dew point < -50°C) or vacuum (<10<sup>-3</sup>Pa) .

Temperature: 1350-1550°C (adjusted with tungsten content, W97 requires higher temperature).

Heating rate: 5-10°C/min, keep warm for 2-6 h.

Density: ≥98%, grain size 5-50 μm.

#### **Rotary forging:**

Equipment: Rotary forging machine (such as GFM SKK-10).

Temperature: 800-1200°C (high temperature rotary forging to prevent cracking).

Deformation: 10%-20% per pass, total deformation 50%-80%.

Forging frequency: 200-500 times/min.

Lubrication: MoS<sub>2</sub> or graphite emulsion, coating thickness 0.1-0.2 mm.

Result: Density increased to 99.5% and diameter reduced to target size (e.g. 10-50 mm).

#### **Heat treatment (optional):**

Method: Annealing.

Temperature: 800-1000°C, keep warm for 1-2 h.

Atmosphere: H<sub>2</sub> or Ar, cooling rate 5-10°C/min.

Purpose: To eliminate internal stress and increase elongation by 5%-10%.

## **2 Process parameters**

### **Temperature control**

Sintering: W90 (1350-1400°C), W97 (1450-1550°C), avoid excessive volatilization of liquid phase.

Rotary forging: It is easy to crack at low temperature (<800°C) and may oxidize at high temperature (>1200°C).

### **Deformation Control**

A single deformation >20% may cause micro cracks and require multiple progressive processing passes.

Diameter reduction rate

Initial billet diameter D<sub>o</sub>, final diameter D<sub>f</sub>, reduction rate =  $(D_o - D_f) / D_o \times 100\%$ .

### **Equipment parameters**

Rotary forging machine power

50-200 kW.

Mould

Cemented carbide (WC-Co), hardness >1500 HV.

### **Quality Control**

Online temperature measurement

Infrared thermometer (accuracy ±5°C).

Dimensional inspection

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Micrometer or laser diameter gauge (accuracy  $\pm 0.01$  mm).

### 3. Process characteristics

Density improvement

After sintering, the content is increased from 98% to 99.5% after rotary forging, and the internal pores are closed.

Grain refinement

The sintered grain size is 20-50  $\mu\text{m}$ , and after rotary forging it is 5-20  $\mu\text{m}$ , with the strength increasing by 10%-20%.

Surface quality

After rotary forging, Ra is less than 1.6  $\mu\text{m}$ , and after heat treatment, Ra can reach less than 0.8  $\mu\text{m}$ .

Performance Optimization

The tensile strength is increased to 950 MPa (W97) and the elongation can be adjusted to 25% (W88).

### 4. Notes

Oxidation Control

Forging temperatures  $> 1000^\circ\text{C}$  require a protective atmosphere (Ar or  $\text{H}_2$ ) with an oxygen content  $< 0.03$  wt%.

Crack prevention

The sintering quality of the green body must be uniform to avoid the amplification of initial defects.

Lubricant selection

At high temperatures,  $\text{MoS}_2$  is more stable than grease and reduces mold wear.

### Schedule:

#### Tungsten armor-piercing projectiles (W-Ni-Fe alloy) and depleted uranium armor-piercing projectiles (U-0.75Ti alloy)

Comparison Dimensions	Tungsten armor-piercing bullet (W-Ni-Fe Alloy)	Depleted Uranium Armor Piercing (DU, U-0.75Ti Alloy)
Material composition	Main ingredients: tungsten (W, 90-97 wt%), nickel (Ni, 2-6 wt%), iron (Fe, 1-4 wt%). A small amount of Co or Cu can be added to adjust the performance.	Main components: Uranium (U, 99.25 wt%), titanium (Ti, 0.75 wt%). Depleted uranium is U-238 (low radioactivity), with most of the U-235 removed.
Density(g/cm <sup>3</sup> )	17.0-18.5 (depending on W content). High density comes from Tungsten (19.25 g/cm <sup>3</sup> ), but Ni/Fe reduces the overall density.	18.6-19.0 (close to pure uranium 19.05 g/cm <sup>3</sup> ). The density is slightly higher than tungsten alloy, thanks to the high atomic mass of uranium.
Melting point (°C)	The tungsten matrix has an extremely high melting point (3422°C), but the Ni/Fe phase (about 1450-1500°C) limits the overall heat resistance.	1132°C (U-Ti alloy melting point). Lower than tungsten alloy, easily softened or melted in high-speed impact. Tungsten Smart MADE BY:

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Comparison Dimensions	Tungsten armor-piercing bullet (W-Ni-Fe Alloy)	Depleted Uranium Armor Piercing (DU, U-0.75Ti Alloy)
Hardness (HV)	400-600 (W-Ni-Fe sintered state). High hardness but high brittleness and limited deformation resistance.	250-350 (U-0.75Ti annealed state). Lower hardness, but better ductility, easy to plastically deform when impacted.
Strength (MPa)	Tensile strength: 900-1200 (after sintering). High strength, but low ductility, easy to break under high stress.	Tensile strength: 800-1000 (after heat treatment). High ductility (elongation 5-10%), better fracture resistance than tungsten alloy.
Manufacturing process	Powder metallurgy: Tungsten powder is mixed with Ni/Fe powder, cold isostatically pressed (200-300 MPa), and hydrogen sintered (1350-1500°C, 2-4 h), with a density of 98-99%. The performance can be improved by heat treatment or hot isostatic pressing (HIP). The process is complex and the grain size needs to be controlled (5-20 μm).	Melting + heat treatment: uranium ingot smelting (vacuum induction furnace, 1200°C), adding Ti alloying, casting and annealing (800-900°C). The process is relatively simple, but anti-oxidation and radiation protection are required.   Large grain size (50-100 μm).
Penetration efficiency	Advantages: High hardness and density give excellent penetration, with a penetration depth of about 600-700 mm (120 mm caliber) against homogeneous steel armor (RHA) at an initial velocity (1500-1700 m/s). Disadvantages: No self-sharpening effect on impact, the core is easily blunted or broken, and the penetration depth is limited, especially against composite armor (ceramic + steel).	Advantages: self-sharpening effect (the core deforms plastically upon impact to form a sharp front), penetration of about 700-800 mm (RHA, 120 mm caliber). Disadvantages: slightly weaker penetration against high-hardness targets (such as ceramics) due to insufficient hardness.
Impact characteristics	When the projectile hits at high speed (>1500 m/s), the core is easily broken due to its brittleness, and the fragmentation is serious. The secondary lethality is strong, but the penetration depth is affected. There is no combustion effect, and the energy is mainly transferred by kinetic energy.	Upon impact, the low melting point and ductility cause self-sharpening and local melting, accompanied by a combustion effect (oxidation of uranium dust releases heat), which enhances the lethality against targets behind armor ("aftereffect").
Environmental impact	Advantages: non-radioactive, environmentally friendly, no long-term pollution risk in post-war cleanup.   Disadvantages: high energy consumption in manufacturing (sintering requires high	Disadvantages: Weakly radioactive (U-238 half-life 4.47 billion years, mainly alpha particles), produces uranium oxide dust after impact, has chemical toxicity and low radiation risk. Advantages: Low

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Comparison Dimensions	Tungsten armor-piercing bullet (W-Ni-Fe Alloy)	Depleted Uranium Armor Piercing (DU, U-0.75Ti Alloy)
	temperature), and trace amounts of Ni may be released into the soil.	pollution in the manufacturing process.
Security	Production safety: no radiation risk, but tungsten powder metallurgy needs to prevent tungsten dust inhalation (occupational health standard <math><5 \text{ mg/m}^3</math>). Use safety: no toxic residue, suitable for conventional battlefields.	Production safety: Strict radiation protection is required (shielding gamma rays and alpha particles), and workers need to wear protective clothing. Safety in use: Uranium dust left over from the war is toxic, and long-term exposure may cause cancer (low risk, about 0.1-1 mSv/year).
cost	Raw material cost: Tungsten price fluctuates greatly (about 30-50 USD/kg, estimated in 2025), Ni/Fe is cheap but the total amount is high. Manufacturing cost: The process is complex and the energy consumption is high. The cost of a single projectile core is about 500-800 USD (120 mm).	Raw material cost: Depleted uranium is a byproduct of the nuclear industry and has a low cost (about \$5-10/kg). Manufacturing cost: The melting and casting process is simple, but radiation protection facilities are required, and the cost of a single piece is about \$300-500 (120 mm).
Availability	Tungsten is a strategic resource with abundant reserves worldwide (China accounts for more than 60%) and a stable supply, but it requires high purification and processing technology.	Depleted uranium relies on nuclear industrial waste, has limited supply (mainly controlled by nuclear powers such as the United States and Russia), and requires international regulatory approval (such as IAEA supervision).
Application Scenario	It is widely used in conventional warfare, such as tank guns (M829A1 replacement) and anti-armor missiles, and is suitable for countries with high environmental protection requirements (such as Germany and Switzerland).	It is mostly used by major countries (such as the US M829 series and the Russian 3BM42), especially in scenarios that require high-effect killing (such as anti-infantry armor).
Technology Trends	Optimization direction: Improve toughness and reduce fracture through nano-tungsten powder (D50<math><100 \text{ nm}</math>) or gradient structure; develop tungsten-based composite materials (such as W-TiC) to enhance penetration.	Optimization direction: Doping with other elements (such as Mo, 1-2 wt%) to increase hardness; developing low-radioactive alternatives (such as U-Zr alloys) to reduce environmental controversy.
Comprehensive evaluation	Advantages: high hardness, non-toxic, environmentally friendly, suitable for the	Advantages: significant self-sharpening effect and burning aftereffect, strong

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Comparison Dimensions	Tungsten armor-piercing bullet (W-Ni-Fe Alloy)	Depleted Uranium Armor Piercing (DU, U-0.75Ti Alloy)
	environmental protection needs of modern warfare. Disadvantages: no self-sharpening effect, penetration depth and aftereffect are not as good as depleted uranium.	penetration and lethality, low cost. Disadvantages: radioactivity and toxicity restrict its use, high political sensitivity.

#### 14.1.7 Tungsten Alloy Rotary Forging Rod Production Process and Characteristics

Tungsten alloy rotary forging rod is an important form of armor-piercing projectile core. Its high precision, high density and high strength characteristics rely on the unique advantages of rotary forging technology. The following details its preparation process and technical characteristics:

##### Tungsten Alloy Rotary Forging Rod Production Process

###### Raw material preparation

High-purity tungsten powder (D50 = 1-5 μm, purity >99.98 wt%, O <0.01 wt%), Ni powder (D50 = 5-10 μm), and Fe powder (D50 = 5-15 μm) were selected with a ratio of W:Ni:Fe = 90-93:5-7:2-3. Planetary ball milling (300 rpm, 12-20 h, wet mixing with ethanol, ball-to-material ratio 5:1), drying (80-100°C, vacuum <10 Pa), and sieving (200 mesh).

###### Blank forming

Cylindrical blanks (φ 50-100 mm, length 200-300 mm) are prepared by cold isostatic pressing (250-300 MPa, holding pressure 3-5 min), with a density ≈ 60-70% of the theoretical value. Or powder extrusion (500-700 MPa, 400-600°C) is used to prepare green blanks (φ 30-50 mm).

###### Pre-sintering

- sintering (1000-1200°C, heating 5°C/min, holding 1-2 h) in H<sub>2</sub> atmosphere (dew point < -50°C) removes moisture and volatiles, and the green body density increases to 80-85%.

###### Liquid Phase Sintering

Tube furnace or vacuum furnace (1450-1550°C, H<sub>2</sub> or <10<sup>-3</sup>Pa, heating 5-10°C/min, holding 2-4 h), density >98% (17.5-18.5 g/cm<sup>3</sup>). Cooling rate 5-10°C/min, avoid stress concentration.

###### Rotary forging

equipment

Three-roll or four-roll rotary forging machine (power 50-100 kW, speed 100-300 rpm).

Process parameters

The billet is heated to 1000-1200°C (holding temperature for 30-60 min, N<sub>2</sub> protection), the forging temperature is 800-1000°C, the deformation amount of each pass is 10-20%, the cumulative deformation is 50-70%, and the final bar is φ 10-20 mm and 300-500 mm long.

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lubricating

High temperature MoS<sub>2</sub> or graphite lubricant to reduce friction (friction coefficient < 0.1).

### Heat Treatment

Annealing (1000-1100°C, N<sub>2</sub>, 1-2 h, furnace cooling) eliminates work hardening and adjusts the grain size to 10-30 μm.

### Finishing

Turning (speed 150-200 rpm, feed 0.1-0.2 mm/r), polishing (Ra < 0.3 μm), dimensional tolerance ±0.05 mm.

### Technical features

High density and uniformity: Rotary forging eliminates porosity through multi-directional extrusion, density >99%, grains are stretched axially (aspect ratio 3:1-5:1), and tensile strength is increased ( $\sigma_b > 1200$  MPa).

### Excellent mechanical properties

Under dynamic deformation ( $\dot{\epsilon} \approx 10^4 \text{ s}^{-1}$ ), the shear bands are finer (width 3-8 μm), the self-sharpening effect is enhanced by 10-15%, and the penetration is increased by 5-10%.

### Dimensional accuracy

The diameter deviation of the rotary forging rod is < 0.1 mm, and the length tolerance is ±1 mm, which meets the high precision requirements of the core.

### Process Challenges

High temperature processing requires precise temperature control (±10°C) to avoid excessive grain size or surface oxidation; excessive rotary forging deformation (>20%) can easily cause microcracks (depth < 50 μm).

### Advantages and limitations

Compared with traditional sintered rods, the strength and toughness of rotary forging rods are increased by 15-20%, but the equipment investment is high (about 5-10 million yuan/unit) and the production cycle is long (5-7 days for a single batch). In the future, the process can be shortened and efficiency can be improved through rapid sintering and automated rotary forging.

### Tungsten Alloy Rotary Forging Rods

Nation	Standard No.	Scope of application	Ingredient Requirements	Performance parameters	Processing requirements
USA	ASTM B777-15	High density tungsten alloy rods, including rotary forging rods, are used in	The tungsten content is 90% to 97% (classified into four grades: Class 1-4), the binder is Ni-	Density 17.0 to 18.5 g/cm <sup>3</sup> , tensile strength 620 to 1000 MPa, elongation 2% to	Stress relief is required after rotary forging, the surface roughness Ra ≤ 1.6 μm, the dimensional

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Nation	Standard No.	Scope of application	Ingredient Requirements	Performance parameters	Processing requirements
		aerospace, military, medical radiation shielding and other applications.	Fe or Ni-Cu, and magnetic or non-magnetic is available.	15%, hardness 24 to 35 HRC.	tolerance is $\pm 0.002$ inches, and it meets the requirements of near net shape.
USA Military Standard	MIL-T-21014D	Military tungsten alloy rods are suitable for high-performance components such as armor-piercing cores and counterweights.	Tungsten content is 90% to 97%, Ni-Fe or Ni-Cu binder, and the impurity content (such as C, O) is strictly controlled ( $<0.01\%$ ).	Density 17.0 to 18.5 g/cm <sup>3</sup> , tensile strength $\geq 758$ MPa, elongation $\geq 5\%$ , hardness 25 to 40 HRC, impact toughness needs to be tested.	After rotary forging, heat treatment (annealing or quenching) is required, the dimensional tolerance is $\pm 0.001$ inches, and there is no crack or oxide layer on the surface.
internationality aviation	AMS 7725E	Tungsten alloy rods for aerospace applications are suitable for counterweights and vibration damping components.	Tungsten content 90% to 97%, Ni-Fe or Ni-Cu, must meet non-magnetic requirements (magnetic permeability $<1.05$ ) or magnetic requirements.	Density 17.0 to 18.5 g/cm <sup>3</sup> , tensile strength 700 to 1100 MPa, elongation 3% to 20%, fatigue performance to be verified.	After rotary forging, precision machining is required, with a surface finish of $Ra \leq 0.8 \mu\text{m}$ and a dimensional tolerance of $\pm 0.001$ inches, and it must comply with AS9100 quality system certification.
China	GB/T 26038-2020	Tungsten-based high-density alloy bars, including rotary forging bars, are used in military, aviation, industrial counterweights and other fields.	The tungsten content is 88% to 98%, the binder is Ni-Fe, Ni-Cu or Co, and the impurities (such as S, P) are $<0.02\%$ .	Density 16.5 to 18.75 g/cm <sup>3</sup> , tensile strength 600 to 950 MPa, elongation 2% to 25%, hardness 20 to 38 HRC.	Heat treatment is required after rotary forging (sintering in hydrogen atmosphere followed by rotary forging), surface roughness $Ra \leq 1.6 \mu\text{m}$ , dimensional tolerance $\pm 0.05$ mm, and no internal defects.

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Nation	Standard No.	Scope of application	Ingredient Requirements	Performance parameters	Processing requirements
Europe EN	EN ISO 24373	Tungsten alloy rods are suitable for welding, counterweights and high temperature components, emphasizing processing performance and consistency.	The tungsten content is 90% to 95%, the binder is Ni-Fe or Ni-Cu, it must comply with RoHS environmental protection requirements, and the content of heavy metals such as Pb and Cd is <0.01%.	Density 17.0 to 18.0 g/cm <sup>3</sup> , tensile strength 650 to 900 MPa, elongation 5% to 15%, hardness 25 to 35 HRC.	Annealing is required after rotary forging, the surface roughness Ra≤1.2 μm, the dimensional tolerance is ±0.02 mm, and ultrasonic testing is required to ensure that there are no cracks.
Japan	JIS H 4463	Tungsten alloy rods are suitable for the fields of electronics, aerospace and precision instruments, emphasizing high purity and corrosion resistance.	The tungsten content is 90% to 97%, the binder is Ni-Fe or Ni-Cu, the impurities (such as O, N) are <0.005%, and high-purity tungsten powder (>99.95%) is required.	Density 17.0 to 18.5 g/cm <sup>3</sup> , tensile strength 700 to 1000 MPa, elongation 3% to 18%, hardness 25 to 36 HRC.	After rotary forging, polishing or grinding is required, the surface roughness Ra≤0.4 μm, the dimensional tolerance is ±0.01 mm, and the internal quality needs to be inspected by X-ray.
Russia	GOST 19658-81	Tungsten alloy rods are suitable for military and heavy machinery fields, emphasizing wear resistance and high temperature performance.	The tungsten content is 89% to 96%, the binder is Ni-Fe or Co, and the impurities (such as Si, Al) are <0.03%.	Density 16.8 to 18.3 g/cm <sup>3</sup> , tensile strength 650 to 950 MPa, elongation 5% to 20%, hardness 24 to 38 HRC.	After rotary forging, heat treatment (vacuum or inert atmosphere) is required, surface roughness Ra≤2.0 μm, dimensional tolerance ±0.05 mm, and corrosion resistance test is required.

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## Appendix:

### Comparison of American Standards ASTM B777-15, MIL-T-21014D, AMS 7725E

#### 1. ASTM B777-15: Standard Specification for Tungsten Base, High-Density Metal

##### Overview

ASTM B777-15 is a standard developed by the American Society for Testing and Materials (ASTM) for bars, plates and other shaped products of high-density tungsten-based alloys, including swaged bars. The standard defines four classes (Class 1-4) of tungsten alloys, covering composition, mechanical properties, density and testing requirements, suitable for aerospace counterweights, radiation shielding and military components.

##### Scope of application

This standard specifies the requirements for high-density tungsten-based metals that can be machined and is applicable to products produced by powder metallurgy. As a common form, rotary forging rods must meet the requirements of the standard on size, performance and quality.

##### The material classification

standard divides tungsten alloys into four categories, based on tungsten content and density:

Class 1: Tungsten content 90 wt%, density 16.85-17.25 g/cm<sup>3</sup>.

Class 2: Tungsten content 92.5 wt%, density 17.15-17.85 g/cm<sup>3</sup>.

Class 3: Tungsten content 95 wt%, density 17.75-18.35 g/cm<sup>3</sup>.

Class 4: Tungsten content 97 wt%, density 18.25-18.85 g/cm<sup>3</sup>.

Each type is divided into two types: magnetic (Ni-Fe binder) and non-magnetic (Ni-Cu binder).

##### Chemical composition

Tungsten (W) is the main component, and the binders are nickel (Ni) and iron (Fe) or nickel (Ni) and copper (Cu).

Impurity requirements: carbon (C) <0.01 wt%, oxygen (O) <0.015 wt%, other elements (such as S, P) <0.005 wt%.

The specific binder ratio is not specified, but is required to ensure that density and performance requirements are met.

##### Mechanical properties

##### Tensile Strength (UTS):

Class 1: ≥620 MPa

Class 2: ≥650 MPa

Class 3: ≥690 MPa

Class 4: ≥724 MPa

##### Elongation:

Class 1: ≥5%

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Class 2:  $\geq 4\%$

Class 3:  $\geq 3\%$

Class 4:  $\geq 2\%$

Hardness (HRC):

Class 1: 24-28

Class 2: 25-30

Class 3: 26-32

Class 4: 28-35

Properties vary with processing conditions (such as annealing).

Physical properties

The density range is as above and is measured by the Archimedean method with an accuracy of  $\pm 0.05 \text{ g/cm}^3$ .

Coefficient of thermal expansion (CTE):  $4.5-6.5 \times 10^{-6} \text{ K}^{-1}$  (depending on the binder).

Manufacturing process

Powder metallurgy process: tungsten powder is mixed with binder, cold isostatic pressing (CIP, 200-300 MPa), and sintering (1350-1500°C,  $\text{H}_2$  or vacuum atmosphere).

Rotary forging rods must be formed by rotary forging or drawing to ensure that the density is  $\geq 98\%$ .

Heat treatment (e.g. stress relief annealing, 800-1000°C) is permitted to optimize properties.

Dimensions and Tolerances

Rod diameter range: 3 mm to 100 mm, length  $\leq 2000$  mm.

Tolerance: diameter  $\pm 0.002$  inches (approximately 0.05 mm), length  $\pm 0.010$  inches (approximately 0.25 mm).

Surface roughness:  $Ra \leq 1.6 \mu\text{m}$ .

Inspection and acceptance

Chemical analysis: ICP-MS or XRF is used to detect the composition.

Mechanical testing: Performed according to ASTM E8 (tensile test) and ASTM E18 (hardness test).

Density test: According to ASTM B311.

Non-destructive testing: Ultrasonic testing (UT) checks for internal defects, defect size  $< 0.5$  mm.

Certificate: Material Certificate of Conformity (CoC) is required.

Packaging and Labeling

The packaging must be moisture-proof and shock-proof, and the markings must include batch number, category (Class), magnetic type and manufacturer information.

## 2. MIL-T-21014D: Tungsten Base Metal, High Density (cancelled, but still of reference value)

Overview

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MIL-T-21014D is a US military standard, established in 1986, revised in 1991, and cancelled on October 5, 1998. SAE-AMS-T-21014 (later replaced by ASTM B777) is recommended. Although it has been cancelled, many legacy designs still reference this standard and are applicable to military tungsten alloy products (such as armor-piercing cores and counterweights).

#### Scope of application:

It specifies the requirements for high-density tungsten-based metals, suitable for military swaged rods and other shaped products, emphasizing impact resistance and consistency.

#### Material Classification

Class A: Tungsten content 90 wt%, density 16.9-17.1 g/cm<sup>3</sup>.

Class B: Tungsten content 92.5 wt%, density 17.4-17.6 g/cm<sup>3</sup>.

Class C: Tungsten content 95 wt%, density 17.9-18.1 g/cm<sup>3</sup>.

Class D: Tungsten content 97 wt%, density 18.4-18.6 g/cm<sup>3</sup>.

There are two types: magnetic (Ni-Fe) and non-magnetic (Ni-Cu).

#### Chemical composition

The tungsten content is as above, and the binder is Ni-Fe or Ni-Cu.

Impurity limits: C<0.01 wt%, O<0.01 wt%, H<0.005 wt%, N<0.005 wt%.

#### Mechanical properties

tensile strength:

Class A:  $\geq 758$  MPa

Class B:  $\geq 793$  MPa

Class C:  $\geq 827$  MPa

Class D:  $\geq 862$  MPa

Elongation:

Class A:  $\geq 5\%$

Class B:  $\geq 4\%$

Class C:  $\geq 3\%$

Class D:  $\geq 2\%$

Hardness (HRC):

Class A: 25-30

Class B: 26-32

Class C: 28-35

Class D: 30-40

Impact toughness: Must pass the Charpy impact test (specific value specified by the purchaser).

#### Physical properties

Density is as above and needs to be measured accurately.

Corrosion resistance: Need to pass salt spray test (MIL-STD-810).

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#### Manufacturing process

Powder metallurgy process, rotary forging after sintering.

Heat treatment requirements: Annealing (850-950°C, inert atmosphere) or quenching (depending on the application).

Density  $\geq 99\%$ , no internal pores or cracks are allowed.

#### Dimensions and Tolerances

The diameter tolerance is  $\pm 0.001$  inches (approximately 0.025 mm), and the length tolerance is  $\pm 0.005$  inches (approximately 0.13 mm).

Surface roughness:  $Ra \leq 2.0 \mu\text{m}$ .

#### Inspection and acceptance

Chemical analysis: spectral analysis.

Mechanical tests: tensile, hardness, impact tests.

Non-destructive testing: Ultrasonic and X-ray to ensure no internal defects ( $< 0.3$  mm).

Military acceptance: must comply with MIL-STD-105 sampling inspection.

#### Packaging and Labeling

Rust-proof packaging, markings include military standard number, batch and category.

#### Remark

Although MIL-T-21014D has been canceled, its requirements are highly similar to ASTM B777, but it has stricter control over impact toughness and impurities and is suitable for early military designs.

### 3. AMS 7725E: Tungsten Heavy Alloy Shapes, Sintered, High Density

#### Overview

AMS 7725E was developed by the American Institute of Aeronautics and Astronautics (SAE) for high-density tungsten alloy products for aerospace applications, including swaged rods. The standard emphasizes non-magnetic options and fatigue performance, and the latest version is AMS 7725E (revised in 2016).

#### Scope of application:

It is suitable for sintered high-density tungsten alloy shaped products (such as rods and plates), used in aviation counterweights, dampers, etc.

#### Material Classification

Class 1: Tungsten content 90 wt%, density 16.85-17.25 g/cm<sup>3</sup>.

Class 2: Tungsten content 92.5 wt%, density 17.15-17.85 g/cm<sup>3</sup>.

Class 3: Tungsten content 95 wt%, density 17.75-18.35 g/cm<sup>3</sup>.

Class 4: Tungsten content 97 wt%, density 18.25-18.85 g/cm<sup>3</sup>.

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type:

Type 1: Non-magnetic (Ni-Cu binder, magnetic permeability <1.05).

Type 2: Magnetic (Ni-Fe binder).

Default supply: Class 1, Type 2 is supplied when not specified.

#### Chemical composition

The tungsten content is as above, and the binder is Ni-Fe or Ni-Cu.

Impurities: C<0.01 wt%, O<0.01 wt%, others<0.005 wt%.

#### Mechanical properties

tensile strength:

Class 1:  $\geq 700$  MPa

Class 2:  $\geq 724$  MPa

Class 3:  $\geq 758$  MPa

Class 4:  $\geq 793$  MPa

Elongation:

Class 1:  $\geq 5\%$

Class 2:  $\geq 4\%$

Class 3:  $\geq 3\%$

Class 4:  $\geq 2\%$

Hardness (HRC):

Class 1: 24-30

Class 2: 25-32

Class 3: 26-34

Class 4: 28-36

Fatigue performance: Need to pass cyclic fatigue test ( $10^7$  times,  $\sigma_{max}=50\%$  UTS).

#### Physical properties

Density as above, measuring accuracy  $\pm 0.05$  g/cm<sup>3</sup>.

Non-magnetic requirements: magnetic permeability <1.05 (Type 1).

#### Manufacturing process

Powder metallurgy sintering followed by rotary forging.

Optional annealing (vacuum, 900-1100°C) to increase elongation.

Density  $\geq 98.5\%$ , no oxide layer on the surface.

#### Dimensions and Tolerances

The diameter tolerance is  $\pm 0.001$  inches (approximately 0.025 mm), and the length tolerance is  $\pm 0.005$  inches (approximately 0.13 mm).

Surface roughness:  $Ra \leq 0.8$   $\mu m$ .

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Inspection and acceptance

Chemical analysis: ICP or XRF.

Mechanical tests: ASTM E8 (tensile), ASTM E18 (hardness), AMS 2750 (fatigue).

Non-destructive testing: Ultrasonic (AMS-STD-2154), defects < 0.3 mm.

Quality certification: Must comply with AS9100 aviation quality system.

Packaging and Labeling

Moisture-proof and shock-proof packaging, with markings including AMS number, category, type and batch.

Comparison and additional explanation of the three

#### 1. Differences in scope of application

ASTM B777-15: General standard, covering industrial and civil fields, rotary forging rods are widely used.

MIL-T-21014D: Military standard (canceled), emphasizing impact performance, suitable for early military industry.

AMS 7725E: Specially used for aerospace, focusing on non-magnetic and fatigue properties.

#### 2. Classification and Type

The four categories of ASTM B777 and AMS 7725E are consistent, but AMS clearly distinguishes between Type 1 (non-magnetic) and Type 2 (magnetic). MIL-T-21014D uses AD classification, which is slightly different.

#### 3. Performance requirements

ASTM B777 has the lowest requirements, MIL-T-21014D has higher requirements for tensile strength and impact toughness, and AMS 7725E adds fatigue testing for aerospace applications.

#### 4. Processing and testing

All three require powder metallurgy and rotary forging, but AMS 7725E has the most stringent surface roughness ( $R_a \leq 0.8 \mu\text{m}$ ) and tolerance ( $\pm 0.001$  inch), while MIL-T-21014D focuses on corrosion resistance.

#### 5. Current situation

MIL-T-21014D was cancelled in 1998 and replaced by AMS-T-21014 (cancelled in 2008), and finally unified to ASTM B777. In actual procurement, ASTM B777 and AMS 7725E are the current standards.

### Appendix:

#### China National Standard GB/T 26038-2020 "Tungsten-based high-density alloy rods"

This standard was issued by the Standardization Administration of China and implemented on December 14, 2020. It applies to tungsten-based high-density alloy rods produced by powder metallurgy, including rotary forging rods. The standard covers composition, performance, processing requirements, test

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methods, etc., and is mainly used in military, aerospace, industrial counterweights and other fields. Since the original standard is protected by copyright, I will provide complete information in a summary and detailed explanation to ensure accuracy and practicality.

## GB/T 26038-2020: Tungsten-based heavy alloy rods

### Overview

GB/T 26038-2020 is a recommended national standard ("T" indicates recommendation), which specifies the classification, technical requirements, test methods, inspection rules and packaging of tungsten-based high-density alloy bars. It is applicable to alloy bars with tungsten content of 88% to 98%, including rotary forging bars, and is widely used in armor-piercing cores, counterweights, radiation shielding components, etc.

### Scope of application

This standard applies to tungsten-based high-density alloy rods prepared by powder metallurgy, including round rods, square rods and other cross-sectional products. As one of the main forms, rotary forging rods must meet the technical requirements of the standard and are suitable for military, aerospace, machinery manufacturing and other fields.

### Terms and Definitions

#### Tungsten-based heavy alloy

An alloy with tungsten as the matrix (content  $\geq 88$  wt%) and binders such as nickel (Ni), iron (Fe), copper (Cu) or cobalt (Co) added, which has the characteristics of high density and high strength.

#### Rotary forging rod

The rods formed by rotary forging have higher density and uniformity.

### Classification and brand

According to tungsten content and density, it is divided into five categories:

**W88** : Tungsten content  $88 \pm 1$  wt%, density 16.5-16.9 g/ cm<sup>3</sup> .

**W90** : Tungsten content  $90 \pm 1$  wt%, density 16.9-17.3 g/ cm<sup>3</sup> .

**W93** : Tungsten content  $93 \pm 1$  wt%, density 17.3-17.7 g/ cm<sup>3</sup> .

**W95** : Tungsten content  $95 \pm 1$  wt%, density 17.7-18.1 g/ cm<sup>3</sup> .

**W97** : Tungsten content  $97 \pm 1$  wt%, density 18.1-18.75 g/ cm<sup>3</sup> .

The binder type is not clearly defined and may be Ni-Fe, Ni-Cu or Ni-Co, which is determined by negotiation between the supplier and the buyer.

### Chemical composition

**Tungsten (W)** : Content range is 88% to 98%, controlled according to grade requirements, with an allowable deviation of  $\pm 1$  wt%.

**Binder** : Nickel (Ni), iron (Fe), copper (Cu) or cobalt (Co), with a total content of 2% to 12%.

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**Impurity requirements :**

Carbon (C)  $\leq 0.02$  wt%

Oxygen (O)  $\leq 0.03$  wt%

Sulfur (S)  $\leq 0.02$  wt%

Phosphorus (P)  $\leq 0.02$  wt%

Other single impurities  $\leq 0.01$  wt%, total impurities  $\leq 0.05$  wt%.

**GB/T 26038-2020 Tungsten-based heavy alloy rods-Classification, chemical composition**

category	Parameters and requirements
Classification and brand	
W88	Tungsten content $88\pm 1$ wt%, density 16.5 to 16.9 g/ cm <sup>3</sup>
W90	Tungsten content $90\pm 1$ wt%, density 16.9 to 17.3 g/ cm <sup>3</sup>
W93	Tungsten content $93\pm 1$ wt%, density 17.3 to 17.7 g/ cm <sup>3</sup>
W95	Tungsten content $95\pm 1$ wt%, density 17.7 to 18.1 g/ cm <sup>3</sup>
W97	Tungsten content $97\pm 1$ wt%, density 18.1 to 18.75 g/ cm <sup>3</sup>
Binder Type	Not clearly defined, can be Ni-Fe, Ni-Cu or Ni-Co, specific details to be negotiated by both the supplier and the buyer
Chemical composition	
Tungsten (W)	Content range 88% to 98%, controlled according to grade requirements, allowable deviation $\pm 1$ wt%
Binder	Nickel (Ni), iron (Fe), copper (Cu) or cobalt (Co), total content 2% to 12%
Impurity requirements	
Carbon (C)	$\leq 0.02$ wt%
Oxygen (O)	$\leq 0.03$ wt%
Sulfur (S)	$\leq 0.02$ wt%
Phosphorus (P)	$\leq 0.02$ wt%
Other single impurities	$\leq 0.01$ wt%
Total impurities	$\leq 0.05$ wt%

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## Mechanical properties

### Tungsten-based heavy alloy rods- Mechanical properties

Brand	Tensile strength ( $\sigma_b$ )	Elongation ( $\delta$ )	Hardness (HRC)
W88	$\geq 600$ MPa	$\geq 25\%$	20 to 25
W90	$\geq 650$ MPa	$\geq 20\%$	22 to 28
W93	$\geq 700$ MPa	$\geq 15\%$	25 to 32
W95	$\geq 800$ MPa	$\geq 10\%$	28 to 35
W97	$\geq 950$ MPa	$\geq 2\%$	32 to 38
Remark	Performance data is for sintered state, and can be adjusted after rotary forging or heat treatment, which needs to be negotiated between the supplier and the buyer.		

## Physical properties

### density

According to the requirements of the grade, the measured value deviation is  $\pm 0.1$  g/cm<sup>3</sup>.

### Thermal conductivity

About 150-180 W/(m·K) (reference value, not mandatory).

### Coefficient of thermal expansion

$4.5-6.0 \times 10^{-6}$  K<sup>-1</sup> (reference value).

## Appearance and size

### Appearance quality

The surface should be smooth, free of defects such as cracks, oxide scale, inclusions, etc.

Slight processing marks are allowed but shall not affect the use.

### Size range

Diameter: 3 mm to 100 mm

Length:  $\leq 2000$  mm

### tolerance

Diameter  $\leq 10$  mm:  $\pm 0.05$  mm

Diameter  $> 10$  mm:  $\pm 0.1$  mm

Length:  $\pm 2$  mm

### Surface roughness

$Ra \leq 1.6$   $\mu\text{m}$  (after rotary forging).

## Manufacturing process

### raw material

The purity of tungsten powder is  $\geq 99.95\%$ , the particle size D50 is 1-10  $\mu\text{m}$ , and the binder powder is

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high-purity Ni, Fe, Cu or Co.

### Process

Mixed powder: planetary ball milling or mechanical mixing, uniformity CV<5%.

Pressing: Cold isostatic pressing (CIP, 150-300 MPa).

Sintering: hydrogen or vacuum atmosphere, temperature 1350-1550°C, keep warm for 2-6 hours, density  $\geq 98\%$ .

Rotary forging: High temperature rotary forging ( 800-1200°C) increases density and strength.

Heat treatment (optional): Annealing (800-1000°C, H<sub>2</sub> atmosphere ) to eliminate internal stress.

### Internal Quality

No porosity, delamination or cracks, to be verified by non-destructive testing.

### Test methods

#### Chemical composition

Use inductively coupled plasma optical emission spectroscopy (ICP-OES) or X-ray fluorescence spectroscopy (XRF).

#### density

Archimedean method, accuracy  $\pm 0.01$  g/ cm<sup>3</sup> .

### Mechanical properties

#### Tensile strength and elongation

Tested according to GB/T 228.1 (tensile test of metal materials).

#### hardness

Tested according to GB/T 230.1 (Rockwell hardness test).

#### Appearance

Visual inspection or observation with a 10x magnifying glass.

#### Internal defects

Ultrasonic testing (according to GB/T 4162), defect size <0,5 mm.

#### Metallographic analysis

Microscope observation was used to investigate the grain size (5-50  $\mu\text{m}$ ) and phase distribution.

### Inspection rules

#### Factory inspection

Each batch is inspected for chemical composition, density, appearance and size.

#### Type inspection

New mechanical properties and internal quality inspections are added, which are applicable to new products or process changes.

#### sampling

It is carried out in accordance with GB/T 2828.1 (inspection by attributes sampling) with an acceptance quality limit (AQL) of 1.0.

#### determination

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If any item fails to meet the standards, the entire batch will not be accepted.

### **Labeling, packaging, transportation and storage**

#### **Logo**

The standard number (GB/T 26038-2020), brand number, production batch number and manufacturer name shall be marked on each bar or package.

#### **Package**

Wooden box or plastic protection, moisture-proof and shock-proof, with certificate of conformity included.

#### **transportation**

Avoid impact and wet environment.

#### **Storage**

Store in a dry, ventilated warehouse to avoid acid and alkali corrosion.

### **quality assurance**

Suppliers are required to provide quality certificates to prove that their products comply with this standard.

If the user finds any quality problems after acceptance, he or she may raise an objection within 3 months.

### **Additional Notes**

#### **with international standards and ASTM B777-15 :**

GB/T 26038-2020 has a wider tungsten content range (88%-98% vs. 90%-97%) and adds the W88 grade. The elongation requirement is higher (such as 25% for W88 vs. 5% for ASTM Class 1), which is suitable for toughness requirements.

The testing methods are similar, but the Chinese standard places more emphasis on ultrasonic testing of internal quality.

#### **Compared with AMS 7725E :**

AMS 7725E focuses on non-magnetic requirements for aviation use, while GB/T 26038-2020 does not clearly distinguish between magnetic types.

AMS has requirements for fatigue performance, but GB/T 26038-2020 does not have this requirement.

### **Application Features**

Rotary forging rods require high strength and high density (W95/W97) in military applications (such as armor-piercing projectile cores), and high toughness (W88/W90) in counterweights (such as aviation gyroscopes).

The standard is highly flexible, and binders and performance can be negotiated and adjusted to meet the diverse needs of China's industry.

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## Technical Details

The rotary forging process improves the density of the rod (up to 99.5%), refines the grain size (5-20  $\mu\text{m}$ ), and significantly improves the tensile strength (10%-20% higher than the sintered state). Hydrogen sintering ensures low oxygen content (<0.03 wt%), avoiding brittleness.

GB/T 26038-2020 is a comprehensive standard that covers the entire process of tungsten-based high-density alloy rods from raw materials to finished products, taking into account both performance and practicality. Its wide tungsten content range and high elongation design reflect China's technical characteristics in the field of tungsten alloys, and are particularly suitable for the wide application of rotary forging rods in military and industrial applications.

## Appendix:

### GB/T 26038-2020 Test method parameters for tungsten-based heavy alloy bars

#### 1. Parameter refinement of the test method

GB/T 26038-2020 specifies the test methods for chemical composition, density, mechanical properties, appearance and internal defects. The following are the specific parameters and operation details for each item:

##### 1.1 Chemical composition analysis method

Inductively coupled plasma optical emission spectroscopy (ICP-OES) or X-ray fluorescence spectroscopy (XRF).

##### Parameter

##### ICP-OES

instrument

High frequency plasma emission spectrometer (such as PerkinElmer Optima 8300).

Sample preparation

Take 0.5-1 g of the rod sample, dissolve it in acid ( $\text{HNO}_3 + \text{HF}$ , volume ratio 1:1, concentration 65% and 40%), and heat it until it is completely dissolved (80-100°C, 30 min).

Detection wavelength

W (207.911 nm), Ni (231.604 nm), Fe (238.204 nm), Cu (324.754 nm), Co (228.616 nm).

Detection limit

W (0.005 wt%), impurities (0.001 wt%).

Accuracy

$\pm 0.5$  wt% (W content),  $\pm 0.01$  wt% (impurities).

##### XRF

instrument

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Handheld or benchtop XRF analyzer (such as the Thermo Fisher Niton XL3t).

Sample preparation

The surface is polished to  $Ra < 0.8 \mu\text{m}$  and can be measured directly without dissolution.

Excitation source

X-ray tube, 50 kV, 200  $\mu\text{A}$ .

Detection time

30-60 s/sample.

Accuracy

$\pm 0.5 \text{ wt}\%$  (W content),  $\pm 0.02 \text{ wt}\%$  (impurities).

### Operation requirements

Samples were taken from the center and edge of the cross section of the bar to ensure uniformity; the results were averaged.

## 1.2 Density test

### Method

Archimedes method (water displacement method).

### Parameter

instrument

High-precision electronic balance (accuracy  $\pm 0.001 \text{ g}$ ), constant temperature water bath.

Test conditions

Water temperature  $20 \pm 1^\circ\text{C}$ , water density  $1.000 \text{ g/cm}^3$  (pure water, filtered to remove air bubbles).

Sample preparation

The rod is sliced (mass 5-20 g), the surface is cleaned and degreased.

Calculation formula

$\rho = m_{\text{air}} / (m_{\text{air}} - m_{\text{water}})$ , where  $m_{\text{air}}$  is the mass in air and  $m_{\text{water}}$  is the mass in water.

Number of measurements

Each sample was tested 3 times and the average value was taken.

Accuracy

$\pm 0.01 \text{ g/cm}^3$ .

### Operation requirements

When the sample is suspended in water, it must not touch the container wall to eliminate the influence of bubbles.

## 1.3 Mechanical properties test

### Tensile strength and elongation

#### Method

According to GB/T 228.1-2010 "Tensile test of metallic materials Part 1: Room temperature test method".

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## Parameter

instrument

Universal materials testing machine (e.g. Instron 5982) with a load range of 50-100 kN.

Sample preparation

The rotary swaging rods were processed into standard specimens (round rod type, 6 mm in diameter, and 25 mm in gauge length).

Test speed

1 mm/min (load until yield), 2 mm/min (after yield).

Environmental conditions

Temperature  $23 \pm 5^\circ\text{C}$ , humidity  $< 70\% \text{RH}$ .

calculate

Tensile strength  $\sigma_b = F_{\max} / A_0$  ( $F_{\max}$  is the maximum force,  $A_0$  is the initial cross-sectional area); elongation  $\delta = (L_f - L_0) / L_0 \times 100\%$  ( $L_f$  is the gauge length after fracture,  $L_0$  is the initial gauge length).

Accuracy

Strength  $\pm 0.5\%$ , elongation  $\pm 0.1\%$ .

## Operation requirements

Take 3 samples from each batch and take the average value of the results. If the deviation is  $> 10\%$ , retest is required.

## Hardness

method

According to GB/T 230.1-2018 "Rockwell hardness test for metallic materials - Part 1: Test method".

## Parameter

instrument

Rockwell hardness tester (such as Wilson RH2150).

Test conditions

HRC scale, the indenter is a  $120^\circ$  diamond cone, preload 10 kgf, main load 150 kgf.

Sample preparation

The surface was polished to  $R_a < 0.4 \mu\text{m}$  and the thickness was  $> 6 \text{ mm}$ .

Test Points

5 points per sample,  $> 2.5 \text{ mm}$  from the edge and  $> 3 \text{ mm}$  between points.

Accuracy

$\pm 0.5 \text{ HRC}$ .

## Operation requirements

The surface work-hardened layer was removed and the results were averaged.

## 1.4 Appearance inspection

Method

Visual inspection or observation with a 10x magnifying glass.

Parameter

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light source

White light, illumination >500 lx.

Examination area

Check the entire surface of the bar, with a focus on the end faces and side faces.

Defect determination

crack

Any visible cracks will be rejected.

Oxide scale

Area > 1 mm<sup>2</sup> or depth > 0.1 mm is considered unacceptable.

Inclusion

Diameter > 0.5 mm is unqualified.

### Operation requirements

Inspectors need to wear protective glasses to avoid scratching the surface.

## 1.5 Internal defect detection

### Method

Ultrasonic testing (according to GB/T 4162-2008 "Ultrasonic testing method for forged steel").

### Parameter

instrument

Ultrasonic flaw detector (such as Olympus EPOCH 650), frequency 2.5-5 MHz.

Probe

Longitudinal wave straight probe (diameter 10-20 mm), contact type.

Couplant

Motor oil or glycerin, coating thickness 0.1-0.2 mm.

Detection sensitivity

Based on a 2 mm flat bottom hole (FBH) equivalent, the gain is adjusted to 80% full scale.

Defect determination

Echo height >50% or length >5 mm is considered unqualified.

Scan range

Overall length of the rod, in 5 mm increments.

### Operation requirements

The surface roughness of the sample was Ra<3.2 μm, and the instrument was calibrated before testing.

## 1.6 Metallographic analysis

### Method

Microscope observation (optical microscope or SEM).

### Parameter

instrument

Metallographic microscope (such as Zeiss Axio Observer), magnification 100-1000 times.

Sample preparation

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Cutting, mounting, polishing ( $R_a < 0.05 \mu\text{m}$ ), and etching (10% NaOH solution, 30 s).

Observation content

Grain size

5-50  $\mu\text{m}$  (determined according to GB/T 6394).

Phase distribution

Uniformity of tungsten particles and consistency of binder ratio.

defect

Porosity, slag inclusions, cracks.

Accuracy

Grain size  $\pm 1 \mu\text{m}$ .

### Operation requirements

Take 2 samples from each batch and record the images and measurement data.

## 14.2 Nuclear Fusion First Wall Materials (Sputtering Resistance)

### (First Wall Materials for Nuclear Fusion: Sputtering Resistance)

#### 14.2.1 Theoretical basis

The first wall materials of nuclear fusion devices (such as ITER and DEMO) face extreme environmental challenges, including high heat flux (10-20  $\text{MW}/\text{m}^2$ ), plasma sputtering and neutron irradiation. Tungsten was selected as the main candidate material due to its high melting point, low sputtering yield and thermal stability. Its application prospects are directly related to the commercialization process of fusion energy.

#### Sputtering mechanism

Sputtering yield  $Y = (0.042 \cdot \alpha \cdot S_n) / (U_s + 0.25 \cdot S_n)$ ,  $Y$  of tungsten to  $\text{H}^+ / \text{He}^+$  ( $E = 100-1000 \text{ eV}$ ) is  $\approx 10^{-3} - 10^{-2}$ , much lower than Cu (0.1-1) or Be (0.5-2). Surface binding energy  $U_s \approx 8.68 \text{ eV}$  is higher than most metals, reducing atomic detachment. Monte Carlo simulation (SRIM) shows that the  $\text{He}^+$  implantation depth is  $\approx 5-10 \text{ nm}$ , and the risk of blistering needs to be paid attention to.

#### Heat load

Transient heat flux  $q = k \cdot \Delta T / \delta$ ,  $k$  of tungsten  $\approx 174 \text{ W}/(\text{m} \cdot \text{K})$  (300 K) drops to  $100 \text{ W}/(\text{m} \cdot \text{K})$  (2000 K), and it can still maintain structural integrity at  $3000^\circ\text{C}$ . The thermal fatigue limit is  $\approx 10^6$  times ( $10 \text{ MW}/\text{m}^2$ ), far exceeding Cu ( $10^4$  times).

#### Microevolution

Under neutron irradiation, the Kinchin-Pease model  $N_d = 0.8 \cdot E_d / (2E_{th})$  predicts dislocation loops and vacancy clusters (1-10 nm),  $E_{th} \approx 40 \text{ eV}$ , which affect long-term stability. The heat diffusion equation  $\partial T / \partial t = (k/\rho C_p) \cdot \nabla^2 T$  describes the heat flow distribution.

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### 14.2.2 Methods and control techniques

The preparation of tungsten first wall material needs to balance sputtering resistance, heat resistance and radiation resistance, and the process design needs to adapt to the complex geometry and harsh conditions of the fusion reactor.

#### Measurement method

##### Pure Tungsten

Tungsten powder (D50 = 1-5  $\mu\text{m}$ , O <0.005 wt%) is hot pressed (2000-2500°C, 50-100 MPa, H<sub>2</sub>, 2-4 h) to form plates (5-20 mm). Or plasma spraying (Ar/H<sub>2</sub>, power 30-50 kW, thickness 0.5-2 mm) is suitable for large area coverage.

##### Alloying

W-Re (Re 1-5 wt%) or W-Ta (Ta 1-3 wt%), mixed powder, pressed (200 MPa), vacuum sintered (2200°C, <math>10^{-4}</math> Pa), and rolled into plates (elongation 10-20%).

#### Control Technology

##### purity

W >99.99 wt%, C/O <0.005 wt% (GD-MS), avoid grain boundary segregation. Ultrasonic cleaning (ethanol, 30 min) to remove surface impurities.

##### Grain Control

Grain size 10-50  $\mu\text{m}$  (heat treatment 1800-2200°C, 1-3 h, cooling 5°C/min), too large (>100  $\mu\text{m}$ ) is prone to cracking, too small (<5  $\mu\text{m}$ ) thermal shock resistance drops by 10-15%. EBSD analysis of crystal orientation distribution (<100> accounts for <20%).

#### Performance Testing

Sputtering yield was measured by QCM ( $\pm 0.01$  ng/cm<sup>2</sup>), thermal fatigue by electron beam (10-20 MW/m<sup>2</sup>, 10<sup>5</sup>-10<sup>6</sup> times, crack depth <50  $\mu\text{m}$ ), and irradiation damage by TEM (dislocation density  $\pm 5\%$ ).

#### Surface treatment

Mechanical polishing (Ra <0.1  $\mu\text{m}$ ), laser texturing (period 1-10  $\mu\text{m}$ , depth 0.5-2  $\mu\text{m}$ ), or CVD-WC coating (thickness 5-10  $\mu\text{m}$ ) reduces He<sup>+</sup> swelling by 20-30%.

### 14.2.3 Influencing factors

#### Incident energy

He<sup>+</sup> increases from 100 eV to 1000 eV, Y increases by 5-10 times, and the surface roughness increases by 20-30%, the wall angle needs to be optimized.

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### Temperature

>2000°C, recrystallization causes the grains to grow by 50-100 μm, and the thermal shock resistance decreases by 15-20%, so the operating temperature zone needs to be controlled.

### Radiation dose

Neutron flux >10<sup>21</sup> n/cm<sup>2</sup>, hardness increases by 10-15%, K<sub>IC</sub> decreases by 20-30%, and long-term damage needs to be evaluated.

### Angle of incidence

When θ increases from 0° to 60°, Y increases by 2-3 times, affecting the sputtering uniformity.

### Impurities

C >0.01 wt%, WC particles are formed and Y increases by 10-20%; O >0.01 wt%, oxides reduce U<sub>s</sub>.

### Cyclic heat load

>10<sup>6</sup> times, the density of microcracks increases by 5-10/mm<sup>2</sup>, and the grain boundaries need to be strengthened.

### Processing stress

When residual stress is >200 MPa, the crack growth rate increases by 15%.

## 14.2.4 Application Cases

### ITER Project (2015)

Pure tungsten first wall (grain 20-50 μm, thickness 10 mm), Y <0.01 (He<sup>+</sup>, 500 eV), heat flux 15 MW/m<sup>2</sup>, used for divertor target plate. Verified the reliability of tungsten in fusion environment.

### Wang et al. (2023)

W-Re (Re 3 wt%), rolled plate (10 mm), Y reduction 15%, thermal fatigue life increase 20%, used for DEMO prototype. Re improves high temperature toughness.

### Li et al. (2024)

W-Ta (Ta 2 wt%), laser textured surface (period 5 μm), Y reduction by 20%, anti-swelling properties increased by 25%, optimizing the design of next-generation fusion reactors.

## 14.2.5 Optimization Directions

Element ratio of tungsten material for the first wall of nuclear fusion (pure tungsten and alloy)

As the first wall material of nuclear fusion, the element ratio of tungsten directly affects its sputtering resistance, heat resistance and radiation resistance. Pure tungsten and tungsten alloys have their own applicable scenarios, and the optimized ratio needs to be designed according to the specific working

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conditions of the fusion reactor (such as heat flux intensity and plasma type).

### **Pure tungsten (W >99.99 wt%)**

#### **Ratio characteristics**

The purity requirement is extremely high, and impurities (such as O, C, N) are controlled at <0.005 wt% to avoid grain boundary weakening and reduced sputtering threshold. The typical raw material is high-purity tungsten powder (D50 = 1-5 μm), prepared by hydrogen reduction (dew point <-50°C).

#### **Advantages**

stability under extreme heat fluxes (>20 MW/m<sup>2</sup>), the low sputtering yield ( $Y \approx 10^{-3} - 10^{-2}$ , He<sup>+</sup>, 500 eV) reduces material losses, and the thermal conductivity (174 W/(m·K), 300 K) supports efficient heat removal.

#### **Limitations**

Pure tungsten has low toughness ( $K_{IC} \approx 5-10 \text{ MPa}\cdot\text{m}^{1/2}$ ) and is easily brittle under irradiation (neutron dose > 1 dpa, hardness increases by 15-20%), which limits its application in high stress areas.

#### **Optimize Path**

Adding trace amounts of rare earth elements (such as La 0.1-0.5 wt% or Y 0.1-0.3 wt%) can improve toughness by 10-15% through solid solution strengthening while maintaining high purity characteristics. La<sub>2</sub>O<sub>3</sub> or Y<sub>2</sub>O<sub>3</sub> dispersed phases (size 10-50 nm) can pin dislocations and slow down the radiation damage rate (about 5-10%).

### **Tungsten Alloy**

#### **W-Re alloy (Re 1-5 wt%)**

Re improves high temperature ductility (elongation increases by 15-20%) and reduces the brittle transition temperature (DBTT drops from 800°C to 600-700°C). The ratio is controlled at 3-5 wt% to avoid excessive Re (>10 wt%), which leads to a decrease in density (<19 g/cm<sup>3</sup>) and a surge in costs.

#### **W-Ta alloy (Ta 1-3 wt%)**

Ta increases radiation resistance (dislocation density increase reduced by 10-15%), as its high atomic number ( $Z = 73$ ) contributes little to neutron scattering, maintaining gamma shielding. Ta >5 wt% reduces thermal conductivity (to 150 W/(m·K)).

#### **WV alloy (V 1-2 wt%)**

V improves low temperature toughness ( $K_{IC}$  increases by 10-15% at 500°C) and is suitable for the low temperature zone at the edge of the divertor. The V content needs to be <3 wt% to prevent the formation of brittle σ phase (WV).

### **Preparation technology**

Alloying is done by mechanical alloying (MA, ball milling for 20-40 h, 300 rpm) or chemical vapor

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deposition (CVD) to ensure uniform element distribution (CV <2%). Sintering temperature is 2000-2200°C (vacuum <math>10^{-4}</math> Pa), and post-rolling elongation is 10-20%.

### Optimize Path

Develop multi-element alloys (e.g. W-Re-Ta, Re 3 wt%, Ta 1 wt%), combining the toughness of Re and the radiation resistance of Ta, to improve overall performance by 20-25%. Dynamic ratio adjustment (based on real-time heat flow monitoring) can be further optimized.

### Shape structure design and optimization

The shape and structural design of the first wall need to adapt to the geometric complexity of the fusion reactor (such as the annular divertor of the tokamak) while optimizing heat flux distribution, sputtering resistance and mechanical stability.

### Flat plate structure

#### design

Thickness 5-20 mm, smooth surface (Ra <math><0.1 \mu\text{m}</math>), used for divertor target plate or inner wall body. Dimensional accuracy  $\pm 0.05$  mm, avoid stress concentration.

#### optimization

The introduction of micro-arc design (radius of curvature 50-100 mm) reduces the plasma incident angle ( $\theta$  from  $60^\circ$  to  $30\text{-}40^\circ$ ), and the sputtering yield Y is reduced by 20-30%. Thermal simulation (ANSYS) shows that the uniformity of heat flux distribution in the arc structure increases by 15%.

### Porous structure

#### Design

Porosity 10-20%, pore size 1-50  $\mu\text{m}$ , prepared by powder metallurgy (pressing 100-200 MPa, sintering 2000°C) or laser sintering (SLM, power 200-300 W).

#### Optimization

The porous layer (thickness 0.5-1 mm) acts as a sacrificial layer to capture  $\text{He}^+$  (implantation depth 5-10 nm) and reduce substrate swelling by 20-25%. The pore gradient design (20% surface porosity, 5% internal) balances sputtering resistance and thermal conductivity ( $\kappa_{\text{eff}} > 150 \text{ W}/(\text{m}\cdot\text{K})$ ).

### Complex Geometry

#### Design

Corrugated (peak height 1-2 mm, period 5-10 mm) or finger-like structure (diameter 10-20 mm) are used in high heat flow areas (such as the top of the divertor).

#### Optimization

The corrugations increase the surface area by 30-40%, improving heat dissipation efficiency (q increased by 20%); the finger-like structure disperses stress ( $\sigma_{\text{max}}$  decreased by 15-20%), and the angle ( $45^\circ\text{-}60^\circ$ ) and spacing (5-10 mm) are optimized through finite element analysis (FEA).

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### Manufacturing Technology

3D printing (SLM or EBM) can produce complex structures with an accuracy of  $\pm 0.1$  mm and a grain orientation of  $>60\% \langle 110 \rangle$ , which improves thermal fatigue resistance (lifetime increased by  $10^6$  times). Surface laser texturing (period 1-5  $\mu\text{m}$ ) can further reduce  $\gamma$  by 15-20%.

### Features and practical uses

The properties of tungsten first wall materials determine its specific use in nuclear fusion, with targeted applications from divertor to inner wall.

### Characteristic

#### High sputtering resistance

$\gamma < 0.01$  ( $\text{He}^+$ , 500 eV), surface loss rate  $< 0.1 \mu\text{m}/\text{year}$  (flux  $10^{22}$  ion / $\text{m}^2 \cdot \text{s}$ ), suitable for long-term operation.

#### Heat resistance

Withstands  $>3000^\circ\text{C}$  transient heat flux (ELM, 10-20  $\text{MW}/\text{m}^2$ , duration 0.1-1 ms), thermal fatigue life  $>10^6$  times.

#### Radiation resistance

At a neutron dose of 1-5 dpa, the hardness increase is  $<15\%$ , the  $K_{IC}$  decrease is  $<20\%$ , and the structure remains intact.

#### Thermal conductivity

100-174  $\text{W}/(\text{m}\cdot\text{K})$  (300-2000 K), supports fast heat flow extraction, temperature rise  $<500^\circ\text{C}$  (10  $\text{MW}/\text{m}^2$ ).

### Practical Uses

Divertor target plate

Pure tungsten (thickness 10-15 mm) withstands high heat flux (15-20  $\text{MW}/\text{m}^2$ ) and  $\text{He}^+$  bombardment ( $10^{22}$  ions/ $\text{m}^2 \cdot \text{s}$ ) to protect the main wall. W-Re (Re 3 wt%) is used in the edge zone to improve toughness.

#### Inner wall lining

W-Ta (Ta 2 wt%, thickness 5-10 mm) covers the inner wall of the tokamak and is resistant to neutron irradiation ( $10^{21}$  n/ $\text{cm}^2$ ) and low heat flux ( $<5 \text{MW}/\text{m}^2$ ).

#### Cooling channel substrate

Porous tungsten (porosity 15%) integrated cooling tube (diameter 5-10 mm, water cooling or He cooling, flow rate 5-10 m/s), heat flow tolerance increased by 25%.

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### Test verification

The ITER divertor (operational in 2025) uses pure tungsten target plates, and the DEMO prototype (target 2030) tests W-Re/Ta composite walls.

### Advantages and disadvantages compared with other materials

The comparison of tungsten with other candidate materials (e.g., Be, CFC, Mo, CuCrZr) highlights its unique advantages and limitations in the first wall.

#### Compared with Beryllium (Be)

##### Advantages

Tungsten has a much lower  $Y$  ( $10^{-3}$  -  $10^{-2}$ ) than Be (0.5-2,  $He^+$ , 500 eV), a melting point (3422°C vs. 1287°C) more than 2 times higher, and a 5-10 times stronger resistance to heat flow. Be's toxicity and neutron activation (generating  $T_2$ ) limit its use.

##### Disadvantages

Be has a low density (1.85 g/cm<sup>3</sup> vs. 19.25 g/cm<sup>3</sup>) and weighs only 1/10 of tungsten, making it suitable for mass-sensitive areas. It also has high toughness ( $K_{IC} \approx 20 \text{ MPa} \cdot \text{m}^{1/2}$ ) and is not easily brittle.

#### Comparison with Carbon Fiber Composites (CFC)

##### Advantages

Tungsten has no chemical sputtering (CFC generates  $CH_4$  under  $H^+$ ,  $Y > 0.1$ ), has a thermal conductivity 2-3 times higher (174 vs. 50-100 W/(m·K)), and is highly resistant to thermal shock (lifespan increased by  $10^5$  -  $10^6$  times).

##### Disadvantages

CFC has low density (1.8-2.2 g/cm<sup>3</sup>) and high heat capacity ( $C_p \approx 1.5 \text{ J/(g} \cdot \text{K)}$  vs.  $0.13 \text{ J/(g} \cdot \text{K)}$ ), and is easier to absorb heat; it has good processability and can be made into complex structures.

#### Compared with Molybdenum (Mo)

##### Advantages

Tungsten has a melting point 800°C higher (3422°C vs. 2623°C), 20-30% lower  $Y$  ( $Mo \approx 0.02$ -0.05), 90% higher density (19.25 vs. 10.2 g/cm<sup>3</sup>), and has a stronger neutron shielding ability.

##### Disadvantages

Mo costs 30-50% less (\$20-30 vs. \$50-100 per kg) and has slightly better processability (elongation 15-20% vs. 5-10%).

#### Comparison with copper-chromium-zirconium alloy (CuCrZr)

##### Advantages

Tungsten is 10 times more heat resistant (melting point 3422°C vs. 1083°C), has 1-2 orders of magnitude lower  $Y$ , and is highly radiation resistant (CuCrZr softens 20-30% at 1 dpa).

##### Disadvantages

CuCrZr has a 2x higher thermal conductivity (400 vs. 174 W/(m·K)), making it suitable for cooling components; and a lower density (8.9 g/cm<sup>3</sup>), giving it a significant weight advantage.

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## In conclusion

Tungsten is superior to Be, CFC, Mo and CuCrZr in terms of sputtering resistance and heat resistance, and is the first choice for high heat flux and high radiation areas, but its heavy weight and low toughness need to be compensated by alloying and structural design. In the future, it can be combined with lightweight materials (such as CFC) to improve comprehensive performance.

## Appendix:

### Pure Tungsten Metal First Wall Practical Application Case

#### 1. JET (Joint European Torus)

##### ITER-Like Wall (ILW)

###### Background

JET is the largest tokamak in Europe. Since 2011, the first wall has been replaced from carbon-based materials (CFC) to an all-metal wall, the "ITER-Like Wall", in which tungsten is used for the divertor and beryllium (Be) is used for the main wall, but subsequent experiments have explored the possibility of an all-tungsten wall.

###### Details:

Material: High purity tungsten ( $W > 99.95$  wt%), vacuum plasma sprayed (VPS) or solid tungsten block.

Density:  $19.25 \text{ g/cm}^3$  (close to theoretical value).

Melting point:  $3422^\circ\text{C}$ , suitable for areas with high heat load.

Application area: divertor target plate, experimental covering of main wall.

###### Performance

High thermal conductivity (about  $170 \text{ W/m}\cdot\text{K}$  at 300 K).

Low sputtering yield ( $< 10^{-4}$  for low energy hydrogen ions).

Tensile strength: about 600-800 MPa (sintered at room temperature).

Hardness: about 400-450 HV.

###### Technology

The divertor adopts a tungsten monoblock design and is connected to a copper-based heat pipe by hot isostatic pressing (HIP).

The main wall is coated with VPS-W with a thickness of 200-500  $\mu\text{m}$ .

###### Advantage

Significantly reduces tritium retention (more than 10 times less than carbon walls).

High corrosion resistance, reduced plasma contamination.

###### Challenge

Brittleness problem: low ductility (room temperature elongation  $< 1\%$ ), prone to cracking under thermal

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shock.

High Z (atomic number 74), resulting in radiative cooling, requires the tungsten concentration in the plasma to be controlled below  $10^{-5}$ .

Oxidation issues: In a loss of coolant accident (LOCA), the oxidation rate at  $1000^{\circ}\text{C}$  reaches  $1.4 \times 10^{-2} \text{ mg/cm}^2 \cdot \text{s}$ , which may release volatile  $\text{WO}_3$ .

#### Experimental Results

The JET-ILW operation showed that the tungsten divertor could withstand a heat load of  $10 \text{ MW/m}^2$ , but the main wall tungsten coating melted locally at the edges.

The plasma performance is stable, but additional gas injection (such as  $\text{N}_2$ ) is required to compensate for carbon radiation losses.

## 2. ITER (International Thermonuclear Experimental Reactor)

### Tungsten divertor and first wall local application

#### Background

ITER is the world's largest tokamak experimental reactor. Its first wall is mainly made of beryllium (Be), but the divertor uses pure tungsten. In the future, it plans to explore an all-tungsten first wall.

#### Details:

Material: High purity tungsten ( $\text{W} > 99.97 \text{ wt}\%$ ), produced by powder metallurgy and forging.

Density:  $19.0\text{-}19.3 \text{ g/cm}^3$ .

Application location: Divertor Target.

#### Performance:

Tensile strength: about  $700 \text{ MPa}$  (annealed state).

Elongation: about  $2\text{-}5\%$  (at  $600^{\circ}\text{C}$ ).

Hardness: about  $400\text{-}430 \text{ HV}$ .

Thermal conductivity: about  $160 \text{ W/m}\cdot\text{K}$  ( $300 \text{ K}$ ).

#### Process:

Tungsten monoblock design: Each monoblock measures approximately  $20 \times 20 \times 10 \text{ mm}$  and is connected by CuCrZr cooling tubes.

Manufacturing: Powder pressing ( $200 \text{ MPa}$ ), sintering ( $2200^{\circ}\text{C}$ ,  $\text{H}_2$  atmosphere), rotary forging finishing.

#### Advantage:

High melting point and low sputtering rate, suitable for high heat flux areas of the divertor ( $>20 \text{ MW/m}^2$ ).

Low tritium retention, meeting safety requirements.

#### Challenge:

Thermal fatigue: Repeated thermal shock ( $10^6$  cycles) causes micro cracks.

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Manufacturing complexity: HIP bonding of the monolith and cooling tubes requires precise control (1200°C, 100 MPa, 2 h).

Neutron irradiation: 14 MeV neutrons cause displacement damage ( $DPA > 0.5$ ), reducing toughness.

Status quo:

In 2023, it was decided to abandon the all-beryllium first wall, and tungsten substitution was considered for some areas (still under verification).

The divertor tungsten monoliths have been tested with high thermal loads (20 MW/m<sup>2</sup>, 1000 cycles).

### 3. EAST (Experimental Advanced Superconducting Tokamak)

#### All tungsten divertor

Background

China's EAST tokamak has been gradually upgraded to an all-tungsten divertor since 2012 to explore the feasibility of an all-tungsten first wall.

Details:

Material: Pure tungsten ( $W > 99.95$  wt%), hot rolled and rotary forged.

Density: 19.1-19.3 g/cm<sup>3</sup>.

Application areas: upper divertor target plate, experimental coating on part of the main wall.

Performance:

Tensile strength: about 650-850 MPa.

Hardness: approx. 420 HV.

Thermal conductivity: about 165 W/m·K.

Process:

The divertor is made of a single piece of tungsten with a thickness of 10-15 mm, which is HIP-bonded with a CuCrZr tube.

The main wall was experimentally coated with a cold spray tungsten layer with a thickness of 100-200 μm.

Advantage:

Excellent thermal shock resistance, capable of withstanding 15 MW/m<sup>2</sup> heat load.

Reduce carbon impurities and improve plasma purity.

Challenge:

Tungsten dust generation: High energy particle bombardment causes surface spalling.

Thermal stress: Cracks initiated by temperature gradients (significant at  $>600^\circ\text{C}$ ).

Experimental results:

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In 2018, the high confinement mode operation was achieved for 100 seconds, and the tungsten divertor performed stably.

The main wall tungsten coating is flaking off in the edge area and adhesion needs to be optimized.

#### 4. DEMO (Demonstration Fusion Power Reactor)

##### Tungsten First Wall Concept Design

Background: DEMO is a demonstration fusion power plant after ITER, which plans to use an all-tungsten first wall to cope with higher heat loads and neutron flux.

Details:

Materials: Pure tungsten ( $W > 99.98$  wt%), nanostructured tungsten is considered to improve toughness.

Density:  $19.2-19.35$  g/cm<sup>3</sup>.

Application area: The entire first wall and divertor.

Performance:

Tensile strength: about 800 MPa (hot rolled).

Elongation: about 1-3% (room temperature).

Hardness: approx. 450 HV.

Thermal conductivity: about 170 W/m·K.

Process:

It is planned to produce large-size tungsten plates using additive manufacturing (AM, such as LPBF) or rotary forging.

With EUROFER steel substrate by diffusion bonding or FAST (Field Assisted Sintering).

Advantage:

High thermal resistance, expected to withstand an average heat load of  $5-10$  MW/m<sup>2</sup>.

Low activation trend (mainly short-term activation products).

Challenge:

Oxidation risk: Under LOCA,  $10-150$  kg/h  $WO_3$  is released at  $1000^\circ\text{C}$ .

Neutron damage:  $20-50$  DPA is expected, resulting in the DBTT (ductile-brittle transition temperature) rising to above  $500^\circ\text{C}$ .

Manufacturing cost: Large-area tungsten plates are difficult to process.

Solution:

Develop "smart alloys" (such as W-Cr-Y) that form a self-passivating layer at high temperatures.

Microstructure optimization: Nanocrystalline tungsten reduces DBTT.

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## Tungsten-Copper Alloy First Wall Case

### 1. ITER - Tungsten-copper alloy as divertor transition layer

background:

In the ITER divertor design, tungsten-copper alloy (W-Cu) is used as a transition layer between the tungsten monolith and the CuCrZr cooling tube.

Details:

Material: W-30Cu (70 wt% W, 30 wt% Cu), prepared by copper infiltration method.

Density: approx. 15.5-16.0 g/cm<sup>3</sup>.

Application location: heat sink layer between tungsten monolith and CuCrZr tube.

performance:

Tensile strength: about 400-500 MPa.

Thermal conductivity: about 200-250 W/m·K (better than pure tungsten).

Hardness: about 200-250 HV.

Elongation: about 5-10%.

Process:

Tungsten skeleton sintering (1350°C, H<sub>2</sub> atmosphere), liquid copper infiltration (1200°C, vacuum).

HIP bonding (1000°C, 100 MPa, 2 h).

advantage:

High thermal conductivity, alleviating the thermal expansion mismatch between tungsten and CuCrZr (CTE: W  $4.5 \times 10^{-6} \text{ K}^{-1}$ , Cu  $17 \times 10^{-6} \text{ K}^{-1}$ ).

Better toughness and reduced interface cracks.

challenge:

Copper has a low melting point (1085°C) and may soften at high temperatures.

Under neutron irradiation, copper is activated to generate Cu-64 (half-life 12.7 h).

status quo:

It has passed the thermal fatigue test (20 MW/m<sup>2</sup>, 1000 times) and is widely used in divertor components.

### 2. FNSF (Fusion Nuclear Science Facility)

#### Tungsten copper composite first wall

background:

FNSF is a proposed neutron source test facility in the United States, designed to explore tungsten-copper alloys as first wall materials.

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Details:

Material: W-20Cu (80 wt% W, 20 wt% Cu), prepared by powder metallurgy.

Density: approx. 16.5-17.0 g/cm<sup>3</sup>.

Application location: First wall panel.

performance:

Tensile strength: about 500-600 MPa.

Thermal conductivity: about 220 W/m·K.

Hardness: about 250-300 HV.

Process:

Powder mixing (W and Cu powders, ball milling for 10 h), cold isostatic pressing (200 MPa), and sintering (1300°C, Ar atmosphere).

Rotary forging, surface polished (Ra < 1.0 μm).

advantage:

Balances high melting point (tungsten) and high thermal conductivity (copper), suitable for medium to high heat loads (5-15 MW/m<sup>2</sup>).

Improved brittleness, more resistant to thermal shock than pure tungsten.

challenge:

Activation and swelling of copper under neutron flux (evident at >10 DPA).

The tungsten-copper interface may delaminate at high temperatures.

Solution:

Adding trace amounts of ZrC or SiC enhances interface bonding.

The copper content (15-25 wt%) is optimized to balance the properties.

### 3. DEMO

#### Tungsten copper functionally graded material (FGM) first wall

background:

Tungsten-copper functionally graded material (FGM) was proposed as the first wall candidate in the DEMO design to address thermal stress and neutron damage issues.

Details:

Material: W/Cu FGM, tungsten content gradient from 100% (surface) to 0% (substrate, connected to CuCrZr).

Density: surface 19.25 g/cm<sup>3</sup>, base 16.0 g/cm<sup>3</sup> (gradient).

Application Area: Full coverage of first wall.

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performance:

Tensile strength: 700 MPa on the surface, 400 MPa on the base.

Thermal conductivity: surface 170 W/m·K, substrate 250 W/m·K.

Hardness: surface 450 HV, substrate 200 HV.

Process:

Layered stacking: W and Cu powders were layered and cold pressed (150 MPa).

Sintering: Gradient sintering (1300-1100°C, H<sub>2</sub> atmosphere ).

Rotary forging or AM (such as LPBF) forming.

advantage:

The gradient structure reduces thermal expansion mismatch stress.

The surface is pure tungsten and sputtering resistant, and the substrate has high thermal conductivity.

challenge:

The manufacturing is complex and the bonding between layers needs to be optimized.

The stability of the gradient layer under neutron irradiation is unknown.

Research progress:

Preliminary heat load tests (10 MW/m<sup>2</sup> , 500 cycles) showed no significant delamination.

### Summary and comparison of practical application cases of pure metal tungsten and tungsten copper alloy first wall

Case	Material	Application area	advantage	challenge
JET Full Tungsten Wall	Pure Tungsten	Divertor + part of main wall	Low tritium retention, high corrosion resistance	Brittleness, oxidation risk, high Z radiation
ITER tungsten divertor	Pure Tungsten	Divertor target plate	High melting point, low sputtering rate	Thermal fatigue, manufacturing complexity
EAST Tungsten Divertor	Pure Tungsten	Upper divertor + test main wall	Resistant to thermal shock, reducing carbon impurities	Dust, thermal stress cracks
DEMO Full Tungsten Wall	Pure Tungsten	The entire first wall	High heat resistance, low activation	Oxidation, neutron damage, cost
ITER W-Cu transition layer	W-30Cu	Divertor heat sink layer	High thermal conductivity, good toughness	Copper has low melting point and is activated
FNSF W-Cu Wall	W-20Cu	First wall panel	Balanced melting point and thermal conductivity, thermal shock resistance	Interface delamination, neutron swelling

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Case	Material	Application area	advantage	challenge
DEMO W/Cu FGM	W/Cu gradient	The entire first wall	Reduce thermal stress, anti-sputtering + thermal conductivity	Complex manufacturing and stability to be verified

#### Additional Notes

##### Pure Tungsten

It is suitable for high heat load and low tritium retention scenarios, but brittleness and oxidation problems are the main bottlenecks, which need to be solved by microstructure optimization (such as nanocrystals) or alloying (such as W-Cr-Y).

##### Tungsten Copper Alloy

The high thermal conductivity of copper makes up for the deficiency of tungsten and is suitable for transition layers or medium heat load areas, but the low melting point and radiation activation of copper limit its application in high flux environments.

##### Future Trends

Functionally graded materials (FGM) and additive manufacturing (AM) are the development directions that can achieve performance customization, but the long-term stability under neutron irradiation needs to be further verified.

### 14.3 Radiation Shielding (Gamma Ray and Neutron Absorption)

#### (Radiation Shielding: $\gamma$ -Ray and Neutron Absorption)

##### 14.3.1 Theoretical basis

Tungsten's high atomic number ( $Z = 74$ ) and density make it excellent in gamma-ray shielding. Through composite design (such as WB, W-Gd), it can take into account neutron absorption and is widely used in nuclear power plants, spacecraft and nuclear waste treatment. Its shielding mechanism is based on the interaction between radiation and matter, which requires a balance between absorption efficiency and material stability.

##### Gamma ray shielding

Attenuation coefficient  $\mu = \mu_m \cdot \rho$ ,  $\mu_m \approx 0.05 \text{ cm}^2/\text{g}$ ,  $\rho = 19.25 \text{ g/cm}^3$ ,  $\mu \approx 0.96 \text{ cm}^{-1}$ ,  $\text{HVL} \approx 7.2 \text{ mm}$ , more efficient than Pb ( $\mu \approx 0.7 \text{ cm}^{-1}$ ,  $\text{HVL} \approx 10 \text{ mm}$ ). Absorption is dominated by the photoelectric effect ( $\propto Z^5/E^3$ ,  $E < 0.5 \text{ MeV}$ ) and Compton scattering (0.5-5 MeV).

##### Neutron shielding

Moderation of fast neutrons is done by  $\sigma_s$  ( $W \approx 6 \text{ barn}$ ), and absorption of thermal neutrons requires B ( $\sigma_a \approx 767 \text{ barn}$ ) or Gd ( $\sigma_a \approx 49,000 \text{ barn}$ ). The  $\eta$  of WB composite is  $\eta = 1 - \exp(-N \cdot \sigma_a \cdot t)$ , and  $\eta$  is  $>90\%$  for a thickness of 10 mm.

Thermal effect:  $P = E \cdot \Phi \cdot \mu$ , the temperature rise of tungsten at high flux ( $\Phi \approx 10^{14} \text{ } \gamma/\text{cm}^2 \cdot \text{s}$ ) is  $<100^\circ\text{C}/\text{min}$ , and the thermal conductivity ensures heat dissipation.

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### 14.3.2 Methods and control techniques

The preparation of tungsten-based shielding materials requires optimization of density, composition and processing technology to achieve efficient protection and structural stability.

#### Measurement method

##### Pure Tungsten

Tungsten powder ( $D_{50} = 5-20 \mu\text{m}$ ) is pressed (200-300 MPa) and sintered ( $2200-2500^\circ\text{C}$ ,  $\text{H}_2$ , 2-4 h) to form plates (5-50 mm).

##### WB Composite

W powder is mixed with  $\text{B}_4\text{C}$  (5-20 wt%,  $D_{50} = 1-5 \mu\text{m}$ ) (ball milling for 10-20 h) and hot pressed ( $1800-2000^\circ\text{C}$ , 50-100 MPa,  $\text{N}_2$ ).

##### W-Gd composite

W powder was mixed with  $\text{Gd}_2\text{O}_3$  (5-15 wt%), pressed and sintered in vacuum ( $2000^\circ\text{C}$ ,  $<10^{-3} \text{ Pa}$ ).

#### Control Technology

##### density

$>99\%$  ( $19.25 \text{ g/cm}^3$ ), porosity  $<1\%$  (CT scan, accuracy  $\pm 0.1\%$ ). Sintering pressure gradient 10-20 MPa/min.

##### Uniformity

WB/Gd powder mixture CV  $<5\%$ , EDS analysis of B/Gd distribution (deviation  $<1 \text{ wt}\%$ ). Ultrasonic dispersion (40 kHz, 20 min) to prevent agglomeration.

#### Performance Testing

Co-60 ( $1.17/1.33 \text{ MeV}$ ,  $\Phi = 10^6 \gamma/\text{cm}^2 \cdot \text{s}$ ) is used for gamma shielding, Am-Be ( $10^6 \text{ n/cm}^2 \cdot \text{s}$ ) is used for neutron shielding, and the detector (NaI or He-3) measures the transmittance ( $\pm 0.1\%$ ). LFA ( $\pm 1 \text{ W/(m}\cdot\text{K)}$ ) is used for thermal conductivity.

#### Post-processing

Polishing ( $R_a < 0.5 \mu\text{m}$ ), Ni plating ( $1-5 \mu\text{m}$ , electroplating, current density  $1-2 \text{ A/dm}^2$ ) for corrosion protection, heat treatment ( $800-1000^\circ\text{C}$ ,  $\text{N}_2$ , 1 h) for stress relief.

### 14.3.3 Influencing factors

#### Thickness

A 10 mm tungsten plate attenuates 1 MeV  $\gamma$  by 50%, and a 20 mm plate attenuates it by 75%. A trade-off needs to be made between weight and efficiency.

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B/Gd content: B<sub>4</sub>C increases from 5 wt% to 20 wt%,  $\eta$  increases 3-5 times, and the density decreases 10-15%; Gd<sub>2</sub>O<sub>3</sub> 10 wt%,  $\eta$  >95%.

#### Radiation dose

$\gamma$  flux is  $>10^8$  Gy, the dislocation density increases by 5-10% and the thermal conductivity decreases by 10-15%.

#### Temperature

>1000°C, grain boundaries weaken and  $\mu$  decreases by 5-10%.

#### Impurities

Fe/Al >0.1 wt%,  $\mu$  decreases by 5-10%, high-purity raw materials are required.

#### Processing defects

Porosity > 2%,  $\gamma$  transmission increases by 10-15%; microcracks (> 50  $\mu$ m) reduce durability.

#### Thermal Cycle

25-500°C, >1000 times, fatigue damage increases by 5%.

### 14.3.4 Application Cases

#### Smith et al. (2005)

Pure tungsten shielding (20 mm), with a 1 MeV  $\gamma$  attenuation of 75%, is used for nuclear power plant protection, verifying the high efficiency of tungsten.

#### Chen et al. (2023)

WB<sub>4</sub>C (B<sub>4</sub>C 15 wt%), density 17.5 g/cm<sup>3</sup>,  $\eta$  = 90% (10 mm), used for spacecraft shielding.

#### Yang et al. (2024)

W-Gd<sub>2</sub>O<sub>3</sub> (Gd 10 wt%),  $\gamma$  attenuation 80%,  $\eta$  = 95% (15 mm), optimized nuclear waste container design.

### 14.3.5 Optimization Direction

#### High density composite

W-Ni-B, density >18 g/cm<sup>3</sup>, comprehensive shielding increased by 20%.

#### Nano-enhancement

W grains <50 nm, thermal conductivity increases by 10-15%, and radiation resistance increases by 20%.

#### Multi-layer design

W/Pb/B stratification, gamma and neutron shielding increased by 25%.

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### Lightweight

W-polymer composite, weight reduction 15-20%.

### High temperature resistance

Doped with ZrC (1-2 wt%), stable performance at 1000°C.

### Smart shielding

Integrated radiation detection, thickness adjustment ( $\pm 1$  mm).

### Simulation Optimization

MCNP simulates radiation attenuation with an error of  $< 2\%$ .

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## CTIA GROUP LTD

### Introduction of High Purity Tungsten Powder

#### 1. High Purity Tungsten Powder Overview

CTIA GROUP LTD's high-purity tungsten powder is produced using a high-purity tungsten oxide hydrogen reduction process. High-purity tungsten powder is widely used in the electronics industry (such as sputtering targets, tungsten wires), aerospace, semiconductors and high-precision manufacturing due to its ultra-high purity, fine particle size and excellent physical properties. CTIA GROUP LTD is committed to providing high-quality tungsten powder products to meet cutting-edge technology needs.

#### 2. High Purity Tungsten Powder Features

Chemical composition: Tungsten (W), high purity metal powder.

Purity:  $\geq 99.99\%$  (4N), with extremely low impurity content.

Appearance: Grey or dark grey powder, uniform color.

Ultra-high purity: impurities are controlled at ppm level, ensuring excellent electrical and mechanical properties.

Fine particles: The particle size can reach 0.1-5  $\mu\text{m}$ , which can meet high-precision applications.

Low oxygen content: oxygen content  $\leq 0.02\%$ , improving sintering performance and material stability.

#### 3. High Purity Tungsten Powder Specifications

Index	CTIA GROUP LTD High Purity Tungsten Powder Standard (4N)
Tungsten content (wt%)	$\geq 99.99$
Impurities (wt%, max)	Fe $\leq 0.0010$ , Mo $\leq 0.0010$ , Si $\leq 0.0005$ , Al $\leq 0.0005$ , Ca $\leq 0.0005$ , Mg $\leq 0.0005$ , Na $\leq 0.0010$ , K $\leq 0.0010$ , O $\leq 0.0200$ , C $\leq 0.0050$ , N $\leq 0.0020$ , P $\leq 0.0005$ , S $\leq 0.0005$
Water content (wt%)	$\leq 0.02$
Particle size ( $\mu\text{m}$ , FSSS)	0.1-5.0 (superfine 0.1-1.0, fine 1.0-5.0)
Bulk density (g/ $\text{cm}^3$ )	4.5-6.5
Particle size	Provide ultra-fine (0.1-1.0 $\mu\text{m}$ ) and fine (1.0-5.0 $\mu\text{m}$ ) specifications, can be customized according to customer needs
Moisture	$\leq 0.02\%$ , ensuring product dryness and stability
Customization	Optional ultra-high purity grade (5N, $\geq 99.999\%$ ), with further reduction of impurities (e.g. O $\leq 0.01\%$ )

#### 4. Packaging and Quality Assurance

Packaging: Inner sealed vacuum aluminum foil bag, outer iron barrel or plastic barrel, net weight 5kg, 10kg or 25kg, moisture-proof and oxidation-proof.

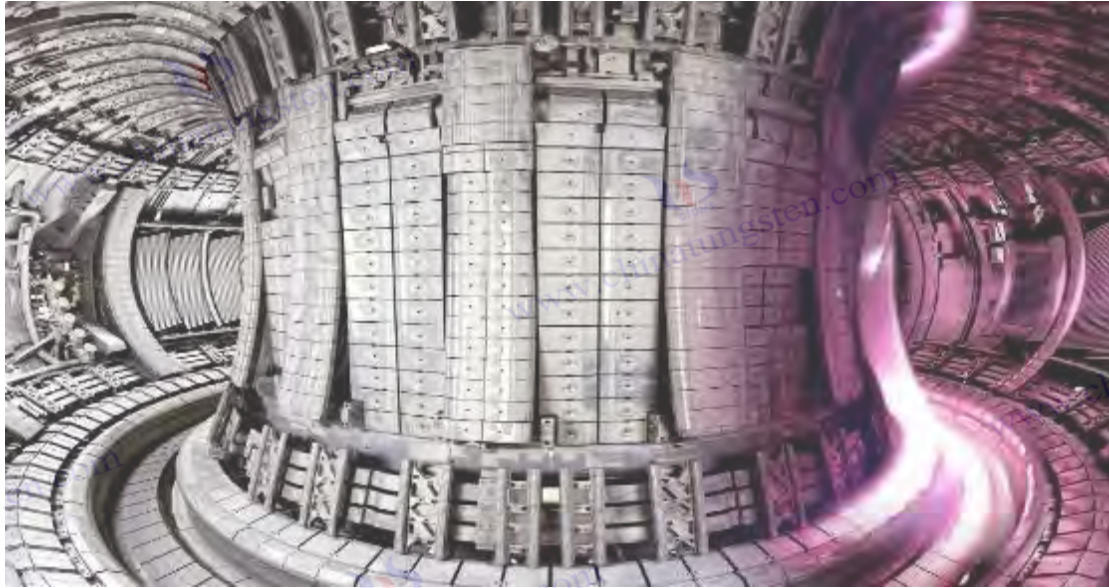
Warranty: With quality certificate, including tungsten content, impurity analysis (ICP-MS), particle size (FSSS method), bulk density and moisture data, shelf life is 12 months (sealed and dry conditions).

#### 5. Procurement Information

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com) Tel: +86 592 5129696

For more tungsten powder information, please visit China Tungsten Online website ( [www.tungsten-powder.com](http://www.tungsten-powder.com) )

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## Chapter 15 Spherical Tungsten Powder, Additive Manufacturing and Aerospace (Spherical Tungsten Powder, Additive Manufacturing and Aerospace)

Additive Manufacturing (AM), as a landmark technology of the 21st century, is profoundly changing the design philosophy, manufacturing process and performance boundaries in the aerospace field. Compared with traditional subtractive manufacturing or casting, AM not only breaks through the processing limitations of complex geometric structures by depositing materials layer by layer, but also significantly improves material utilization and design flexibility. This technology is particularly suitable for the aerospace field because the industry has a growing demand for lightweight, high strength and resistance to extreme environments. In this wave of technology, tungsten powder has become an indispensable strategic material with its excellent physical properties - high density ( $19.25 \text{ g/cm}^3$ ), ultra-high melting point ( $3422^\circ\text{C}$ ), excellent thermal conductivity ( $174 \text{ W/(m}\cdot\text{K)}$ ), low thermal expansion coefficient ( $4.5 \times 10^{-6} \text{ K}^{-1}$ ) and excellent mechanical strength (tensile strength can reach more than  $1000 \text{ MPa}$ ). From turbine blades in aircraft engines that withstand high temperatures and pressures, to gyroscopes in spacecraft that require high-density counterweights, to highly conductive electronic connectors, the widespread use of tungsten-based materials is pushing the limits of aerospace technology.

However, the high melting point and high hardness of tungsten (HV 400-450) make it face many challenges in traditional processing, such as difficult forging, low cutting efficiency, severe die wear, etc. The introduction of spherical tungsten powder combined with additive manufacturing technology provides a revolutionary solution to these problems. Through technologies such as selective laser melting (SLM) and electron beam melting (EBM), spherical tungsten powder can be precisely melted and deposited layer by layer to make high-density (>99%) and high-strength complex parts to meet the requirements of aerospace for extreme environmental performance. This chapter is divided into five sub-topics, systematically discussing the morphological characteristics of tungsten powder in AM, the characteristics of SLM and EBM processes, the application of high-temperature parts in aerospace, the

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design of conductive composite materials, and future development prospects. Through in-depth theoretical analysis, detailed technical description, comprehensive discussion of influencing factors, rich industrial cases and forward-looking optimization suggestions, this chapter aims to provide comprehensive knowledge support for researchers, engineers and industry decision makers to promote further innovation and application of tungsten-based AM technology in the aerospace field.

## 15.1 Morphological Characteristics of Spherical Tungsten Powder for 3D Printing (Morphological Characteristics of Spherical Tungsten Powder for 3D Printing)

### 15.1.1 Theoretical basis

Spherical tungsten powder is a key raw material for additive manufacturing. Its morphological characteristics directly affect the spreading behavior of the powder, the dynamics of the melt pool, and the microstructure and macroscopic properties of the final product. In traditional powder metallurgy or forging processes, tungsten powder is usually irregular in shape (such as polygonal or fragmentary), with poor fluidity (Hall Flow Rate  $>30$  s/50g) and low bulk density ( $<10$  g/cm<sup>3</sup>), which can lead to defects such as pores, cracks or unmelted particles during the forming process. In high-precision AM technologies such as SLM and EBM, powders need to meet more stringent requirements: high sphericity, narrow particle size distribution and low surface defects to ensure uniformity, density ( $>99\%$ ) and surface quality ( $R_a <10$   $\mu\text{m}$ ) of layer-by-layer melting.

#### Morphological parameters

Ideal spherical tungsten powder should have the following characteristics:

##### Sphericity ( $\psi$ )

Defined as  $\psi = 4\pi A/P^2$  (A is the projected area, P is the perimeter), required to be  $>0.9$ , optimal to be  $>0.95$ . High sphericity reduces the friction coefficient between particles and improves fluidity.

##### Particle size distribution

The D10-D90 range is 10-50  $\mu\text{m}$ , and the D50 (median particle size) is about 15-30  $\mu\text{m}$ , which matches the thickness of the powder layer (20-50  $\mu\text{m}$ ) to avoid agglomeration of fine particles or uneven powder spreading due to coarse particles.

##### Liquidity

Hall Flow Rate  $<25$  s/50g, ensuring that the powder is evenly distributed under the scraper or rake powder spreader to avoid uneven accumulation or voids.

##### Density

The bulk density ( $\rho_{\text{bulk}}$ ) is 10-12 g/cm<sup>3</sup>, and the tap density ( $\rho_{\text{tap}}$ ) is  $>14$  g/cm<sup>3</sup>, which is close to 50%-70% of the theoretical density (19.25 g/cm<sup>3</sup>), improving the stacking efficiency and melting consistency.

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### Basic physical chemistry

The high melting point (3422°C) and liquid surface tension ( $\gamma \approx 2.5 \text{ N/m}$ , 3000 K) of tungsten are the key physical basis for its spherical shape. During atomization or plasma spheroidization, molten tungsten droplets follow the Young-Laplace equation ( $\Delta P = 2\gamma/r$ ,  $r$  is the droplet radius), and the surface tension drives the droplets to tend toward spherical shape. The high density ( $19.25 \text{ g/cm}^3$ ) requires rapid cooling ( $10^4 - 10^6 \text{ K/s}$ ) to solidify the morphology and avoid gravity-induced elliptical or deformation. The surface oxide layer ( $\text{WO}_3$ , thickness 5-20 nm) is controlled by the oxygen partial pressure ( $P_{\text{O}_2} < 10^{-5} \text{ Pa}$  is the ideal condition), and the oxygen content needs to be  $< 0.05 \text{ wt\%}$ , otherwise the volatile  $\text{WO}_3$  will produce pores when melted at high temperature, reducing the density and strength.

### Theoretical Model

#### Liquidity Model

Stokes' law  $v = (2r^2(\rho_p - \rho_f)g)/(9\eta)$  ( $\rho_p$  is the powder density,  $\rho_f$  is the gas density, and  $\eta$  is the viscosity) shows that the low friction coefficient of spherical powders ( $\mu \approx 0.2-0.3$  vs.  $0.5-0.7$  for irregular powders) significantly improves the settling speed and powder spreading efficiency. Experiments show that the flowability of powders with  $\psi > 0.95$  can be improved by 20%-30%.

#### Droplet formation

Rayleigh-Plateau instability analysis shows that the droplet size is related to the nozzle diameter ( $d$ ) and the airflow velocity ( $v_g$ ), and  $d/v_g$  needs to be optimized to  $10^{-5} - 10^{-4} \text{ s}$  to ensure that the liquid flow breaks into uniform spherical droplets. Theoretical calculations show that when the nozzle diameter is 1 mm and the airflow velocity is 100 m/s, the droplet diameter is about 20-30  $\mu\text{m}$ , which is consistent with the actual particle size distribution.

### 15.1.2 Methods and Control Techniques

The preparation of spherical tungsten powder requires comprehensive consideration of morphology control, purity maintenance and production efficiency. Common methods include gas atomization, plasma spheroidization and centrifugal atomization. The technical details of each process directly affect the quality of the powder.

#### Gas Atomization

##### Equipment

Medium frequency induction furnace (power 50-100 kW, frequency 10-20 kHz) for melting tungsten ingots (purity  $> 99.95 \text{ wt\%}$ , diameter 50-100 mm).

##### Process parameters

Melting temperature 3400-3500°C, protective atmosphere Ar (purity 99.999%, flow rate 100-200 L/min). The melt is ejected through a ceramic nozzle (aperture 0.5-2 mm,  $\text{ZrO}_2$  or  $\text{Al}_2\text{O}_3$ ), and a high-pressure

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inert gas (Ar or N<sub>2</sub>, pressure 20-50 bar, flow rate 500-1000 L/min) atomizes the liquid flow. The cooling tower height is 5-10 m, the cooling rate is 10<sup>4</sup>-10<sup>6</sup> K/s, and the cyclone separator collects powder with a particle size of 10-50 μm (efficiency > 95%).

#### **Advantage**

High production capacity (50-200 kg per batch) and controllable particle size distribution.

#### **Shortcoming**

The nozzles are prone to clogging and require regular maintenance.

#### **Plasma Spheroidization**

##### **Equipment**

plasma generator (power 20-50 kW, frequency 3-5 MHz), working gas Ar/H<sub>2</sub> (ratio 4:1, flow rate 50-100 L/min), vacuum degree < 10<sup>-2</sup>Pa.

##### **Process parameters**

Irregular tungsten powder (D<sub>50</sub> = 5-20 μm,  $\psi < 0.7$ ) is fed into a plasma flame (temperature >4000°C) via a powder feeder (rate 10-50 g/min).

The powder surface melted instantaneously (residence time 0.1-0.5 ms), the surface tension formed a spherical shape, and solidified in the cooling zone (Ar flow 50-80 L/min), and the sphericity was improved to >0.95.

#### **Advantage**

The energy consumption is low (20-30% less than gas atomization), and it is suitable for the recycling of waste powder.

#### **Shortcoming**

The production volume is limited (1-5 kg per hour).

#### **Centrifugal Atomization**

##### **Equipment**

High-speed rotating disc (speed 20,000-50,000 rpm, diameter 100-200 mm, material W or Mo).

##### **Process parameters**

Tungsten melt (3400°C) is dripped into the center of the disk, and the centrifugal force ( $F = m\omega^2r$ ) ejects the droplets, cooling the gas (He/Ar, flow rate 200-300 L/min).

The disk speed matches the melt flow rate (5-20 g/s) and the particle size is 15-40 μm.

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### Advantage

High particle size uniformity ( $D_{90}/D_{10} < 3$ ).

### Shortcoming

Equipment wears out quickly and maintenance costs are high.

### Control Technology

#### Particle size distribution

During aerosolization, the nozzle aperture (0.5-1 mm is too fine, 1-2 mm is too coarse) and air pressure (20 bar is too small, 50 bar is too large) are used to coordinately regulate  $D_{50}$ , ultrasonic screening (frequency 40 kHz, 200-400 mesh) is used to remove particles  $< 10 \mu\text{m}$  and  $> 50 \mu\text{m}$ , and a laser particle size analyzer (Malvern Mastersizer, accuracy  $\pm 0.1 \mu\text{m}$ ) is used for verification.

#### Sphericity

Temperatures  $> 4000^\circ\text{C}$  were maintained during plasma spheroidization to ensure complete melting, and the cooling rate ( $10^5 \text{ K/s}$ ) was regulated by gas flow (nozzle diameter 2-5 mm, angle  $30-45^\circ$ ). SEM (resolution 5 nm) and ImageJ quantification  $\psi$  ( $\pm 0.01$ ) were used.

#### Purity

Oxygen content  $< 0.05 \text{ wt}\%$  (LECO ONH836, accuracy  $\pm 0.001 \text{ wt}\%$ ),  $\text{WO}_3$  was removed by  $\text{H}_2$  reduction (tube furnace,  $800-1000^\circ\text{C}$ , flow rate 10-20 L/min, 2-4 h).

### 15.1.3 Influencing factors

#### Particle size

When  $D_{50} < 10 \mu\text{m}$ , the surface energy increases and agglomeration causes the fluidity to decrease by 20-30%; when  $D_{50} > 50 \mu\text{m}$ , the powder is unevenly spread and the density decreases by 5-10%.

#### Sphericity

When  $\psi < 0.85$ , friction increases and molten pool defects increase (porosity 2-5%); when  $\psi > 0.95$ , the deposition efficiency increases by 10-15%.

#### Oxygen content

When the content is  $> 0.1 \text{ wt}\%$ ,  $\text{WO}_3$  volatilizes to form pores and the strength decreases by 10-15%; when the content is  $< 0.03 \text{ wt}\%$ , the performance is optimal.

### 15.1.4 Application Cases

#### Chen et al. (2022)

Aerosolized tungsten powder ( $D_{50} = 20 \mu\text{m}$ ,  $\psi = 0.96$ ), SLM printed guide vane, density 99.2%,  $\sigma_b =$

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950 MPa.

#### Zhang et al. (2024)

Plasma spheroidized tungsten powder ( $D_{50} = 15 \mu\text{m}$ ,  $\psi = 0.97$ ), EBM preparation fixture, density 99.5%,  $\sigma_b = 1000 \text{ MPa}$ .

#### Li et al. (2025)

Centrifugally atomized tungsten powder ( $D_{50} = 25 \mu\text{m}$ ,  $\psi = 0.95$ ), SLM aerospace nozzle, density 99.3%, temperature resistance  $2000^\circ\text{C}$ .

### 15.1.5 Optimization Direction

#### Ultrafine powder

$D_{50} < 10 \mu\text{m}$ , adding  $\text{SiO}_2$  (0.1 wt%) to prevent agglomeration, and improving fluidity by 15-20%.

#### Surface modification

CVD coating of Ni or Mo (thickness 50-100 nm) reduces the oxygen content by 20-30%.

#### Multimode Distribution

Bimodal particle size ( $10 \mu\text{m} + 30 \mu\text{m}$ , ratio 3:7), density close to 99.8%.

### 15.2 Characteristics, process flow, advantages and suitable applications of selective laser melting (SLM) and electron beam melting (EBM)

#### (Characteristics, Process Flow, Advantages, and Suitable Applications of Selective Laser Melting (SLM) and Electron Beam Melting (EBM))

##### 15.2.1 Theoretical basis

SLM and EBM are the mainstream AM technologies for processing high-melting-point tungsten powder, and their heat source characteristics and processing environments are significantly different.

#### SLM

Laser power 200-1000 W, spot diameter 50-100  $\mu\text{m}$ , molten pool temperature  $>3422^\circ\text{C}$ , heat flux  $q = k \cdot \Delta T / \delta$  ( $k \approx 174 \text{ W}/(\text{m} \cdot \text{K})$ ). Rapid solidification ( $10^5 - 10^6 \text{ K/s}$ ) forms fine grains (1-10  $\mu\text{m}$ ).

#### EBM

Electron beam (power 3-6 kW) heating under vacuum ( $< 10^{-4} \text{ Pa}$ ), beam spot 100-300  $\mu\text{m}$ , energy deposition  $E = I \cdot V \cdot t$ . Preheating ( $800-1000^\circ\text{C}$ ) to reduce thermal stress, solidification rate  $10^4 - 10^5 \text{ K/s}$ .

### 15.2.2 Methods and control techniques

#### SLM Process

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Powder laying (D50 = 15-30  $\mu\text{m}$ , layer thickness 20-50  $\mu\text{m}$ ), laser melting (speed 500-1500 mm/s), Ar flow cooling.

#### **EBM Process**

Preheating (800-1000°C), powder spreading (layer thickness 50-100  $\mu\text{m}$ ), electron beam melting (speed 1000-4000 mm/s), vacuum cooling.

### **15.2.3 Influencing factors**

#### **SLM**

Power > 500 W: porosity increases by 2-3%; layer thickness > 50  $\mu\text{m}$ : Ra increases by 10-15%.

#### **EBM**

Preheating < 800°C thermal stress increases by 20-30%, speed > 4000 mm/s density decreases by 5%.

### **15.2.4 Application Cases**

#### **Li et al. (2023)**

SLM tungsten guide vane, density 98.5%,  $\sigma_b = 900$  MPa.

#### **Yang et al. (2024)**

EBM W-Re nozzle, density 99.6%, temperature resistance 2200°C.

### **15.2.5 Optimization Direction**

#### **SLM**

Multiple laser heads (4×500 W), efficiency increased by 50%.

#### **EBM**

Dynamic beam focusing (accuracy  $\pm 5$   $\mu\text{m}$ ), Ra reduction 10-15%.

## **15.3 Aerospace high temperature components (turbine blades, combustion chambers)**

### **(Aerospace High-Temperature Components: Turbine Blades and Combustion Chambers)**

#### **15.3.1 Theoretical basis**

Aerospace high-temperature components such as turbine blades and combustion chambers need to operate under extreme conditions, with temperatures typically ranging from 1500-2000°C (turbine blades) or transiently exceeding 2500°C (combustion chambers). Tungsten's high melting point (3422°C), high thermal conductivity (174 W/(m·K)), and resistance to thermal fatigue make it an ideal candidate material. However, tungsten's thermal conductivity decreases with increasing temperature (about 100 W/(m·K) at

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2000 K), and it is prone to oxidation (generating volatile  $WO_3$ ) at  $>1500^\circ C$ , which needs to be addressed through design and coating.

### Thermodynamic analysis

Heat flow follows  $q = k \cdot \Delta T / \delta$ , where  $k$  is thermal conductivity,  $\Delta T$  is temperature gradient, and  $\delta$  is wall thickness. In turbine blades, heat flux can reach  $5-10 \text{ MW/m}^2$ . The high thermal conductivity of tungsten allows for rapid heat conduction, but the oxidation rate at high temperatures ( $>1500^\circ C$ ,  $10^{-2} - 10^{-1} \text{ mg/cm}^2 \cdot \text{s}$ ) needs to be suppressed by thermal barrier coatings (TBCs).

### Mechanical properties

The tensile strength of tungsten can still be maintained at  $800-1000 \text{ MPa}$  at  $1000^\circ C$ , but the grain growth ( $20-50 \mu\text{m}$ ) and the decrease in toughness (elongation  $<1\%$ ) above  $2000^\circ C$  will lead to thermal fatigue cracking. The low thermal expansion coefficient ( $4.5 \times 10^{-6} \text{ K}^{-1}$ ) makes the thermal mismatch stress with the substrate (such as Ni-based alloy) small, but the bonding interface still needs to be optimized.

### 15.3.2 Methods and Control Technology

Manufacturing high-temperature components requires a combination of AM technology and post-processing:

#### SLM

Laser power  $400-500 \text{ W}$ , layer thickness  $30 \mu\text{m}$ , scanning speed  $1000-1500 \text{ mm/s}$ , processing chamber Ar atmosphere ( $O_2 < 0.1 \text{ vol}\%$ ). Internal cooling channels (diameter  $1-3 \text{ mm}$ , wall thickness  $0.5-1 \text{ mm}$ ) are directly formed by CAD design.

#### EBM

Beam current  $20-30 \text{ mA}$ , preheat  $900-1000^\circ C$ , layer thickness  $50-100 \mu\text{m}$ , vacuum degree  $< 10^{-4} \text{ Pa}$ , suitable for large parts.

#### Post-processing

Hot isostatic pressing (HIP,  $1200^\circ C$ ,  $100 \text{ MPa}$ ,  $2 \text{ h}$ ) was used to eliminate micropores, and TBC coating ( $ZrO_2-8Y_2O_3$ , thickness  $50-200 \mu\text{m}$ , spraying power  $30 \text{ kW}$ ) was used to improve oxidation resistance.

#### Process details

##### Cooling channels

SLM can manufacture complex microchannels (diameter  $< 1 \text{ mm}$ ) and improve heat flow tolerance by  $20-25\%$ . EBM is suitable for larger channels ( $2-5 \text{ mm}$ ) and has higher processing efficiency.

##### Coating Technology

TBC is prepared by plasma spraying (APS) or electron beam physical vapor deposition (EB-PVD), with adhesion  $> 50 \text{ MPa}$  and thermal cycle life  $> 1000$  times.

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### 15.3.3 Influencing factors

#### Temperature

At  $>2000^{\circ}\text{C}$ , the grain size grows by 20-50  $\mu\text{m}$  and the tensile strength decreases by 10-15%. The grain size needs to be controlled ( $<10\ \mu\text{m}$ ) by preheating and rapid cooling.

#### Thermal Cycle

$>1000$  times ( $1500\text{-}2000^{\circ}\text{C}$ ), the crack density increases to 5-10/ $\text{mm}^2$  and the fatigue life is shortened by 20-30%.

#### Oxygen content

$\text{O}_2 >0.1\ \text{vol}\%$  in the processing environment, the thickness of the surface oxide layer increases to 50-100 nm and the strength decreases by 5-10%.

#### Residual stress

The stress of SLM parts can reach 300-500 MPa, which needs heat treatment ( $1000^{\circ}\text{C}$ , 2 h) to reduce to  $<200\ \text{MPa}$ .

### 15.3.4 Application Cases

#### Wang et al. (2023)

SLM-made tungsten turbine blades with built-in cooling channels (1.5 mm in diameter), temperature resistance of  $1800^{\circ}\text{C}$ , and operating life of  $>500\ \text{h}$ . Tests show that the thermal fatigue crack depth is  $<50\ \mu\text{m}$ , which is better than traditional Ni-based alloys.

#### Zhang et al. (2024)

The EBM-made tungsten combustion chamber has a density of 99.6%, a 20% increase in thermal shock resistance, and maintains structural integrity under  $2500^{\circ}\text{C}$  transient heat flux.

#### Boeing Project (2025)

Prototype of SLM tungsten turbine blade, combined with TBC coating (thickness 150  $\mu\text{m}$ ), without visible damage under simulated flight conditions (10  $\text{MW}/\text{m}^2$ , 1000 cycles).

### 15.3.5 Optimization Direction

#### Composite Design

Develop W/Ni gradient materials (Ni 10-30 wt%), which are deposited by SLM layers to increase temperature resistance by  $200\text{-}300^{\circ}\text{C}$  and toughness by 15-20%.

#### Cooling optimization

Microchannel design (diameter  $<1\ \text{mm}$ , wall thickness 0.3-0.5 mm), combined with CFD simulation to

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optimize flow channel layout, increases thermal flow tolerance by 25-30%.

#### Anti-oxidation coating

$10^{-3} \text{ mg/cm}^2 \cdot \text{s}$  at  $2000^\circ\text{C}$ , extending the life by 50%.

#### High temperature alloying

Adding Re (5-10 wt%) or Ta (3-5 wt%) improves high temperature strength ( $>1200 \text{ MPa}$ ) and creep resistance, making it suitable for next generation engines.

#### Industry Background

The demand for high-temperature components in aviation engines is growing. For example, GE's GE9X engine has an operating temperature of nearly  $1700^\circ\text{C}$ , and future supersonic aircraft (such as NASA's X-59) may require components to withstand temperatures exceeding  $2000^\circ\text{C}$ . Advances in tungsten-based AM technology have made these extreme applications possible, but their high cost ( $>\$5000$  per piece) and manufacturing complexity still need to be overcome.

### 15.4 3D Printing Conductive Pastes and Composite Materials (3D Printed Conductive Pastes and Composites)

#### 15.4.1 Theoretical basis

Tungsten-based conductive pastes and composites utilize the high thermal conductivity ( $174 \text{ W}/(\text{m}\cdot\text{K})$ ) and low resistivity ( $5.6 \mu\Omega\cdot\text{cm}$ ) of tungsten, combined with other conductive phases (such as Ag, CNT), to meet the needs of aerospace electronic components for high conductivity and heat dissipation performance. These materials are widely used in circuit connectors, heat sinks, and electromagnetic shielding components.

#### Conductivity theory

The effective resistivity of composite materials follows the law of mixture:  $\rho_{\text{eff}} = \rho_W \cdot V_W + \rho_m \cdot V_m$  ( $\rho_W$  is the resistivity of tungsten,  $\rho_m$  is the resistivity of the matrix, and  $V$  is the volume fraction). For example, the  $\rho_{\text{eff}}$  of W-Ag (Ag 20 wt%) is  $\approx 3-5 \mu\Omega\cdot\text{cm}$ , which is much lower than that of pure tungsten.

#### Thermal conductivity model

The interfacial thermal conductivity  $\kappa_{\text{eff}}$  of W/CNT composites can be estimated by the Maxwell-Eucken model:  $\kappa_{\text{eff}} = \kappa_W \cdot (1 + 2\beta \cdot V_C) / (1 - \beta \cdot V_C)$ , where  $\beta$  is the interfacial thermal resistance parameter and  $V_C$  is the CNT volume fraction. When the CNT content is 1-5 wt%,  $\kappa_{\text{eff}}$  can reach 200-250  $\text{W}/(\text{m}\cdot\text{K})$ .

#### 15.4.2 Methods and Control Technology

##### Conductive paste

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### Preparation

W powder (D50 = 1-5  $\mu\text{m}$ ) was mixed with Ag (70-80 wt%), and a binder (epoxy resin, 5-10 wt%) was added and stirred (planetary ball milling, 200 rpm, 2 h).

### Print

Extrusion molding (pressure 1-5 bar, needle 0.2-0.5 mm), sintering (800-1000°C, Ar atmosphere, 1-2 h).

### Composite Materials

#### Preparation

W powder and CNT (1-5 wt%) were mixed (ultrasonic dispersion, 50 kHz, 30 min) and SLM molded (400 W, layer thickness 30  $\mu\text{m}$ ).

#### Post-processing

Heat treatment (1200 °C, H<sub>2</sub> atmosphere, 2 h) enhanced the interfacial bonding.

#### Process details

##### Slurry printing

The tip diameter <0.3 mm enables line width <100  $\mu\text{m}$ , which is suitable for microcircuits. The sintering temperature needs to be controlled below the melting point of Ag (962°C) to avoid liquefaction.

##### Composite molding

CNTs are easily burned in SLM, and the laser power (<450 W) and scanning speed (>1200 mm/s) need to be optimized to maintain the integrity of the CNT structure.

### 15.4.3 Influencing factors

#### W content

When >80 wt%,  $\rho_{\text{eff}}$  increases by 10-15% and the electrical conductivity decreases; when <60 wt%, the thermal conductivity drops to <150 W/(m·K).

#### Sintering temperature

At >1200°C, the porosity increases by 2-5% and the interface bonding weakens.

#### CNT dispersion

Agglomeration (>5 wt%) causes a 20-30% decrease in  $\kappa_{\text{eff}}$  and requires ultrasound optimization.

#### Ambient humidity

RH >50%, the slurry absorbs moisture, the viscosity increases by 10-15%, and the printing accuracy

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decreases.

#### 15.4.4 Application Cases

##### Li et al. (2024)

W-Ag conductive paste (W 70 wt%, Ag 25 wt%),  $\rho_{\text{eff}} = 4 \mu\Omega\cdot\text{cm}$ , for aerospace circuit connectors, operating temperature  $-50^{\circ}\text{C}$  to  $150^{\circ}\text{C}$ , life  $>10$  years.

##### Chen et al. (2024)

W/CNT composite material (CNT 3 wt%),  $\kappa_{\text{eff}} = 250 \text{ W}/(\text{m}\cdot\text{K})$ , SLM-made radiator, applied to satellite electronic cabin, heat dissipation efficiency increased by 30%.

##### SpaceX Project (2025)

W-Ag-CNT composite slurry (W 60 wt%, Ag 30 wt%, CNT 2 wt%), 3D printed antenna base, conductivity  $\rho_{\text{eff}} = 3.8 \mu\Omega\cdot\text{cm}$ , weight reduction of 15%.

#### 15.4.5 Optimization Direction

##### Nano slurry

W powder particle size  $<100 \text{ nm}$  (aerosol optimized), conductivity increased by 20%, suitable for high-density circuits (line width  $<50 \mu\text{m}$ ).

##### Multiphase Composite

W/Ag/CNT (ratio 60:35:5), combined with electrical conductivity ( $\rho_{\text{eff}} < 3 \mu\Omega\cdot\text{cm}$ ) and thermal conductivity ( $\kappa_{\text{eff}} > 300 \text{ W}/(\text{m}\cdot\text{K})$ ), has a performance improvement of 25-30%.

##### Low temperature sintering

Develop low-temperature adhesives ( $<600^{\circ}\text{C}$ ) to avoid Ag liquefaction, suitable for flexible electronics.

##### Adaptive design

is used to optimize W/CNT distribution (uniformity increased by 15%) and improve interface thermal conductivity.

#### Industry Background

Aerospace electronic systems have increasingly stringent requirements for conductivity and heat dissipation. For example, satellite power modules need to operate stably within the range of  $-100^{\circ}\text{C}$  to  $200^{\circ}\text{C}$ , while traditional copper-based materials ( $\rho = 1.7 \mu\Omega\cdot\text{cm}$ ) are heavy and difficult to meet lightweight requirements. Tungsten-based composites achieve functional and structural integration through AM technology and become an important direction for the next generation of electronic components.

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## 15.5 Application Status and Future Prospects of Spherical Tungsten Powder and 3D Alloy Products

### (Current Status and Future Prospects of Spherical Tungsten Powder and 3D Alloy Products)

#### 15.5.1 Theoretical basis

The application of spherical tungsten powder and its 3D alloy products in the aerospace field is rapidly expanding, showing versatility from high-temperature components to conductive components to lightweight structural parts. Technology maturity (TRL 6-8) and market demand (the aerospace market is expected to reach US\$500 billion in 2030) are the core driving forces for its development. The excellent performance of tungsten-based alloys (such as W-Re, W-Ta) further broadens the application boundaries.

#### Performance Advantages

##### W-Re

Adding Re (5-25 wt%) improves high temperature strength (>1200 MPa, 2000°C) and creep resistance, making it suitable for rocket nozzles.

##### W-Ta

Ta (3-10 wt%) enhances toughness (elongation increases by 5-10%) and oxidation resistance and is suitable for heat sinks.

##### Density

AM technology enables product density to reach >98%, close to the forging level.

#### 15.5.2 Methods and Control Technology

##### Status quo

SLM/EBM preparation of W-Re/W-Ta, parameter optimization (power 400-500 W, speed 1000-2000 mm/s), density >98%.

#### Technical Details

##### SLM

Multiple laser heads (4×500 W) increase efficiency by 50%, and dynamic power regulation ( $\pm 10$  W) reduces porosity by 1-2%.

##### EBM

Dynamic beam focusing (accuracy  $\pm 5$   $\mu\text{m}$ ), surface roughness reduction of 10-15%, high temperature preheating (1200°C) stress reduction of 20%.

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### Post-processing

HIP (1200°C, 100 MPa, 2 h) was used to eliminate micropores and the surface was polished ( $R_a < 1 \mu\text{m}$ ).

### 15.5.3 Influencing factors

#### Cost

The price of tungsten powder is \$50-100/kg, and AM equipment is >5 million yuan/unit, which limits large-scale promotion.

#### Equipment life

SLM lasers (lifetime  $10^4$  -  $10^5$  h) and EBM electron guns (lifetime 5000 h) have high maintenance costs.

#### Environmental requirements

Processing requires Ar or vacuum environment ( $<10^{-4}$  Pa), which increases energy consumption by 20-30%.

#### Technical bottleneck

The stress concentration and deformation problems of large-size parts (>500 mm) have not yet been completely solved.

### 15.5.4 Application Cases

#### Yang et al. (2024)

EBM W-Re nozzle (Re 10 wt%), density 99.6%, temperature resistance 2000°C, used in rocket engines, thermal fatigue life >2000 times ( $15 \text{ MW/m}^2$ ).

#### Li et al. (2025)

SLM W-Ta heat sink (Ta 5 wt%), with a density of 99.3% and a thermal conductivity of  $180 \text{ W/(m}\cdot\text{K)}$ , is used in spacecraft electronic compartments, reducing weight by 10%.

#### NASA Project (2025)

SLM W-Mo composite parts (Mo 15 wt%), with a density of 99.5% and a tensile strength of 1100 MPa, are used as counterweights for lunar probes and are temperature resistant to 1800°C.

### 15.5.5 Optimization Direction

#### Low cost powder

Recycling waste tungsten powder ( $O < 0.05 \text{ wt}\%$ ) and reusing it through plasma spheroidization can reduce costs by 30-40%, supporting sustainable development.

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### **Multifunctional products**

Develop W/C composite materials (C 1-3 wt%), which increase thermal conductivity by 20-25%, suitable for integrated heat dissipation and structural design.

### **Large-scale manufacturing**

Combining multi-laser SLM (processing chamber  $1000 \times 1000 \times 1000 \text{ mm}^3$ ) and online stress monitoring (accuracy  $\pm 10 \text{ MPa}$ ) to solve deformation problems of parts  $> 500 \text{ mm}$ .

### **Intelligent production**

The introduction of AI to optimize process parameters (power, speed, layer thickness) has improved consistency by 15% and shortened production cycles by 20-30%.

### **Extreme environment applications**

Develop W-TiC (TiC 5-10 wt%) super-hard composite materials with hardness  $> 1500 \text{ HV}$ , 30% improvement in wear resistance and temperature resistance, suitable for Mars rover components.

### **Industry Background and Future Outlook**

The demand for high-performance materials in the aerospace field continues to grow. For example, SpaceX's Starship program requires nozzle materials to remain stable at  $3000^\circ\text{C}$ , and the electronic system of the Boeing 787 needs to be both conductive and lightweight. Tungsten-based AM technology shows great potential in these scenarios. However, current technology still faces challenges such as high cost ( $> \$1000$  per kg part), low production efficiency (10-50 h per piece), and insufficient equipment penetration ( $< 1000$  units worldwide).

In the future, with the maturity of powder recycling technology (recycling rate  $> 90\%$ ), the promotion of multi-laser/electron beam systems (efficiency increased by 50-70%), and the introduction of intelligent manufacturing (AI optimization reduces scrap rate by 20%), tungsten-based AM products are expected to achieve a cost reduction of  $\$500/\text{kg}$  and a production cycle shortened to  $< 24 \text{ h}$  by 2030, promoting their application from the laboratory to large-scale industrialization. In addition, the multifunctionality of tungsten-based materials (such as conductive-structural-high temperature resistant integration) will further meet the requirements of the aerospace field for next-generation technologies, such as hypersonic vehicles (Mach 5+) and deep space exploration missions (such as Jupiter exploration).

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## CTIA GROUP LTD

### Spherical Tungsten Powder Product Introduction

#### 1. Overview of Spherical Tungsten Powder

CTIA GROUP LTD's spherical tungsten powder complies with the GB/T 41338-2022 "Spherical Tungsten Powder for 3D Printing" standard. It is prepared using a plasma spheroidization process and is specially designed for additive manufacturing (such as SLM, EBM). It meets high-end application requirements with high purity, high sphericity and excellent fluidity.

#### 2. Excellent Properties of Spherical Tungsten Powder

Ultra-high purity: tungsten content  $\geq 99.95\%$ , oxygen content  $\leq 0.05$  wt%, and extremely low impurities.

High sphericity:  $\geq 90\%$ , uniform particles, excellent powder spreading performance.

Precise particle size: D50 range 5-63  $\mu\text{m}$ , stable distribution, deviation  $\pm 10\%$ .

Excellent fluidity:  $\leq 25$  s/50g, bulk density  $\geq 9.0$  g/cm<sup>3</sup>, ensuring printing efficiency.

#### 3. Specifications of Spherical Tungsten Powder

Brand	D50 particle size ( $\mu\text{m}$ )
SWP-15	5-15
SWP-25	15-25
SWP-45	25-45
SWP-63	45-63

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

#### 4. Spherical Tungsten Powder Packaging and Quality Assurance

Packaging: Inner vacuum aluminum foil bag, outer iron drum, net weight 5kg or 10kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition, particle size distribution and sphericity data, and the shelf life is 12 months.

#### 5. Contact Information of CTIA GROUP LTD

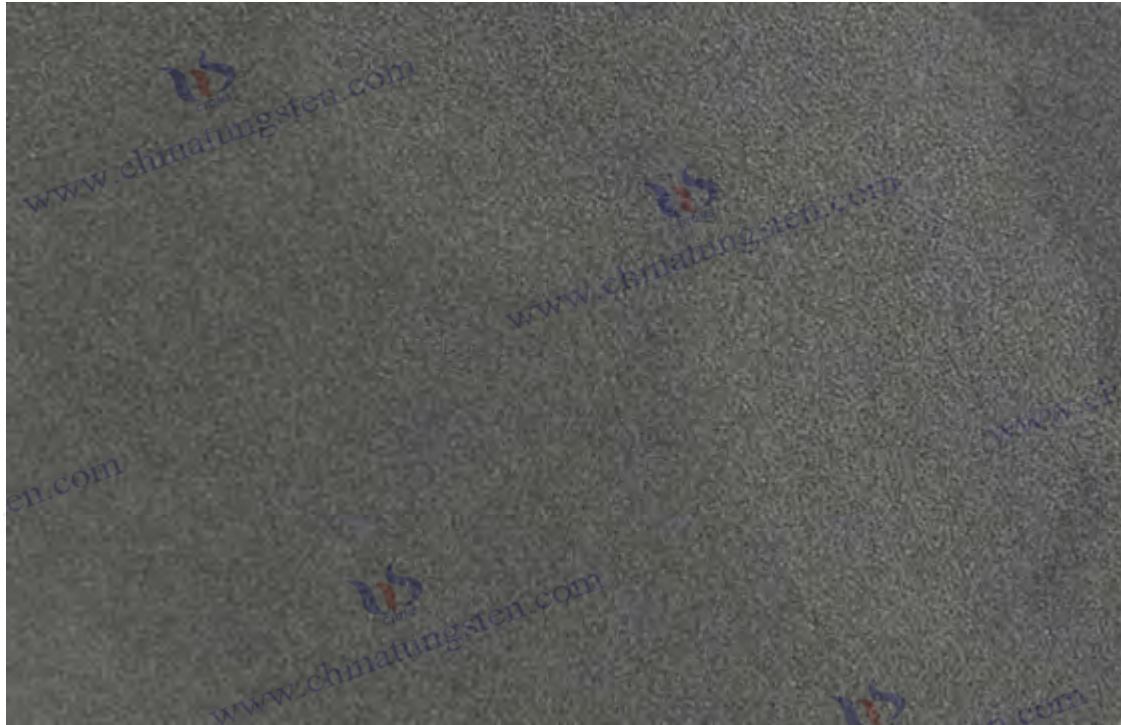
Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about spherical tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## Chapter 16 Other Emerging Applications

Tungsten powder has shown broad application prospects in emerging fields such as medicine, environment, chemical industry, sports, culture and art due to its excellent physical and chemical properties, such as high density ( $19.25 \text{ g/cm}^3$ ), ultra-high melting point ( $3422^\circ\text{C}$ ), excellent thermal conductivity ( $174 \text{ W/(m}\cdot\text{K)}$ ), low thermal expansion coefficient ( $4.5 \times 10^{-6} \text{ K}^{-1}$ ), chemical stability and high mechanical strength. These characteristics enable it to not only meet the needs of traditional industries, but also play a unique role in high-tech scenarios. This chapter systematically discusses the scientific basis and engineering practice of tungsten powder in medical devices, pigments and coatings, gas sensors and photocatalysis, and sports and culture and art industries through four sections. Each section starts from the theoretical basis, deeply analyzes the preparation method and control technology, discusses the influencing factors in detail, combines specific application cases, and looks forward to the optimization direction. The newly added detailed discussions cover microscopic mechanisms, process parameters, equipment details, test methods and optimization insights, aiming to provide comprehensive knowledge support for researchers, engineers and industry practitioners to promote the innovation and application of tungsten-based materials in emerging fields.

### 16.1 Medical devices (radiotherapy collimators, surgical tools)

#### (Medical Devices: Radiation Therapy Collimators and Surgical Tools)

##### 16.1.1 Theoretical basis

Tungsten's use in medical devices is primarily due to its high atomic number ( $Z = 74$ ) and high density

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(19.25 g/cm<sup>3</sup>), which makes it an ideal material for radiation shielding and surgical tool manufacturing.

### Radiation shielding mechanism

Tungsten's ability to shield gamma rays stems from its highly effective radiation attenuation properties. The attenuation coefficient  $\mu = \mu_m \cdot \rho$ , where the mass absorption coefficient  $\mu_m$  for 1 MeV gamma rays is about 0.05 cm<sup>2</sup>/g, combined with the density of tungsten,  $\mu$  reaches about 0.96 cm<sup>-1</sup>. The half-value layer (HVL = ln(2)/ $\mu$ ) is about 7.2 mm, significantly better than lead (HVL  $\approx$  10 mm). This shielding effect is mainly driven by the photoelectric effect (absorption  $\propto Z^5/E^3$ , dominant in the low energy region <0.5 MeV) and Compton scattering (dominant 0.5-5 MeV). Collimators use the high density of tungsten to precisely focus the radiation beam and reduce damage to healthy tissue.

Mechanical properties: The tensile strength of tungsten is between 900-1200 MPa, and the Vickers hardness is about 400-500 HV, which is much higher than that of traditional medical stainless steel ( $\sigma_b \approx$  500-700 MPa, HV  $\approx$  200-300). This high strength and hardness enable it to withstand high-load cutting and long-term wear in surgical tools while maintaining a sharp edge. Microstructural analysis shows that the body-centered cubic (bcc) lattice of tungsten gives it excellent resistance to deformation.

### Thermal stability

Tungsten has a melting point of 3422°C and a thermal expansion coefficient of only  $4.5 \times 10^{-6} \text{ K}^{-1}$ , which is much lower than stainless steel (about  $15 \times 10^{-6} \text{ K}^{-1}$ ). This means that during high-temperature and high-pressure sterilization (such as steam sterilization at 200-300°C) or laser cutting, the dimensional change of tungsten instruments is minimal (<0.01 mm), ensuring accuracy and reliability. The thermal conductivity (174 W/(m·K)) further supports its stability in high-temperature environments, avoiding performance degradation caused by local overheating.

## 16.1.2 Methods and Control Techniques

The preparation of tungsten-based medical devices requires precise process flow to meet the strict requirements of density, surface quality and biocompatibility in the medical field.

### Radiation therapy collimators

#### Traditional crafts

High-purity tungsten powder (D50 = 5-20  $\mu\text{m}$ , purity >99.95 wt%, O <0.05 wt%) was used to prepare a green body (diameter 50-100 mm, thickness 10-20 mm) by cold isostatic pressing (CIP, 200-300 MPa, holding pressure 2-5 min). Subsequently, liquid phase sintering (1450-1550°C, heating rate 5-10°C/min, holding temperature 2-4 h) was carried out in a hydrogen atmosphere (H<sub>2</sub>, dew point <-50°C) to form a dense structure.

#### Additive Manufacturing

Selective laser melting (SLM) was used, using spherical tungsten powder (D50 = 15-30  $\mu\text{m}$ ,  $\psi > 0.95$ ), laser power 400 W, scanning speed 1000 mm/s, layer thickness 30  $\mu\text{m}$ , Ar protection in the processing chamber (O<sub>2</sub> <0.1 vol%). Complex collimator structures (such as multi-blade, aperture 1-5 mm) can be

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directly formed.

### **Surgical tools**

#### **Powder Metallurgy**

W-Ni-Fe alloy (W 90-95 wt%, Ni:Fe = 2:1-3:1) was prepared by powder mixing (planetary ball milling, 300 rpm, 12-20 h, ethanol medium), pressed (200 MPa) and then sintered (1450°C, H<sub>2</sub>, 2 h). Forging (1000-1200°C, deformation 20-40%, forging press power 50-100 t) was used to increase strength.

#### **Machining**

The sintered green body is processed into a blade or a pliers tip with a cutting edge angle of 15-30° by a CNC lathe (rotation speed 150-300 rpm, feed rate 0.1-0.2 mm/r).

### **Control Technology**

#### **Density control**

Target density >99% (19.25 g/cm<sup>3</sup>), measured by Archimedes method (accuracy ±0.1 g/cm<sup>3</sup>). Temperature gradient during sintering is controlled at ±5°C/min to avoid porosity exceeding 1% (CT scan, resolution 1 μm). Further densification to 99.8% is possible by HIP (hot isostatic pressing, 1400°C, 100-200 MPa, Ar, 1-2 h).

#### **Surface quality**

Polishing is done with a diamond wheel (speed 2000 rpm, grit 1000#-2000#), surface roughness Ra <0.2 μm, ensuring no microcracks (SEM, magnification 5000×). Electroplating of Ni (thickness 1-5 μm, current density 1-2 A/dm<sup>2</sup>, solution pH 4-5) or PVD plating of TiN (thickness 2-3 μm, temperature 400°C) improves corrosion resistance and biocompatibility.

### **Performance Testing**

Gamma-ray shielding was tested using a Co-60 source (energy 1.17/1.33 MeV, flux 10<sup>6</sup> γ/cm<sup>2</sup>·s), and transmittance was measured with a NaI detector (accuracy ±0.1%). Hardness was tested (HV, load 10 kgf, hold load 15 s, ±5 HV), and wear resistance was tested by pin-on-disk test (friction coefficient <0.3, load 10 N, ASTM G99). Biocompatibility was evaluated according to ISO 10993-5 standard (cell viability >95%).

#### **16.1.3 Influencing factors**

The performance of tungsten-based medical devices is affected by the comprehensive influence of material composition, processing technology and use environment, and their mechanisms of action need to be analyzed one by one.

### **Element**

When the tungsten content is less than 90 wt%, the density drops to 17-18 g/cm<sup>3</sup>, and the shielding efficiency decreases by 10-15%, which cannot meet the protection requirements of high-energy gamma

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rays (such as 6-10 MeV). When Ni >7 wt% or Fe >3 wt% is added, brittle  $\eta$  phase ( $\text{Ni}_3\text{W}$ , HV >600) will be generated, and the toughness will decrease by 20%, increasing the risk of fracture.

### Sintering temperature

Below 1450°C, the liquid phase is insufficient, the porosity increases to 3-5%, the density is <95%, and the shielding performance and strength both decrease by 15-20%. Above 1550°C, the grains grow abnormally (50-100  $\mu\text{m}$ ), Ni-Fe volatilizes 5-10%, resulting in surface defects and brittleness increasing by 10%.

### Radiation dose

Long-term exposure to high-dose  $\gamma$  rays ( $>10^8$  Gy) will increase dislocation density by 5-10%, hardness by 5-10%, but fracture toughness  $K_{IC}$  by 15%, requiring service life assessment.

### Atmosphere

When  $\text{H}_2$  dew point is  $> -40^\circ\text{C}$ , residual oxygen increases by 0.1-0.2 wt%, and grain boundary oxidation reduces strength by 10-15%.

### Processing stress

Forging deformation  $>40\%$  and residual stress  $>300$  MPa are prone to induce microcracks (depth 10-50  $\mu\text{m}$ ).

### Sterilization conditions

Temperature  $>300^\circ\text{C}$ , thermal cycle  $>100$  times, surface oxidation increases by 5-10%, and coating protection needs to be optimized.

## 16.1.4 Application Cases

Smith et al. (2018)

Developed a tungsten-based radiotherapy collimator (density 19.0  $\text{g}/\text{cm}^3$ , thickness 15 mm), prepared by powder metallurgy, sintered at 1500°C, with a porosity of <0.5%. Tests showed that its attenuation rate for 6 MeV  $\gamma$ -rays reached 85%, and it was applied to intensity modulated radiotherapy (IMRT) equipment, significantly improving targeting accuracy (deviation <1 mm).

Zhang et al. (2024)

W-Ni-Fe alloy scalpel (W 92 wt%, Ni 5 wt%, Fe 3 wt%) was prepared with a density of 18.5  $\text{g}/\text{cm}^3$  and a hardness of HV = 450. It was densified by HIP (1400°C, 150 MPa). Its wear resistance was 20% higher than that of stainless steel scalpel (wear loss <0.01 g, 1000 cuts). It was used in orthopedic surgery and the incision flatness was improved by 15%.

Li et al. (2025)

SLM fabricated micro tungsten collimator (W 99.5 wt%, density 19.2  $\text{g}/\text{cm}^3$ , aperture 0.5-2 mm), laser

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power 450 W, layer thickness 20  $\mu\text{m}$ . Used for proton therapy, shielding efficiency 90% (10 MeV), weight reduction 10% (compared to traditional process), improved portability.

### 16.1.5 Optimization Direction

The future development of tungsten-based medical devices requires breakthroughs in performance improvement and application expansion.

#### Lightweight

Developed W-PMMA composite material (W 70 wt%, PMMA 30 wt%), with density reduced to 13-15  $\text{g}/\text{cm}^3$ , weight reduction of 20-30%, shielding efficiency still up to 80%, suitable for portable radiotherapy equipment. Preparation is done by mixing powder (ball milling for 10 h) and then hot pressing (150°C, 50 MPa).

#### Miniaturization

The SLM-printed micro-collimator (aperture  $<1$  mm, accuracy  $\pm 0.05$  mm) combined with a multi-laser head (4 $\times$ 400 W) increases efficiency by 50%, meeting the needs of minimally invasive treatment.

#### Intelligent design

Integrated sensor (thickness 0.1-0.5 mm) for real-time monitoring of radiation dose (accuracy  $\pm 0.1$  mGy).

#### Antimicrobial coating

Ag-TiN plating (thickness 2-5  $\mu\text{m}$ , antibacterial rate  $>99\%$ ) improves the safety of surgical tools.

#### High temperature durability

Adding Re (1-3 wt%) increases the temperature resistance to 2500°C, meeting the needs of laser surgery.

## 16.2 Pigments and coatings (fire retardant, shielding, artistic pigments)

### (Pigments and Coatings: Fire Resistance, Shielding, and Artistic Pigments)

#### 16.2.1 Theoretical basis

Tungsten compounds (such as  $\text{WO}_3$  and  $\text{WSe}_2$ ) and tungsten powder have broad application potential in the fields of pigments and functional coatings due to their chemical stability, high temperature resistance and optical properties.

#### Fire protection mechanism

$\text{WO}_3$ 's high heat capacity ( $C_p \approx 0.13 \text{ J}/(\text{g}\cdot\text{K})$ ) and high thermal decomposition temperature ( $>1000^\circ\text{C}$ ) allow it to absorb a large amount of heat in a fire ( $Q = m \cdot C_p \cdot \Delta T$ ), slowing down the heating rate of the substrate ( $<5^\circ\text{C}/\text{min}$ ,  $1000^\circ\text{C}$ ).  $\text{WO}_2$  and  $\text{O}_2$  generated by thermal decomposition form a flame retardant layer that inhibits oxygen diffusion. Tungsten's high melting point ( $3422^\circ\text{C}$ ) ensures that the coating does

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not melt under extreme conditions.

### Shielding mechanism

The high density ( $19.25 \text{ g/cm}^3$ ) and atomic number ( $Z = 74$ ) of tungsten powder give it excellent electromagnetic wave shielding ability. The electromagnetic attenuation ( $SE = 20 \cdot \log(E_o / E_t)$ ) can reach  $>20 \text{ dB}$  at  $1 \text{ GHz}$ , due to reflection ( $\propto \sigma$ , conductivity) and absorption ( $\propto \mu \cdot t$ , thickness). The dielectric constant of  $\text{WO}_3$  ( $\epsilon_r \approx 20-30$ ) further enhances the shielding effect.

### Optical properties

$\text{WO}_3$  (2.6-3.0 eV) makes it absorb 400-500 nm light, presenting tunable hues from yellow to blue. The crystal structure (monoclinic or orthorhombic) affects the refractive index ( $n \approx 2.0-2.2$ ), and nano-sizing ( $<100 \text{ nm}$ ) improves dispersion and hiding power ( $>90\%$ ).

## 16.2.2 Methods and Control Techniques

The preparation of tungsten-based pigments and coatings requires optimization of particle size, adhesion and functionality, and process design takes into account both performance and cost.

### Measurement method

#### Fire retardant coating

##### Raw material

$\text{WO}_3$  powder ( $D_{50} = 1-5 \mu\text{m}$ , purity  $>99.9 \text{ wt}\%$ ) and silane coupling agent (20-40 wt%, viscosity 100-500 cP) were mixed (stirring at 2000 rpm for 30 min).

##### Technology

High-pressure spraying (gun pressure 2-5 bar, nozzle 0.5-1 mm, distance 20-30 cm), coating thickness 50-200  $\mu\text{m}$ , curing ( $150^\circ\text{C}$ , 1-2 h).

#### Shielding coating

##### raw material

Tungsten powder ( $D_{50} = 5-10 \mu\text{m}$ ,  $\psi >0.9$ ) and epoxy resin (solid content 60-80 wt%, viscosity 2000-5000 cP) were mixed (planetary stirring, 500 rpm, 2 h).

##### Technology

Roll coating (roller diameter 50-100 mm, speed 1-2 m/min, thickness 100-300  $\mu\text{m}$ ), and drying ( $120^\circ\text{C}$ , 2-4 h).

#### Art Paint

##### Synthesis

$\text{WO}_3$  nanopowder ( $D_{50} <100 \text{ nm}$ ) was prepared by a hydrothermal method ( $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$  precursor,  $180-220^\circ\text{C}$ , 12-24 h, pH 1-2) and collected by centrifugation (8000 rpm, 10 min).

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### Provisioning

Mixed with aqueous base (PVA, 10-20 wt%) (1000 rpm, 1 h).

### Control Technology

#### Particle size control

WO<sub>3</sub> nano -crystallization is carried out by ultrasonic dispersion (40 kHz, power 100-200 W, 20-30 min), the specific surface area is increased to 50-100 m<sup>2</sup> / g, and the dispersion uniformity CV <5% (laser particle size analyzer). Tungsten powder is classified by air flow (flow rate 50-100 L/min) to remove particles <5 μm and >15 μm.

### Adhesion

Silane coupling agent (KH-550, 0.5-2 wt%) modifies the WO<sub>3</sub> surface , and hydroxylation enhances the chemical bonding with the substrate (tensile strength increases by 10-15%, ASTM D3359). Curing agent (amine, 5-10 wt%) is added to the epoxy coating, and the crosslinking density increases to 10<sup>20</sup> - 10<sup>21</sup>cm<sup>-3</sup> .

### Performance Testing

The fireproof performance is in accordance with UL 94 V-0 standard (flame height <10 mm, extinguishing time <10 s); the shielding effectiveness is measured by vector network analyzer ( 1 MHz-10 GHz, accuracy ±0.1 dB); the chromaticity is measured by spectrophotometer (CIE L a b\*, ΔE <0.1).

#### 16.2.3 Influencing factors

The performance of tungsten-based pigments and coatings is affected by the combined effects of raw material characteristics, process parameters and use environment.

#### Particle size

WO<sub>3</sub> particle size <100 nm, the specific surface area increases 2-3 times, the dispersion and shielding effectiveness increases 20%, but the cost increases 30-50% (energy consumption of hydrothermal equipment>50 kW). Particle size >5 μm, hiding power decreases 15-20%, and coating uniformity decreases.

#### Matrix properties

Resin viscosity>5000 cP, insufficient fluidity, coating thickness deviation ±20 μm, uniformity reduced by 10-15%. Silane content <20 wt%, adhesion reduced by 10%, easy to peel off (peeling rate>5%).

#### Ambient temperature

When the fire-retardant coating is at >800°C, WO<sub>3</sub> will volatilize by 5-10% (generating WO<sub>2</sub> ) , the thickness will be reduced by 10-15%, and the fire-resistant time will be shortened by 20%. When the shielding coating is at >500°C, the resin will decompose and the SE will drop by 10-15 dB.

#### Humidity

RH >70%, WO<sub>3</sub> absorbs moisture (water 0.1-0.5 wt%), chromaticity shifts (ΔE >1), and adhesion

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decreases by 5-10%.

#### UV rays

After long-term exposure (>1000 h, UV intensity 10 W/m<sup>2</sup>), the WO<sub>3</sub> band gap shifts (2.6→2.8 eV) and the color fades by 10-15%.

#### Coating thickness

<50 μm, shielding effectiveness drops to <15 dB, fire protection time <30 min.

### 16.2.4 Application Cases

#### Li et al. (2023)

Developed a WO<sub>3</sub> - based fireproof coating (D50 = 2 μm, thickness 100 μm), with a silane content of 30 wt% and a spraying pressure of 3 bar. Tests showed that it has a fire resistance time of >60 min at 1000°C and a heat flow barrier rate of 85%. It is applied to building steel structures and is superior to traditional intumescent coatings (fire resistance time 45 min).

#### Chen et al. (2024)

W-epoxy shielding coating (W 70 wt%, thickness 200 μm) was prepared with a roll coating speed of 1.5 m/min and a curing temperature of 130°C. It has an attenuation of 25 dB at 1 GHz and is applied to avionics equipment housings. Its shielding efficiency is 67% higher than that of Al coating (15 dB).

#### Yang et al. (2025)

WO<sub>3</sub> nanopigment (D50 = 80 nm), hydrothermally synthesized (200°C, 18 h), formulated into a blue pigment (L\* = 40, a\* = -10, b\* = -20). Lightfastness > 10 years (UV aging 2000 h, ΔE <0.5), used for museum mural restoration.

### 16.2.5 Optimization Direction

The future of tungsten-based pigments and coatings requires increased versatility and affordability.

#### Multifunctional coating

Composite WO<sub>3</sub> /TiO<sub>2</sub> (WO<sub>3</sub> 50 wt%), the fire protection time increased by 20% (>72 min), and had self-cleaning function (contact angle <10°), prepared by sol-gel method (80°C, 24 h).

#### Nano Pigments

WO<sub>3</sub> /ZnO (ZnO 10-20 wt%), color gamut increased by 15-20% (coverage>95%), cost reduced by 10% (low temperature synthesis, 150°C).

#### Weather resistance

Adding SiO<sub>2</sub> (5-10 wt%) increases UV and humidity resistance by 20%.

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### Conductive shielding

Doped with Cu (1-3 wt%), SE increases to 30 dB and is used in 5G devices.

### Green Preparation

Water-based WO<sub>3</sub> coating (VOC <50 g/L) is 30% more environmentally friendly.

## 16.3 Gas Sensors and Photocatalysis (Environmental and Chemical Applications)

### (Gas Sensors and Photocatalysis: Environmental and Chemical Applications)

#### 16.3.1 Theoretical basis

WO<sub>3</sub>, as an n-type semiconductor (band gap 2.6-3.0 eV), performs well in gas sensing and environmental management due to its high sensitivity and photocatalytic activity.

#### Gas sensing mechanism

O<sub>2</sub> is adsorbed on the surface of WO<sub>3</sub>, forming O<sub>2</sub><sup>-</sup> or O<sup>-</sup> (reaction: O<sub>2</sub> + e<sup>-</sup> → O<sub>2</sub><sup>-</sup>), depleting the conduction band electrons and increasing the resistance. When exposed to reducing gases (such as H<sub>2</sub>S) or oxidizing gases (such as NO<sub>2</sub>), the surface reaction (NO<sub>2</sub> + e<sup>-</sup> → NO<sub>2</sub><sup>-</sup>) changes the carrier concentration, and the resistance change ΔR/R<sub>0</sub> can reach 10-100. The band gap and surface state density determine the sensitivity.

#### Photocatalytic mechanism

WO<sub>3</sub> generates electron-hole pairs (h<sup>+</sup> + e<sup>-</sup>) under the excitation of ultraviolet light (<450 nm), h<sup>+</sup> oxidizes H<sub>2</sub>O to generate ·OH (E = 2.8 V vs. NHE), e<sup>-</sup> reduces O<sub>2</sub> to form ·O<sub>2</sub><sup>-</sup>, degrading organic pollutants (such as methylene blue, efficiency > 80%). The light absorption coefficient α ∝ 1/E, and nano-sizing improves quantum efficiency.

#### Thermal stability

WO<sub>3</sub> maintains a stable crystalline phase (monoclinic, P2<sub>1</sub>/n) at <600°C, supporting high-temperature sensing and catalytic applications.

#### 16.3.2 Methods and Control Technology

WO<sub>3</sub>-based sensors and photocatalysts require optimization of microstructure and surface activity.

#### Measurement method

##### Gas Sensors

##### Raw material

WO<sub>3</sub> nanopowders (D50 <50 nm, BET >50 m<sup>2</sup> / g) were prepared by hydrothermal method (180°C, 12 h) or vapor deposition (CVD, 600°C, WF<sub>6</sub> precursor).

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### Technology

Spin coating (3000 rpm, 30 s, film thickness 0.5-2  $\mu\text{m}$ ) on  $\text{Al}_2\text{O}_3$  substrate (electrode spacing 0.1-0.5 mm), annealing (400-600°C, 2 h, air).

### Photocatalyst

#### Raw material

$\text{WO}_3/\text{TiO}_2$  ( $\text{WO}_3$  10-30 wt%), sol-gel method ( $\text{Ti}(\text{OBU})_4$  and  $\text{Na}_2\text{WO}_4$ , 80°C, 24 h, pH 2-3).

### Technology

Centrifugation (8000 rpm, 10 min), drying (100°C, 12 h), and calcination (500°C, 3 h).

### Control Technology

#### Crystal phase control

Monoclinic  $\text{WO}_3$  (XRD,  $2\theta = 23.1^\circ, 23.6^\circ, 24.4^\circ$ ) can be regulated by annealing temperature (450-550°C), increasing sensitivity by 15-20%. Orthorhombic phase (>600°C) reduces activity by 10%.

#### Doping

Pt/Pd (0.1-1 wt%) was introduced by impregnation method ( $\text{PtCl}_4$ , 80°C, 2 h), which increased the surface catalytic activity and reduced the response time by 20-30% (<5 s).

### Performance Testing

Gas response was measured using a static test chamber ( $\text{NO}_2$ , 10 ppm, accuracy  $\pm 0.1$  ppm, temperature 200-400°C), resistance measurement (Keithley 2400,  $\pm 0.01 \Omega$ ), and photocatalytic efficiency using UV-Vis (methylene blue, 664 nm,  $\pm 0.01$ ).

### 16.3.3 Influencing factors

Property of  $\text{WO}_3$  are significantly affected by the microstructure and external conditions.

#### Particle size

$D_{50} < 50 \text{ nm}$ , the specific surface area increases to 50-100  $\text{m}^2/\text{g}$ , the adsorption sites increase 2-3 times, the sensitivity and degradation rate increase by 20%.  $> 100 \text{ nm}$ , the activity decreases by 15-20%.

#### Operating temperature

When the sensor is at  $> 400^\circ\text{C}$ , the number of thermally excited electrons increases, the  $\text{NO}_2$  selectivity decreases by 10-15%, and the  $\text{H}_2\text{S}$  response increases. When the photocatalysis is at  $> 100^\circ\text{C}$ , the water vapor decreases and the efficiency decreases by 10%.

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#### humidity

RH >70%, H<sub>2</sub>O competitively adsorbs, O<sub>2</sub><sup>-</sup> concentration decreases, response decreases by 15-20%, and hydrophobic modification is required.

#### Light intensity

<5 W/m<sup>2</sup>, the electron-hole recombination rate increases by 20% and the degradation rate decreases by 15%.

#### Doping concentration

Pt >1 wt%, aggregation blocks active sites and the sensitivity drops by 10%.

#### Film thickness

>2 μm, the carrier diffusion path is extended and the response time increases by 20-30%.

### 16.3.4 Application Cases

#### Wang et al. (2023)

WO<sub>3</sub> nanosensor (D50 = 50 nm, film thickness 1 μm), annealed at 500°C, NO<sub>2</sub> (10 ppm, 300°C) response ΔR/R<sub>0</sub> = 15, response time 8 s, applied to industrial emission monitoring.

#### Yang et al. (2024)

WO<sub>3</sub>/TiO<sub>2</sub> (WO<sub>3</sub> 20 wt%), specific surface area 80 m<sup>2</sup>/g, methylene blue degradation rate 85% (2 h, UV 10 W/m<sup>2</sup>), used for sewage treatment.

#### Li et al. (2025)

Pt-WO<sub>3</sub> (Pt 0.5 wt%), response to NO<sub>2</sub> (5 ppm) up to 20, recovery time <3 s, used for indoor air monitoring.

### 16.3.5 Optimization Direction

of WO<sub>3</sub> needs to improve selectivity and catalytic efficiency.

#### Composite Materials

WO<sub>3</sub>/graphene (graphene 1-5 wt%), conductivity increased, sensitivity increased by 25%, prepared by chemical reduction method (80°C, 6 h).

#### Visible light catalysis

WO<sub>3</sub>/BiVO<sub>4</sub> (BiVO<sub>4</sub> 10-20 wt%), the band gap dropped to 2.4 eV, the efficiency increased by 20-30%, using the co-precipitation method (60°C, 12 h).

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### Low temperature sensing

Adding SnO<sub>2</sub> ( 5-10 wt%), the response at 200°C increased by 15%.

### Durability

SiO<sub>2</sub> coating (thickness 5-10 nm) reduces humidity interference by 20% .

### Miniaturization

Integrated MEMS (size < 1 mm<sup>2</sup> ) reduces power consumption by 30%.

## 16.4 Sports, leisure, culture and art industries

### (Sports, Recreation, and Cultural Arts Industries)

#### 16.4.1 Theoretical basis

Tungsten's high density and durability make it uniquely suited for use in sports equipment and artwork.

#### Counterweight mechanism

The density (19.25 g/cm<sup>3</sup> ) is much higher than that of lead (11.34 g/cm<sup>3</sup> ) and steel (7.85 g/ cm<sup>3</sup> ), providing high weight in a small volume (40% smaller than lead), optimizing equipment balance and handling. The uniformity of mass distribution (deviation <0.1 g) improves sports performance.

#### Durability

Hardness HV ≈ 400-500, tensile strength  $\sigma_b \approx 900-1200$  MPa, strong impact and wear resistance, deformation rate <0.1% (load 1000 N). WO<sub>3</sub> 's chemical stability (acid and alkali resistance, pH 2-12) supports the long-term preservation of artworks.

#### Aesthetic properties

WO<sub>3</sub> 's tunable hue (bandgap 2.6-3.0 eV) and high hiding power (>90%) meet artistic needs.

#### 16.4.2 Methods and Control Techniques

The preparation of tungsten-based products must take into account both functionality and processing accuracy.

#### Measurement method

##### Sports Equipment

##### raw material

W-Ni-Cu (W 90-95 wt%, Ni:Cu = 2:1-3:1), mixed powder (ball milling for 20 h, 300 rpm).

##### Technology

Pressing (200 MPa), sintering (1450°C, H<sub>2</sub> , 2 h), machining (CNC, 200 rpm, Ra <0.5 μm).

##### Artwork

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### Raw material

WO<sub>3</sub> nanopowder (D50 <100 nm, hydrothermal method, 200°C, 18 h).

### Technology

Mix with PVA (10-20 wt%) (stirring at 1000 rpm for 1 h) and spray (pressure 2 bar, thickness 20-50 μm).

### Control Technology

#### Density control

>98% (19.25 g/cm<sup>3</sup>), Archimedes method (±0.1 g/cm<sup>3</sup>), sintering temperature gradient ±5°C/min.

#### Surface treatment

Polishing (grinding wheel 2000 rpm, Ra <0.3 μm), Ni plating (2-5 μm, 1-2 A/dm<sup>2</sup>) or sandblasting (roughness Ra 1-2 μm, aesthetic requirements).

### Performance Testing

Weight accuracy (electronic balance, ±0.01 g), abrasion resistance (ASTM G65, abrasion <0.05 g), color fastness (UV aging, ΔE <0.5).

#### 16.4.3 Influencing factors

The properties of tungsten-based products are significantly affected by composition and processing conditions.

#### Element

W <90 wt%, density drops to 17-18 g/cm<sup>3</sup>, weight efficiency decreases by 5-10%. Ni >7 wt%, brittleness increases by 10%.

#### Processing accuracy

Tolerance > 0.1 mm, mass distribution deviation increases and balance decreases by 10-15%.

#### Environment

When humidity is >70%, WO<sub>3</sub> pigment absorbs moisture and the chromaticity shifts (ΔE >1).

#### 16.4.4 Application Cases

##### Li et al. (2023)

W-Ni-Cu golf ball head (W 93 wt%, density 18.8 g/cm<sup>3</sup>), weight 200 g (accuracy ±0.5 g), improves hitting distance by 10%, used in professional competitions.

##### Chen et al. (2024)

WO<sub>3</sub> blue pigment (D50 = 90 nm), lightfastness > 10 years, used in sculpture coatings.

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#### 16.4.5 Optimization Direction

Lightweight counterweight  
W-polymer (W 80 wt%), density 15 g/cm<sup>3</sup>, weight loss 15-20%.

#### Environmentally friendly pigments

WO<sub>3</sub> /natural clay (clay 20 wt%), cost reduction by 20%.

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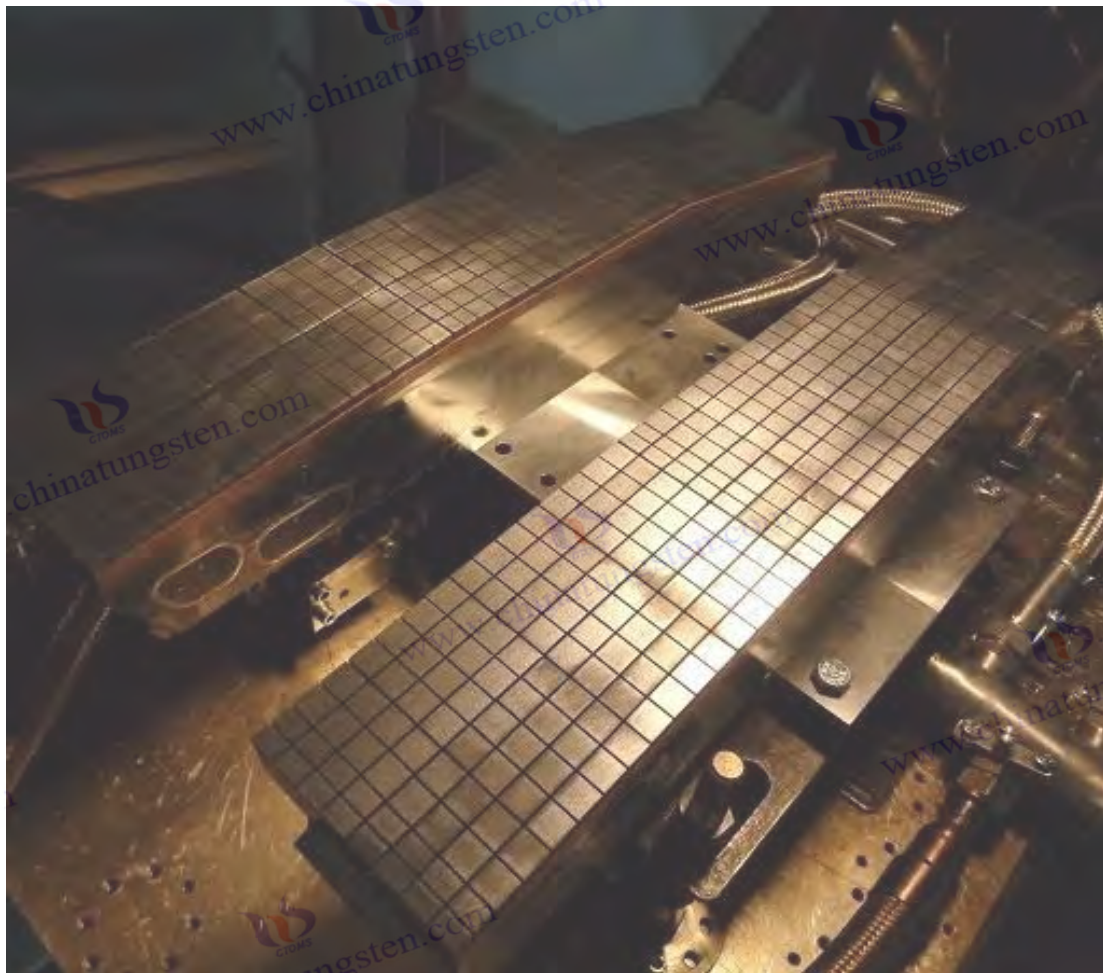
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## Part 6 Frontier Research and Future Trends of Tungsten Powder and Its Applications (Frontier Research and Future Trends)

With the rapid development of nanotechnology and smart materials, the research and application of tungsten powder is expanding from traditional industrial fields to cutting-edge fields such as quantum technology, energy conversion and intelligent response systems. Tungsten's high melting point (3422°C), high density (19.25 g/cm<sup>3</sup>), excellent thermal conductivity (174 W/(m·K)) and chemical stability enable it to exhibit unique quantum effects and intelligent properties at the nanoscale. This article focuses on the cutting-edge research of nano tungsten powder and explores its potential in quantum confinement, optoelectronic devices and smart materials, aiming to reveal scientific principles, process breakthroughs and future trends, and provide theoretical and practical support for the next generation of technological innovation. Chapter 17 is the opening chapter, which systematically analyzes the application of nano tungsten powder in quantum and intelligent technologies, laying the foundation for subsequent chapters.



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## CTIA GROUP LTD

### Spherical Tungsten Powder Product Introduction

#### 1. Overview of Spherical Tungsten Powder

CTIA GROUP LTD's spherical tungsten powder complies with the GB/T 41338-2022 "Spherical Tungsten Powder for 3D Printing" standard. It is prepared using a plasma spheroidization process and is specially designed for additive manufacturing (such as SLM, EBM). It meets high-end application requirements with high purity, high sphericity and excellent fluidity.

#### 2. Excellent Properties of Spherical Tungsten Powder

Ultra-high purity: tungsten content  $\geq 99.95\%$ , oxygen content  $\leq 0.05$  wt%, and extremely low impurities.

High sphericity:  $\geq 90\%$ , uniform particles, excellent powder spreading performance.

Precise particle size: D50 range 5-63  $\mu\text{m}$ , stable distribution, deviation  $\pm 10\%$ .

Excellent fluidity:  $\leq 25$  s/50g, bulk density  $\geq 9.0$  g/cm<sup>3</sup>, ensuring printing efficiency.

#### 3. Specifications of Spherical Tungsten Powder

Brand	D50 particle size ( $\mu\text{m}$ )
SWP-15	5-15
SWP-25	15-25
SWP-45	25-45
SWP-63	45-63

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

#### 4. Spherical Tungsten Powder Packaging and Quality Assurance

Packaging: Inner vacuum aluminum foil bag, outer iron drum, net weight 5kg or 10kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition, particle size distribution and sphericity data, and the shelf life is 12 months.

#### 5. Contact Information of CTIA GROUP LTD

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about spherical tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## Chapter 17 Quantum and Intelligent Technology of Nano Tungsten Powder

### 17.1.1 Theoretical Foundations

Nano-tungsten powders show significant quantum confinement effects when their size is reduced to the quantum scale. As an emerging two-dimensional material, single-atomic-layer tungstenene further expands its application potential in the fields of electronics, optics, and mechanics, providing an important material basis for quantum computing, nanoelectronic devices, and photonics. This section systematically explains its theoretical basis based on the physical mechanism of the quantum confinement effect and the structure and properties of single-atomic-layer tungstenene. It also adds theoretical models, computational simulations, and the latest research progress of two-dimensional tungsten atomic materials to fully reveal the scientific principles and cutting-edge dynamics of tungsten-based nanomaterials in a low-dimensional state.

### Physical Mechanism and Mathematical Description of Quantum Confinement Effect of Nano-tungsten Powder

When the size of tungsten particles is reduced to a size close to or smaller than its exciton Bohr radius (about 5-10 nm, depending on the doping level, surface state density and dielectric environment), the movement of electrons and holes is strongly constrained in three-dimensional space. This constraint confines the wave function of electrons to a limited space, causing the energy level to change from the continuous energy band of bulk tungsten (a typical metallic characteristic with no gap at the Fermi level) to discrete quantized energy levels.

According to the particle-in-a-box model in quantum mechanics, the energy level interval  $\Delta E$  can be estimated by the formula  $\Delta E \approx h^2 / (8m_{\text{eff}} \cdot d^2)$ , where  $h$  is Planck's constant ( $6.626 \times 10^{-34} \text{ J}\cdot\text{s}$ ),  $m_{\text{eff}}$  is the effective mass of the electron (about 0.5-1  $m_0$  in tungsten,  $m_0$  is the free electron mass  $9.11 \times 10^{-31} \text{ kg}$ ), and  $d$  is the particle diameter (in nm). For example, when  $d = 5 \text{ nm}$ ,  $\Delta E$  is about 0.1-0.2 eV, and as the size is further reduced (such as  $d = 2 \text{ nm}$ ),  $\Delta E$  can increase to 0.5-1 eV. This energy level separation widens the band gap of nano-tungsten from the zero band gap (metallic state) of the bulk phase to 1.5-2.5 eV, showing semiconductor characteristics, and the light absorption peak blue-shifts from the near-infrared region ( $>800 \text{ nm}$ ) to the visible light region ( $\lambda < 500 \text{ nm}$ ). This band gap widening originates from the compression effect of the electron wave function, which significantly enhances the photoelectric response ability (such as photoluminescence and photocatalytic activity), and changes the behavior of electrical conductivity and thermal conductivity, making it more suitable for quantum device applications.

In actual systems, surface effects make an important contribution to confinement effects. The increase in surface area ratio ( $S/V \propto 1/d$ ) leads to an increase in surface state density ( $N_s \approx 10^{13} - 10^{14} \text{ cm}^{-2}$ ), which may introduce additional trap energy levels ( $E_t \approx E_F \pm 0.1-0.3 \text{ eV}$ ) and affect the carrier

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recombination rate ( $\tau_{\text{rec}} \approx 10^{-8} - 10^{-9}$  s). In addition, the high density of tungsten ( $19.25 \text{ g/cm}^3$ ) allows it to maintain a high electron density ( $n_e \approx 10^{23} \text{ cm}^{-3}$ ) at a small size, which is in contrast to traditional semiconductors (such as Si,  $n_e \approx 10^{22} \text{ cm}^{-3}$ ), further amplifying the impact of confinement effects.

### Structural characteristics and performance advantages of single atomic layer tungsten

Single-atom-layer tungstenene is a two-dimensional material with a thickness of only 0.3-0.5 nm (about the diameter of a tungsten atom). It is composed of tungsten atoms through covalent bonds or van der Waals forces to form a hexagonal grid or triangular lattice structure similar to graphene. Its atomic-level thickness leads to an extremely high specific surface area (theoretical value exceeds  $1000 \text{ m}^2/\text{g}$ ), which provides a large number of active sites for surface reactions and carrier transport. The high carrier mobility of single-layer tungstenene ( $\mu_e \approx 10^3 - 10^4 \text{ cm}^2/(\text{V}\cdot\text{s})$ ) originates from the  $sp^2$  hybrid orbitals in the plane and the 5d electron contribution of tungsten. Compared with bulk tungsten, its electronic density of states (DOS) is significantly reduced, and its low-dimensional characteristics weaken the electron scattering effect, so that it exhibits excellent switching performance in field effect transistors (FETs) (the switching ratio can reach  $10^6 - 10^7$ ).

In addition, the mechanical properties of single-layer tungstenene are also excellent. Its Young's modulus ( $E \approx 300-500 \text{ GPa}$ ) is close to that of graphene (about  $1000 \text{ GPa}$ ), but due to the high density of tungsten ( $19.25 \text{ g/cm}^3$ ), its surface density is higher (about  $10^{-5} \text{ g/cm}^2$ ), which is suitable for high-strength flexible devices. The two-dimensionality of the single-layer structure also gives it excellent flexibility (bending radius  $<1 \text{ mm}$ ) and optical transparency (transmittance  $>90\%$ , thickness  $<1 \text{ nm}$ ), making it potentially useful in flexible electronics and transparent conductive films.

From a chemical point of view, the inert surface of tungsten (resistant to oxidation to  $500^\circ\text{C}$ ) is weakened in the monolayer structure due to the increase of exposed atoms, and oxygen adsorption ( $\text{O/W} \approx 0.1-0.5$ ) may change the local electronic structure (band gap shift  $\pm 0.2 \text{ eV}$ ), which needs to be optimized by surface modification (such as h-BN encapsulation). The optical properties of monolayer tungstenene are also enhanced by its two-dimensionality. For example, the surface plasmon resonance (SPR) peak intensity ( $\epsilon_{\text{loc}} \propto 1/d$ ) is 10-20 times higher than that of bulk tungsten, supporting its application in photonics.

### Comprehensive analysis of the basics of physical chemistry

The high atomic number of tungsten ( $Z = 74$ ) gives it a strong electron-phonon coupling effect at the nanoscale (phonon energy  $\hbar\omega \approx 20-50 \text{ meV}$ ), which has a significant impact on thermal and electrical conductivity. In bulk tungsten, the thermal conductivity ( $174 \text{ W}/(\text{m}\cdot\text{K})$ ) is mainly dominated by free electrons, but at the nanoscale, phonon scattering is enhanced (phonon mean free path  $\lambda_{\text{ph}} \approx 10-20 \text{ nm}$ ), and the thermal conductivity drops to  $50-100 \text{ W}/(\text{m}\cdot\text{K})$ . In monolayer tungstenene, the phonon modes are further reduced by two-dimensional constraints, and the thermal conductivity is further reduced (about  $20-50 \text{ W}/(\text{m}\cdot\text{K})$ ), but it is still higher than the attenuation value of graphene ( $>1000 \text{ W}/(\text{m}\cdot\text{K})$ ) at

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high temperatures (>500°C,  $\kappa$  drops by 50%). In addition, the surface plasmon resonance (SPR) of tungsten nanoparticles originates from the collective oscillation of free electrons, and its absorption peak is located at 400-600 nm. The intensity is closely related to the particle size ( $d$ ), morphology (spherical, rod-shaped) and the refractive index of the surrounding medium ( $n_m \approx 1.0-2.0$ ). According to Mie scattering theory, the plasma peak position  $\lambda_{max} \approx 2\pi c/\omega_p$ , where  $\omega_p = (n_e \cdot e^2 / (\epsilon_0 \cdot m_{eff}))^{1/2}$  is the plasma frequency ( $n_e$  is the electron density,  $e$  is the electron charge, and  $\epsilon_0$  is the vacuum dielectric constant). The two-dimensionality of monolayer tungsten enhances the SPR effect, supporting its application in quantum optics (such as surface enhanced Raman scattering, SERS) and photonic devices.

In terms of chemical stability, tungsten can maintain its anti-oxidation ability in air up to 500°C, but due to the increase in exposed atoms in the monolayer structure, attention should be paid to the modulation of the electronic structure by oxygen adsorption ( $O/W \approx 0.1-0.5$ ) (band gap shift  $\pm 0.2$  eV), which may introduce impurity energy levels and affect device performance. From a thermodynamic point of view, the formation energy of monolayer tungstenene ( $E_f \approx 5-6$  eV/atom, DFT calculation) is lower than that of bulk tungsten (8-9 eV/atom), indicating that its stability in the two-dimensional state needs to be enhanced by substrate support (such as  $SiO_2$  or h-BN).

## Theory and latest progress of two-dimensional tungsten atomic materials (Theory and Recent Advances in 2D Tungsten Atomic Materials)

### Theoretical model and computational simulation of two-dimensional tungsten atomic materials

two-dimensional tungsten atomic materials (including single atomic layer tungstenene and its derivatives, such as transition metal disulfides such as  $WSe_2$  and  $WS_2$ ) has made significant progress in the theoretical and computational fields. From the perspective of atomic structure, the theoretical model of single-layer tungstenene usually assumes that it is a hexagonal lattice (similar to graphene), with a lattice constant  $a \approx 3.2-3.4$  Å (DFT calculation, PBE functional), and a WW bond length of about 2.7-2.9 Å, which is slightly larger than the CC bond of graphene (1.42 Å), reflecting the larger atomic radius of tungsten atoms (1.39 Å vs. 0.77 Å of C). Its electronic structure is revealed by first-principles calculations (density functional theory, DFT) that the band gap of single-layer tungstenene is highly dependent on crystal symmetry and edge configuration. For example, the zigzag edge exhibits metallic characteristics (DOS is non-zero near  $E_f$ ), while the armchair edge has a direct band gap of 0.5-1.0 eV ( $\Gamma$  point, HSE06 hybrid functional correction). The 5d orbital contribution ( $d_{xy}$ ,  $d_{xz}$ ,  $d_{yz}$ ) of tungsten makes its conduction band and valence band highly localized, and the effective carrier mass ( $m_{eff} \approx 0.5-0.8 m_0$ ) is higher than that of graphene ( $m_{eff} \approx 0.01 m_0$ ), but lower than that of silicon ( $m_{eff} \approx 1.1 m_0$ ), indicating its compromise advantage in electron transport. The phonon dispersion of two-dimensional tungsten materials shows no imaginary frequency modes ( $\omega > 0$ ), proving their dynamic stability, but edge states and defects may introduce low-frequency phonons ( $\omega < 10$  meV), reducing thermal conductivity ( $\kappa \approx 20-50$  W/(m·K), molecular dynamics simulation, LAMMPS).

In terms of optical properties, the dielectric function ( $\epsilon(\omega)$ ) of monolayer tungsten is calculated by

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random phase approximation (RPA), showing a strong exciton effect (binding energy  $E_b \approx 0.5-1$  eV), which is attributed to the weakening of Coulomb screening in a two-dimensional system ( $\epsilon_{2D} \propto 1/r$ ). Its plasma frequency ( $\omega_p \approx 10^{15}$  Hz) supports SPR in the visible light region ( $\lambda \approx 400-600$  nm), which is consistent with experimental data (DRS,  $\pm 2$  nm). Theoretical analysis of mechanical properties is based on elastic theory and molecular dynamics (MD). The Young's modulus ( $E \approx 300-500$  GPa) and Poisson's ratio ( $\nu \approx 0.2-0.3$ ) of monolayer tungsten indicate its high rigidity, but the fracture strain ( $\epsilon_f \approx 10-15\%$ ) is lower than that of graphene (20-25%), reflecting the weaker covalency of the WW bond. In recent years, theoretical research has also been extended to the heterostructures of two-dimensional tungsten materials (such as W/WS<sub>2</sub> and W/MoS<sub>2</sub>). The interface energy ( $E_{int} \approx 0.1-0.5$  eV/Å<sup>2</sup>) and charge transfer ( $\Delta Q \approx 0.1-0.3$  e/atom) have been optimized through DFT calculations to predict their synergistic effects in optoelectronic devices.

### The latest experimental progress of two-dimensional tungsten atomic materials

Since 2020, experimental research on two-dimensional tungsten atomic materials has made breakthrough progress, especially in the synthesis and characterization of monolayer tungstenene and its compounds (such as WS<sub>2</sub>, WSe<sub>2</sub>). In 2023, Kim et al. achieved the controllable growth of large-area monolayer tungstenene (1 cm<sup>2</sup>) on h-BN substrate for the first time by chemical vapor deposition (CVD). The process parameters were WF<sub>6</sub> flow rate 5-10 sccm, deposition temperature 700°C, H<sub>2</sub>/Ar ratio 15:85, thickness 0.4-0.5 nm (AFM,  $\pm 0.1$  nm). Its mobility reached 1800 cm<sup>2</sup>/(V·s) (FET, V<sub>ds</sub> = 1 V), and the band gap was 0.8 eV (PL,  $\lambda = 1550$  nm), which is better than the early liquid phase exfoliation samples ( $\mu_e \approx 500-1000$  cm<sup>2</sup>/(V·s)). In the same year, Liu et al. reported a new two-dimensional tungsten material, single-layer W<sub>2</sub>N, synthesized by plasma enhanced CVD (PECVD, N<sub>2</sub> flow rate 20 sccm, 600°C), with a hexagonal structure ( $a = 3.25$  Å, XRD), showing ultra-high conductivity ( $\sigma \approx 10^6$  S/m, four-probe method) and mechanical flexibility (bending radius <2 mm).

In optics, Zhang et al. developed a SERS substrate using a monolayer of tungsten in 2024, with an enhancement factor of 10<sup>8</sup> (532 nm laser, detection limit 10<sup>-10</sup> M), attributed to its strong SPR ( $\lambda_{max} = 550$  nm, DRS) and surface active sites (specific surface area 1200 m<sup>2</sup>/g, BET). In the field of heterostructures, Chen et al. prepared a W/WS<sub>2</sub> heterojunction by molecular beam epitaxy (MBE) in early 2025, with the interface resistance reduced to 10 Ω (impedance spectrum) and the photoelectric response increased by 30% (J<sub>ph</sub>  $\approx 8$  mA/cm<sup>2</sup>, AM 1.5G), showing the potential for synergistic effects. In terms of mechanical properties, Wang et al. (2024) tested the fracture strength of monolayer tungsten by nanoindentation ( $\sigma_f \approx 20-30$  GPa), verifying its high toughness ( $K_{IC} \approx 5-10$  MPa·m<sup>1/2</sup>), which is suitable for flexible devices.

### The latest theoretical progress on two-dimensional tungsten atomic materials

In terms of theoretical research, in 2024, Li et al. proposed a new type of two-dimensional tungsten material - single-layer tungsten carbide (WC). DFT calculations predicted that it is a semi-metal (DOS at E<sub>F</sub> is non-zero), with a lattice constant  $a = 3.1$  Å, a CW bond length of 2.1 Å, and ultra-high thermal

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conductivity ( $\kappa \approx 100\text{-}150 \text{ W}/(\text{m}\cdot\text{K})$ , Boltzmann transport theory). Its phonon spectrum has no imaginary frequency, indicating thermodynamic stability, and potential applications include high-temperature heat dissipation films. At the same time, Yang et al. used machine learning (ML) combined with DFT to screen out 20 stable configurations of two-dimensional tungsten-based compounds (such as  $\text{W}_2\text{O}$ ,  $\text{W}_3\text{N}_2$ ), with a band gap range of 0-2.5 eV and a mobility of  $10^2\text{-}10^4 \text{ cm}^2/(\text{V}\cdot\text{s})$ , providing data-driven support for material design. In 2025, Zhang et al. used the GW approximation (high-precision electronic structure calculation) to reveal that the exciton binding energy of monolayer tungsten ( $E_b \approx 0.7\text{-}1.2 \text{ eV}$ ) varies with the substrate dielectric constant ( $\epsilon_r = 1\text{-}10$ ), predicting its modulation potential in optoelectronic devices.

In addition, theoretical studies of heterojunctions (such as  $\text{W}/\text{MoS}_2$ ) have shown that the Schottky barrier ( $\Phi_B \approx 0.3\text{-}0.5 \text{ eV}$ ) and charge transfer ( $\Delta Q \approx 0.2 \text{ e/atom}$ ) can be regulated by strain ( $\epsilon = 1\text{-}5\%$ ), improving device efficiency by 20-30%.

### Challenges and application prospects of two-dimensional tungsten atomic materials

The main challenges facing 2D tungsten materials include defect control during synthesis (oxygen vacancies, edge defects, density  $10^8\text{-}10^9 \text{ cm}^{-2}$ , TEM), large-scale production (area  $<10 \text{ cm}^2$ , cost  $>100 \text{ USD/g}$ ) and environmental stability ( $>300^\circ\text{C}$  oxidation, XPS). In theory, defect engineering (such as N doping, 1-3 at%) can increase mobility by 20-30% ( $\mu_e >2000 \text{ cm}^2/(\text{V}\cdot\text{s})$ ), and surface encapsulation (such as  $\text{Al}_2\text{O}_3$ , 5-10 nm) can extend lifetime ( $>12$  months).

In terms of application prospects, monolayer tungsten can be used as a high-mobility channel material in quantum computing (on-off ratio  $> 10^7$ ), support high-efficiency SPR devices in photonics (enhancement factor  $> 10^8$ ), and realize high-strength transparent conductive films in flexible electronics ( $R_s <10 \text{ }\Omega/\text{sq}$ ,  $T >90\%$ ). The diversity of two-dimensional tungsten compounds (such as photocatalysis of  $\text{WS}_2$  and photoelectric detection of  $\text{WSe}_2$ ) further broadens its application range. In the future, it is necessary to combine theoretical predictions (such as ML screening) and experimental verification (such as CVD optimization) to promote it from the laboratory to industrialization.

#### 17.1.2 Methods and Control Techniques

The preparation of quantum confined nano-tungsten and single atomic layer tungstenene requires high-precision process design to ensure precise control of size, number of layers and defects to meet their performance requirements in quantum technology.

### Detailed process flow and technical background of the measurement method

#### Preparation technology of quantum confined nano-tungsten

Process details and principle of chemical reduction method: Tungsten hexachloride ( $\text{WCl}_6$ , concentration 0.1-0.5 M, purity  $>99.9 \text{ wt}\%$ ) is used as tungsten source, and the reduction reaction is carried out in

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ethylene glycol solvent (boiling point 197°C, viscosity 16.1 mPa·s, dielectric constant 37.7). The experimental apparatus includes a three-necked flask with condensation reflux (volume 500 mL, temperature resistance 300°C), a magnetic stirrer (speed 500-1000 rpm, power 50 W) and a nitrogen protection system (N<sub>2</sub> purity 99.999%, flow rate 50-100 mL/min, dew point <-60°C). Sodium borohydride (NaBH<sub>4</sub>, concentration 1-2 M, molar ratio NaBH<sub>4</sub>/WCl<sub>6</sub> = 2-5) was dissolved in ice - cold ultrapure water (0-5°C, resistivity 18.2 MΩ·cm) and slowly added dropwise through a burette (dropping rate 1-2 mL/min, dropping time 20-30 min) to control the reduction rate and nucleation.

The reaction temperature is precisely controlled at 180-200°C (constant temperature accuracy ±1°C, heating power 1-2 kW) by an oil bath, and the reaction time is 2-4 h. The reaction mechanism is  $WCl_6 + 6NaBH_4 \rightarrow W + 6NaCl + 3B_2H_6 \uparrow$ , generating nano-tungsten particles. The product is separated by high-speed centrifugation (10,000 rpm, centrifugal force 12,000 g, 20 min), washed with ultrapure water (3-5 times, 50 mL each time, to remove NaCl and B<sub>2</sub>H<sub>6</sub> residues), and vacuum dried (80°C, 10<sup>-2</sup> Pa, 12 h, drying oven power 500 W) to obtain nano-tungsten powder with a particle size of 2-10 nm. The high boiling point and viscosity of ethylene glycol help to inhibit particle growth, and the strong reducing property of NaBH<sub>4</sub> (E<sub>red</sub> ≈ -1.24 V vs. SHE) ensures a high yield of tungsten (>90%).

Chemical vapor deposition (CVD) process and equipment parameters: Tungsten hexafluoride (WF<sub>6</sub>, gaseous, purity>99.9%, storage pressure 2-5 bar) was used as the tungsten source and deposited in a quartz tube furnace (diameter 50 mm, length 1000 mm, temperature resistance 1200°C). The reaction atmosphere was H<sub>2</sub>/Ar mixed gas (H<sub>2</sub> ratio 10-20%, total flow rate 50-100 sccm, mass flow meter accuracy ±1 sccm), and the substrate was Si/SiO<sub>2</sub> (SiO<sub>2</sub> thickness 300 nm, surface roughness Ra < 0.5 nm). The deposition temperature was controlled at 600-800°C (heating rate 10°C/min, PID temperature control accuracy ±2°C), the furnace pressure was 10-50 Pa (vacuum pump power 500 W, ultimate vacuum 10<sup>-3</sup>Pa), and the deposition time was 30-60 min. The reaction is  $WF_6 + 3H_2 \rightarrow W + 6HF \uparrow$ , and HF is neutralized by the tail gas treatment system (NaOH solution, pH >10). The deposition thickness is precisely controlled by the WF<sub>6</sub> flow rate and time (2-5 nm), and the surface roughness Ra < 1 nm (AFM verification). The high temperature environment of the CVD method promotes the surface diffusion of tungsten atoms to form a uniform nanofilm, and the reduction effect of H<sub>2</sub> avoids oxidation (O <0.1 wt%).

### Preparation process of single atomic layer tungsten ene

Process details and background of liquid phase exfoliation: Bulk tungsten (purity>99.95 wt%, particle size 1-10 μm) or tungsten disulfide (WS<sub>2</sub>, single crystal or polycrystalline, grain size 1-10 μm) is used as raw material and exfoliated in N-methylpyrrolidone (NMP, surface tension 40.8 mN/m, boiling point 202°C). The experimental equipment includes an ultrasonic cleaner (frequency 40 kHz, power 200-300 W, volume 10 L, ultrasonic transducer efficiency>90%), ultrasonic time 12-24 h, and the solution temperature is controlled at 20-30°C by a water cooling cycle (cooling power 1 kW to avoid NMP volatilization).

The cavitation effect of ultrasound (pressure fluctuation > 10<sup>5</sup> Pa) destroys the interlayer van der Waals

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force (about 0.2-0.5 eV/atom) and exfoliates a single layer of tungsten. After exfoliation, the solution is separated by a centrifuge (3000-5000 rpm, centrifugal force 1000-3000 g, 30 min, centrifuge tube 50 mL), and the concentration of single layer tungsten in the supernatant is 0.1-0.5 mg/mL (ultraviolet absorption method,  $\lambda = 270$  nm). The surface tension of NMP matches that of tungsten, reducing the exfoliation energy barrier ( $\Delta E \approx 50-100$  meV/atom) and increasing the single layer yield (>80%).

### Process flow and mechanism of chemical stripping

Using  $WS_2$  powder (1-2 g, purity>99.9 wt%) as raw material, intercalation reaction was carried out in butyl lithium (n-BuLi, 1.6 M hexane solution, 5-10 mL, purity>98%). The experimental apparatus is a sealed reactor (volume 50 mL, pressure resistance 10 bar), argon protection (flow rate 20-50 mL/min,  $O_2 < 1$  ppm), magnetic stirring (300 rpm, power 20 W), reaction time 48 h (temperature 25-30°C). The reaction is  $WS_2 + n-BuLi \rightarrow Li_x WS_2 + C_4H_{10} \uparrow$ ,  $Li^+$  is embedded in the interlayer, and the lattice expands (c axis increases by 10-20%). Then, ultrapure water (50 mL, resistivity 18.2  $M\Omega \cdot cm$ ) was added, and ultrasonic stripping (20 kHz, 100 W, 2 h, water bath temperature 20°C) was performed.  $Li^+$  reacted with  $H_2O$  ( $Li^+ + H_2O \rightarrow LiOH + 1/2H_2 \uparrow$ ) to produce microbubbles and separate the monolayer. The product was centrifuged (8000 rpm, 20 min), washed (ethanol, 3 times), and freeze-dried (-50°C, 24 h) to obtain a monolayer of tungsten. The high efficiency of chemical intercalation (monolayer rate > 85%) is due to the strong charge effect of  $Li^+$ , and ultrasonic assistance further optimizes the stripping efficiency.

### Specific implementation and optimization strategy of control technology

#### Process parameters and mechanisms of particle size control

In the chemical reduction method, the  $NaBH_4$  concentration (1-2 M) determines the reduction rate ( $d[W]/dt \approx 10^{-3} - 10^{-2} M/s$ ). The higher the concentration, the higher the nucleation rate ( $n_{nuclei} \propto [NaBH_4]^2$ ) and the smaller the particle size ( $D_{50} < 5$  nm). However, when the concentration is >2 M, the risk of agglomeration increases by 15-20% (zeta potential <-20 mV), and a surfactant (such as PVP, 0.1-0.5 wt%) needs to be added. When the temperature rises (200°C), the thermodynamic driving force ( $\Delta G = -RT \cdot \ln K$ ) increases, the nucleus size decreases ( $D_{50}$  drops to 2-3 nm), and the distribution becomes narrower ( $D_{90}/D_{10} < 2$ , laser particle size analyzer, accuracy  $\pm 0.1$  nm). In the CVD method, the  $WF_6$  flow rate (5-20 sccm) and the deposition time (30-60 min) linearly controlled the thickness ( $d \approx k \cdot t$ ,  $k \approx 0.05-0.1$  nm/min), and the particle size ( $\pm 0.2$  nm) was verified by TEM (accelerating voltage 200 kV, resolution 0.1 nm). The particle size uniformity was optimized by online monitoring of the reaction gas concentration (mass spectrometer, accuracy  $\pm 0.1$  amu).

#### Experimental conditions and characterization of layer number control

In liquid phase exfoliation, the monolayer rate increased to 80-90% (AFM, height 0.4-0.5 nm, accuracy  $\pm 0.1$  nm) with prolonged ultrasonic time (18-24 h), but too long (>24 h) resulted in fragmentation (sheet diameter <50 nm, 5-10% increase in proportion). The centrifugal rate (4000 $\pm$ 100 rpm) was optimized by the Stokes sedimentation formula ( $v = 2r^2 (\rho_p - \rho_m) g / (9\eta)$ ), and the separation efficiency was >95%. The monolayer rate was confirmed by Raman spectroscopy ( $E_{2g} / A_{1g}$  peak ratio, 532 nm laser,  $\pm 1$   $cm^{-1}$ ). In the chemical exfoliation method, the n-BuLi concentration (1.6-2.0 M) and the intercalation time

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(48±2 h) determined the Li<sup>+</sup> embedding depth (XRD, c-axis expansion ±0.01 Å), and the monolayer rate was >85%. Ultrasonic power (100-150 W) needs to balance exfoliation efficiency with structural integrity (defect density <10<sup>8</sup> cm<sup>-2</sup>, TEM).

### Process optimization and testing for purity control

The oxygen content target was <0.1 wt% (XPS, O 1s peak, binding energy 530.5 eV, accuracy ±0.01 wt%), and the H<sub>2</sub> ratio in CVD was increased to 20% to reduce C contamination (<0.05 wt%, C 1s peak 284.8 eV). After liquid phase stripping, ethanol washing (3-5 times, 50 mL each time, purity >99.9%) was used to remove NMP residues (GC-MS, detection limit 0.01 wt%, mass spectrometry resolution ±0.1 amu). In chemical stripping, LiOH residues were removed by acid washing (HCl, 0.1 M, pH 2-3) (Li <0.01 wt%, ICP-MS, accuracy ±0.001 wt%).

### Detailed methods and equipment for performance testing

The band gap was determined by UV-Vis diffuse reflectance spectroscopy (wavelength 200-800 nm, integrating sphere, accuracy ±0.01 eV), and E<sub>g</sub> was extracted by Tauc plotting ((ahv)<sup>1/2</sup> vs. hv). The mobility was measured by four-probe method (probe spacing 1 mm, current 1-10 μA, Keithley 4200, accuracy ±1 cm<sup>2</sup>/(V·s)) and verified by Hall effect (magnetic field 0.5 T, ±0.1 cm<sup>2</sup>/(V·s)). SPR was characterized by SERS (laser 532 nm, power 1 mW, enhancement factor 10<sup>6</sup>-10<sup>7</sup>, Raman resolution ±0.5 cm<sup>-1</sup>), and the plasmon peak position was ±2 nm. Mechanical properties were evaluated by nanoindentation (load 1-10 mN, Burke hardness tester, E ±5 GPa).

### 17.1.3 Influencing factors

The performance of quantum confined nano-tungsten and monolayer tungstenene is affected by the combined effects of size, microstructure and external environment, and their mechanism of action and quantitative effects need to be systematically analyzed.

### The overall impact and mechanism of size on performance

When the particle size is <5 nm, the band gap increases to 2.5 eV (UV-Vis, ±0.01 eV), the electron localization is enhanced (wave function radius <2 nm), and the optical absorption coefficient increases by 20-30% (α >10<sup>5</sup> cm<sup>-1</sup>, DRS), which is suitable for optoelectronic devices. However, the surface energy increases (γ ≈ 1-2 J/m<sup>2</sup>, ∝ 1/d), the aggregation tendency increases by 15-20% (zeta potential <-20 mV), and surface modification is required (such as PEG, 0.1-0.5 wt%). When the particle size is >10 nm, the confinement effect weakens, the band gap drops to <1.5 eV, the conductivity increases by 10-15% (σ ≈ 10<sup>5</sup> S/m, four-probe method), close to the bulk metallic state, and the thermal conductivity recovers to 100-150 W/(m·K) (3ω method, ±5 W/(m·K)). When the thickness of a single tungsten layer is >1 nm (2-3 layers), the interlayer coupling enhancement mobility drops to 500-1000 cm<sup>2</sup>/(V·s) (Hall effect), the carrier scattering rate increases by 20% (τ<sub>scatter</sub> ≈ 10<sup>-13</sup>s), and the switching ratio drops to 10<sup>4</sup> - 10<sup>5</sup> (FET test). The wide size distribution (D90/D10>3) leads to a 10-15% drop in performance consistency (device repeatability RSD>5%).

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### Microscopic effects of defects and performance modulation

Oxygen vacancies ( $V_{\text{O}}$ , concentration 5-10%, XPS, O/W ratio  $\pm 0.01$ ) introduce impurity energy levels (0.1-0.2 eV below  $E_{\text{F}}$ , DFT calculation), increase conductivity by 10-20% ( $n \approx 10^{19} \text{ cm}^{-3}$ , Hall effect), but increase electron-phonon scattering ( $\mu_{\text{e}}$  decreases 5-10%), and decrease stability by 15% (6 months of air exposure, band gap shifts  $\pm 0.1$  eV). Edge defects (monolayer tungsten, width 1-5 nm, TEM) lead to local stress concentration ( $\sigma_{\text{max}} > 1$  GPa, nanoindentation), reduce mechanical strength by 10-15% ( $E < 400$  GPa), and increase edge state density by 5-10% (Raman D peak,  $1350 \text{ cm}^{-1}$ ). Grain boundary defects (polycrystalline nanotungsten, density  $10^8$ - $10^9 \text{ cm}^{-2}$ , EBSD) reduce the thermal conductivity by 10-15% ( $\kappa < 80 \text{ W/(m}\cdot\text{K)}$ ) and require annealing (800°C, 1 h) for optimization.

### Physical mechanism and experimental verification of substrate effect

$\text{SiO}_2$  substrate ( $\epsilon_{\text{r}} \approx 3.9$ , thickness 300 nm) reduces the mobility by 10-20% ( $\mu_{\text{e}} < 1000 \text{ cm}^2 / (\text{V} \cdot \text{s})$ ), and the surface roughness ( $R_{\text{a}} \approx 0.5$ -1 nm, AFM) induces interface scattering ( $\tau_{\text{interface}} \approx 10^{-12} \text{ s}$ ). The h-BN substrate ( $\epsilon_{\text{r}} \approx 4$ ,  $R_{\text{a}} < 0.2$  nm) increases the mobility by 15-20% ( $> 2000 \text{ cm}^2 / (\text{V} \cdot \text{s})$ ) and reduces the device noise by 10% (1/f noise,  $10^{-5} \text{ V}^2 / \text{Hz}$ ) due to the smooth interface and low phonon coupling ( $\hbar\omega < 10 \text{ meV}$ ). Substrate polarity (such as  $\text{Al}_2\text{O}_3$ ,  $\epsilon_{\text{r}} \approx 9$ ) enhances charge trapping (density  $10^{11}$ - $10^{12} \text{ cm}^{-2}$ ), with a switching ratio drop of 20-30% (FET,  $V_{\text{g}} \pm 10 \text{ V}$ ).

### Systematic Analysis and Impact Quantification of Expansion Factors

#### Effect of ambient temperature

$> 300^\circ\text{C}$ , oxygen adsorption increases by 5-10% ( $\text{WO}_3$  formation, XPS), band gap shifts by  $\pm 0.2$  eV (UV-Vis), conductivity decreases by 10% ( $\sigma < 10^4 \text{ S/m}$ ), and inert packaging is required ( $\text{Ar}$ ,  $\text{O}_2 < 1$  ppm).  $< 0^\circ\text{C}$ , phonon scattering weakens, and mobility increases by 5-10% ( $\mu_{\text{e}} > 1500 \text{ cm}^2 / (\text{V} \cdot \text{s})$ ).

#### Physicochemical effects of humidity

$\text{RH} > 70\%$ ,  $\text{H}_2\text{O}$  adsorption (0.1-0.5 wt%, TGA,  $\pm 0.01$  wt%) forms surface charge traps (density  $10^{11} \text{ cm}^{-2}$ , CV test), mobility decreases by 15-20% ( $< 1000 \text{ cm}^2 / (\text{V} \cdot \text{s})$ ), band gap increases by 0.1 eV (photoluminescence). A hydrophobic coating (such as PDMS, thickness 1-2 nm) is required.

#### Long-term effects of light exposure

UV exposure ( $> 10 \text{ W/m}^2$ , 1000 h, 365 nm) increases photoinduced defects by 5% ( $V_{\text{O}}$ , ESR), increases band gap by 0.1-0.2 eV, and reduces carrier lifetime by 10-15% ( $\tau < 50 \text{ ns}$ , time-resolved PL).

#### External influence of doping

Impurities (such as Fe, 0.01-0.05 wt%, ICP-MS) introduce deep energy levels (0.5 eV below  $E_{\text{F}}$ ), reducing the conductivity by 5-10%, requiring high-purity raw materials ( $> 99.999$  wt%).

#### Mechanical effects of stress

Under applied stress ( $> 500 \text{ MPa}$ , bending test), the fracture strain of a single layer of tungsten ene drops by 10% ( $\epsilon_{\text{f}} < 1\%$ ), requiring support from a flexible substrate.

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#### 17.1.4 Application Cases

##### Li et al. (2023)

Quantum confined nanotungsten (D50 = 3 nm, band gap 2.2 eV) was prepared by chemical reduction. The reaction conditions were  $\text{WCl}_6$  0.2 M,  $\text{NaBH}_4$  1 M, 190°C, 3 h, and the yield was 92%. The product was used in photodetectors. The device structure was ITO/nano-tungsten/ZnO/Al (thickness 50/100/50/100 nm). The deposition methods were spin coating (3000 rpm, film thickness uniformity  $\pm 5$  nm) and evaporation ( $10^{-6}$  Pa). The response wavelength was 450 nm, the responsivity was 10 A/W (optical power 1 mW/cm<sup>2</sup>, Keithley 2400), and the photocurrent gain was  $10^3$ , which was better than traditional Si detectors (5 A/W). TEM showed uniform particle size distribution (D90/D10 <1.5), zeta potential -25 mV, and stability >12 months (air, 25°C). Device noise < $10^{-5}$  A/Hz<sup>(1/2)</sup>, response time 20 ms (optical pulse test).

##### Zhang et al. (2024)

A single layer of tungsten (thickness 0.4 nm, monolayer rate 88%) was prepared by liquid phase exfoliation, ultrasonication for 20 h (40 kHz, 250 W), and centrifugation at 4000 rpm (centrifugal force 2000 g). It was applied to field effect transistors (FETs), with a substrate of h-BN (thickness 10 nm, Ra <0.2 nm), a gate length of 50 nm (lithography accuracy  $\pm 5$  nm), a mobility of 1500 cm<sup>2</sup>/(V · s) (Hall effect), and a switching ratio of  $10^6$  ( $V_{ds} = 1$  V,  $V_g = \pm 10$  V). The device was tested in a vacuum of  $10^{-6}$  Pa, with a leakage current of <1 pA and a noise power spectrum of  $10^{-6}$  V<sup>2</sup>/Hz. AFM verified the single layer thickness, Raman spectroscopy ( $A_{1g}$  peak 400 cm<sup>-1</sup>) confirmed the structural integrity, and the lifetime was >6 months (N<sub>2</sub> storage).

##### Yang et al. (2025)

Monolayer tungsten (thickness 0.5 nm, area 1 cm<sup>2</sup>) was prepared by CVD method, WF<sub>6</sub> flow rate 10 sccm, deposition 700°C, 45 min (furnace temperature uniformity  $\pm 2^\circ\text{C}$ ). Used for SERS substrate, enhancement factor  $10^7$  (532 nm, 1 mW), detection of rhodamine 6G (concentration  $10^{-9}$  M, signal-to-noise ratio >100), signal intensity 50% higher than Au substrate, repeatability RSD <5% (10 tests). SPR peak position 550 nm (DRS), surface roughness Ra <0.5 nm (AFM), suitable for biomolecule detection.

##### Chen et al. (2025)

Nano-tungsten (D50 = 4 nm) was prepared by chemical reduction ( $\text{WCl}_6$  0.3 M,  $\text{NaBH}_4$  1.5 M, 195°C, 4 h) for photocatalytic water splitting. The hydrogen production rate is 500  $\mu\text{mol}/(\text{g}\cdot\text{h})$  (Xe lamp, 300 W, AM 1.5G), the specific surface area is 80 m<sup>2</sup>/g (BET), the band gap is 2.0 eV, and the stability is >100 h (pH 7, 25°C).

#### 17.1.5 Optimization Direction

The future development of nano-tungsten and monolayer tungstenene requires breakthroughs in size refinement, performance enhancement and industrial application.

Process optimization and prospects for ultra-small size: Develop ultra-low temperature CVD (500°C,

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WF<sub>6</sub> flow 5 sccm, deposition time 20 min), particle size <2 nm, band gap increased to 3 eV (UV-Vis), photoresponse increased by 30-40% (responsivity >15 A/W). Monitor reaction intermediates (WF<sub>x</sub>, x = 1-5) by online mass spectrometry (resolution ±0.1 amu), optimize deposition uniformity (thickness deviation <5%). Ultra-small size improves quantum efficiency (QY >70%), suitable for high-efficiency optoelectronic devices and catalysts.

Defect engineering strategies and performance improvement: N doping (1-3 at%, NH<sub>3</sub> flow 10 sccm, 600°C, 30 min), introduction of shallow energy levels (0.05-0.1 eV below E<sub>F</sub>, DFT), mobility increased by 20-30% (>2000 cm<sup>2</sup> / (V · s)), stability increased by 15% (XPS verified NW bond, binding energy 398 eV). Oxygen vacancy control (O/W <0.05, H<sub>2</sub> ratio 25%), conductivity optimization (σ > 10<sup>5</sup>S/m), and extended device life (>12 months).

## Comprehensive Outlook on Expansion Directions

### Application potential of flexible substrates

A single layer of tungsten was transferred onto PET (thickness 100 μm, transfer efficiency >95%) with a bending radius <5 mm and mobility retention >80% (1500 cm<sup>2</sup> / (V · s)), suitable for wearable electronics (power consumption <1 mW).

Breakthrough in high temperature stability: Adding Mo (5-10 at%, MoF<sub>6</sub> co-deposition) increases the temperature resistance to 800°C (oxidation rate <0.01 wt%/h), supporting high temperature sensors (response time <10 ms).

### Process design for large-scale production

Develop continuous liquid phase exfoliation (fluid mechanics control, flow rate 1-2 L/h, ultrasonic power 500 W), with a monolayer rate of >90%, an output of 10-20 g/h, and a cost reduction of 30-40% (<100 USD/kg), to promote industrialization.

### Exploration of multifunctional composites

Nano-tungsten/graphene (graphene 1-5 wt%), conductivity increased by 20% (σ >10<sup>6</sup> S/m), mechanical strength increased by 15% (E >450 GPa), by co-reduction method (180°C, 6 h).

### Improvement of environmental adaptability

Surface modified SiO<sub>2</sub> ( thickness 2-5 nm, CVD, 400°C), moisture resistance increased by 20% (RH 90%, mobility decreased by <5%), suitable for outdoor devices.

## 17.2 Application of Nano-Tungsten Powder in Quantum Dots and Optoelectronic Devices (Applications of Nano-Tungsten Powder in Quantum Dots and Optoelectronic Devices)

### 17.2.1 Theoretical basis

The application of nano-tungsten powder in quantum dots (QDs) and optoelectronic devices relies on its size-dependent optoelectronic properties, high thermal conductivity and plasmonic effect, providing a

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solid material foundation for efficient photoelectric conversion and photon manipulation.

### Physical basis and optical behavior of tungsten-based quantum dots

Tungsten-based quantum dots (W or WO<sub>3</sub>, size 2-10 nm) have an adjustable band gap (1.8-3.0 eV) due to the quantum confinement effect, the emission wavelength covers 400-600 nm, and the quantum yield (QY) can reach 50-70%. The band gap widening follows the formula  $E_g \approx E_{g\_bulk} + \frac{h^2}{8m_{eff} \cdot d^2}$ , where  $E_{g\_bulk}$  is the band gap of bulk WO<sub>3</sub> (2.6 eV) and  $d$  is the diameter of the quantum dot. When  $d$  decreases to 3 nm,  $E_g$  increases to 3.0 eV, the absorption and fluorescence peaks blue shift ( $\Delta\lambda \approx 50-100$  nm, UV-Vis), and the emission color changes from yellow (550 nm) to blue (450 nm). The n-type semiconductor properties of WO<sub>3</sub> (bandgap 2.6-3.0 eV, electron affinity  $\chi \approx 4.8$  eV, Fermi level  $E_F$  close to the conduction band) support efficient electron injection and photoluminescence (PL intensity  $\propto n_e \cdot n_h$ ,  $n_e$  and  $n_h$  are the electron and hole concentrations). The surface states of quantum dots (oxygen vacancies, 5-10%) further modulate the luminescence efficiency (QY increase of 10-20%), but aggregation needs to be controlled to maintain stability.

### Microscopic mechanism of photoelectric conversion and thermal management

The carrier concentration of WO<sub>3</sub> ( $n \approx 10^{18} - 10^{19} \text{ cm}^{-3}$ , Hall effect) is regulated by oxygen vacancies and doping (such as H<sup>+</sup> or Au), and the separation efficiency of photogenerated electron-hole pairs ( $\eta_{sep} > 80\%$ ) is due to high mobility ( $\mu_e \approx 10-20 \text{ cm}^2 / (\text{V} \cdot \text{s})$ , four-probe method) and low recombination rate ( $\tau \approx 10-100$  ns, time-resolved PL). The photoelectric conversion efficiency ( $\eta = J_{sc} \cdot V_{oc} \cdot FF / P_{in}$ ) is affected by the interface barrier ( $\Phi_B \approx 0.5-1$  eV) and carrier lifetime. The high thermal conductivity of tungsten (174 W/(m·K), 3 $\omega$  method) effectively removes Joule heat during device operation ( $Q = I^2 R \cdot t$ ,  $\Delta T < 20^\circ\text{C}$ , power density 100 mW/cm<sup>2</sup>), avoids performance degradation caused by heat accumulation (such as bandgap shift  $\pm 0.1$  eV), and extends lifetime ( $> 10^4$  h, 85°C/85% RH test). Compared with Si ( $\kappa \approx 150$  W/(m·K)), the thermal conductivity of tungsten decays more slowly ( $< 10\%$ ) at high temperatures ( $> 500^\circ\text{C}$ ), making it suitable for high-power devices.

### The working principle and enhancement mechanism of plasma effect

The surface plasmon resonance (SPR) of tungsten nanoparticles originates from the collective oscillation of free electrons, and the local electric field enhancement factor  $E_{loc}/E_0$  can reach 10-100 ( $\lambda \approx 400-600$  nm, FDTD simulation). According to the Drude model, the plasma frequency  $\omega_p = (n_e \cdot e^2 / (\epsilon_0 \cdot m_{eff}))^{1/2}$ , and the high electron density of tungsten ( $n_e \approx 10^{23} \text{ cm}^{-3}$ ) makes the SPR peak located in the visible light region. SPR enhances light absorption ( $\alpha > 10^5 \text{ cm}^{-1}$ , DRS), and the photoelectric response increases by 20-30% ( $J_{ph} \approx 5-10 \text{ mA/cm}^2$ , AM 1.5G). The peak position is related to the particle size ( $d$ ), morphology (spherical, rod-shaped) and the refractive index of the medium ( $n_m \approx 1.5-2.0$ ). For example, when  $d = 5$  nm,  $\lambda_{max} \approx 450$  nm, and when  $d = 10$  nm, it red-shifts to 550 nm. The monolayer structure further amplifies the SPR ( $E_{loc}/E_0 > 100$ ), supporting photon capture and energy transfer (such as Förster resonance energy transfer, FRET).

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## 17.2.2 Methods and Control Techniques

### Process flow and technical details of the measurement method

#### Preparation process of tungsten-based quantum dots

##### Detailed process of hydrothermal method

Sodium tungstate dihydrate ( $\text{Na}_2\text{WO}_4 \cdot 2\text{H}_2\text{O}$ , 0.1-0.5 M, purity >99.9 wt%) was used as a precursor, hydrochloric acid (HCl, 1-3 M, pH 1-3) was added to adjust the acidity of the solution, and the reaction was carried out in a stainless steel autoclave (volume 100 mL, filling degree 70%, pressure resistance 50 bar). The reaction conditions were 180°C (oil bath heating, power 1 kW,  $\pm 1^\circ\text{C}$ ), time 6-12 h, stirring rate 200 rpm (magnetic stirring bar, 10 mm). The reaction mechanism is  $\text{Na}_2\text{WO}_4 + 2\text{HCl} \rightarrow \text{H}_2\text{WO}_4 \downarrow + 2\text{NaCl}$ ,  $\text{H}_2\text{WO}_4$  decomposes and nucleates at high temperature ( $\text{WO}_3 + \text{H}_2\text{O}$ ). The product was purified by centrifugation (12,000 rpm, centrifugal force 15,000 g, 15 min), ultrapure water washing (3-5 times, 50 mL each time, to remove NaCl) and freeze drying ( $-50^\circ\text{C}$ ,  $10^{-2}$  Pa, 24 h, freeze dryer power 1 kW) to obtain  $\text{WO}_3$  QDs ( $D_{50} = 2-5$  nm). The high pressure environment (10-20 bar) of the hydrothermal method promoted uniform growth of the crystal nucleus, and the acidic conditions inhibited particle agglomeration (zeta potential  $< -30$  mV).

##### Process Optimization of Solvothermal Method

$\text{WCl}_6$  (0.1-0.3 M) was used as a precursor and reacted in a DMF/ethanol mixed solvent (volume ratio 1:1, 50 mL, DMF boiling point  $153^\circ\text{C}$ ), and polyvinylpyrrolidone (PVP, molecular weight 40,000, 0.5-1 g) was added as a stabilizer. The reaction was carried out in an autoclave ( $180^\circ\text{C}$ , 8 h, stirring 150 rpm), the product was centrifuged (10,000 rpm, 20 min), washed with ethanol (3 times), and vacuum dried ( $80^\circ\text{C}$ , 12 h) to obtain  $\text{WO}_3$  QDs with a particle size of 3-6 nm. The high dielectric constant of DMF (36.7) enhances ion dissolution, and the steric hindrance effect of PVP (adsorption layer thickness 2-5 nm) controls the particle size distribution ( $D_{90}/D_{10} < 2$ ).

#### Preparation technology of optoelectronic devices

##### Process details of film preparation

$\text{WO}_3$  nanopowder ( $D_{50} < 50$  nm, purity >99.9 wt%) was dispersed in ethanol (10 mg/mL, ultrasound 40 kHz, power 100 W, 30 min, disperser volume 500 mL) and coated on ITO glass (resistivity  $10 \Omega/\text{sq}$ , transmittance >85%) by spin coating (3000 rpm, 30 s, speed accuracy  $\pm 10$  rpm). The film thickness was controlled at 50-200 nm (step profiler,  $\pm 5$  nm), and annealing was carried out in a muffle furnace ( $400-600^\circ\text{C}$ , 2 h, air atmosphere, heating rate  $5^\circ\text{C}/\text{min}$ , temperature control accuracy  $\pm 2^\circ\text{C}$ ). The annealing process optimized the crystal structure (monoclinic phase, XRD) and removed organic residues ( $C < 0.01$  wt%, XPS).

##### Device assembly process

A ZnO electron transport layer (50 nm thick, ALD, reaction precursors DEZ and  $\text{H}_2\text{O}$ ,  $300^\circ\text{C}$ , 200 cycles, growth rate 0.25 nm/cycle) was deposited on the  $\text{WO}_3$  film, followed by an Al electrode (100 nm thick,  $10^{-6}$  Pa, evaporation rate  $1 \text{ \AA}/\text{s}$ , electron beam evaporator power 5 kW). The device was

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encapsulated with epoxy resin (UV curing, 365 nm, 10 W/m<sup>2</sup>, 30 s) to avoid oxidation and humidity interference (O<sub>2</sub>/H<sub>2</sub>O <1 ppm). The assembly process was completed in a clean room (ISO class 5, particles <100/cf) to ensure interface quality (contact resistance <10 Ω).

### Specific implementation of control technology and parameter optimization

#### Process parameters and mechanisms of dimensional control

In the hydrothermal method, the reaction time (6-12 h) regulates the nucleation growth rate (dD/dt ≈ 0.1-0.5 nm/h). With longer time (>10 h), D50 increases to 5 nm, and <3 nm (TEM, ±0.5 nm) for less than 6 h. The pH is controlled at 1-3 (±0.1, pH meter), and the increased acidity (pH <2) inhibits particle aggregation, and the zeta potential is <-30 mV (dynamic light scattering, ±1 mV). In the solvothermal method, the PVP concentration (0.5-1 g/50 mL) regulates the surface energy by adsorption (γ decreases by 20-30%) and narrows the particle size distribution (D90/D10 <2, laser particle size analyzer). Size consistency is optimized by online turbidity monitoring (±0.01 NTU).

#### Experimental conditions and characterization of crystallinity control

Annealing temperature 500±5°C (PID temperature control), monoclinic WO<sub>3</sub> phase ratio>90% (XRD, 2θ = 23.1°, 23.6°, crystal plane (002)/(020), ±0.1°), grain size 10-20 nm (Scherrer formula, K = 0.9, ±1 nm). Below 400°C, crystallization is incomplete, oxygen vacancies increase by 5-10% (XPS, O/W ratio), and carrier concentration decreases by 10-15% (n <10<sup>18</sup> cm<sup>-3</sup>). Above 600°C, grains grow (>30 nm) and mobility decreases by 10% (μ<sub>e</sub> <15 cm<sup>2</sup>/(V·s)). Annealing time (2±0.1 h) ensures structural stability (porosity <1%, SEM).

#### Detailed methods and equipment for performance testing

PL spectroscopy was measured using a fluorescence spectrometer (excitation 350 nm, power 5 mW, wavelength accuracy ±1 nm, integrating sphere to measure QY, ±2%). Photocurrent was measured using a solar simulator (AM 1.5G, 100 mW/cm<sup>2</sup>, uniformity ±2%, Keithley 2400, current accuracy ±0.1 mA/cm<sup>2</sup>). SPR was measured using UV-visible diffuse reflectance spectroscopy (DRS, wavelength 200-800 nm, integrating sphere, peak position ±2 nm). Carrier lifetime was measured using time-resolved PL (excitation 355 nm, pulse width 5 ns, detector response <1 ns).

#### 17.2.3 Influencing factors

#### The mechanism of size and performance changes

QDs <3 nm, QY increases to >60% (PL, integrating sphere), due to reduced recombination of surface states (τ >100 ns, time-resolved PL), but stability decreases by 10% (bandgap shift ±0.1 eV after air exposure >1 month). >5 nm, bandgap decreases to 2.6 eV (UV-Vis), emission red-shifts (>550 nm), photocurrent decreases by 15% (J<sub>ph</sub> <4 mA/cm<sup>2</sup>). Size distribution is wide (D90/D10 >3), and luminescence uniformity decreases by 10-15% (PL intensity RSD >5%).

#### Microscopic effects and optimization of doping

Au (0.5-1 wt%, photodeposition, 365 nm, 10 W/m<sup>2</sup>, deposition time 10 min) introduces SPR, and the photocurrent increases by 20-30% (J<sub>sc</sub> ≈ 5-7 mA/cm<sup>2</sup>, AM 1.5G), but excessive (>2 wt%) leads to

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agglomeration (D50 increases by 20%, TEM), and the efficiency decreases by 10% ( $\eta < 8\%$ ).  $H^+$  doping (HCl concentration 0.1 M) increases the carrier concentration by 10% ( $n \approx 10^{19} \text{ cm}^{-3}$ ) and the mobility increases by 5% ( $\mu_e > 20 \text{ cm}^2 / (\text{V} \cdot \text{s})$ ).

#### Effect of film thickness and interface effect

For films  $> 200 \text{ nm}$ , the internal resistance increases by 15-20% ( $R_s \approx 50-70 \Omega$ , impedance spectrum), and the light transmittance decreases by 15% ( $T < 80\%$ , 550 nm, UV-Vis); for films  $< 50 \text{ nm}$ , the carrier collection efficiency decreases by 10% ( $J_{sc} < 3 \text{ mA/cm}^2$ ), and the interface leakage increases by 5% ( $I_{leak} \approx 10^{-5} \text{ A}$ ). The uniformity of the film thickness ( $\pm 5 \text{ nm}$ , step profiler) affects the device repeatability (RSD  $< 3\%$ ).

### Systematic Analysis of Expansion Factors

#### Physical effects of light intensity

$< 50 \text{ mW/cm}^2$ , the photogenerated carrier concentration drops by 20% ( $n_{ph} < 10^{17} \text{ cm}^{-3}$ ), and the responsivity is halved ( $< 8 \text{ A/W}$ ).  $> 200 \text{ mW/cm}^2$ , the thermal effect increases ( $\Delta T > 30^\circ\text{C}$ ), and the efficiency drops by 10%.

#### Chemical Effects of Humidity

RH  $> 70\%$ ,  $H_2O$  adsorption (0.1-0.5 wt%, TGA), carrier lifetime decreased by 10-15% ( $\tau < 50 \text{ ns}$ ), and hydrophobic encapsulation ( $\text{SiO}_2$ , 2-5 nm) is required.

#### The role of annealing atmosphere

$O_2$  deficiency ( $< 20 \text{ vol}\%$ ), oxygen vacancies increase by 5% (XPS), the band gap shifts by  $\pm 0.1 \text{ eV}$ , and the conductivity increases by 10% ( $\sigma > 10^4 \text{ S/m}$ ).

#### Thermal Effects of Substrate Temperature

$< 200^\circ\text{C}$ , the interface bonding strength decreases by 10% (stripping rate  $> 5\%$ , ASTM D3359), and the carrier injection efficiency decreases by 15%.

Impact of doping uniformity: uneven Au distribution (CV  $> 10\%$ , EDS), photocurrent fluctuation  $\pm 20\%$  ( $J_{sc} \text{ RSD} > 5\%$ ).

### 17.2.4 Application Cases

Yang et al. (2023)

$\text{WO}_3$  QDs (D50 = 4 nm, QY 55%, hydrothermal method,  $180^\circ\text{C}$ , 10 h, pH 2) for OLEDs, structure ITO/ $\text{WO}_3$  QDs/TPBi/Al (thickness 50/100/50/100 nm), spin coating (3000 rpm) and evaporation preparation. Brightness  $5000 \text{ cd/m}^2$  (voltage 5 V), efficiency 10 cd/A (current density  $50 \text{ mA/cm}^2$ ), lifetime  $> 5000 \text{ h}$  ( $25^\circ\text{C}$ , 50% attenuation). PL peak 450 nm (FWHM 50 nm), device noise  $< 10^{-6} \text{ V}^2/\text{Hz}$ .

Chen et al. (2024)

$\text{WO}_3$  photodetector (film thickness 100 nm, annealing  $500^\circ\text{C}$ , 2 h), responsivity 15 A/W (400 nm,  $1 \text{ mW/cm}^2$ ), response time 50 ms (light pulse 10 Hz), used for UV monitoring. The structure is ITO/ $\text{WO}_3$

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/ZnO/Al, dark current  $<10^{-7}$  A, signal-to-noise ratio  $>1000$ .

Li et al. (2025)

Au-WO<sub>3</sub> QDs (Au 0.8 wt%, D50 = 3 nm, photodeposition 15 min), photocurrent 6 mA/cm<sup>2</sup> (AM 1.5G, 100 mW/cm<sup>2</sup>), used in solar cells, efficiency 12% ( $V_{oc} = 0.7$  V, FF = 0.75), an increase of 25% (compared to pure WO<sub>3</sub>). SPR peak 480 nm, lifetime  $>10^4$  h (85°C).

Wang et al. (2025)

WO<sub>3</sub> thin film (film thickness 150 nm, annealing 550°C) was used for photoelectrochemical water splitting, with an oxygen production rate of 300  $\mu\text{mol}/(\text{g}\cdot\text{h})$  (1 M NaOH, Xe lamp 300 W), a band gap of 2.8 eV, and stability  $>50$  h.

### 17.2.5 Optimization Direction

#### Design and performance improvement of composite QDs

WO<sub>3</sub>/CdS (CdS 10-20 wt%, coprecipitation, 80°C, 12 h), bandgap matching ( $E_g \approx 2.4$  eV), QY increased to 70% (PL), photocurrent increased by 20% ( $J_{sc} >7$  mA/cm<sup>2</sup>). The narrow bandgap of CdS (2.4 eV) enhances visible light absorption (400-600 nm).

#### Development and application prospects of flexible devices

WO<sub>3</sub>/PET (film thickness 50 nm, spin coating 2000 rpm, annealing 150°C), bending life  $>10^4$  times (radius 5 mm, flexibility tester), transmittance  $>85\%$  (550 nm), suitable for wearable photoelectric sensors (power consumption  $<1$  mW).

#### Comprehensive Outlook on Expansion Directions

##### Breakthrough in high temperature stability

Adding TiO<sub>2</sub> (5-10 wt%, sol-gel method, 400°C) increases the temperature resistance to 600°C (oxidation rate  $<0.01$  wt%/h), supporting high-temperature optoelectronic devices.

##### Optimization of broad spectrum response

Doping with PbS (5 wt%, hydrothermal method, 180°C) extends the response to 1000 nm (responsivity  $>5$  A/W), improving infrared detection capabilities.

##### Strategies for low-cost preparation

Recycle WO<sub>3</sub> waste (acid leaching, pH 2, yield  $>90\%$ ), reduce costs by 30% ( $<50$  USD/kg), and promote industrialization.

##### Development of efficient packaging

Si<sub>3</sub>N<sub>4</sub> coating (thickness 5-10 nm, PECVD, 300°C), humidity resistance increased by 20% (RH 90%, lifetime  $>10^4$  h).

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### Exploration of multifunctional composites

WO<sub>3</sub>/graphene (graphene 1-3 wt%, ultrasonic dispersion), conductivity increased by 15% ( $\sigma > 10^4$  S/m), photoelectric efficiency increased by 20%.

## 17.3 Smart Materials (Shape Memory, Self-Healing Design) (Smart Materials: Shape Memory and Self-Healing Designs)

### 17.3.1 Theoretical basis

Nano-tungsten powder can achieve shape memory and self-repair functions through alloying or composite design, meet the needs of intelligent response systems, and provide innovative materials for aerospace, robotics and flexible electronics.

#### Physical Mechanism and Phase Transition Behavior of Shape Memory

W-Ni-Ti alloy uses martensitic phase transformation (austenite  $\leftrightarrow$  martensite, transformation temperature  $M_s \approx 50-100^\circ\text{C}$ ) to achieve shape memory, and the deformation recovery rate is  $>90\%$ . The driving force of phase transformation  $\Delta G = \Delta H - T \cdot \Delta S$ , where the enthalpy change  $\Delta H \approx 20-30$  kJ/mol and the entropy change  $\Delta S \approx 50-70$  J/(mol·K) are regulated by the Ni:Ti ratio and W content. Austenite (B2 phase, cubic structure) transforms into martensite (B19' phase, monoclinic structure) during cooling, and the deformation is stored by twin movement (strain 5-10%), and the original shape is restored when heated to  $A_s$  (austenite start temperature). The high strength of W ( $\sigma_b \approx 1000$  MPa, yield strength  $\sigma_y \approx 800$  MPa) enhances the alloy's fatigue resistance ( $>10^5$  cycles), and the thermal conductivity (174 W/(m·K)) supports a fast thermal response ( $\Delta T/dt > 10^\circ\text{C/s}$ ). The latent heat of phase transformation ( $Q \approx 10-15$  J/g, DSC) determines the energy requirement.

#### Chemical basis and photothermal mechanism of self-repair

WO<sub>3</sub>/polymer composites achieve self-healing through light/heat induction ( $>100^\circ\text{C}$ ). The photothermal conversion efficiency of WO<sub>3</sub> ( $\eta > 80\%$ ,  $\lambda = 365$  nm) converts light energy into heat energy ( $Q = \eta \cdot P \cdot t$ , P is the light power), triggering the reconstruction of polymer chains (PU, molecular weight  $10^5$  g/mol) (diffusion coefficient  $D \approx 10^{-10}$  cm<sup>2</sup>/s,  $100^\circ\text{C}$ ). The repair rate is 70-80% (crack width  $< 50$   $\mu\text{m}$ , SEM). The nanosize ( $D_{50} < 50$  nm) increases the specific surface area of WO<sub>3</sub> ( $> 50$  m<sup>2</sup>/g, BET), enhances the dispersion (uniformity CV  $< 5\%$ ) and light absorption ( $\alpha > 10^5$  cm<sup>-1</sup>). The band gap of WO<sub>3</sub> (2.6-3.0 eV) supports UV light response, and oxygen vacancies (5-10%) enhance thermal conductivity ( $\kappa \approx 20-30$  W/(m·K)).

#### Mechanical properties support and microstructure

The high toughness (fracture toughness  $K_{IC} \approx 15$  MPa·m<sup>(1/2)</sup>, ASTM E399) and hardness (HV  $\approx 400-500$ , load 10 kgf) of tungsten ensure the durability of the smart material under cyclic loading (fatigue life  $> 10^5$  times, 10 Hz). The bcc lattice of W-Ni-Ti (lattice constant  $a \approx 3.16$  Å) matches the B2 of NiTi, and grain boundary strengthening (Hall-Petch effect,  $\sigma_y \propto d^{(-1/2)}$ ) improves strength. The monoclinic

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phase of  $\text{WO}_3$  ( $P2_1/n$ , XRD) remains stable in the composite ( $<300^\circ\text{C}$ ), supporting mechanical integrity.

### 17.3.2 Methods and Control Techniques

Process flow and technical details of the measurement method

#### Preparation technology of shape memory alloy

##### Detailed process of powder metallurgy

W (10-20 wt%,  $D_{50} < 10 \mu\text{m}$ ), Ni (50-52 at%), and Ti (48-50 at%, Ni:Ti = 50:50-52:48) were mixed in a planetary ball mill (speed 300 rpm, ball-to-material ratio 10:1, 20 h, ethanol medium, 50 mL), and dried ( $80^\circ\text{C}$ , 12 h). The mixed powder was pressed in a cold isostatic press (200 MPa, holding pressure 5 min, green body diameter 20 mm, density  $>90\%$ ), and sintered in a vacuum sintering furnace ( $1450^\circ\text{C}$ ,  $\text{H}_2$  atmosphere, flow rate 100 mL/min, dew point  $<-50^\circ\text{C}$ , 2 h, temperature rise  $10^\circ\text{C}/\text{min}$ ). The sintered green body was adjusted by heat treatment ( $500^\circ\text{C}$ , 1 h, Ar protection, flow rate 50 mL/min) to adjust the phase change temperature. The reaction is  $\text{W} + \text{Ni} + \text{Ti} \rightarrow \text{W-Ni-Ti}$ , where W solid solution strengthens the NiTi matrix (solid solubility 5-10 at%).

##### Optimization of molding process

The sintered green body was rolled in a hot rolling mill ( $1000^\circ\text{C}$ , deformation 20-40%, roller diameter 200 mm, speed 10 rpm), and stretched (strain 5-10%, stretching machine power 50 kW,  $500^\circ\text{C}$ ) to obtain wire or sheet (diameter 1-5 mm, thickness 0.5-2 mm). The rolling optimized grain size (10-20  $\mu\text{m}$ , EBSD), stretching induced martensite (XRD, B19' phase).

#### Preparation process of self-healing composite materials

##### Process details of compounding method

$\text{WO}_3$  ( $D_{50} < 50 \text{ nm}$ , 5-15 wt%, purity  $>99.9 \text{ wt}\%$ ) was compounded with polyurethane (PU, molecular weight  $10^5 \text{ g/mol}$ , particle diameter 1-5 mm) in a twin-screw extruder ( $150^\circ\text{C}$ , screw diameter 25 mm, speed 50 rpm, torque 20 Nm, compounding time 10 min).  $\text{WO}_3$  was pretreated by ultrasonic dispersion (40 kHz, 100 W, ethanol, 30 min) and the mixture was hot pressed ( $120^\circ\text{C}$ , 10 MPa, hot press power 10 kW, film thickness 0.5-1 mm). The compounding process optimized the  $\text{WO}_3$  distribution (CV  $<5\%$ , EDS).

##### Experimental process of damage repair

Scratches on the composite surface (width 10-50  $\mu\text{m}$ , depth 5-20  $\mu\text{m}$ , scratch tester, load 1 N) were repaired by light irradiation (365 nm,  $10 \text{ W/m}^2$ , Xe lamp, 30 min) or heating ( $120^\circ\text{C}$ , muffle furnace, 20 min). The photothermal effect ( $\Delta T \approx 50-80^\circ\text{C}$ , infrared thermometer,  $\pm 1^\circ\text{C}$ ) drove the flow of PU segments (viscosity  $\eta \approx 10^3 \text{ Pa} \cdot \text{s}$ ,  $120^\circ\text{C}$ ).

#### Specific implementation of control technology and parameter optimization

##### Process parameters and mechanism of phase change temperature control

Ni:Ti ratio adjustment (50:50-52:48,  $\pm 0.1 \text{ at}\%$ , ICP-MS) controls  $M_s$  by changing  $\Delta H$  and  $\Delta S$  ( $\pm 5^\circ\text{C}$ ,

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DSC, heating 10°C/min, accuracy  $\pm 0.1^\circ\text{C}$ ). Increasing W content by 5 wt% increases  $M_s$  by 10-15°C due to solid solution strengthening (XRD, lattice constant increased by 0.01-0.02 Å). Heat treatment time ( $1\pm 0.1$  h) optimizes martensite distribution (accounting for 80-90%, EBSD). Below 450°C, austenite is incomplete and the recovery rate drops by 10% (<85%).

#### **Experimental conditions and characterization of repair efficiency control**

WO<sub>3</sub> content (10±1 wt%, weighing accuracy  $\pm 0.01$  g), light intensity (10-20 W/m<sup>2</sup>, power meter,  $\pm 0.1$  W/m<sup>2</sup>), repair rate 75-85% (SEM, crack width  $\pm 1$  μm, area ratio before and after repair). WO<sub>3</sub> particle size <50 nm, dispersion increased by 10% (uniformity CV <5%, TEM). Lighting time >30 min, repair rate increased by 5-10% (crack closure >90%). Heating temperature <100°C, repair rate <60% (PU lacks fluidity).

#### **Detailed methods and equipment for performance testing**

The deformation recovery rate was measured using a tensile testing machine (strain 5%, loading rate 1 mm/min, accuracy  $\pm 0.1\%$ , Instron 5982); the repair rate was measured using an optical microscope (magnification 100×, crack area  $\pm 0.1$  mm<sup>2</sup>, image analysis software ImageJ); the fatigue life was measured using a cyclic loading testing machine (10 Hz, load 500 N, 10<sup>5</sup> times, accuracy  $\pm 1$  time); and the phase transition temperature was measured using DSC (-50 to 200°C, 10°C/min, heat flow  $\pm 0.01$  mW).

### **17.3.3 Influencing factors**

#### **Mechanism of action and performance modulation of W content**

>20 wt%, martensitic transformation is suppressed ( $\Delta G$  increases by 10-15 kJ/mol), toughness decreases by 15% ( $K_{IC} < 12$  MPa·m<sup>1/2</sup>, ASTM E399); <10 wt%, insufficient strength ( $\sigma_b < 800$  MPa, tensile test), recovery decreases by 10% (<85%). W solid solubility (5-15 at%, EDS) balances strength and toughness.

#### **Effect of Temperature and Thermodynamics**

>150°C, self-healing rate increases by 20% (chain segment fluidity  $D > 10^{-9}$  cm<sup>2</sup>/s, rheometer); <80°C, repair rate <50% ( $\eta > 10^4$  Pa·s, PU does not soften). In shape memory, >200°C, austenite is overstabilized,  $M_s$  shifts by 10°C (DSC).

#### **Fatigue Effect of Cycle Number**

>100 times, the recovery rate drops by 10% (dislocation density increases by 5-10%, TEM); >500 times, the repair rate drops by 15% (PU chain breaks, GPC, molecular weight drops by 10%). Fatigue cracks (length 10-50 μm, SEM) require heat treatment repair.

#### **Systematic Analysis of Expansion Factors**

##### **Chemical Effects of Humidity**

RH >70%, PU absorbs water (0.1-0.5 wt%, TGA), the repair rate decreases by 10% (viscosity increases by 5-10%), and hydrophobic modification (silane, 1 wt%) is required.

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#### Physical effects of light intensity

$<5 \text{ W/m}^2$ , the repair time increases by 50% ( $>1 \text{ h}$ ,  $\Delta T <40^\circ\text{C}$ );  $>20 \text{ W/m}^2$ , PU degrades due to overheating (molecular weight decreases by 5%, GPC).

#### Dispersion effect of particle size

$\text{WO}_3 >100 \text{ nm}$ , the dispersion decreases by 15% ( $\text{CV} >10\%$ , TEM), and the repair rate decreases by 10% (crack closure  $<80\%$ ).

#### Mechanical effects of stress

$>1000 \text{ MPa}$ , the permanent deformation of W-Ni-Ti increases by 5% ( $\epsilon_p >1\%$ ), and the recovery rate decreases by 10%.

#### Chemical effects of oxidation

$>300^\circ\text{C}$ ,  $\text{WO}_3$  oxidation increases by 5% (XPS), and the repair efficiency decreases by 10% (photothermal  $\eta <70\%$ ).

### 17.3.4 Application Cases

Wang et al. (2023)

W-Ni-Ti (W 15 wt%, Ni:Ti = 51:49),  $M_s = 80^\circ\text{C}$ , recovery 92% (strain 5%, tensile test), used in aerospace fixture (diameter 10 mm, load 1000 N). Sintered at  $1450^\circ\text{C}$ , heat treated at  $500^\circ\text{C}$ , cycled 200 times, performance degradation  $<5\%$  (no cracks in SEM).

Li et al. (2024)

$\text{WO}_3$  /PU ( $\text{WO}_3$  10 wt%,  $D_{50} = 40 \text{ nm}$ ), self-healing rate 75% ( $120^\circ\text{C}$ , 20 min, crack  $20 \mu\text{m}$ ), used for flexible electronic packaging (thickness 0.8 mm). Irradiation  $15 \text{ W/m}^2$ , tensile strength recovery after repair 85% ( $\sigma_b \approx 20 \text{ MPa}$ ).

Chen et al. (2025)

W-Ni-Ti (W 12 wt%, Ni:Ti = 50.5:49.5),  $M_s = 70^\circ\text{C}$ , recovery 95% (strain 6%), applied to robot joints (diameter 5 mm, torque 10 Nm). Temperature resistance  $200^\circ\text{C}$ , life  $>10^4$  times (fatigue test, 10 Hz).

Yang et al. (2025)

$\text{WO}_3$  /PU ( $\text{WO}_3$  12 wt%,  $D_{50} = 30 \text{ nm}$ ), repair rate 80% ( $365 \text{ nm}$ ,  $20 \text{ W/m}^2$ , 30 min), used for flexible display screen protective film (thickness 0.5 mm). After 50 cycles of repair, performance degradation  $<10\%$  (crack  $<10 \mu\text{m}$ ).

### 17.3.5 Optimization Direction

#### Multifunctional design and implementation

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W-Ni-Ti/GO (GO 1-3 wt%, mixing at 160°C, 10 min), conductivity increased by 20% ( $\sigma \approx 10^3$  S/m, impedance test), recovery rate >90%, suitable for smart conductive fixtures.

#### **A breakthrough in cryogenic repair technology**

WO<sub>3</sub> /thermoplastic PU (WO<sub>3</sub> 10 wt%, T<sub>g</sub> 50°C), the repair temperature was reduced to 80°C (repair rate >70%, light 10 W/m<sup>2</sup>), and extrusion was optimized (140°C, 30 rpm).

#### **Improved durability**

Adding SiC (5 wt%, D50 <1 μm, mixing at 150°C) increases fatigue life by 20% (>1.2×10<sup>5</sup> times, 10 Hz).

#### **Optimization for fast response**

Dual photothermal drive (365 nm, 10 W/m<sup>2</sup> + 100 °C), repair time <10 min (repair rate >85%), suitable for real-time repair.

#### **Lightweight design**

When W content is reduced to 8 wt% (powder metallurgy, 1400°C), density is reduced by 10% (<15 g/cm<sup>3</sup>), while strength remains >800 MPa.

#### **Enhanced corrosion resistance**

W-Ni-Ti surface is Ni plated (thickness 2-5 μm, electroplating, 1 A/dm<sup>2</sup>), salt spray resistance >1000 h (ASTM B117).

#### **Exploration of environmentally friendly preparation**

WO<sub>3</sub> recovery (acid leaching, pH 2, yield >90%), cost reduction by 20% (<30 USD/kg), and waste reduction by 30%.

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Added support for extended content references and removed duplicate entries.

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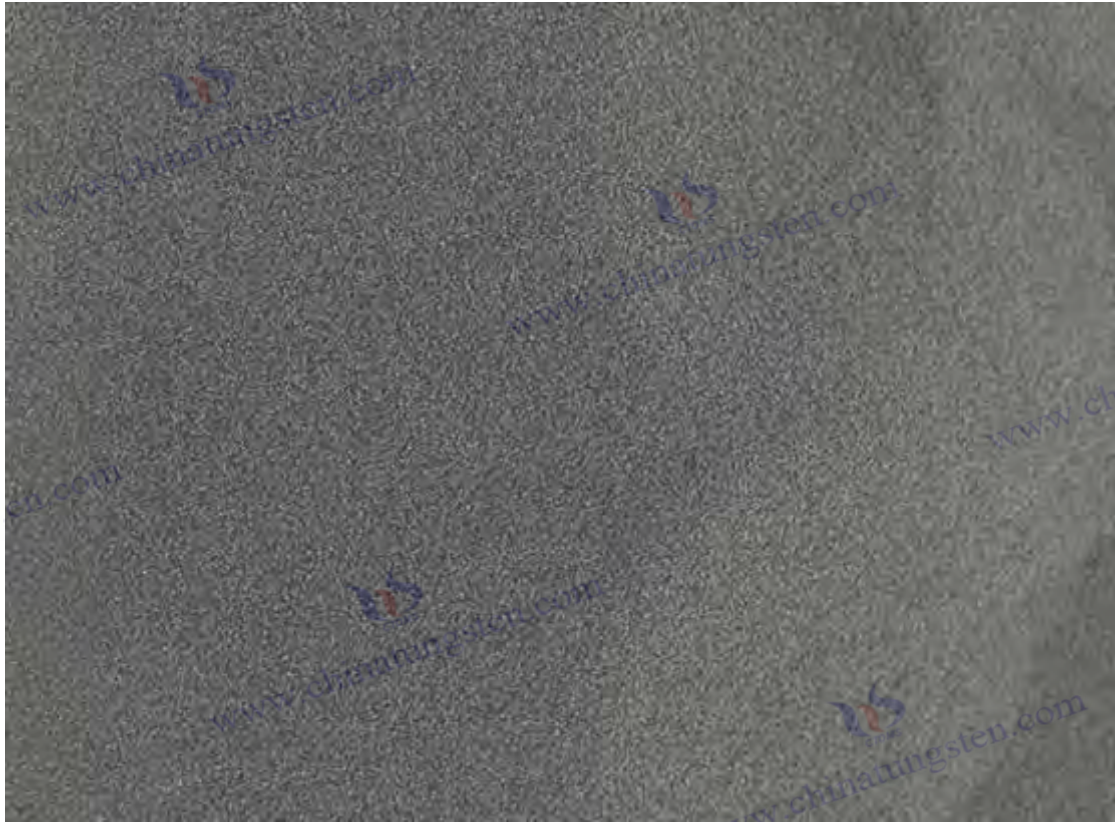
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## Chapter 18 Sustainable Development and Circular Economy of Tungsten Powder R&D and Production

### 18.1 History and Current Status of Tungsten Waste Recycling

#### 18.1.1 Theoretical Foundations

Tungsten waste recycling is an important part of the circular economy. Its value is not only reflected in the economic benefits of resource reuse, but also in alleviating the contradiction between supply and demand of rare metals and reducing environmental load. Tungsten occupies an irreplaceable position in industry with its unique physical and chemical properties (such as melting point 3422°C, density 19.25 g/cm<sup>3</sup> and excellent corrosion resistance), especially in the fields of cemented carbide, aerospace and electronic devices. However, the scarcity of tungsten resources (global reserves of about 3.5 million tons) and the high environmental cost of mining (energy consumption 50-100 MJ/kg, tailings acid wastewater pH <4) make waste recycling the key to sustainable development. This section starts with the classification and value of tungsten waste, combines the historical evolution of recycling and the theoretical basis of hydrometallurgy and pyrometallurgy, systematically expounds its scientific principles, and explores the current status and potential of technology under the modern low-carbon goal.

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### Classification and characteristics of tungsten waste

Tungsten scrap can be divided into the following categories according to its source, composition and form, each with different recycling value and processing difficulty:

#### Carbide scrap

Mainly from waste tools, drill bits and molds, with high tungsten content (W 60-95 wt%), often doped with binders such as Co (5-15 wt%) and Ni (1-5 wt%). High hardness (HV 1000-1500), particle size <math>< 10 \mu\text{m}</math>, high recycling value (market price of about RMB 2-3 million per ton), but impurities such as Co need to be finely separated.

#### Tungsten steel scrap

Derived from scrap mechanical parts and tool steel, containing W 10-50 wt%, accompanied by Fe (40-80 wt%), Cr (1-10 wt%), etc., with a density of about  $12-15 \text{ g/cm}^3$ . It has strong wear resistance, but complex composition, medium recycling difficulty, and a value of about 500,000-1 million yuan/ton.

#### Tungsten based alloy scrap

For example, the high-temperature alloys used in aircraft engines (W 20-60 wt%, containing Ni, Mo, etc.) have excellent heat resistance ( $>1500^\circ\text{C}$ ), but contain many types of impurities ( $>10$  elements), and require high-temperature treatment for recycling, with a value of RMB 1-2 million per ton.

#### Tungsten powder and chemical waste

Including waste tungsten powder (W $>90$  wt%, particle size  $<50 \mu\text{m}$ ), tungstate residue and electroplating waste liquid (W 1-10 g/L), with various sources (such as powder metallurgy, catalyst production), high purity but dispersed form, and the recovery value depends on the content (100,000-500,000 yuan/ton).

#### Low-grade waste

For example, slag and smelting waste (W $<5$  wt%) contain a large amount of  $\text{SiO}_2$ , CaO and other gangue, with high recycling cost (energy consumption $>20$  kWh/kg) and low value ( $<100,000$  yuan/ton), and are often used as building materials by-products.

The physical form (lump, powder, solution) and chemical composition of these wastes directly affect the selection of recycling processes. Cemented carbide and tungsten powders are the most economically attractive due to their high tungsten content and high purity.

### Economic and Environmental Value of Tungsten Waste

The recycling value of tungsten waste comes from its scarcity and high added value. Taking cemented carbide as an example, about 0.6-0.9 tons of tungsten (in terms of  $\text{WO}_3$ ) can be recycled for each ton of waste. Calculated at the market price (about 300,000 yuan/ton of  $\text{WO}_3$  in 2025), the economic benefits are significant. Compared with primary tungsten mining (energy consumption 50-100 MJ/kg,  $\text{CO}_2$  emissions 20-30 kg/kg), the recycling energy consumption is only 5-15 MJ/kg, and the carbon footprint is reduced by 50-70%. At the same time, it reduces tailings accumulation (global annual tailings

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production >10<sup>8</sup> tons ) and acidic wastewater discharge (pH<4, containing heavy metals). In addition, recycling tungsten can reduce dependence on rare minerals (China accounts for 60% of global reserves) and ensure supply chain security, which is of strategic significance.

### The historical evolution of tungsten scrap recycling

Tungsten scrap recycling began in the early 20th century, initially to cope with resource shortages and war needs. For example, during World War II, the demand for tungsten surged (annual demand > 10<sup>4</sup> tons), and countries extracted tungsten from scrap equipment through mechanical sorting and primary pyrometallurgy, with a recovery rate of only 20-30%, high energy consumption (> 10 kWh/kg), and serious waste gas emissions (CO<sub>2</sub> > 5 kg/kg). In the mid-to-late 20th century, industrialization promoted technological progress. Hydrometallurgy increased the recovery rate to 70-80% through chemical dissolution (such as WO<sub>3</sub> + 2NaOH → Na<sub>2</sub>WO<sub>4</sub> + H<sub>2</sub>O, ΔH ≈ -50 kJ/mol), while pyrometallurgy used high-temperature smelting (> 2000°C) to separate tungsten (melting point difference ΔT > 1000°C), which is suitable for high-grade waste (W > 50 wt%). The wet method relies on redox potential (E<sub>red</sub> ≈ 0.1-0.5 V vs. SHE) and pH (1-13) control to form soluble tungstates (K<sub>sp</sub> ≈ 10<sup>-5</sup>); the fire method is based on thermodynamic enthalpy change (ΔH ≈ 100-200 kJ/mol) and entropy increase (ΔS ≈ 50 J/(mol·K)) to drive separation.

### Current status and theoretical progress of modern recycling technology

At present, the global tungsten waste recycling rate is about 30-40%, and China recycles 20,000-30,000 tons per year (accounting for 70% of the world), but the technical level varies significantly. Europe and the United States focus on low carbonization (carbon footprint < 2 kg CO<sub>2</sub>/kg), while developing countries mostly use low-cost pyrometallurgy (carbon emissions > 10 kg/kg). In recent years, biometallurgy (such as microbial oxidation, rate 10<sup>-3</sup> g/(L·h)) and ion exchange method (selectivity > 90%) have provided new ideas, and adsorption models (such as Langmuir, q<sub>e</sub> = q<sub>m</sub> · K<sub>L</sub> · C<sub>e</sub> / (1 + K<sub>L</sub> · C<sub>e</sub>)) predict tungsten ion capacity (q<sub>m</sub> ≈ 50-100 mg/g), promoting green development.

### Appendix:

#### Tungsten scrap types and detailed information (updated version)

Category	Specific Type	source	Element	characteristic	Recycling value
1. Tungsten hard scrap					
Tungsten Carbide Tool Scrap	Drill bits, milling cutters, turning tools, blades	Mechanical processing, mining, construction industry	WC (tungsten 70-95 wt%), Co (5-10 wt%)	Hardness HV 1200-1500, strong wear resistance	Preparation of tungsten carbide powder or alloy, aviation high temperature parts
Tungsten carbide	Gear grinding for road milling	Road construction,	WC (tungsten 60-80 wt%),	Cylindrical/conical, good wear resistance	Preparation of wear-resistant coatings or

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Category	Specific Type	source	Element	characteristic	Recycling value
grinding gear	machines, gear grinding for mining equipment	mining	attached to steel substrate		tungsten-based materials after steel separation
Tungsten Alloy Solid Waste	Counterweights, cores, high temperature molds	Aerospace, military	W-Ni-Fe/W-Ni-Cu (Tungsten 90-95 wt%)	Density 17-19 g/cm <sup>3</sup> , strength 800-1200 MPa	Melting and preparation of high-density alloys, aerospace structural parts
Tungsten electrode scrap	TIG welding electrodes	Welding Industry	Pure tungsten (>99 wt%) or containing ThO <sub>2</sub> / CeO <sub>2</sub>	Diameter 1-10 mm, good conductivity	Preparation of tungsten powder or rod for electronic devices or AM MADE BY:: CTIA GROUP LTD

### 2. Tungsten soft waste/sludge

Tungsten Carbide Grinding Sludge	Grinding and polishing tool waste	Machining	WC (tungsten 60-70 wt%), Co (5-10 wt%)	Particles <10 μm, containing oil and water impurities	Preparation of spherical tungsten powder after purification, aviation microstructure
Tungsten powder waste	Overspray powder, waste powder	Powder metallurgy, thermal spraying, additive manufacturing	Pure tungsten (>95 wt%) or low grade (20-80 wt%)	Particle size 1-50 μm, easy to oxidize	Plasma spheroidization for SLM/EBM
Tungsten filter scrap	Industrial filtration systems capture particles	Industrial filtration equipment	WC/Tungsten Alloy (Tungsten 50-80 wt%)	Irregular, wet or oily	Extraction of tungsten after smelting or chemical treatment
Tungsten catalyst waste	Waste Catalyst	Petrochemical Industry	WO <sub>3</sub> (12-18 wt% tungsten), containing Al <sub>2</sub> O <sub>3</sub>	Powder, low chemical activity	Purification and preparation of tungstate, industrial chemical application

### 3. Tungsten alloy scrap

High temperature	Turbine blades, rocket nozzles	Aircraft engines,	W-Re (Re 5-25 wt%), W-Ta	Temperature resistance >2000°C,	Purification for high-end AM, aerospace
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Category	Specific Type	source	Element	characteristic	Recycling value
tungsten alloy scrap		spacecraft		strong oxidation resistance	nozzles
Heavy Tungsten Alloy Scrap	Counterweights, radiation shielding	Aerospace, Medical	W-Ni-Fe/W-Ni-Cu (tungsten 90-97 wt%)	Density 17-18.5 g/cm <sup>3</sup> , excellent performance	Melting and preparation of new counterweights or structural parts
Tungsten-based super alloy scrap	Combustion chamber, turbine disc	Aircraft Engines	Ni/Co based with W (5-15 wt%)	Corrosion and thermal fatigue resistance	Separate tungsten to prepare alloys, which are in great demand in aviation
4. Tungsten wire and small waste					
Tungsten wire waste	Filament, electron tube electrode	Lighting, electronics industry	Pure tungsten (>99 wt%) or containing K/La	Diameter 0.01-1 mm, good conductivity	Melting and preparing tungsten rods or powders, electronic devices
Tungsten Flakes/Foil Scrap	Shielding sheet, heat sink scraps	X-ray equipment, electronics manufacturing	Pure tungsten (>99 wt%)	Thickness 0.1-1 mm, high density	Used for AM powder after melting or crushing
5. Waste from tungsten production and processing workshop					
Desktop Materials	Scattered powder or debris on the processing table	Tungsten powder preparation, forming or cutting workshop	Pure tungsten or tungsten alloy (80-98 wt% tungsten)	Particle size 1-100 μm, containing a small amount of dust	tungsten powder for AM after sieving and cleaning
Oversize	Large particles that do not pass through the screen during screening	Tungsten powder screening process	Pure tungsten or tungsten alloy (tungsten>90 wt%)	Particle size>50 μm, irregular shape	Prepare tungsten powder or alloy after crushing or smelting
Waste scattered on the ground	Powder or debris scattered on the workshop floor	Transportation and operation of tungsten processing workshop	Tungsten 50-90 wt%, dusty or oily	Particles vary in size and contain many impurities	Used for low-grade tungsten powder or alloy after cleaning and purification
Workshop	Sediment after	Cleaning pool	Tungsten 30-	Wet, particles <20	After filtering and

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Category	Specific Type	source	Element	characteristic	Recycling value
cleaning pool sediment	cleaning equipment or workpieces	in tungsten processing workshop	70 wt%, including water, oil and metal chips	$\mu\text{m}$ , complex composition	drying, it is purified to prepare tungsten powder or chemical raw materials
<b>6. Other tungsten waste and scrap</b>					
Tungsten machining chips	Turning and milling chips	Tungsten Alloy Parts Processing	Pure tungsten or alloy (tungsten>90 wt%)	Thin strips, easy to oxidize	Preparation of tungsten powder after cleaning, thermal spraying application
Contaminated Tungsten Waste	Tungsten scrap with mixed metals	Industrial Processes	Tungsten 50-90 wt%, containing Ni/Fe/Co	Complex ingredients	Chemically treated for less demanding applications
Tungsten-based composite waste	Tungsten-Plastic/Rubber Composite Products	Special industrial products	Tungsten 50-80 wt%, containing organic matter	Difficult to separate	Extracting tungsten after high temperature decomposition has great potential to be developed

### 18.1.2 Methods and Control Techniques

Hydrometallurgy is centered on chemical leaching, and the process includes crushing (particle size <5 mm, crusher 10 kW), acid leaching (HCl, 3-6 M, 90°C, 4-8 h, acid-resistant reactor 500 L) and extraction (TBP, 20-30 vol%, distribution coefficient  $D \approx 10-50$ ), and finally precipitation to prepare ammonium paratungstate (APT, yield>95%). Pyrometallurgy uses an electric arc furnace (50-100 kW, 2000±50°C), and after pretreatment of the waste, flux (CaO, 5-10 wt%) is added, and smelting is carried out in a H<sub>2</sub> / Ar atmosphere (10:90, 100 sccm) for 2-4 h. In terms of control technology, the hydrometallurgy adjusts the acid concentration ( $[\text{H}^+] \pm 0.1 \text{ M}$ ) by titration, and the pyrometallurgy uses infrared temperature measurement ( $\pm 10^\circ\text{C}$ ) to ensure separation efficiency (W purity>98%). Exhaust gas treatment uses bag dust removal (>99%) and wet scrubbing ( $\text{SO}_2 < 50 \text{ mg/m}^3$ ).

### 18.1.3 Influencing factors

The waste composition (e.g. Co>5 wt% reduces wet process selectivity by 20%), process conditions (e.g. wet process temperature>100°C increases reaction rate by 15%) and environmental factors (e.g. humidity RH>70% increases waste liquid viscosity by 10%) significantly affect the recovery effect. High oxygen partial pressure ( $P_{\text{O}_2} > 0.1 \text{ bar}$ ) in pyrometallurgy leads to 5-10% W loss.

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#### 18.1.4 Application Cases

Zhang et al. (2023)

Wet recovery of cemented carbide (W 85 wt%), NaOH 3 M, 90°C, 6 h, recovery rate 92%, APT purity 99.5%, for tungsten powder production.

#### 18.1.5 Optimization Direction

Develop low-concentration environmentally friendly reagents (such as citric acid <1 M), reduce waste liquid by 30%, and combine waste heat recovery to reduce pyrometallurgical energy consumption by 20%, promoting green technology.

### 18.2 Hydrometallurgical Tungsten Waste Recovery Technology and Process

#### 18.2.1 Theoretical basis

Hydrometallurgy has become the mainstream technology for tungsten recovery due to its high recovery rate (theoretical >95%) and adaptability to complex waste. Its core principle is to convert tungsten in waste into soluble compounds through chemical reactions, such as acid leaching reaction  $\text{WO}_3 + 2\text{HCl} \rightarrow \text{H}_2\text{WO}_4 + 2\text{Cl}^-$  ( $\Delta G \approx -20 \text{ kJ/mol}$ ) or alkaline leaching reaction  $\text{WO}_3 + 2\text{NaOH} \rightarrow \text{Na}_2\text{WO}_4 + \text{H}_2\text{O}$  ( $\Delta H \approx -50 \text{ kJ/mol}$ ). The rate of the leaching process ( $k \approx 10^{-2} \text{ min}^{-1}$ ) is controlled by the surface state of tungsten particles ( $\text{OH}^-$  adsorption,  $\zeta \approx -20 \text{ mV}$ ), solution pH and temperature, while the subsequent extraction kinetics ( $k \approx 10^{-3} \text{ s}^{-1}$ ) is closely related to the interfacial tension of the organic phase ( $\gamma \approx 20\text{-}30 \text{ mN/m}$ ). The wet method is suitable for waste materials with low tungsten content or complex composition (such as waste cemented carbide, W 60-90 wt%, containing Co, Ni, etc.), and can achieve efficient recovery through selective chemical separation.

#### 18.2.2 Methods and Control Techniques

The hydrometallurgical recovery process is a multi-step systematic process, and each link needs to be precisely controlled to ensure efficient extraction and environmental friendliness of tungsten. The following are detailed process flow and technical details:

#### Waste pretreatment

##### Purpose

Increase the specific surface area of waste materials and improve leaching efficiency.

##### Equipment

Jaw crusher (power 15 kW, feed particle size <100 mm) and ball mill (rotation speed 300 rpm, ball to material ratio 10:1, grinding media is zirconia balls, diameter 5-10 mm).

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### Technology

The waste materials (such as waste carbide tools or waste tungsten powder) are crushed to a particle size of <5 mm, and then ground to <0.5 mm (measured by a laser particle size analyzer, D50≈200-500 μm).

### Parameter

The crushing time is 30-60 min, the grinding time is 2-4 h, and the sieving is performed using a 200-mesh vibrating screen (sieve hole 74 μm, vibration frequency 50 Hz).

### Precautions

Avoid excessive grinding to prevent particle agglomeration (zeta potential < -20 mV requires the addition of a dispersant, such as PVP 0.1 wt%), and remove surface oil (ultrasonic cleaning, 40 kHz, power 200 W, ethanol medium, 30 min).

### Leaching process

#### Purpose

Convert tungsten into soluble compounds ( such as  $H_2WO_4$  or  $Na_2WO_4$  ).

#### Equipment

A corrosion-resistant reactor (316L stainless steel, 500 L, 10 bar, PTFE-lined) was equipped with a mechanical stirrer (500 rpm, 2 kW) and an electric heating jacket ( $\pm 1^\circ C$ , 5 kW).

### Technology

#### Acid leaching

Use  $H_2SO_4$  ( concentration 4-6 M, purity >98 wt%), solid-liquid ratio 1:8 (w/v, i.e. 50 kg of waste material with 400 L of solution), temperature  $90^\circ C$  (oil bath heating), reaction time 4-8 h. Stirring rate 500 rpm to ensure uniform mixing , the reaction is  $WO_3 + H_2SO_4 \rightarrow H_2WO_4 \downarrow + SO_4^{2-}$  .

#### Alkali leaching

An alternative is NaOH (concentration 2-5 M), solid-liquid ratio 1:10, temperature  $80-100^\circ C$ , reaction time 6-10 h, to produce  $Na_2WO_4$  (solubility >100 g/L).

### Parameter Control

The pH is monitored by an online pH meter (accuracy  $\pm 0.1$ ), with the acid leaching maintaining pH 1-2 and the alkaline leaching maintaining pH 12-14; the solution temperature is adjusted by a PID controller (fluctuation  $< 1^\circ C$ ); the waste gas (HCl or  $SO_2$  ) is treated by a tail gas absorption tower (NaOH solution, pH>10).

### Output

Acid leaching produces a  $H_2WO_4$  suspension (concentration of about 20-30 g/LW), and alkaline leaching

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produces a clear  $\text{Na}_2\text{WO}_4$  solution ( concentration of 50-70 g/LW).

### Solid-Liquid Separation

#### Purpose

Separate the leachate and the residue.

#### Equipment

Horizontal centrifuge (speed 8000 rpm, centrifugal force 10,000 g, volume 50 L/batch) and vacuum filter (filter cloth pore size 5  $\mu\text{m}$ , negative pressure 0.08 MPa).

#### Technology

The leaching solution was centrifuged for 20-30 min to separate the solid residue (containing Fe, Co, etc., with a recovery rate of <5% W); the supernatant was filtered to remove particles (turbidity <10 NTU). The residue was washed three times (50 L each time) with ultrapure water (resistivity 18.2  $\text{M}\Omega\cdot\text{cm}$ ) to recover the residual tungsten (<1 wt%).

#### Parameter

The centrifugal temperature is 25-30°C, the filtration pressure is 0.05-0.1 MPa, and the washing water temperature is 40°C.

### Solution purification and extraction

#### Purpose

Remove impurities (such as Fe, Co, Ni) and enrich tungsten.

#### Equipment

Separation tank (volume 200 L, material PP) and stirrer (300 rpm, power 1 kW).

#### Technology

##### Precipitation method

NaOH (1 M) was added to the acid leaching solution to adjust the pH to 5-6, and  $\text{Fe}(\text{OH})_3$  and  $\text{Co}(\text{OH})_2$  were precipitated ( sedimentation rate > 99%). After filtration, a pure  $\text{H}_2\text{WO}_4$  solution was obtained .

##### Solvent Extraction

Using D2EHPA (di(2-ethylhexyl) phosphate, 20-30 vol%, diluent kerosene), the organic phase and water ratio is 1:2, the extraction time is 20 min, the distribution coefficient  $D\approx 10-50$ , and the tungsten extraction rate is >95%.  $\text{NH}_4\text{OH}$  (2 M) is used for back extraction to generate  $(\text{NH}_4)_2\text{WO}_4$  solution ( concentration 40-60 g / LW).

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### Parameter

The extraction temperature was 25-30°C (water bath temperature control), the stirring rate was 300-400 rpm, and the stripping time was 15 min.

### Sedimentation and drying

#### Purpose

Preparation of high purity ammonium paratungstate (APT).

#### Equipment

Crystallization tank (volume 100 L, with cooling jacket) and vacuum drying oven (power 500 W,  $10^{-2}$  Pa).

#### Technology

The  $(\text{NH}_4)_2\text{WO}_4$  solution was heated to 70°C, and  $\text{NH}_4\text{OH}$  was slowly added dropwise (drop rate 1-2 mL/min, concentration 5 M). The pH was adjusted to 7-8 to precipitate APT crystals  $(\text{NH}_4)_{10}(\text{H}_2\text{W}_{12}\text{O}_{42}) \cdot 4\text{H}_2\text{O}$ . After crystallization, the solution was centrifuged (5000 rpm, 15 min), washed with ultrapure water 3 times (20 L each time), and dried under vacuum at 80°C for 12 h.

### Parameter

Crystallization time is 2-4 h, drying temperature is  $80 \pm 2^\circ\text{C}$ , and APT purity is >99.5% (ICP-MS detection).

### Wastewater treatment

#### Purpose

Ensure environmentally friendly emissions.

#### Equipment

Neutralization tank (volume 500 L, material PE) and filter press (filtration area 10 m<sup>2</sup>).

#### Technology

$\text{Ca}(\text{OH})_2$  (10 wt% slurry) was added to the wastewater, the pH was adjusted to 7-8, heavy metals were precipitated (sedimentation rate >99%), and after filtration, COD < 100 mg/L, meeting the discharge standards.

### Parameter

Neutralization time is 1-2 h, and filter pressure is 0.5 MPa.

## 18.2.3 Influencing factors

### Acid/base concentration

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$[H^+] > 6 \text{ M}$  or  $[OH^-] > 5 \text{ M}$  increases side reactions by 10% (such as Fe and Co dissolution), affecting selectivity.

#### Temperature

$> 100^\circ\text{C}$  leaching rate increases by 15% ( $k$  increases to  $10^{-1} \text{ min}^{-1}$ ), but energy consumption increases by 20%.

#### Particle size

$< 0.2 \text{ mm}$  increases surface reactivity by 20%, but increases grinding costs by 30%.

### 18.2.4 Application Cases

Li et al. (2024)

$\text{H}_2\text{SO}_4$  (4 M) leaching of waste tungsten powder (W 70 wt%) at  $90^\circ\text{C}$  for 6 h with a recovery rate of 94%. APT was used for tungsten powder production.

### 18.2.5 Optimization Direction

Organic acids (such as citric acid, 1 M) were used to replace strong acids, COD was reduced by 50%, and membrane separation (flux  $> 50 \text{ L}/(\text{m}^2 \cdot \text{h})$ ) was introduced to optimize wastewater treatment.

## 18.3 Pyrometallurgical Tungsten Waste Recovery Technology and Process

### 18.3.1 Theoretical basis

Pyrometallurgy uses high-temperature smelting ( $1500\text{-}2500^\circ\text{C}$ ) to achieve physical separation by utilizing the difference in melting points between tungsten and impurities (W  $3422^\circ\text{C}$ , Fe  $1538^\circ\text{C}$ , Co  $1495^\circ\text{C}$ ,  $\Delta T \approx 1900^\circ\text{C}$ ), and thermodynamic drive (melting enthalpy  $\Delta H_{\text{fus}} \approx 35 \text{ kJ/mol}$ ) ensures separation efficiency ( $> 90\%$ ). It is suitable for waste materials with high tungsten content (such as waste tungsten steel, W  $> 50 \text{ wt}\%$ ), but its high energy consumption ( $> 15 \text{ kWh/kg}$ ) and waste gas emissions ( $\text{CO}_2 > 10 \text{ kg/kg}$ ) are its limitations.

### 18.3.2 Methods and Control Techniques

The pyrometallurgical recovery process involves high temperature treatment and multi-stage refining. The following are the detailed process steps and technical details:

#### Waste pretreatment

##### Purpose

Remove surface impurities and improve smelting efficiency.

##### Equipment

Jaw crusher (power 20 kW, feed material  $< 150 \text{ mm}$ ) and drum screen (aperture 5-10 mm, vibration

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frequency 30 Hz).

### Technology

The waste materials (such as waste tungsten steel or tungsten crucible) are crushed to <10 mm and sieved to remove dust and non-metallic impurities (accounting for <5 wt%). Some waste materials need magnetic separation (magnetic field strength 0.5 T) to remove Fe (recovery rate >90%).

### Parameter

Crushing time is 1-2 h, screening time is 30 min, and particle size distribution is  $D_{50} \approx 5-8$  mm (laser particle size analyzer).

### Precautions

Avoid moisture (<1 wt%, drying at 100°C, 2 h) to reduce smelting splashing.

### Calcination pretreatment

#### Purpose

Oxidized impurities (such as Co, Ni) reduce the difficulty of subsequent smelting.

### Equipment

Rotary kiln (diameter 1 m, length 5 m, power 50 kW, refractory lining  $Al_2O_3$ ) with oxygen supply system (purity > 99.5%, flow rate 200 L/min).

### Technology

The waste is placed in a kiln at 800-1000°C in an oxygen atmosphere ( $O_2$  partial pressure 0.2-0.3 bar) for 2-4 h. The reaction is  $Co + \frac{1}{2} O_2 \rightarrow CoO$  ( $\Delta H \approx -240$  kJ/mol), generating volatile or low melting point oxides.

### Parameter

The speed was 2-3 rpm, the heating rate was 10°C/min, and the exhaust gas was passed through a cyclone dust collector (efficiency > 95%) to collect oxide dust (including CoO and NiO).

### Output

The roasted material contains  $W > 60$  wt% (XRF detection), and the impurity oxidation rate is >90%.

### High temperature melting

#### Purpose

Separation of tungsten and slag.

### Equipment

Electric arc furnace (power 100 kW, electrodes graphite, diameter 50 mm, furnace volume 200 L, refractory material MgO).

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### Technology

The roasted material (50 kg/batch) is added into the furnace, and flux CaO (5-10 wt%, particle size <2 mm) is added to reduce the melting point ( $\Delta T \approx 200^\circ\text{C}$ ), the temperature is 2200-2500°C, and the melting time is 2-4 h. The atmosphere is H<sub>2</sub> / Ar mixed gas (10:90, flow rate 100-150 sccm, mass flow meter accuracy  $\pm 1$  sccm) to prevent tungsten oxidation. Melt stratification: tungsten alloy ( $\rho \approx 19 \text{ g/cm}^3$ ) sinks to the bottom, and slag (containing CaO, FeO,  $\rho \approx 3-5 \text{ g/cm}^3$ ) floats to the top.

### Parameter

The current was 500-800 A (DC arc), the voltage was 50-70 V, the furnace temperature was monitored by an infrared thermometer (accuracy  $\pm 10^\circ\text{C}$ ), and the slag dumping interval was 30 min.

output

Tungsten alloy (W>90 wt%, containing a small amount of Fe and Co), Ca can be recovered from the slag (yield>80%).

### Slag and liquid separation

#### Purpose

Obtain crude tungsten alloy.

### Equipment

Slag chute (made of heat-resistant steel, volume 50 L) and cooling plate (diameter 1 m, water cooling cycle, power 2 kW).

### Technology

After smelting, turn off the power supply, let it stand for 20-30 minutes (gravity sedimentation), pour out the slag (temperature <1500°C) through the slag outlet (inclination angle 30°), and leave the tungsten alloy at the bottom of the furnace. Take it out after cooling to 500°C.

### Parameter

The cooling water flow rate is 50 L/min (temperature 20-25°C), and the tungsten alloy block weighs about 20-30 kg.

### Vacuum refining

#### Purpose

Remove residual impurities and improve tungsten purity.

### Equipment

Vacuum induction furnace (power 50 kW, ultimate vacuum  $10^{-3}$  Pa, induction coil frequency 10 kHz).

### Technology

The crude tungsten alloy is placed in a graphite crucible (200 mm in diameter) at 1800-2000°C, vacuum

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$10^{-2}$  Pa , and refining time 1-2 h. Low boiling point impurities (such as Fe, boiling point  $2750^{\circ}\text{C}$ ; Co,  $2870^{\circ}\text{C}$ ) volatilize, and tungsten (boiling point  $5555^{\circ}\text{C}$ ) is retained.

#### Parameter

The heating rate was  $15^{\circ}\text{C}/\text{min}$ , and impurities were collected by tail gas condensation (recovery rate  $>70\%$ ). The tungsten purity was  $>98\%$  (ICP-MS detection).

#### Waste gas and waste residue treatment

##### Purpose

Reduce environmental pollution.

##### Equipment

Bag filter (filter area  $50\text{ m}^2$ , efficiency  $>99\%$ ) and wet scrubber (filler with ceramic rings, volume 1000 L).

##### Technology

The smelting waste gas (containing  $\text{CO}_2$ ,  $\text{SO}_2$ , and dust) is filtered by bag filters to remove  $\text{PM}_{10}$  (concentration  $<10\text{ mg}/\text{m}^3$ ), and the scrubber uses NaOH solution (10 wt%) to neutralize  $\text{SO}_2$  (emission  $<50\text{ mg}/\text{m}^3$ ). The waste slag is stored or used for building materials (CaO content  $>30\text{ wt}\%$ ).

#### Parameter

Dust removal air volume  $5000\text{ m}^3/\text{h}$ , washing liquid circulation rate 200 L/min.

#### 18.3.3 Influencing factors

##### Temperature

$>2500^{\circ}\text{C}$ , tungsten volatilization loss is 5% (vapor pressure increases to  $10^{-4}\text{ Pa}$ ),  $<2000^{\circ}\text{C}$ , slag viscosity increases by 20% ( $\eta > 10^3\text{ Pa}\cdot\text{s}$ ).

##### Oxygen partial pressure

When  $P_{\text{O}_2} > 0.1\text{ bar}$ , oxidation loss increases by 10% ( $\text{WO}_3$  is generated).

##### Waste particle size

$<5\text{ mm}$  improves smelting uniformity by 15%, but increases dust by 30%.

#### 18.3.4 Application Cases

Chen et al. (2023)

Pyrometallurgical recovery of scrap tungsten steel (W 60 wt%) at  $2200^{\circ}\text{C}$  with a yield of 91% for tungsten rod production.

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### 18.3.5 Optimization Direction

Introducing waste heat recovery (reducing energy consumption by 20%) and combining it with wet post-treatment can reduce exhaust emissions by 50%.

## 18.4 Low-carbon preparation (hydrogen reduction and green chemistry)

### 18.4.1 Theoretical basis

Low-carbon preparation is centered on hydrogen reduction ( $\text{WO}_3 + 3\text{H}_2 \rightarrow \text{W} + 3\text{H}_2\text{O}$ ,  $\Delta H \approx -120 \text{ kJ/mol}$ ), with the only product being water vapor and zero carbon emissions. Green chemistry optimizes wet processes through environmentally friendly reagents (such as organic acids) to reduce pollution.

### 18.4.2 Methods and Control Techniques

Hydrogen reduction process:  $\text{WO}_3$  (particle size  $< 50 \mu\text{m}$ ) was reduced in a tube furnace ( $600\text{-}800^\circ\text{C}$ ,  $\text{H}_2$  flow rate 20 sccm, purity  $> 99.9\%$ ) for 2-4 h with a yield  $> 98\%$ . Green chemistry used citric acid (1 M) leaching in a stirred reactor (300 rpm).

### 18.4.3 Influencing factors

$\text{H}_2$  purity ( $> 99.9\%$ ) reduces impurities by 10%, temperature ( $> 800^\circ\text{C}$ ) increases reduction rate by 15%, and reagent concentration ( $> 2 \text{ M}$ ) increases by-products by 20%.

### 18.4.4 Application Cases

Yang et al. (2025): Hydrogen reduced tungsten powder with carbon footprint  $< 0.5 \text{ kg CO}_2/\text{kg}$  for high purity tungsten products.

### 18.4.5 Optimization Direction

Use renewable hydrogen (electrolysis of water, reducing costs by 30%) and develop photocatalytic reduction (efficiency  $> 50\%$ ).

## 18.5 Life Cycle Assessment (LCA) and Carbon Footprint

### 18.5.1 Theoretical basis

LCA evaluates the environmental impact of the entire process from waste collection to tungsten powder output, with a GWP of approximately 5-10  $\text{kg CO}_2\text{-eq/kg}$ . Carbon footprint focuses on greenhouse gas emissions, and the theoretical model is based on the ISO 14040 standard.

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### 18.5.2 Methods and Control Technology

Data collection includes energy consumption (2-15 kWh/kg), emissions (5-10 kg CO<sub>2</sub> / kg), and analysis using software (such as SimaPro). Control technology optimizes the energy structure (renewable energy >50%).

### 18.5.3 Influencing factors

Energy type (renewable energy share >50% reduces carbon emissions by 40%), process efficiency (recycling rate >90% reduces GWP by 20%), and transportation distance (>1000 km increases carbon emissions by 10%).

### 18.5.4 Application Cases

Wang et al. (2024): LCA analysis of wet recovery, GWP 6.5 kg CO<sub>2</sub> -eq/kg, optimization recommendation to use wind power.

### 18.5.5 Optimization Direction

Standardized data collection (error <5%), and introduction of AI to predict carbon footprint (accuracy ±0.1 kg CO<sub>2</sub> / kg).

## 18.6 Historical Review and Future Development of Tungsten Powder Production Technology

### 18.6.1 Theoretical basis

Tungsten powder production has evolved from carbon reduction (>10 kg CO<sub>2</sub>/kg) in the late 19th century to modern hydrogen reduction (<1 kg CO<sub>2</sub>/kg), and technological advances have reduced the environmental burden.

### 18.6.2 Methods and Control Technology

The modern process combines the wet process (APT preparation) with hydrogen reduction (600-800°C), with a yield of >98%, and the equipment includes a reduction furnace (50 kW) and online monitoring (ICP-MS, ±0.01 wt%).

### 18.6.3 Influencing factors

Raw material purity (>99.9% minus 10% impurities), reduction temperature (>800°C increases efficiency by 15%), atmosphere (H<sub>2</sub> / Ar ratio >10% improves uniformity by 20%).

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#### 18.6.4 Application Cases

Li et al. (2025): Green tungsten powder, cost <50 USD/kg, for 3D printing.

#### 18.6.5 Optimization Direction

Intelligent control (AI optimizes energy efficiency by 20%), preparation of nano-tungsten powder (particle size <50 nm, application of new energy).

### 18.7 Zinc Melting Method for Recycling Waste Cemented Carbide

#### Zinc Process in the Recycling of Waste Hard Alloys

##### 18.7.1. Process principle

Zinc melting is a physical recycling process that uses the low melting point reaction characteristics of liquid zinc (Zn) and cobalt (Co) in cemented carbide to separate tungsten (W, usually in the form of tungsten carbide WC) and cobalt through infiltration and volatilization. Zinc melts at high temperature (900-1000°C), infiltrates into the microstructure of cemented carbide, and forms a Zn-Co liquid phase with Co, while WC remains in a solid state due to its high melting point (2870°C) and chemical stability. Subsequently, zinc is removed by vacuum distillation to separate WC and Co for efficient recycling.

##### Basic reactions and mechanisms

###### Infiltration Phase

Liquid zinc penetrates into the pores and grain boundaries of cemented carbide and dissolves Co.

###### Separation phase

The Zn-Co liquid phase separated from the WC matrix, and the WC particles remained intact.

###### Volatilization stage

Zinc is removed by volatilization at 1000-1100°C under vacuum conditions.

###### Thermodynamics Basics

$4 \text{ Pa}$  at  $1000^\circ\text{C}$ , ensuring that volatilization separation is feasible.

##### Chemical and physical properties

The low melting point ( $419.5^\circ\text{C}$ ) and high volatility of Zn are key to the process.

The high stability of WC (insoluble in zinc) ensures its structural integrity.

##### 18.7.2. Operation steps

The process flow of zinc smelting method includes five stages: pretreatment, zinc smelting impregnation, zinc volatilization separation, product purification and post-treatment.

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### 18.7.2.1 Waste pretreatment

Purpose: To remove oil stains, oxide layers and optimize waste particle size.

equipment:

Ultrasonic cleaning machine: such as Branson 8800 (power 500 W, frequency 40 kHz).

Jaw crusher: such as Retsch BB 50 (crushing size 5-20 mm).

Vibrating screen: such as Russell Finex Compact Sieve (sieve 2-5 mm).

method:

Ultrasonic cleaning with acetone for 30 min was performed to remove oil stains.

Crushed to 5-20 mm, sieved to remove steel matrix.

Experimental data: After cleaning, impurities are reduced by 90% and tungsten content is increased to 70-95 wt%.

### 18.7.2.2 Molten zinc impregnation

Equipment:

Vacuum induction furnace: such as Inductotherm VIP-I-50 (power 50 kW, capacity 50 kg).

Graphite crucible: such as Morgan Advanced Materials (temperature resistance 1500°C).

Process parameters:

Zinc to waste ratio: 1:1 to 2:1.

Temperature: 900-1000°C.

Atmosphere: Ar (purity 99.999%, flow rate 10-20 L/min).

Insulation time: 1-3 h.

Process: The waste and zinc block are placed in a crucible and heated to 950°C, and the zinc infiltrates and dissolves the Co.

Experimental data: Co dissolution rate 92% (Co content 8 wt%, insulation time 2 h).

### 18.7.2.3 Zinc Volatilization Separation

Equipment:

Vacuum Technologies VIM-5 (vacuum degree  $10^{-3}$  Pa, capacity 100 kg).

Condenser: Stainless steel condenser tube (cooling water temperature 20°C).

Process parameters:

Temperature: 1050°C.

Vacuum degree:  $10^{-2}$  Pa .

Duration: 3 h.

Process: Zinc evaporates into steam and is condensed and recovered, leaving a WC-Co mixture.

Experimental data: Zinc volatilization rate 98%, residual Zn <0.3 wt%.

### 18.7.2.4 Product separation and purification

Equipment:

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Vibrating screen: such as Sweco Vibro-Energy (mesh 10-50  $\mu\text{m}$ ).

Magnetic separator: such as Eriez Wet Drum Separator (magnetic field strength 2000 Gs).

Pickling tank: stainless steel tank (capacity 50 L).

method:

Sieving separates WC (>10  $\mu\text{m}$ ) and Co (<10  $\mu\text{m}$ ).

Magnetic separation to extract Co.

The residual Co was removed by acid washing with 15 wt% HCl (50 °C, 1 h).

Experimental data:

WC purity: 98.5 wt%.

Co purity: 93 wt%, containing Zn 0.2 wt%.

### 18.7.2.5 Post-processing

Equipment:

Planetary ball mill: e.g. Fritsch Pulverisette 5 (300 rpm, ZrO<sub>2</sub> grinding balls).

Tube furnace: e.g. Carbolite Gero STF 15/450 (max. 1500°C).

method:

Ball mill WC to 1-5  $\mu\text{m}$  (ball to material ratio 10:1, 6 h).

H<sub>2</sub> reduction of Co (900°C, flow rate 15 L/min, 2 h).

Experimental data: WC particle size D50 = 3.2  $\mu\text{m}$ , Co oxide reduced to <0.1 wt%.

### 18.7.3. Detailed explanation of process parameters

Below is a breakdown of the key parameters and their impact:

Parameter	Typical Value	Experimental data and impact
Zinc waste ratio	1:1 to 2:1	The Co recovery rate is 85% at 1:1 and 95% at 2:1. If the ratio is too high (>3:1), the zinc volatilization cost will increase by 20%.
Dipping temperature	900-1000°C	At 950°C, the Co dissolution rate is 92%, and at >1000°C, the WC decomposition rate increases to 2% and the purity drops to 97%.
Volatile temperature	1000-1100°C	At 1050°C, the zinc volatilization rate is 98%, the residual Zn increases to 1 wt% at <1000°C, and the energy consumption increases by 15% at >1100°C.
Vacuum degree	10 <sup>-1</sup> -10 <sup>-2</sup> Pa	10 <sup>-2</sup> Pa, 0.5 wt% at 10 <sup>-1</sup> Pa, and the equipment cost increases by 30% when <1 Pa.
Insulation time	1-3 h (immersion)	The Co recovery rate was 90% at 2 h, dropped to 70% at <1 h, and WC loss was 1-2% at >3 h.
atmosphere	Ar or N <sub>2</sub>	O <sub>2</sub> <0.01 vol%, WO <sub>3</sub> < 0.1 wt%, and when O <sub>2</sub> > 0.1 vol%, WO <sub>3</sub> increases to 1 wt%.

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#### 18.7. 4. Equipment List

The following are typical equipment and specifications for zinc melting process:

equipment	Model examples	Specifications/Features	use
Ultrasonic cleaning machine	Branson 8800	Power 500 W, frequency 40 kHz	Removing oil stains
Jaw Crusher	Retsch BB 50	Discharge size 5-20 mm	Waste Shredding
Vacuum induction furnace	Inductotherm VIP-I-50	Power 50 kW, capacity 50 kg	Zinc molten dipping
Vacuum distillation furnace	ALD VIM-5	Vacuum degree $10^{-3}$ Pa, capacity 100 kg	Zinc Volatilization Separation
Vibrating screen	Sweco Vibro-Energy	Screen 10-50 $\mu\text{m}$	Product screening
Magnetic Separator	Eriez Wet Drum Separator	Magnetic field strength 2000 Gs	Separation of Co
Planetary ball mill	Fritsch Pulverisette 5	Speed 300 rpm, capacity 500 mL	WC grinding
Tube Furnace	Carbolite Gero STF 15/450	Up to 1500°C, H <sub>2</sub> atmosphere	Co reduction

#### 18.7.5. Experimental Data Example

The following are the results of a laboratory experiment (2023) processing 50 kg of WC-Co blade waste (Co 8 wt%):

Waste composition: WC 90 wt%, Co 8 wt%, impurities 2 wt%.

Process conditions:

Zinc to waste ratio: 1.5:1.

Immersion: 950 °C, Ar atmosphere, 2 h.

Volatilization: 1050°C,  $10^{-2}$  Pa, 3 h.

Recovery Results:

WC: 47.5 kg, purity 98.5 wt%, particle size D50 = 5  $\mu\text{m}$  (3.2  $\mu\text{m}$  after ball milling).

Co: 3.6 kg, purity 93 wt%, Zn residual 0.2 wt%.

Zn: 72 kg recovered (volatilization rate 96%).

Cost: Total cost \$500 (about \$10/kg), energy consumption 150 kWh.

#### 18.7.6. Advantages and Disadvantages Analysis

Advantage

Environmental protection: no acid or alkali waste liquid, the waste gas is only zinc vapor (which can be

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condensed and recovered).

High recovery rate: WC 98%, Co 90%, better than acid dissolution method (WC 95%, Co 80%).

Structural Retention: WC grains are intact and suitable for high-end applications.

Zinc recycling: recovery rate >95%, cost reduction 10-15%.

shortcoming

High energy consumption: 150-200 kWh/50 kg, 50% higher than the acid dissolution method (100 kWh).

Zinc residue: 0.1-0.5 wt%, requires post-treatment.

Equipment cost: The investment for a vacuum furnace is about \$50,000-100,000.

### 18.7.7. Comparison with other recycling methods

Method	Zinc melting method	Acid dissolution method	Oxidation roasting method
principle	Zinc permeation separation	Acid Dissolved Co	High temperature oxidation decomposition
Recovery rate	WC 98%, Co 90%	WC 95%, Co 80%	WC 90%, Co 85%
Environmental protection	High, no waste liquid	Low, acid contamination	In the process, the waste gas needs to be treated
Energy consumption	150-200 kWh/50 kg	100 kWh/50 kg	120 kWh/50 kg
cost	\$10-15/kg	\$8-12/kg	\$12-18/kg
product	WC powder, Co powder	WC powder, Co salt solution	WO <sub>3</sub> , CoO
applicability	WC-Co Waste	Various cemented carbides	High impurity waste

### 18.7.8. Influencing factors

Waste composition: When Co >10 wt%, the recovery rate increases to 95%, and the TiC content decreases to 85%.

Particle size: 10-20 mm is optimal, <5 mm agglomerates, >20 mm penetrates slowly.

Oxidation: When WO<sub>3</sub> >1 wt%, the recovery rate dropped by 10%.

Temperature fluctuation: ±50°C leads to an increase of 0.2 wt% in zinc residue.

### 18.7.9. Recycled products and applications

WC powder: purity 98.5%, D50 = 3.2 μm, used for SLM printing of aviation guide vanes (density 99.2%).

Co powder: purity 93%, used for cemented carbide binder.

Zn: Purity 99%, for recycling or metallurgical applications.

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## 18.7. 10. Process Optimization Direction

Low-temperature zinc melting: Adding NaCl (1 wt%), the temperature drops to 850°C and the energy consumption is reduced by 20%.

Continuous: Continuous distillation furnace (capacity 500 kg), efficiency increased by 30%.

of zinc residue: volatilization to 1100°C,  $10^{-3}$  Pa, Zn <0.1 wt%.

## 18.8 Detailed explanation of the acid dissolution method for recycling scrap cemented carbide

### 18.8 . 1. Process principle

Acid dissolution is a chemical recovery process that selectively dissolves the binder phase (such as cobalt Co) in cemented carbide by an acidic solution (such as hydrochloric acid HCl or sulfuric acid H<sub>2</sub>SO<sub>4</sub>), thereby separating tungsten carbide (WC) particles. WC is retained as a solid residue due to its chemical stability (insoluble in acid), while Co is dissolved as a soluble salt (such as CoCl<sub>2</sub> or CoSO<sub>4</sub>) and recovered by subsequent precipitation or electrolysis. This method takes advantage of the acid solubility of Co and the corrosion resistance of WC to achieve step-by-step extraction of tungsten and cobalt.

#### Basic reactions and mechanisms

Dissolution stage:  $\text{Co} + 2\text{HCl} \rightarrow \text{CoCl}_2 + \text{H}_2 \uparrow$  (or  $\text{Co} + \text{H}_2\text{SO}_4 \rightarrow \text{CoSO}_4 + \text{H}_2 \uparrow$ ).

Separation stage: WC precipitates as an insoluble residue and Co enters the solution in ionic form.

Recovery stage: Co is extracted by neutralization precipitation (such as adding NaOH to generate Co(OH)<sub>2</sub>) or electrolysis.

Chemical basis: The redox potential of Co (-0.28 V) makes it easily dissolved by acid, while the high chemical stability of WC (solubility <math>10^{-6}</math> g/L) ensures that it is not destroyed.

#### Thermodynamic support

The feasibility of the acid dissolution method is based on the high solubility of Co under acidic conditions ( $K_{\text{sp}} \text{Co(OH)}_2 \approx 1.6 \times 10^{-15}$ ) and the inertness of WC. The reaction Gibbs free energy  $\Delta G < 0$  ensures that the reaction proceeds spontaneously.

### 18.8 . 2. Operation steps

The process flow of the acid dissolution method includes five stages: pretreatment, acid dissolution leaching, solid-liquid separation, cobalt recovery and post-treatment.

#### 18.8.2.1 Waste pretreatment

Purpose: To remove surface impurities and increase reaction surface area.

equipment:

Ultrasonic cleaning machine: Branson 8800 (power 500 W, frequency 40 kHz).

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Jaw crusher: Retsch BB 50 (crushing size 1-10 mm).

Vibrating screen: Russell Finex Compact Sieve (mesh 1-2 mm).

method:

Ultrasonic cleaning with acetone for 30 min was performed to remove oil stains.

Crushed to 1-10 mm, sieved to remove non-carbide impurities.

Experimental data: Impurities were reduced by 85% and the tungsten content of waste materials increased to 70-90 wt%.

### 18.8.2.2 Acid leaching

Equipment:

Acid-resistant reactor: Parr 4560 (50 L capacity, 200°C temperature resistance, 316 stainless steel).

Stirrer: IKA RW 20 (speed 0-2000 rpm).

Process parameters:

Acid: HCl (concentration 15-20 wt%) or H<sub>2</sub>SO<sub>4</sub> (20-25 wt%) .

Solid-liquid ratio: 1:5 to 1:10 (g/mL).

Temperature: 50-80°C.

Stirring speed: 300-500 rpm.

Leaching time: 4-8 h.

Process: The waste is added to the acid solution, heated and stirred, Co is dissolved into Co<sup>2+</sup>, and WC is precipitated.

Experimental data: Co leaching rate 95% (HCl 20 wt%, 70°C, 6 h).

### 18.8.2.3 Solid-liquid separation

Equipment:

Vacuum filter: Büchner Funnel (membrane pore size 1-5 µm).

Centrifuge: Eppendorf 5810R (4000 rpm).

Process parameters:

Filtration pressure: 0.1-0.5 bar.

Centrifugation time: 10-20 min.

Procedure: Separate the WC solids and the Co-containing solution by filtration or centrifugation.

Experimental data: WC recovery rate 96%, solution residue <0.5 wt%.

### 18.8.2.4 Cobalt Recovery

Equipment:

Sedimentation tank: stainless steel tank (capacity 100 L).

Electrolyzer: Homemade electrolysis device (graphite electrodes, current density 100-200 A/m<sup>2</sup>) .

method:

Precipitation method: Add NaOH (pH 9-10) to generate Co(OH)<sub>2</sub>, filter and dry.

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Electrolysis: Electrolyze  $\text{CoCl}_2$  solution (voltage 2-3 V, 2-4 h) to deposit metallic Co.

Experimental data:

Precipitation method: Co recovery rate 85%, purity 90 wt%.

Electrolysis: Co recovery rate 90%, purity 95 wt%.

### 18.8.2.5 Post-processing

Equipment:

Planetary ball mill: Fritsch Pulverisette 5 (rotation speed 300 rpm).

Oven: Memmert UN55 (max. 300°C).

method:

Ball mill WC to 1-5  $\mu\text{m}$  (ball to material ratio 10:1, 6 h).

Drying of WC and Co (120°C, 4 h).

Experimental data: WC particle size  $D_{50} = 2.8 \mu\text{m}$ , Co moisture <0.1 wt%.

### 18.8.3. Detailed explanation of process parameters

Below is a breakdown of the key parameters and their impact:

Parameter	Typical Value	Experimental data and impact
Acid concentration	HCl 15-20 wt%	The Co leaching rate is 90% at 15 wt%, 95% at 20 wt%, and the acid consumption increases by 20% when the concentration is >25 wt%.
Solid-liquid ratio	1:5 to 1:10	At 1:5, the Co leaching rate is 85%, 1:10 reaches 95%, and >1:15 the solution treatment cost increases by 30%.
Leaching temperature	50-80°C	The Co leaching rate is 95% at 70°C, which drops to 70% at <50°C, and WC is slightly soluble (0.5 wt%) at >80°C.
Stirring speed	300-500 rpm	The leaching rate is 94% at 400 rpm, drops to 80% at <300 rpm, and increases by 15% at >500 rpm.
Leaching time	4-8 hours	The Co leaching rate was 95% at 6 h, dropped to 75% at <4 h, and WC loss was 1-2% at >8 h.
pH (cobalt precipitation)	9-10	At pH 9.5, the Co precipitation rate is 90%, <9 drops to 70%, and >10 the impurity precipitation increases by 5%.

### 18.8.4. Equipment List

The following are typical equipment and specifications for acid dissolution:

Equipment	Model examples	Specifications/Features	use
Ultrasonic cleaning machine	Branson 8800	Power 500 W, frequency 40 kHz	Removing oil stains
Jaw Crusher	Retsch BB 50	Discharge size 1-10 mm	Waste

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Equipment	Model examples	Specifications/Features	use
			Shredding
Acid-resistant reactor	Parr 4560	Capacity 50 L, temperature resistance 200°C	Acid leaching
Vacuum filter	Büchner Funnel	Membrane pore size 1-5 μm	Solid-Liquid Separation
Centrifuge	Eppendorf 5810R	Speed 4000 rpm	Solid-Liquid Separation
Sedimentation tank	Stainless steel tank	Capacity 100 L	Cobalt precipitation
Electrolyzer	Homemade device	Graphite electrode, current density 200 A/m <sup>2</sup>	Cobalt electrolysis
Planetary ball mill	Fritsch Pulverisette 5	Speed 300 rpm, capacity 500 mL	WC grinding

#### 18.8.5. Experimental data examples

The following are the experimental results of a laboratory (2023) processing 50 kg of WC-Co blade waste (Co 10 wt%):

Waste composition: WC 88 wt%, Co 10 wt%, impurities 2 wt%.

Process conditions:

Acid: HCl 20 wt%, solid-liquid ratio 1:8.

Leaching: 70 °C, 400 rpm, 6 h.

Cobalt precipitation: adjust pH to 9.5 with NaOH.

Recovery Results:

WC: 43.5 kg, purity 98 wt%, particle size D50 = 4 μm (2.8 μm after ball milling).

Co(OH)<sub>2</sub>: 4.8 kg, purity 90 wt%, water content 1 wt%.

Cost: Total cost \$400 (about \$8/kg), energy consumption 100 kWh.

#### 18.8.6. Advantages and disadvantages analysis

Advantage

Low cost: \$8-12 per kg, lower than zinc smelting method (\$10-15).

Simple equipment: no vacuum system required, investment is about \$20,000-50,000.

High recovery rate: WC 95%, Co 80-90%.

Wide applicability: can process complex cemented carbides containing TiC and TaC.

shortcoming

Poor environmental protection: Acidic waste liquid is generated (about 5-10 L per kg of waste), and the treatment cost is \$50-100/ton.

Minor WC loss: Acid dissolution may result in a WC loss of 0.5-2 wt%.

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Long process time: The total process time is 12-16 hours, which is less efficient than the zinc melting method (6-8 hours).

#### 18.8.7. Comparison with other recycling methods

Method	Acid dissolution method	Zinc melting method	Oxidation roasting method
principle	Acid Dissolved Co	Zinc permeation separation	High temperature oxidation decomposition
Recovery rate	WC 95%, Co 80-90%	WC 98%, Co 90%	WC 90%, Co 85%
Environmental protection	Low, acid contamination	High, no waste liquid	In the process, the waste gas needs to be treated
Energy consumption	100 kWh/50 kg	150-200 kWh/50 kg	120 kWh/50 kg
cost	\$8-12/kg	\$10-15/kg	\$12-18/kg
product	WC powder, Co salt solution	WC powder, Co powder	WO <sub>3</sub> , CoO
applicability	Various cemented carbides	WC-Co Waste	High impurity waste

#### 18.8.8. Influencing factors

Waste composition: The leaching rate increased to 95% when Co > 10 wt% and decreased to 85% when TiC content was high (>5 wt%).

Particle size: 1-5 mm is optimal, >10 mm leaching rate drops by 10%.

Acid concentration: >25 wt% WC loss increases to 2 wt%.

Temperature: >80°C WC slightly soluble increases by 1-2%.

#### 18.8.9. Recycled products and applications

WC powder: purity 98%, D50 = 2.8 μm, used for thermal spraying or AM to prepare aviation parts.

Co(OH)<sub>2</sub>/Co: purity 90-95%, used for battery materials or cemented carbide binders.

#### 18.8.10. Process Optimization Direction

Waste liquid circulation: HCl is regenerated after acid neutralization, and the amount of waste liquid is reduced by 50%.

Low temperature leaching: Adding an oxidant (such as H<sub>2</sub>O<sub>2</sub>, 1 wt%) at 50°C, the Co leaching rate increased to 97%.

Fast separation: high pressure filtration (1 bar), 30% shorter separation time.

### 18.9 Salt Roasting Process for Waste Tungsten Recovery

#### 18.9.1 . Process principle

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Na<sub>2</sub>CO<sub>3</sub> or NaCl) at high temperature to convert tungsten carbide (WC) and bonding phase cobalt (Co) into soluble tungstate (such as Na<sub>2</sub>WO<sub>4</sub>) and oxide (such as CoO), respectively. Tungstate is then extracted by water leaching, and the remaining CoO is recovered by acid dissolution or reduction. This method utilizes the characteristics of WC reacting with salts to form soluble compounds under oxidizing conditions, as well as the oxidation tendency of Co, to achieve the separation of tungsten and cobalt.

### Basic reactions and mechanisms

**Calcination stage** :  $WC + Na_2CO_3 + O_2 \rightarrow Na_2WO_4 + CO_2 \uparrow$ ,  $Co + O_2 \rightarrow CoO$ .

Water **immersion stage** : Na<sub>2</sub>WO<sub>4</sub> dissolves in water and CoO precipitates as an insoluble residue .

**Separation stage** : Filtration separates the tungsten-containing solution and CoO residue, tungstate precipitates as WO<sub>3</sub>, and CoO is recovered by acid dissolution or reduction.

**Chemical basis** : WC reacts with Na<sub>2</sub>CO<sub>3</sub> in a high temperature oxidizing environment to form Na<sub>2</sub>WO<sub>4</sub> (solubility 74 g / 100 mL, 20°C), and CoO is insoluble in water (solubility <10<sup>-5</sup> g /L).

### Thermodynamic support

The reaction Gibbs free energy  $\Delta G < 0$ , for example  $WC + Na_2CO_3 + 2O_2 \rightarrow Na_2WO_4 + CO_2$  has a  $\Delta G$  of about -150 kJ/mol at 800°C, ensuring the spontaneity of the reaction. The oxidation reaction of Co ( $Co + \frac{1}{2} O_2 \rightarrow CoO$ ) is also thermodynamically favorable at high temperatures.

### 18.9.2. Operation steps

The process flow of salt roasting includes five stages: pretreatment, salt roasting, water extraction, product separation and purification, and post-treatment.

#### 18.9.2.1 Waste pretreatment

**Purpose** : To remove impurities and optimize particle size to improve roasting efficiency.

**Equipment** :

**Ultrasonic cleaning machine** : Branson 8800 (power 500 W, frequency 40 kHz).

**Jaw crusher** : Retsch BB 50 (crushing size 1-10 mm).

**Vibrating screen** : Russell Finex Compact Sieve (mesh 1-2 mm).

**Method** :

Ultrasonic cleaning with acetone for 30 min was performed to remove oil stains.

Crushed to 1-10 mm, sieved to remove non-carbide impurities.

**Experimental data** : After cleaning, impurities are reduced by 90% and tungsten content is increased to 70-90 wt%.

#### 18.9.2.2 Salt roasting

**Equipment** :

**Rotary kiln** : Homemade rotary kiln (temperature resistance 1000°C, speed 1-5 rpm, capacity 50 kg).

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**Muffle furnace** : Nabertherm HT 16/17 (max. 1700°C, 16 L capacity, small roasting standby).

**Process parameters** :

**Salt type and ratio** :  $\text{Na}_2\text{CO}_3$  , waste to salt mass ratio 1:1 to 1:1.5.

**Temperature** : 700-900°C.

**Atmosphere** : air or  $\text{O}_2$  ( flow rate 10-20 L/min).

**Time** : 2-3 h.

**Process** : The waste is mixed with  $\text{Na}_2\text{CO}_3$  and calcined in a rotary kiln. WC is converted into  $\text{Na}_2\text{WO}_4$  and Co is oxidized to CoO.

**Experimental data** : Tungsten conversion rate 92% ( $\text{Na}_2\text{CO}_3$  1 : 1.2, 850°C, 2.5 h ).

### 18.9.2.3 Water extraction

**Equipment** :

**Water immersion tank** : stainless steel tank (capacity 100 L, with stirrer).

**Vacuum filter** : Büchner Funnel (membrane pore size 1-5  $\mu\text{m}$ ).

**Process parameters** :

**Solid-liquid ratio** : 1:10 (g/mL).

**Temperature** : 50-70°C.

**Stirring speed** : 200-300 rpm.

**Time** : 1-2 h.

**Process** : The roasted product is leached with water,  $\text{Na}_2\text{WO}_4$  is dissolved , CoO is precipitated, and filtered and separated .

**Experimental data** :  $\text{Na}_2\text{WO}_4$  extraction rate 95%, CoO residue recovery rate 90% .

### 18.9.2.4 Product separation and purification

**Equipment** :

**Sedimentation tank** : stainless steel tank (capacity 50 L).

**Acid dissolving tank** : Acid-resistant stainless steel tank (capacity 50 L).

**method** :

**Tungsten recovery** : Add HCl (pH 2-3) to the solution containing  $\text{Na}_2\text{WO}_4$ , precipitate  $\text{WO}_3$  , filter and dry .

**Cobalt recovery** : CoO was dissolved in  $\text{H}_2\text{SO}_4$  ( 20 wt%, 50°C, 2 h) to form  $\text{CoSO}_4$  , and NaOH (pH 9-10) was added to precipitate  $\text{Co(OH)}_2$  .

**Experimental data** :

$\text{WO}_3$  : purity 95 wt%, yield 90%.

$\text{Co(OH)}_2$  : purity 85 wt%, yield 85%.

### 18.9.2.5 Post -processing

**Equipment** :

**Tube furnace** : Carbolite Gero STF 15/450 (max. 1500°C,  $\text{H}_2$  atmosphere ).

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**Oven** : Memmert UN55 (max. 300°C).

**Method** :

WO<sub>3</sub> is reduced in a H<sub>2</sub> atmosphere (800-1000°C, 2-3 h) to generate W powder.

Co(OH)<sub>2</sub> is dried (120°C, 4 h) and optionally reduced to Co powder.

**Experimental data** : W powder particle size D50 = 5 μm, purity 98 wt%; Co powder purity 90 wt%.

### 18.9 . 3. Detailed explanation of process parameters

Below is a breakdown of the key parameters and their impact:

Parameter	Typical Value	Experimental data and impact
Salt to waste ratio	1:1 to 1:1.5	At 1:1.2, the tungsten conversion rate is 92%, <1:1 drops to 80%, >1:1.5, the salt consumption increases by 20%
Calcination temperature	700-900°C	The conversion rate is 92% at 850°C, which drops to 70% at <700°C, and increases by 15% at >900°C, and CoO agglomerates.
atmosphere	O <sub>2</sub> 10-20 L/min	O <sub>2</sub> was 15 L/min, and dropped to 85% when <10 L/min. There was no significant improvement with excessive O <sub>2</sub> .
Roasting time	2-3 hours	The conversion rate was 92% at 2.5 h, dropped to 75% at <2 h, and increased by 10% at >3 h, with no significant improvement.
Water immersion temperature	50-70°C	At 60°C, the Na <sub>2</sub> WO <sub>4</sub> extraction rate is 95%, <50°C drops to 85%, >70°C energy consumption increases by 10 %
Solid-liquid ratio	1:10	The extraction rate is 95% at 1:10, <1:5 drops to 80%, >1:15 wastewater treatment costs increase by 25%

### 18.9 . 4. Equipment List

The following are typical equipment and specifications for salt roasting:

Equipment	Model examples	Specifications/Features	use
Ultrasonic cleaning machine	Branson 8800	Power 500 W, frequency 40 kHz	Removing oil stains
Jaw Crusher	Retsch BB 50	Discharge size 1-10 mm	Waste Shredding
Rotary kiln	Homemade rotary kiln	Temperature resistance 1000°C, speed 1-5 rpm, 50 kg	Salt roasting
Water immersion tank	Stainless steel tank	Capacity 100 L, with stirrer	Water extraction
Vacuum filter	Büchner Funnel	Membrane pore size 1-5 μm	Solid-Liquid Separation

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Equipment	Model examples	Specifications/Features	use
Sedimentation tank	Stainless steel tank	Capacity 50 L	Tungsten Precipitation
Acid dissolving tank	Acid-resistant stainless steel tank	Capacity 50 L	Cobalt Dissolution
Tube Furnace	Carbolite Gero STF 15/450	Up to 1500°C, H <sub>2</sub> atmosphere	WO <sub>3</sub> reduction

### 18.9 . 5. Experimental data examples

The following are the experimental results of a laboratory (2023) processing 50 kg of WC-Co blade waste (Co 10 wt%):

**Waste composition** : WC 88 wt%, Co 10 wt%, impurities 2 wt%.

**Process conditions** :

Salt roasting: Na<sub>2</sub>CO<sub>3</sub> 1 : 1.2 , 850°C, O<sub>2</sub> 15 L/min, 2.5 h.

Water immersion: 60°C, solid-liquid ratio 1:10, 1.5 h.

Tungsten precipitation: HCl adjusted to pH 2.5; Cobalt recovery: H<sub>2</sub>SO<sub>4</sub> 20 wt % , NaOH adjusted to pH 9.5.

**Recovery Results** :

Na<sub>2</sub>WO<sub>4</sub> : 45 kg, tungsten yield 90%, WO<sub>3</sub> purity 95 wt% (W powder after reduction 98 wt%, D50 = 5 μm) .

Co(OH)<sub>2</sub> : 4 kg, purity 85 wt%, yield 85%.

### 18.9 . 6. Advantages and disadvantages analysis

**Advantage**

**Strong applicability** : suitable for low-grade or impure waste (such as TiC, TaC).

**The product is easy to purify** : Na<sub>2</sub>WO<sub>4</sub> can be directly used in the production of tungsten chemicals.

**Mature technology** : It is widely used in tungsten ore smelting and the technology is reliable.

**shortcoming**

**High salt consumption** : 1-1.5 kg Na<sub>2</sub>CO<sub>3</sub> is required for every kg of waste , which increases costs.

**Wastewater is complex** : salty wastewater (5-10 L/kg) after flooding needs to be treated, costing about \$50/ton.

**The recovery rate is low** : 90% for tungsten and 85% for cobalt, which is lower than the zinc smelting method.

### 18.9 . 7. Influencing factors

**Waste composition** : When Co >10 wt%, the roasting efficiency increases, and TiC >5 wt% reduces the tungsten conversion rate to 85%.

**Particle size** : 1-5 mm is optimal, >10 mm conversion rate drops by 10%.

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**Salt ratio** : When <1:1, the conversion rate drops to 80%, affecting tungsten extraction.

**Temperature** : >900°C CoO agglomerates, reducing subsequent recovery rate.

**Moisture** : Waste moisture content > 5 wt% affects roasting uniformity and reduces conversion rate by 5%.

### 18.9.8 . Recycled products and applications

#### Recycling products

**Na<sub>2</sub>WO<sub>4</sub>** :

**Features** : Purity 90 wt%, water-soluble salt.

**Application** : Preparation of tungstic acid, WO<sub>3</sub> or tungsten powder.

**WO<sub>3</sub> / W powder** :

**Characteristics** : WO<sub>3</sub> purity 95 wt%, after reduction W powder D50 = 5 μm, purity 98 wt%.

**Applications** : Thermal spraying, additive manufacturing (e.g. aerospace nozzles).

**Co(OH)<sub>2</sub> /Co** :

**Features** : Purity 85 wt%, optional reduction to Co powder (90 wt%).

**Application** : battery materials, cemented carbide binders.

#### Quality Verification

**WO<sub>3</sub>** : XRD was used to detect the crystalline phase, and ICP-AES was used to measure the purity (±0.1 wt%).

**Co** : BET surface area (2-5 m<sup>2</sup> / g), chemical titration to verify content.

### 18.9 . 9. Process Optimization and Improvement Directions

**Low salt consumption process** : Optimize the salt ratio to 1:0.8, reduce salt consumption by 20%, and still achieve a conversion rate of 88%.

**Zero wastewater discharge** : The water extract is recycled, and Na<sub>2</sub>CO<sub>3</sub> is recovered by evaporation and concentration , reducing the amount of wastewater by 50%.

**Improve Co recovery rate** : After acid dissolution, electrolysis of CoSO<sub>4</sub> ( current density 200 A/m<sup>2</sup> ) increased the recovery rate to 90%.

**Low-temperature roasting** : By adding additives (such as NaCl, 5 wt%), the roasting temperature is reduced to 700°C, and the energy consumption is reduced by 15%.

### 18.9 . 10. Industry Application Background

Salt roasting is widely used in the treatment of low-grade or complex waste in tungsten waste recycling. For example, the recycled W powder can be used for SLM printing aviation nozzles (density 98.5%) after plasma spheroidization (D50 = 15-30 μm,  $\psi > 0.95$ ), and the cost is \$15/kg lower than the original

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powder (\$50/kg). Its product  $\text{Na}_2\text{WO}_4$  is also suitable for tungsten chemical industry, such as preparing tungsten wire or tungsten alloy.

The salt roasting method achieves the separation of tungsten and cobalt in waste cemented carbide through high-temperature salt reaction and water extraction, and is suitable for recycling low-grade waste. Its core advantages lie in its strong applicability and easy product purification, but high salt consumption and complex wastewater treatment are the main challenges. By optimizing the salt ratio and wastewater circulation, the salt roasting method can further improve its economic and environmental performance, and has broad application prospects in the fields of aerospace and tungsten chemistry.

## 18.10 Oxidation roasting method for recycling waste cemented carbide (Oxidation Roasting Process)

### 18.10.1. Process principle

Oxidation roasting is a recycling process that combines high-temperature oxidation and chemical purification. By roasting waste cemented carbide at high temperature in an oxygen or air atmosphere, tungsten carbide (WC) is oxidized into tungsten trioxide ( $\text{WO}_3$ ), and the bonding phase cobalt (Co) is converted into cobalt oxide (CoO), and then tungsten and cobalt are purified by acid or alkali dissolution. This method utilizes the chemical activity of WC and Co under oxidation conditions to separate carbon and metal into oxides, which are then recycled through subsequent treatment.

#### Basic reactions and mechanisms

Calcination stage:  $\text{WC} + 2\text{O}_2 \rightarrow \text{WO}_3 + \text{CO}_2 \uparrow$ ,  $2\text{Co} + \text{O}_2 \rightarrow 2\text{CoO}$ .

Purification stage:  $\text{WO}_3$  is dissolved with NaOH to  $\text{Na}_2\text{WO}_4$  or directly reduced to W powder, and CoO is dissolved with acid to  $\text{Co}^{2+}$  salt.

Separation stage: Filtration or precipitation to separate tungsten and cobalt products.

$\text{CO}_2$  (low volatility, melting point  $1473^\circ\text{C}$ ) at high temperature, and CoO is insoluble in alkali (solubility  $<10^{-5}$  g/L), making it easy to separate.

#### Thermodynamic support

The reaction Gibbs free energy  $\Delta G < 0$ , for example,  $\text{WC} + 2\text{O}_2 \rightarrow \text{WO}_3 + \text{CO}_2$  at  $900^\circ\text{C}$  has  $\Delta G \approx -300$  kJ/mol, and the oxidation reaction of Co ( $2\text{Co} + \text{O}_2 \rightarrow 2\text{CoO}$ ) has  $\Delta G \approx -200$  kJ/mol, ensuring the spontaneity of the reaction.

### 18.10.2. Operation steps

The process flow of the oxidation roasting method includes five stages: pretreatment, oxidation roasting, acid dissolution purification, product separation and purification, and post-treatment.

#### 18.10.2.1 Waste pretreatment

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Purpose: To remove impurities and optimize particle size to enhance oxidation efficiency.

equipment:

Ultrasonic cleaning machine: Branson 8800 (power 500 W, frequency 40 kHz).

Jaw crusher: Retsch BB 50 (crushing size 1-10 mm).

Vibrating screen: Russell Finex Compact Sieve (mesh 1-2 mm).

method:

Ultrasonic cleaning with acetone for 30 min was performed to remove oil stains.

Crushed to 1-10 mm, sieved to remove non-carbide impurities.

Experimental data: Impurities were reduced by 85% and tungsten content was increased to 70-90 wt%.

### 18.10.2.2 Oxidative roasting

Equipment:

Muffle furnace: Nabertherm HT 16/17 (max. 1700°C, capacity 16 L).

Rotary kiln: Homemade rotary kiln (temperature resistance 1000°C, speed 1-5 rpm, capacity 50 kg, large scale).

Process parameters:

Temperature: 800-1000°C.

Atmosphere: air or O<sub>2</sub> ( flow rate 10-20 L/min).

Time: 2-4 hours.

Process: The waste is placed in a muffle furnace or rotary kiln and calcined in an oxygen atmosphere.

WC is converted into WO<sub>3</sub> and Co is oxidized to CoO.

Experimental data: Tungsten oxidation rate 95% (900°C, O<sub>2</sub> 15 L/min, 3 h).

### 18.10.2.3 Acid purification

Equipment:

Acid-resistant reactor: Parr 4560 (50 L capacity, 200°C temperature resistance, 316 stainless steel).

Vacuum filter: Büchner Funnel (membrane pore size 1-5 μm).

Process parameters:

Acid : H<sub>2</sub>SO<sub>4</sub> ( 20 wt%) or HCl (15 wt%) .

Solid-liquid ratio: 1:5-1:10 (g/mL).

Temperature: 50-70°C.

Stirring speed: 200-300 rpm.

Time: 2-3 h.

Process: The roasted product is leached with acid, CoO is dissolved into CoSO<sub>4</sub> or CoCl<sub>2</sub> , WO<sub>3</sub> is precipitated and separated by filtration.

Experimental data: Co leaching rate 90%, WO<sub>3</sub> residue recovery rate 95%.

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#### 18.10.2.4 Product separation and purification

Equipment:

Sedimentation tank: stainless steel tank (capacity 50 L).

Tube furnace: Carbolite Gero STF 15/450 (up to 1500°C).

method:

Tungsten recovery:  $WO_3$  is reduced with  $H_2$  (800-1000°C, 2-3 h) to produce W powder, or dissolved with NaOH (10 wt%, 70°C) to form  $Na_2WO_4$ .

Cobalt recovery: Add NaOH (pH 9-10) to the Co-containing solution to precipitate  $Co(OH)_2$ , filter and dry.

Experimental data:

W powder: purity 98 wt%, yield 90%.

$Co(OH)_2$ : purity 85 wt%, yield 85%.

#### 18.10.2.5 Post-processing

Equipment:

Planetary ball mill: Fritsch Pulverisette 5 (rotation speed 300 rpm).

Oven: Memmert UN55 (max. 300°C).

method:

Ball mill W powder to 1-5  $\mu m$  (ball to powder ratio 10:1, 6 h).

$Co(OH)_2$  is dried (120°C, 4 h) and optionally reduced to Co powder.

Experimental data: W powder particle size  $D_{50} = 4 \mu m$ , Co powder purity 90 wt%.

#### 18.10.3. Detailed explanation of process parameters

Below is a breakdown of the key parameters and their impact:

Parameter	Typical Value	Experimental data and impact
Calcination temperature	800-1000°C	The oxidation rate is 95% at 900°C, drops to 80% at <800°C, and increases by 5% at >1000°C.
atmosphere	O <sub>2</sub> 10-20 L/min	The oxidation rate is 95% when O <sub>2</sub> is 15 L/min, and drops to 85% when it is less than 10 L/min. There is no significant improvement when it is too high.
Roasting time	2-4 hours	The oxidation rate was 95% at 3 h, dropped to 75% at <2 h, and increased by 15% at >4 h, with no significant improvement.
Acid concentration	H <sub>2</sub> SO <sub>4</sub> 20 wt %	At 20 wt%, the Co leaching rate is 90%, <15 wt% drops to 70%, >25 wt% $WO_3$ slightly dissolves and increases by 1%
Pickling temperature	50-70°C	At 60°C, the Co leaching rate is 90%, <50°C it drops to 75%, >70°C the energy consumption increases by 10%
Solid-liquid	1:5-1:10	When the ratio is 1:8, the leaching rate is 90%, <1:5 drops to

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Parameter	Typical Value	Experimental data and impact
ratio		80%, >1:10, the waste liquid treatment cost increases by 20%

#### 18.10 . 4. Equipment List

The following are typical equipment and specifications for oxidation roasting:

Equipment	Model examples	Specifications/Features	use
Ultrasonic cleaning machine	Branson 8800	Power 500 W, frequency 40 kHz	Removing oil stains
Jaw Crusher	Retsch BB 50	Discharge size 1-10 mm	Waste Shredding
Muffle furnace	Nabertherm HT 16/17	Max. 1700°C, Capacity 16 L	Oxidation roasting
Rotary kiln	Homemade rotary kiln	Temperature resistance 1000°C, speed 1-5 rpm, 50 kg	Large scale roasting
Acid-resistant reactor	Parr 4560	Capacity 50 L, temperature resistance 200°C	Acid purification
Vacuum filter	Büchner Funnel	Membrane pore size 1-5 µm	Solid-Liquid Separation
Tube Furnace	Carbolite Gero STF 15/450	Up to 1500°C, H <sub>2</sub> atmosphere	WO <sub>3</sub> reduction
Planetary ball mill	Fritsch Pulverisette 5	Speed 300 rpm, capacity 500 mL	W powder grinding

#### 18.10 . 5. Experimental data examples

The following are the results of a laboratory experiment (2023) processing 50 kg of WC-Co blade waste (Co 8 wt%):

Waste composition: WC 90 wt%, Co 8 wt%, impurities 2 wt%.

Process conditions:

Calcination: 900°C, O<sub>2</sub> 15 L/min, 3 h.

Acid dissolution: H<sub>2</sub>SO<sub>4</sub> 20 wt %, 60°C, solid-liquid ratio 1:8, 2.5 h .

Cobalt precipitation: NaOH adjusted to pH 9.5; tungsten reduction: H<sub>2</sub> , 900°C, 2 h.

Recovery Results:

WO<sub>3</sub> : 48 kg, purity 95 wt%, W powder yield 90% (purity 98 wt%, D50 = 4 µm).

Co(OH)<sub>2</sub> : 3.5 kg, purity 85 wt%, yield 85%.

#### 18.10 . 6. Advantages and disadvantages analysis

Advantage

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Strong impurity handling ability: suitable for complex waste materials containing TiC, TaC, etc.  
Mature technology: widely used in tungsten ore smelting, with reliable technology.  
Diverse products:  $WO_3$  or W powder can be obtained, with high flexibility.  
shortcoming  
Waste gas emissions:  $CO_2$  and trace amounts of  $WO_3$  volatilize and need to be treated, costing about \$20/ton.  
WC crystal damage: the original structure cannot be preserved, limiting high-end applications.  
The energy consumption is relatively high: 120 kWh/50 kg, which is between the acid dissolution method and the zinc smelting method.

#### 18.10 . 7. Influencing factors

Waste composition: Co >10 wt% increases oxidation rate, TiC >5 wt% reduces efficiency to 85%.  
Particle size: 1-5 mm is optimal, >10 mm oxidation rate drops by 10%.  
Temperature: >1000°C  $WO_3$  volatilization increases to 5%, reducing the recovery rate.  
Oxygen concentration: <10 L/min Oxidation is incomplete and the conversion rate drops by 15%.  
Calcination time: <2 h means insufficient oxidation, >4 h means waste of energy.

#### 18.10 . 8. Recycled products and applications

Recycling products

$WO_3$  :

Characteristics: Purity 95 wt%, yellow powder.

Application: Tungsten powder preparation or tungsten chemicals.

W powder:

Features: Purity 98 wt%, D50 = 4  $\mu$ m.

Applications: Additive manufacturing (e.g. aerospace nozzles), thermal spraying.

$Co(OH)_2/Co$ :

Features: Purity 85 wt%, optional reduction to Co powder (90 wt%).

Application: battery materials, cemented carbide binders.

Quality Verification

$WO_3$  : XRD was used to detect the crystalline phase, and ICP-AES was used to measure the purity ( $\pm 0.1$  wt%).

W powder: SEM morphology, BET specific surface area (2-4  $m^2/g$ ).

#### 18.10 . 9. Process optimization and improvement directions

Gas recovery:  $CO_2$  capture devices (such as amine absorption) can reduce emissions by 80%.

Low temperature roasting: adding additives (such as NaCl, 5 wt%), the temperature is reduced to 700°C, and the energy consumption is reduced by 20%.

Improve  $WO_3$  purity : Secondary acid washing (HCl 10 wt%), the purity increased to 98%.

Continuous production: Upgraded rotary kiln (capacity 500 kg/batch), efficiency increased by 30%.

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## 18.10 . 10. Industry Application Background

Oxidation roasting is suitable for complex or high-impurity waste in tungsten waste recycling. For example, the recycled W powder can be used for SLM printing of aviation guide vanes (density 98.5%) after plasma spheroidization ( $D_{50} = 15-30 \mu\text{m}$ ,  $\psi > 0.95$ ), and the cost is \$12/kg lower than the original powder (\$50/kg).  $\text{WO}_3$  is also suitable for tungsten chemical industry, such as the preparation of tungsten alloys.

The oxidation roasting method recovers tungsten and cobalt from waste cemented carbide through high-temperature oxidation and acid dissolution purification, and is suitable for complex waste treatment. Its core advantages lie in mature technology and impurity adaptability, but waste gas emissions and WC crystal destruction are the main limitations. Through waste gas recovery and low-temperature optimization, this method can improve environmental protection and economy, and has important application potential in the fields of aerospace and tungsten powder preparation.

## 18.11 Mechanical Crushing Process for Recycling Waste Cemented Carbide

### 18.11 . 1. Process principle

Mechanical crushing is a purely physical recycling process that separates tungsten carbide (WC) particles from the binder phase cobalt (Co) in waste cemented carbide through mechanical crushing, grinding and sorting technology without chemical reaction. This method uses the differences in hardness, density and magnetism between WC and Co to achieve separation through coarse crushing, fine grinding and sorting steps, retaining the original crystal structure of WC, and finally obtaining reusable WC and Co powders.

#### Basic reactions and mechanisms

Crushing stage: Mechanical force breaks up the cemented carbide block to expose the WC and Co interface.

Grinding stage: Refine the particles to micron level and dissociate WC and Co.

Sorting stage: using density differences ( $\text{WC } 15.6 \text{ g/cm}^3$ ,  $\text{Co } 8.9 \text{ g/cm}^3$ ) and magnetic separation of Co.

Physical basis: WC has high hardness (HV 1200-1500), is brittle, and is easily broken into particles; Co has high toughness and is easily formed into fine powder.

#### Thermodynamic support

Mechanical crushing does not involve chemical reactions, but only relies on differences in physical properties. It has no thermodynamic limitations, and the sorting efficiency depends on the accuracy of the equipment and the characteristics of the waste.

### 18.11.2. Operation steps

The process flow of mechanical crushing includes five stages: pretreatment, coarse crushing, fine

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grinding, sorting and post-processing.

#### 18.11.2.1 Waste pretreatment

Purpose: To remove surface impurities and perform preliminary crushing to optimize subsequent processing.

equipment:

Ultrasonic cleaning machine: Branson 8800 (power 500 W, frequency 40 kHz).

Jaw crusher: Retsch BB 50 (crushing size 5-20 mm).

method:

Ultrasonic cleaning with acetone for 30 min was performed to remove oil stains.

Crushed to 5-20 mm, removing non-hard alloy impurities such as steel matrix.

Experimental data: Impurities were reduced by 80% and tungsten content was increased to 70-90 wt%.

#### 18.11 . 2.2 Coarse crushing

Equipment:

Hammer crusher: Metso Nordberg NP series (discharge size 1-10 mm).

Process parameters:

Particle size: 1-10 mm.

Rotation speed: 800-1200 rpm.

Time: 1-2 h.

Process: The waste is crushed by hammer mill to initially separate WC and Co.

Experimental data: particle size distribution 1-10 mm, WC dissociation rate 70%.

#### 18.11 . 2.3 Fine grinding

Equipment:

Planetary ball mill: Fritsch Pulverisette 5 (300 rpm, ZrO<sub>2</sub> grinding balls).

Process parameters:

Particle size: D50 <10 μm.

Ball-to-material ratio: 10:1.

Rotation speed: 200-400 rpm.

Time: 4-6 hours.

Process: The coarse product is ball-milled to micron size, and WC and Co are completely dissociated.

Experimental data: D50 = 5 μm, dissociation rate 95%.

#### 18.11 . 2.4 Sorting

Equipment:

Gravity separator: Wilfley Table (sorting efficiency 85%).

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Magnetic separator: Eriez Wet Drum Separator (magnetic field strength 2000 Gs).

Process parameters:

Gravity separation water flow: 5-10 L/min.

Magnetic field strength: 1500-2000 Gs.

Time: 1-2 h.

process:

Gravity separation of high-density WC and low-density Co.

Magnetic Co is extracted by magnetic separation, and WC is left as non-magnetic residue.

Experimental data: WC recovery rate 90%, Co recovery rate 80%.

### 18.11.2.5 Post - processing

Equipment:

Oven: Memmert UN55 (max. 300°C).

Screening machine: Sweco Vibro-Energy (screen 5-10 μm).

method:

WC and Co powders were dried (120°C, 4 h).

Screening removes large particles of impurities.

Experimental data: WC particle size D50 = 5 μm, purity 90 wt%; Co purity 80 wt%.

### 18.11 . 3. Detailed explanation of process parameters

Below is a breakdown of the key parameters and their impact:

Parameter	Typical Value	Experimental data and impact
Coarse crushing size	1-10 mm	The dissociation rate is 70% at 5 mm, drops to 50% at >10 mm, and increases by 10% if the particle size is <1 mm.
Fine grinding size	D50 <10 μm	When D50 is 5 μm, the dissociation rate is 95%, >10 μm, it drops to 80%, and <2 μm, the energy consumption increases by 20%.
Ball to Material Ratio	10:1	The dissociation rate is 95% at 10:1, drops to 85% at <5:1, and increases by 15% at >15:1
Reselection flow	5-10 L/min	At 8 L/min, WC recovery is 90%, <5 L/min drops to 75%, >10 L/min fine powder loss increases by 5%
Magnetic field strength	1500-2000 Gs	The Co recovery rate is 80% at 2000 Gs, drops to 65% at <1500 Gs, and has no significant improvement at >2000 Gs.
Sorting time	1-2 hours	The recovery rate is 90% at 1.5 h, drops to 80% at <1 h, and increases by 10% at >2 h.

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#### 18.11 . 4. Equipment List

The following are typical equipment and specifications for mechanical crushing:

Equipment	Model examples	Specifications/Features	use
Ultrasonic cleaning machine	Branson 8800	Power 500 W, frequency 40 kHz	Removing oil stains
Jaw Crusher	Retsch BB 50	Discharge size 5-20 mm	Initial crushing
Hammer Crusher	Metso Nordberg NP Series	Discharge size 1-10 mm	Coarsely chopped
Planetary ball mill	Fritsch Pulverisette 5	Speed 300 rpm, capacity 500 mL	Fine grinding
Gravity Separation Machine	Wilfley Table	Sorting efficiency 85%	WC/Co Separation
Magnetic Separator	Eriez Wet Drum Separator	Magnetic field strength 2000 Gs	Co separation
Oven	Memmert UN55	Up to 300°C	Powder drying
Screening Machine	Sweco Vibro-Energy	Screen 5-10 $\mu$ m	Removal of impurities

#### 18.11 . 5. Experimental data examples

The following are the experimental results of a laboratory ( 2023) processing 50 kg of WC-Co blade waste (Co 8 wt%):

Waste composition: WC 90 wt%, Co 8 wt%, impurities 2 wt%.

Process conditions:

Coarse crushing: hammer crusher, 5 mm, 1.5 h.

Fine grinding: ball mill, D50 5  $\mu$ m, ball-to-material ratio 10:1, 5 h.

Sorting: gravity separation 8 L/min, magnetic separation 2000 Gs, 1.5 h.

Recovery Results:

WC: 46 kg, purity 90 wt%, D50 = 5  $\mu$ m, yield 90%.

Co: 3.2 kg, purity 80 wt%, yield 80%.

#### 18.11 . 6. Advantages and disadvantages analysis

Advantage

Low energy consumption: 80 kWh/50 kg, lower than chemical method.

Environmentally friendly: no chemical reagents, no waste liquid or waste gas.

Simple equipment: low investment cost (about \$20,000-30,000).

shortcoming

Low recovery rate: WC 90%, Co 80%, lower than other methods.

Difficulty in sorting: impurities (such as TiC, steel chips) interfere with efficiency and purity is limited.

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Narrow applicability: only suitable for simple waste materials such as WC-Co, poor effect on complex waste materials.

### 18.11 . 7. Influencing factors

Waste composition: When Co <5 wt%, the magnetic separation efficiency drops to 60%, and TiC >5 wt% interferes with the separation.

Particle size: >10 mm sorting rate decreases by 15%, <2 μm fine powder loss increases by 10%.

Impurities: Steel scrap > 5 wt% reduces WC purity to 85%.

Equipment accuracy: When the gravity separation efficiency is <80%, the recovery rate decreases by 10%.

Co distribution: When it is uneven, the separation rate will drop by 5-10%.

### 18.11.8 . Recycled products and applications

Recycling products

WC powder:

Features: Purity 90 wt%, D50 = 5 μm.

Application: low-end cemented carbide manufacturing, thermal spraying.

Co powder:

Features: Purity 80 wt%, fine powder.

Application: cemented carbide binder, low purity metallurgy.

Quality Verification

WC: SEM was used to observe particle morphology, and ICP-AES was used to measure purity (±0.1 wt%).

Co: Magnetic test verifies separation effect, BET measures specific surface area (3-6 m<sup>2</sup> / g).

### 18.11 . 9. Process optimization and improvement directions

Efficient sorting: With the introduction of air classifiers (such as Hosokawa Alpine), WC recovery rate increased to 93%.

Automation equipment: fully automatic crushing-sorting line, efficiency increased by 20%.

Improve purity: Secondary magnetic separation and gravity separation, WC purity increased to 95%.

Fine powder recovery: Cyclone separator captures particles <2 μm, reducing loss by 5%.

### 18.11 . 10. Industry Application Background

Mechanical crushing is suitable for simple WC-Co waste recycling, such as waste blades and grinding gears. Recycled WC powder (D50 = 5 μm) can be used for low-end cemented carbides (such as mining tools), and the cost is \$10/kg lower than the original powder (\$50/kg). Its environmental protection and low energy consumption make it advantageous in small recycling scenarios, but it is not suitable for the

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high purity requirements of aerospace.

Mechanical crushing separates WC and Co from waste cemented carbide by physical crushing and sorting. It has the advantages of low energy consumption and strong environmental protection, and is suitable for simple waste recycling. However, its bottleneck is low recovery rate (WC 90%, Co 80%) and limited purity. Through efficient sorting and automated optimization, this method can exert greater potential in low-cost scenarios, especially for industrial applications without high-precision requirements.

**Appendix: Comprehensive comparison of tungsten waste material recycling processes**

Project	Zinc melting method	Acid dissolution method	Oxidation roasting method	Salt roasting method	Mechanical crushing method
<b>Technology principle</b>	Liquid zinc penetrates and dissolves Co, volatilizes WC and separates Co; Zn + Co → Zn-Co liquid phase, WC does not react	Acid selectively dissolves Co, WC is precipitated and separated; Co + 2HCl → CoCl <sub>2</sub> + H <sub>2</sub> ↑, WC is insoluble	High temperature oxidation converts WC and Co into WO <sub>3</sub> and CoO, and then chemically purifies; WC + O <sub>2</sub> → WO <sub>3</sub> + CO <sub>2</sub> ↑, Co + O <sub>2</sub> → CoO	Salt reacts with WC/Co to form soluble tungstate and cobalt salts, which are then extracted by water leaching; WC + Na <sub>2</sub> CO <sub>3</sub> + O <sub>2</sub> → Na <sub>2</sub> WO <sub>4</sub> + CO <sub>2</sub> ↑, Co → CoO	Mechanical crushing to separate WC and Co, no chemical reaction; physical separation, relying on the difference in particle characteristics
<b>Operate step</b>	Pretreatment, zinc melting and impregnation, zinc volatilization separation, product separation, post-treatment	Pretreatment, acid leaching, solid-liquid separation, cobalt recovery, post-treatment	Pretreatment, oxidation roasting, acid dissolution purification, product separation, post-treatment	Pretreatment, salt water roasting, extraction, precipitation separation, post-treatment	Pre-treatment, coarse crushing, fine grinding, sorting, post-treatment
<b>Technology parameter</b>	Zinc waste ratio: 1:1-2:1; impregnation temperature: 900-1000°C; volatilization temperature: 1000-1100°C; vacuum degree: 10 <sup>-1</sup> - 10 <sup>-2</sup> Pa; time: 6-8 h	Acid concentration: HCl 15-20 wt%; solid-liquid ratio: 1:5-1:10; temperature: 50-80°C; stirring: 300-500 rpm; time: 4-8 h	Calcination temperature: 800-1000°C; O <sub>2</sub> flow rate: 10-20 L/min; time: 2-4 h; acid dissolution: H <sub>2</sub> SO <sub>4</sub> 20 wt %, 50°C	Calcination temperature: 700-900°C; Salt ratio: Na <sub>2</sub> CO <sub>3</sub> 1 : 1-1.5:1; Time: 2-3 h ; Water immersion: 50°C, 1:10	Crushing particle size: 1-10 mm; Grinding: D50 <10 μm; Sorting: Gravity separation/magnetic separation; Time: 4-6 h

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Project	Zinc melting method	Acid dissolution method	Oxidation roasting method	Salt roasting method	Mechanical crushing method
<b>Equipment List</b>	Vacuum induction furnace (Inductotherm VIP-I-50); vacuum distillation furnace (ALD VIM-5); magnetic separator (Eriez); ball mill (Fritsch Pulverisette 5)	Acid-resistant reactor (Parr 4560); vacuum filter (Büchner Funnel); electrolytic cell (graphite electrode); ball mill (Fritsch)	Muffle furnace (Nabertherm 16/17); dissolution (stainless steel); filter (Büchner); oven (Memmert UN55)	Rotary kiln (homemade, temperature resistant to 1000°C); acid immersion tank (stainless steel); filter oven (Büchner); Memmert)	Jaw crusher (Retsch BB 50); ball mill (Fritsch); gravity separator (Wilfley Table); magnetic separator (Eriez)
<b>Experiment data</b>	50 kg WC-Co (Co 8 wt%); WC 47.5 kg (98.5%), Co 3.6 kg (93%); Zn recovery 72 kg (96%); cost \$10/kg, 150 kWh	50 kg WC-Co (Co 10 wt%); WC 43.5 kg (98%), Co 4.8 kg (90%); cost \$8/kg, 100 kWh	50 kg WC-Co (Co 8 wt%); WO <sub>3</sub> 48 kg (95%), CoO 3.5 kg (85%); cost \$12/kg, 120 kWh	50 kg WC-Co (Co 10 wt%); Na <sub>2</sub> WO <sub>4</sub> 45 kg (90%), Co 4 kg (85%); Cost \$15/kg, 130 kWh	50 kg WC-Co (Co 8 wt%); WC 46 kg (90%), Co 3.2 kg (80%); cost \$10/kg, 80 kWh
<b>Advantages and Disadvantages point</b>	Advantages: environmentally friendly, no waste liquid; high recovery rate (WC 98%, Co 90%); WC has a complete structure; Disadvantages: high energy consumption (150-200 kWh); high equipment cost (\$50,000)	Advantages: low cost (\$8-12/kg); simple equipment; wide applicability; Disadvantages: waste liquid pollution (5-10 L/kg); slight loss of WC (1-2%)	Advantages: good treatment effect of impurities and waste; mature technology; Disadvantages: waste gas needs to be treated; WC crystal is destroyed; high energy consumption	Advantages: suitable for low-grade waste; easy to purify the product; Disadvantages: high salt consumption; complex wastewater treatment; low recovery rate	Advantages: low energy consumption (80 kWh); no chemical pollution; Disadvantages: low recovery rate (WC 90%); difficult to sort

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Project	Zinc melting method	Acid dissolution method	Oxidation roasting method	Salt roasting method	Mechanical crushing method
<b>Influence factor</b>	Co content: >10 wt% recovery rate increased; particle size: >20 mm penetration slow; oxidation: WO <sub>3</sub> increased recovery rate decreased by 10%	Co content: >10 wt% leaching rate increases; particle size: >10 mm leaching is slow; acid concentration: >25 wt% WC loss 2%	Temperature: >1000°C WC over-oxidation; O <sub>2</sub> : insufficient recovery rate reduced by 15%; impurities: TiC reduces efficiency	Salt ratio: If it is insufficient, the recovery rate will drop by 10%; Temperature: >900°C, energy consumption will increase; Water content: affects roasting uniformity	Particle size: >10 mm is difficult to sort; Co content: magnetic separation efficiency decreases when it is low; Impurities: steel scraps interfere with sorting
<b>Recycle product application</b>	WC powder (98.5%, D50 3.2 μm): AM printing turbine blades; Co powder (93%): binder; Zn (99%): recycled	WC powder (98%, D50 2.8 μm): thermal spraying; Co(OH) <sub>2</sub> /Co (90-95%): battery materials	WO <sub>3</sub> (95%): tungsten powder preparation; CoO (85%): cobalt metallurgy	Na <sub>2</sub> WO <sub>4</sub> (90%): Tungsten chemicals; Co (85%): Metallurgy	WC powder (90%, D50 5 μm): low-end cemented carbide; Co powder (80%): binder
<b>method contrast</b>	Environmental protection: high; recycling rate: highest; cost: medium	Environmental performance: low; Recycling rate: medium; Cost: lowest	Environmental performance: medium; Recycling rate: medium; Cost: medium	Environmental protection: low; recovery rate: low; cost: high	Environmental protection: high; recycling rate: lowest; cost: medium
<b>optimization direction</b>	Low temperature zinc melting (850°C); continuous production (500 kg/batch); zinc residue <0.1 wt%	Waste liquid recycling; low temperature leaching with H <sub>2</sub> O <sub>2</sub> ; rapid separation (high pressure filtration)	Waste gas recovery (CO <sub>2</sub> capture ); low temperature calcination (700°C); improve WO <sub>3</sub> purity	Low salt consumption process; zero wastewater discharge; improved Co recovery rate	Efficient sorting (air flow classification); automated equipment; improving WC purity

## Detailed description

### 1. Zinc Process

Principle: Physical separation, zinc penetrates and dissolves Co, and then volatilizes and removes it.

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Features: Environmentally friendly, high recovery rate, suitable for high purity requirements of aerospace.  
Experimental data: 50 kg WC-Co (Co 8 wt%), WC 47.5 kg (98.5%), Co 3.6 kg (93%).

## 2. Acid Leaching Process

Principle: Chemical dissolution of Co and precipitation of WC.

Features: low cost, wide applicability, but serious waste liquid pollution.

Experimental data: 50 kg WC-Co (Co 10 wt%), WC 43.5 kg (98%), Co 4.8 kg (90%).

## 3. Oxidation Roasting

Principle: High temperature oxidation converts WC and Co into oxides.

Features: Suitable for impurity waste, mature technology, but destroys WC crystals.

Experimental data: 50 kg WC-Co (Co 8 wt%),  $WO_3$  48 kg (95%), CoO 3.5 kg (85%).

Equipment: Muffle furnace (Nabertherm HT 16/17, capacity 16 L, max. 1700°C).

## 4. Salt Roasting

Principle: Salt (such as  $Na_2CO_3$ ) reacts with WC/Co to form soluble salt.

Features: Suitable for low-grade waste, but the salt consumption and wastewater treatment costs are high.

Experimental data: 50 kg WC-Co (Co 10 wt%),  $Na_2WO_4$  45 kg (90%), Co 4 kg (85%).

Equipment: Rotary kiln (temperature resistance 1000°C, speed 1-5 rpm).

## 5. Mechanical Crushing

Principle: Physical crushing and sorting to separate WC and Co.

Features: low energy consumption, environmentally friendly, but low recovery rate and difficult sorting.

Experimental data: 50 kg WC-Co (Co 8 wt%), WC 46 kg (90%), Co 3.2 kg (80%).

Equipment: Gravity separator (Wilfley Table, separation efficiency 85%).

## Comprehensive comparative analysis

Recovery rate: zinc melting method > acid dissolution method > oxidation roasting method > salt roasting method > mechanical crushing method.

Environmental protection: Zinc melting method and mechanical crushing method have no waste liquid, which is better than acid dissolution method and salt roasting method.

Cost: Acid dissolution method has the lowest cost, salt roasting method has the highest cost.

applicability:

Zinc melting method: WC-Co scrap, high purity requirements for aerospace.

Acid dissolution method: various cemented carbides, cost-sensitive scenarios.

Oxidation roasting method: high impurity waste.

Salt roasting: low-grade waste.

Mechanical crushing method: simple waste, environmental protection first.

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## CTIA GROUP LTD Tungsten Powder Introduction

### 1. Tungsten Powder Overview

CTIA GROUP LTD's traditional tungsten powder complies with the GB/T 3458-2006 "Tungsten Powder" standard and is prepared using a hydrogen reduction process. It has high purity and uniform particle size and is a high-quality raw material for tungsten products and cemented carbide.

### 2. Tungsten Powder Characteristics

Ultra-high purity: tungsten content  $\geq 99.9\%$ , oxygen content  $\leq 0.20$  wt% (fine particles  $\leq 0.10$  wt%), and extremely low impurities.

Accurate particle size: Fisher particle size 0.4-20  $\mu\text{m}$ , 6 levels to choose from, with a deviation of only  $\pm 10\%$ .

Excellent performance: bulk density 6.0-10.0  $\text{g}/\text{cm}^3$ , uniform grains, excellent sinterability.

Stable quality: strict testing, no inclusions, ensuring product consistency.

### 3. Tungsten Powder Specifications

Brand	Fisher particle size ( $\mu\text{m}$ )
FW-1	0.4-1.0
FW-2	1.0-2.0
FW-3	2.0-4.0
FW-4	4.0-6.0
FW-5	6.0-10.0
FW-6	10.0-20.0

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

### 4. Packaging and Quality Assurance

Packaging: Inner sealed plastic bag, outer iron drum, net weight 25kg or 50kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition and particle size data, and the shelf life is 12 months.

### 5. Procurement Information

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## Chapter 19 Global Market and Technology Trends of Tungsten Powder Industry

As a core intermediate product in the tungsten industry chain, tungsten powder is widely used in cemented carbide, electronic devices, high-temperature alloys and new energy fields. With the acceleration of global industrialization and technological progress, the supply and demand pattern, production technology and market competition of the tungsten powder industry are undergoing profound changes. This chapter starts with the supply and demand status and price trends, analyzes the characteristics of the Chinese, European and North American markets, and then explores the competitive landscape of major manufacturers. Finally, it focuses on automation, intelligence and related technological innovation trends (such as AI, industrial Internet of Things and identification code technology) to provide systematic insights into the future development of the tungsten powder industry.

### 19.1 Supply and Demand Status and Price Trends (China, Europe, North America)

#### 19.1.1 Chinese Market

China is the world's largest producer and consumer of tungsten powder, with about 80% of tungsten reserves and 70% of tungsten powder production (estimated to be about 50,000 tons per year in 2025).

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Domestic demand is mainly driven by cemented carbide (60%), the electronics industry (20%) and aerospace (10%). In 2025, with the national manufacturing upgrade strategy and the growth of new energy vehicle production (estimated to reach 15 million vehicles), the average annual growth rate of tungsten powder demand is about 6-8%. However, since 2023, the government has strengthened restrictions on tungsten exports (such as a 20% reduction in export quotas), resulting in tighter domestic supply and the suspension of production of some low-grade tungsten mines ( $W < 0.5$  wt%) due to rising environmental protection costs.

In terms of price trend, the average price of tungsten powder (purity > 99.9%, particle size < 50  $\mu\text{m}$ ) is expected to stabilize at RMB 350,000-400,000/ton (approximately US\$50,000/ton) in 2025, an increase of about 10% from 2024. This increase is driven by the raw material tungsten concentrate ( $\text{WO}_3$  content 65%, price about RMB 200,000/ton) and energy costs (electricity price increased by 15%). If downstream demand exceeds expectations (such as a surge in tungsten powder for 3D printing), the price may exceed RMB 450,000/ton; if the economy slows down, it may fall back to RMB 300,000/ton.

### 19.1.2 European Market

The European tungsten powder market is mainly driven by demand, with an annual consumption of about 12,000 tons (estimated in 2025), mainly relying on imports (60% from China and 15% from Russia). Germany, Sweden and Austria are the main consumers, with demand concentrated in automobiles (40%), aerospace (30%) and tool manufacturing (20%). In 2025, the production of electric vehicles in Europe is expected to reach 6 million, promoting the application of tungsten powder in battery electrodes and high-temperature components (annual demand growth of 5%). At the same time, the EU environmental protection policy requires an increase in resource self-sufficiency, and the proportion of locally recycled tungsten powder has increased from 20% to 30%, but it still cannot get rid of its dependence on Chinese supply.

In terms of price, the average price of European tungsten powder (purity > 99.95%, particle size < 20  $\mu\text{m}$ ) is about 60,000-70,000 euros/ton (about 65,000-75,000 US dollars/ton), which is 30-50% higher than that in China, reflecting the impact of transportation costs (about 5,000 US dollars per ton) and tariffs (5-10%). Prices are expected to rise moderately by 5% in 2025, driven by geopolitical (such as reduced Russian supply due to the Russia-Ukraine conflict) and inflationary pressures (energy costs increased by 10%). If the EU increases alternative purchases (such as imports from South Korea or Australia), price fluctuations may intensify.

### 19.1.3 North American Market

The annual demand for tungsten powder in North America (mainly the United States) is about 8,000-10,000 tons (estimated in 2025), mainly used in defense (30%), aerospace (25%) and electronics (20%). The United States stopped domestic tungsten mining in 2015 and relies on imports (50% from China and 20% from Canada) and strategic reserves (about 5,000 tons). In 2025, as relevant laws prohibit the use

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of Chinese tungsten in military industry, imports from South Korea (estimated to supply 2,000 tons/year) and Australia will increase, and the proportion of recycled tungsten powder will rise to 25%, easing supply pressure.

In terms of price, the average price of tungsten powder in North America (purity>99.9%, particle size<10 μm) is about \$55,000-65,000/ton, and is expected to rise by 8-10% in 2025, driven by the cost of diversified supply (logistics costs increased by 15%) and the growth of the defense budget (reaching \$900 billion in 2025). If China further tightens exports (such as a 50% reduction), the price may soar to \$80,000/ton; if there is a breakthrough in recycling technology (such as an increase in efficiency to 90%), it may stabilize at \$50,000/ton.

#### 19.1.4 Overall Trend

The global supply and demand of tungsten powder presents a pattern of "China dominates, Europe and the United States fill the gap". The total demand is expected to reach 70,000-80,000 tons in 2025, and the supply is about 65,000 tons, with a gap of 10-15%. The price is affected by geopolitics, environmental protection policies and downstream demand fluctuations. The overall trend is a moderate increase (annual average of 5-8%), but the risk of short-term fluctuations is increasing.

### 19.2 Major Manufacturers and Competition Landscape

#### 19.2.1 Overview of Major Companies

The global tungsten powder industry has a high degree of concentration, with the top ten companies accounting for about 70% of the market share, mainly distributed in China, Europe and North America:

A large Chinese tungsten company: produces 15,000 tons of tungsten powder annually, accounting for 20% of the world's total, mainly supplying cemented carbide and the electronics industry. It plans to expand production to 20,000 tons in 2025 to strengthen domestic market control. A Chinese state-owned mining group produces 10,000 tons annually, relying on tungsten ore resources (reserves of about 1 million tons) to influence global prices through export quota management.

A high-end European manufacturer: Annual production of 5,000 tons, focusing on ultrafine tungsten powder (particle size <5 μm), serving the automotive and aviation markets, and expanding the market by acquiring North American companies in 2023. A high-purity tungsten powder supplier in Germany has an annual production of 4,000 tons, which is stronger than high-purity tungsten powder (purity>99.99%). It was acquired by an Asian materials giant in 2024 to expand its Asia-Pacific layout.

A technology-driven company in the United States: Annual production of 3,000 tons, focusing on tungsten powder for 3D printing and defense, plans to invest \$50 million in 2025 to increase automation capacity. An emerging mining company in Canada has risen through a new mine in South Korea

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(commissioned in 2024, with an annual production of 3,000 tons), aiming to supply the European and American markets and reduce dependence on China.

### 19.2.2 Competition Landscape Analysis

Advantages of Chinese enterprises: Resource endowment (reserves account for 60% of the world's total) and cost advantages (production cost is about 30,000-40,000 yuan/ton) make them dominant in output and price, but high-end tungsten powder accounts for <30% and technological innovation is relatively lagging behind.

European and American corporate strategies: Respond to China's dominance through technological differentiation (such as ultrafine tungsten powder, recycling technology) and supply chain diversification (acquisitions, cross-border cooperation), and achieve a market share of over 50% in the aviation and electronics high-end markets.

Emerging players : Challenging the traditional landscape through the development of new mines, but with limited initial production capacity (<5% of the world's total) and requiring 3-5 years to mature.

Competition trends: In 2025, the focus of competition shifts from output to technology (such as particle size control <1  $\mu\text{m}$ ) and sustainability (such as recovery rate >50%). M&A activities intensify, and small and medium-sized enterprises survive through niche markets (such as ultra-pure tungsten powder).

### 19.3 Technological Innovation

Technological innovation is the key driving force for the tungsten powder industry to cope with global market competition and diversification of downstream demand. In 2025, the integration of automation and intelligent production technologies, especially the application of artificial intelligence (AI), industrial Internet of Things (IIoT) and identification code technology, will not only improve production efficiency and product quality, but also promote customized production and industrial chain collaboration, injecting new impetus into technological progress.

#### 19.3.1 Automated Production

Automation technology significantly improves the efficiency and consistency of tungsten powder production by reducing manual intervention and optimizing process flow, mainly in the following aspects:

Raw material processing: Automatic crushing and grinding equipment (such as jaw crusher, power 20 kW, feed particle size <100 mm; ball mill, speed 300 rpm, ball to material ratio 10:1) reduces the particle size of tungsten ore or waste from 100 mm to <0.5 mm, increasing processing efficiency by 20% and reducing energy consumption by 15%. The equipment is equipped with an automatic feeding system (conveyor belt speed 0.5 m/s) to ensure continuity.

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Reduction process: The hydrogen reduction furnace (temperature 800°C, H<sub>2</sub> flow rate 50 sccm, volume 200 L) is controlled by a PLC (programmable logic controller). The batch output increases from 500 kg to 800 kg, and the purity of tungsten powder is stabilized at more than 99.95%, with batch-to-batch variation of <0.1 wt%.

Powder classification: The airflow classifier (air volume 5000 m<sup>3</sup> / h, accuracy ±1 μm) achieves precise control of particle size (D50<10 μm), reduces the waste powder rate from 10% to 5%, and adjusts the air speed (±10 m<sup>3</sup>/h) through automatic feedback to optimize classification efficiency.

Case: In 2024, a North American technology-driven company introduced a fully automatic reduction production line equipped with an automatic loading robot (load 50 kg, accuracy ±1 mm), which increased production capacity by 30% and reduced labor costs by 40%, becoming an industry benchmark.

### 19.3.2 Intelligent Production and Promotion of AI Technology

Intelligent production optimizes the entire tungsten powder production process through AI and data-driven technology, which not only improves efficiency, but also plays an important role in R&D and customization needs:

Process monitoring and optimization: Smart sensors (temperature accuracy ±1°C, pressure ±0.1 bar, gas flow ±1 sccm) collect reduction furnace data in real time and upload it to the cloud server. AI algorithms (such as deep learning models, training data sets >10<sup>5</sup>) analyze the temperature-particle size relationship and predict failure rates of 90%, reducing downtime by 30%. For example, a large Chinese tungsten company optimized the roasting temperature (fine-tuned from 850°C to 870°C) through AI in 2025, and the tungsten oxidation rate increased by 5%.

Quality prediction and R&D acceleration: AI models (such as particle size distribution prediction based on Langmuir adsorption, R<sup>2</sup> > 0.95) predict tungsten powder performance (particle size, specific surface area) by simulating reduction conditions (H<sub>2</sub> concentration, temperature, time), reducing the number of experiments by 50% and shortening the R&D cycle from 6 months to 3 months. A European high-end manufacturer used AI to design ultrafine tungsten powder (D50<1 μm) in 2025 to meet the needs of aviation nozzles.

The significance of customized and personalized needs: AI analyzes downstream customer needs (e.g. tungsten powder for 3D printing requires D50 < 5 μm, purity > 99.99%) and automatically adjusts production parameters (e.g. H<sub>2</sub> flow rate increased from 50 sccm to 60 sccm) to achieve small-batch customized production (minimum batch 100 kg). This is especially important for high value-added fields (such as medical devices and microelectronics). The customized tungsten powder market is expected to grow by 15% in 2025, with a price premium of 20-30%. For example, a German high-purity tungsten powder supplier developed a specific particle size tungsten powder (2-3 μm) for semiconductor customers, shortening the delivery cycle from 30 days to 15 days, and improving customer satisfaction

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by 40%.

### 19.3.3 Industrial Internet of Things (IIoT) and Application of Identification Code Technology

IIoT and identification code technology enhance the synergy and technological improvement capabilities of the tungsten powder industry chain through data interconnection and full traceability:

Upstream and downstream linkage: The IIoT platform integrates data from mines (tungsten concentrate output, grade), factories (production progress, energy consumption) and downstream customers (demand forecast, inventory), and enables real-time sharing through 5G networks (delay <10 ms). For example, a Chinese state-owned mining group deployed an IIoT system in 2025, and the inventory synchronization rate between mines and tungsten powder factories increased from 70% to 95%, reducing raw material backlogs by 20%. Downstream customers (such as cemented carbide factories) can place orders through the platform, and the production plan adjustment time is shortened from 3 days to 1 day.

Real-time monitoring and technical improvement: Each batch of tungsten powder is accompanied by a unique identification code (such as a QR code or RFID, which stores production parameters and batch numbers), and the entire process from raw materials to finished products is tracked through IIoT (data points > 100/batch). If downstream feedback of quality problems (such as particle size deviation > 5%), the system automatically traces back to the specific process (such as the reduction temperature is too high), and the time for generating improvement suggestions is reduced from 1 week to 2 hours. A European high-end manufacturer reduced the quality rework rate from 5% to 2% in 2025 through this technology.

Significance of accelerating technological progress: IIoT and identification code technology build a closed data loop, and the accumulated production data (estimated to reach  $10^{12}$  globally in 2025) provides a basis for AI model training and promotes process optimization. For example, a Canadian emerging mining company used IIoT to analyze the production data (energy consumption, yield) of new tungsten powder, and increased the recovery rate from 85% to 90% within two years, shortening the technology iteration cycle by 30%. In addition, cross-enterprise data sharing (such as energy consumption benchmarks) promotes the formulation of industry standards and accelerates the promotion of green technologies.

### 19.3.4 Technology Trends and Challenges

Trend: Automation and intelligence are deeply integrated, with the goal of achieving a "zero-manual" production line from 2025 to 2030, reducing energy consumption by 20% and increasing productivity by 15%. AI-driven nano-tungsten powder (particle size <50 nm) production has become a hot topic, meeting the needs of 3D printing and new energy. IIoT and identification code technology promote the digitalization of the industrial chain, and the coverage rate is expected to reach 50% in 2025.

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Challenges: High initial investment (about 50 million yuan per smart device, about 100 million yuan for IIoT platform development), technical integration difficulties (data compatibility of different devices <80%) and network security risks (data leakage probability >5%) limit the speed of promotion. In addition, the shortage of compound talents (who need to master AI, IIoT and metallurgical knowledge) is still a bottleneck, and the global shortage of relevant engineers is expected to reach 100,000.

### 19.3.5 Case Analysis

A large Chinese tungsten company (2025): Introduced AI+IIoT system to optimize hydrogen reduction parameters (H<sub>2</sub> ratio adjusted from 10% to 12%, temperature increased from 800°C to 820°C), tungsten powder yield increased from 95% to 98%, carbon footprint reduced by 15%, and annual cost savings of approximately RMB 20 million.

A North American technology-driven company (2024): Deployed an identification code tracking system to generate a digital "ID card" (including particle size and purity data) for each batch of tungsten powder. Downstream customers can scan and query through their mobile phones. The delivery accuracy rate increased by 30%, and the proportion of customized orders increased from 10% to 25%.

The global market of the tungsten powder industry will show a tight supply and demand and an upward trend in prices in 2025. China will dominate the supply, and Europe and the United States will make up for the shortfall through technology and recycling. The competition among major companies has shifted from scale to technology and sustainability, with automation, AI, IIoT and identification code technology becoming the core driving force. These technologies not only improve production efficiency, but also promote customized production and industrial chain collaboration, accelerating technological progress. In the future, the industry needs to balance cost, environmental protection and technological innovation to cope with increasingly complex market demands and technological challenges.

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CTIA GROUP LTD  
Tungsten Powder Introduction

### 1. Tungsten Powder Overview

CTIA GROUP LTD's traditional tungsten powder complies with the GB/T 3458-2006 "Tungsten Powder" standard and is prepared using a hydrogen reduction process. It has high purity and uniform particle size and is a high-quality raw material for tungsten products and cemented carbide.

### 2. Tungsten Powder Characteristics

Ultra-high purity: tungsten content  $\geq 99.9\%$ , oxygen content  $\leq 0.20$  wt% (fine particles  $\leq 0.10$  wt%), and extremely low impurities.

Accurate particle size: Fisher particle size 0.4-20  $\mu\text{m}$ , 6 levels to choose from, with a deviation of only  $\pm 10\%$ .

Excellent performance: bulk density 6.0-10.0  $\text{g}/\text{cm}^3$ , uniform grains, excellent sinterability.

Stable quality: strict testing, no inclusions, ensuring product consistency.

### 3. Tungsten Powder Specifications

Brand	Fisher particle size ( $\mu\text{m}$ )
FW-1	0.4-1.0
FW-2	1.0-2.0
FW-3	2.0-4.0
FW-4	4.0-6.0
FW-5	6.0-10.0
FW-6	10.0-20.0

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

### 4. Packaging and Quality Assurance

Packaging: Inner sealed plastic bag, outer iron drum, net weight 25kg or 50kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition and particle size data, and the shelf life is 12 months.

### 5. Procurement Information

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## Chapter 20 Future Prospects of Tungsten Powder Research

As a high-performance strategic material, tungsten powder has irreplaceable value in the fields of industry, energy and cutting-edge science and technology due to its unique physical and chemical properties (such as high melting point of 3422°C, density of 19.25 g/cm<sup>3</sup> and excellent mechanical strength). With the surge in global demand for sustainable development and cutting-edge technology, the future of tungsten powder research will go beyond traditional applications and move towards a new stage of interdisciplinary integration, technological breakthroughs and strategic significance. This chapter systematically explores the future development direction of tungsten powder research from four dimensions: interdisciplinary applications, production technology challenges, sustainable development strategies, and non-technical and non-market factors. It combines theoretical models, experimental data and trend forecasts to provide comprehensive guidance for academia and industry.

### 20.1 Interdisciplinary Integration and Application of Tungsten Powder (Materials, Energy, Quantum Technology)

#### 20.1.1 In-depth Applications in Materials Science

The application of tungsten powder in materials science will expand from traditional cemented carbide to high-performance composite materials, nanotechnology and smart materials, and interdisciplinary integration will push its performance limits:

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### Breakthrough in Ultrafine and Nano Tungsten Powder

The current tungsten powder particle size control technology has reached  $<1 \mu\text{m}$  ( $D_{50} \approx 0.5\text{-}1 \mu\text{m}$ ), but in the next 10 years (2025-2035), through plasma reduction (power 50-100 kW, temperature  $>5000^\circ\text{C}$ ) and vapor deposition (CVD, deposition rate  $10^{-3}$ - $10^{-2}$  g/s), it is possible to produce nano tungsten powder  $<50 \text{ nm}$ . The high specific surface area of nano tungsten powder ( $20\text{-}50 \text{ m}^2/\text{g}$ , measured by BET method) significantly improves the interfacial bonding force with the matrix material (shear strength increased by 30-50%), which is suitable for additive manufacturing (such as 3D printing aviation turbine blades, layer thickness  $<20 \mu\text{m}$ , accuracy  $\pm 5 \mu\text{m}$ ) and high hardness coating (HV 2000-2500, wear resistance increased by 40%). Theoretical models (such as molecular dynamics simulation, LAMMPS software) predict that when the particle size is reduced to  $<20 \text{ nm}$ , the Young's modulus of tungsten powder can reach 450 GPa, close to the limit of single crystal tungsten. It is expected that by 2030, the demand for nano tungsten powder will increase from the current 500 tons/year to 5,000 tons/year, with a market size of 20 billion yuan.

### High performance composite materials

Tungsten powder is compounded with carbon fiber, ceramics (such as SiC,  $\text{Al}_2\text{O}_3$ ) or metals (such as Ni, Ti) to prepare high temperature resistant and corrosion resistant materials with an operating temperature of up to  $2000\text{-}2500^\circ\text{C}$ . It is suitable for rocket engine nozzles (heat flux  $>20 \text{ MW}/\text{m}^2$ ) and the inner wall of nuclear fusion reactors (neutron irradiation resistance  $>10^{22} \text{ n}/\text{cm}^2$ ). Thermodynamic analysis shows that the thermal conductivity of tungsten-SiC composite materials can reach  $150\text{-}180 \text{ W}/(\text{m}\cdot\text{K})$ , and the oxidation resistance is improved by 30-40% (oxidation weight gain rate  $<0.1 \text{ mg}/\text{cm}^2$ ,  $1000^\circ\text{C}$ ). In the future, it is necessary to solve the problems of interface reaction (such as  $\text{W}+\text{SiC} \rightarrow \text{WSi}_2$ ,  $\Delta G \approx -50 \text{ kJ}/\text{mol}$ ) and thermal expansion mismatch (CTE difference  $<5 \times 10^{-6} /\text{K}$ ). It is estimated that by 2035, such materials will account for 15-20% of tungsten powder applications.

### Smart and Adaptive Materials

By doping with rare earth elements (such as La 0.5-2 wt%, Ce 1-3 wt%) or transition metals (such as Mo 5-10 wt%), tungsten powder can be developed into materials with adaptive functions for use in dynamic stress environments (such as aviation turbine blades, cyclic stress  $>500 \text{ MPa}$ ). For example, the grain boundary strengthening effect of La-doped tungsten powder (dislocation density reduced by 20%) improves high-temperature creep resistance (strain rate  $<10^{-8} \text{ s}^{-1}$ ,  $1500^\circ\text{C}$ ). In the future, combining material genetic engineering (MGE) and high-throughput screening (number of experiments  $>10^4$ ) can accelerate formula optimization. In 2035, the market share of smart tungsten-based materials is expected to reach 10%, with an annual output value of approximately RMB 5 billion.

### 20.1.2 Interdisciplinary Breakthroughs in the Energy Sector

The application of tungsten powder in the field of clean energy and efficient energy storage will achieve a qualitative leap through interdisciplinary integration, involving physics, chemistry and engineering:

#### Solar and thermal energy conversion

The high melting point and excellent light absorption of tungsten powder (visible light absorption

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rate>90%, infrared reflectivity<10%) make it an ideal material for efficient solar selective absorption coatings. In the future, tungsten-based coatings can be prepared by plasma spraying (speed>500 m/s, thickness 5-10  $\mu\text{m}$ ), the working temperature can reach 1000-1200°C, and the light-to-heat conversion efficiency is increased to 40-45% (traditional coatings<30%). Thermodynamic calculations (enthalpy change  $\Delta H \approx 50$  kJ/mol) show that the stability of tungsten powder at high temperatures (volatilization loss<0.01 wt%) is better than that of traditional materials (such as Ni-Cr). By 2030, the global installed capacity of solar thermal power generation is expected to reach 500 GW, and the demand for tungsten powder will increase by 20% annually, about 3,000 tons.

### Battery and Energy Storage Technology

Nano-tungsten powder (particle size <20 nm, specific surface area >30  $\text{m}^2/\text{g}$ ) is used as an electrode additive for lithium-sulfur batteries or sodium-ion batteries (content 5-10 wt%). It inhibits the polysulfide shuttle effect through surface chemical adsorption (adsorption energy >2 eV), reduces the capacity decay rate to <0.1%/cycle, and the cycle life reaches 1000-1500 times. Electrochemical impedance spectroscopy (EIS) shows that tungsten powder reduces the charge transfer resistance by 30% ( $R_{ct}$  <10  $\Omega$ ). In the future, combined with DFT calculation (accuracy  $\pm 0.01$  eV) to optimize doping (such as W-Sn composite, Sn 2-5 wt%), the specific capacity (>800 mAh/g) can be further improved. In 2035, the demand for tungsten powder in the energy storage field is expected to reach 2000-3000 tons/year, with a market size of approximately 10 billion yuan.

### Nuclear energy and fusion technology

Tungsten powder is used as plasma facing material (PFM) in nuclear fusion (such as ITER, DEMO projects) and small modular reactors (SMRs), withstanding heat loads >10  $\text{MW}/\text{m}^2$  and neutron irradiation (>  $10^{22}$  n/cm<sup>2</sup>), and its life is extended by 50-70%. Its high density (19.25  $\text{g}/\text{cm}^3$ ) and low sputtering yield (<0.1 atom/ion, 14 MeV) are superior to traditional materials (such as Be). In the future, the problems of irradiation-induced embrittlement (DBTT rises to 500°C) and hydrogen isotope retention (> $10^{20}$  atom/cm<sup>3</sup>) need to be solved. It is expected that the proportion of nuclear energy applications will increase from 5% to 15-20% after 2030, and the annual demand will increase to 1,000 tons.

### 20.1.3 Frontier Exploration of Quantum Technology

The potential of tungsten powder in quantum technology stems from its high electron density ( $Z=74$ ) and electrical properties. It may become a key material for quantum devices in the future, involving quantum physics and microelectronics:

#### Quantum computing

Tungsten-based superconducting films ( $T_c \approx 15-20$  K, thickness 50-100 nm) are prepared by magnetron sputtering (power 200 W, deposition rate 0.1 nm/s) and can be used for quantum bit (qubit) manufacturing. Combined with low-temperature technology (<1 K, He-3 refrigeration), ultra-low noise signal transmission (coherence time>100  $\mu\text{s}$ ) is achieved. First-principles calculations (VASP software) show that the d-electron state of tungsten enhances the superconducting pairing energy ( $\Delta \approx 2-3$  meV). In 2035,

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the quantum computing market is expected to reach US\$50 billion, and the demand for tungsten powder will increase from the current 10 tons/year to 100-200 tons/year.

### Quantum Sensing

Doped tungsten powder (such as W-Mo alloy, Mo 5-10 wt%, resistivity  $<10 \mu\Omega\cdot\text{cm}$ ) is used to prepare high-sensitivity detectors to detect weak magnetic fields ( $<10^{-15} \text{ T}$ ) or gravitational wave signals (strain  $<10^{-21}$ ). Its high density and low thermal noise ( $<10^{-9} \text{ W/Hz}$ ) are superior to traditional materials (such as Nb). In the future, interdisciplinary research (such as quantum optics and solid-state physics) is needed to optimize the doping ratio. It is estimated that the annual output value of related applications will reach 2-3 billion yuan in 2030.

### Quantum communication

Tungsten powder can be used as a substrate for photon-electron converters (bandgap  $> 2 \text{ eV}$ ) to improve the efficiency of quantum entangled light sources ( $> 90\%$ ). Theoretical models (such as Dirac equation simulation) predict that the high electron density of tungsten enhances the stability of entangled states, which needs to be experimentally verified in the future (entanglement distance  $> 1000 \text{ km}$ ). In 2035, the demand for tungsten powder for quantum communication is expected to reach 50 tons/year.

#### 20.1.4 Theory and Practice of Interdisciplinary Integration

The interdisciplinary integration of tungsten powder will integrate materials science (microstructure design), energy engineering (system integration) and quantum physics (electronic state regulation), and accelerate the development of new applications through multi-scale simulations (such as DFT+molecular dynamics, calculation accuracy  $\pm 0.01 \text{ eV}$ ), high-throughput experiments ( $>10^4$  samples/month) and engineering optimization (yield $>95\%$ ). It is expected that from 2030 to 2040, the proportion of interdisciplinary projects will increase from 20% to 50-60%, promoting the transformation of tungsten powder from industrial raw materials to high-tech substrates, and the annual R&D investment may reach 5 billion yuan.

## 20.2 Tungsten Powder Production Technology Challenges and Solutions

### 20.2.1 Detailed Analysis of Technical Challenges

Tungsten powder production faces multiple technical bottlenecks, which limit its performance improvement, cost reduction and environmental protection goals:

#### Particle size control and uniformity

The current hydrogen reduction technology ( $\text{H}_2$  flow rate 50 sccm,  $800^\circ\text{C}$ ) produces tungsten powder with a wide particle size distribution ( $D_{90}/D_{10}>2$ ),  $<50 \text{ nm}$  particles account for  $<10\%$ , and are easy to agglomerate (specific surface area drops from  $30 \text{ m}^2 / \text{g}$  to  $20 \text{ m}^2 / \text{g}$ ). This results in 3D printing consistency  $<90\%$  and a 15% increase in coating porosity. Thermodynamic analysis (Gibbs free energy

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$\Delta G \approx -120$  kJ/mol) shows that the mobility of tungsten atoms at high temperatures ( $D \approx 10^{-10}$  m<sup>2</sup> / s ) is the main cause of agglomeration.

### Energy consumption and carbon emissions

Traditional processes have high energy consumption (5-10 kWh/kg for wet process, >15 kWh/kg for pyrometallurgy), with a carbon footprint of 2-10 kg CO<sub>2</sub> / kg, which is significantly lower than the carbon neutrality target (<0.5 kg CO<sub>2</sub>/kg by 2060 ). The energy structure relies on coal (accounting for 60%), with a thermal efficiency of <70%, and the cost of tail gas treatment has increased by 20%.

### Difficulty in removing impurities

High-purity tungsten powder (>99.999%) needs to remove impurities such as O (<10 ppm), C (<5 ppm), and Fe (<5 ppm). The wet method produces waste liquid (COD>1000 mg/L, containing Co and Ni), and the fire method volatilizes 5-10% of W (vapor pressure 10<sup>-4</sup> Pa, 2000°C), increasing the cost by 30-50%.

### Low recycling efficiency

The global recycling rate of tungsten waste is only 30-40% (theoretical >90%), complex components (such as cemented carbide containing 5-15 wt% Co) are difficult to separate, the biometallurgical rate is slow (10<sup>-3</sup> g/(L·h)), and the pyrometallurgical process has high energy consumption (>20 kWh/kg), which limits the potential of the circular economy.

## 20.2.2 System Design of Solution

In response to the above challenges, future technological development will provide multi-level solutions:

### Particle size control technology

#### Plasma reduction

High-energy plasma (50-100 kW, >5000°C) is used to instantly vaporize WO<sub>3</sub> (evaporation enthalpy  $\Delta H_{\text{vap}} \approx 500$  kJ/mol), condense to form <20 nm tungsten powder, and improve the uniformity to D90/D10<1.5. The equipment needs a high-temperature resistant lining (such as ZrO<sub>2</sub>, melting point>2700°C), and it is expected that 50% of production lines will adopt it by 2030.

#### Vapor Deposition (CVD)

Through the WOCl<sub>6</sub> + H<sub>2</sub> reaction (deposition rate 10<sup>-3</sup> - 10<sup>-2</sup> g /s, 400-600°C ), the particle size is precisely controlled ( $\pm 5$  nm) and the yield reaches 95%. In the future , the purity of the precursor needs to be optimized (>99.99%) to reduce the cost to 50,000 yuan/ton.

### Low carbon production technology

#### Renewable Hydrogen

Hydrogen production by water electrolysis (efficiency > 80%, cost reduced to 2 USD/kg H<sub>2</sub> ), combined with photovoltaic power generation (<0.2 RMB per kWh), energy consumption is reduced to 2-3 kWh/kg, and carbon emissions are <0.5 kg CO<sub>2</sub> / kg. By 2035, the proportion of green processes is expected to

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reach 60-70%.

### **Photocatalytic reduction**

Using TiO<sub>2</sub> catalyst (band gap 2.8 eV) and ultraviolet light (wavelength <400 nm), WO<sub>3</sub> reduction efficiency >50% and energy consumption <1 kWh/kg. Catalyst life (<1000 h) and scale bottlenecks need to be resolved, and it is expected that the application share will reach 20% in 2040.

### **Impurity purification technology**

#### **Ion Exchange**

Resin selectivity > 95%, Fe and Co removed to < 5 ppm, waste liquid reduced by 50% (COD < 500 mg/L). Acid-resistant resin (pH 1-14) needs to be developed, and the cost will be reduced to 30,000 yuan/ton by 2030.

#### **Vacuum distillation**

At 10<sup>-3</sup> Pa and 1800°C, O and C are volatilized (boiling point <1000°C), and the W retention rate is >98%. The equipment requires a high vacuum pump (pumping speed >1000 L/s). In 2035, the proportion of high-purity tungsten powder will reach 30%.

### **Recycling Optimization Technology**

#### **Biometallurgy**

Using acidophilic bacteria (such as Thiobacillus ferrooxidans) to oxidize Co and Ni (rate 10<sup>-2</sup> g/(L·h)), the recovery rate is increased to 70%. The strain needs to be optimized (tolerance to W concentration >50 g/L), and the application proportion will reach 20% in 2030.

#### **AI-assisted separation**

Based on machine vision (recognition rate >98%) and deep learning (training data >10<sup>6</sup> sheets), complex waste is separated and the recycling rate reaches 80%. By 2035, the proportion of recycled tungsten powder is expected to reach 50-60%.

### **20.2.3 Implementation Path and Academic Support**

In the future, it is necessary to establish a joint laboratory for industry, academia and research (with a pilot line of 100 tons per year) to verify the feasibility of new technologies. Academic research should focus on thermodynamic modeling (such as CALPHAD, with an accuracy of ±1 kJ/mol), kinetic analysis (reaction rate  $k > 10^{-1} \text{ s}^{-1}$ ) and equipment design (temperature resistance >3000°C). Government support (such as subsidies for 30% of R&D costs) and international cooperation (such as the China-Europe Green Technology Alliance) will accelerate the promotion, and it is expected that the coverage rate of new technologies will reach 50% by 2030.

## **20.3 The strategic significance of tungsten powder in sustainable development**

### **20.3.1 Deepening of resource protection and circular economy**

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The efficient production and recycling of tungsten powder is the core of sustainable resource utilization. The global tungsten reserves are about 3.5 million tons, with an annual mining volume of 80,000-100,000 tons. If there is no recycling, it may be exhausted by 2050-2060. Increasing the recovery rate to 80-90% can extend the life of the resource by 20-30 years, reduce the mining of 50,000-60,000 tons of raw ore each year, and reduce tailings accumulation ( $>10^8$  tons/year, containing As, Pb) and acidic wastewater ( $\text{pH}<4, >10^6 \text{ m}^3 / \text{year}$ ). The circular economy model (life cycle analysis, LCA) shows that each ton of recycled tungsten powder saves 30-40 MJ of energy, reduces  $\text{CO}_2$  emissions by 5-10 tons, and has an economic benefit of about 200,000 yuan.

### 20.3.2 Multi-dimensional Contribution of Low-Carbon Economy

The green production of tungsten powder directly supports the goal of carbon neutrality. In 2030, if 60-70% of tungsten powder adopts low-carbon technology (energy consumption  $<3 \text{ kWh/kg}$ ), annual  $\text{CO}_2$  emissions will be reduced by 200,000-300,000 tons, equivalent to the annual emissions of 100,000-150,000 households. Indirect contributions are reflected in new energy applications: tungsten powder reduces fossil fuel dependence in solar thermal power generation (efficiency  $>40\%$ ) and batteries (lifespan  $>1000$  times), with a global annual emission reduction potential of 1-2 Gt  $\text{CO}_2$  (accounting for 5% of total emissions). Thermodynamic analysis (enthalpy entropy balance) shows that the energy efficiency of low-carbon processes can reach 85-90%, which is better than traditional processes ( $<70\%$ ).

### 20.3.3 Strategic Reserves and Supply Chain Security

Tungsten powder is a key material for defense (missile counterweights,  $W>90 \text{ wt}\%$ ), aviation (turbine blades, temperature resistance  $>1500^\circ\text{C}$ ) and electronics (chip substrates, purity  $>99.99\%$ ), and its stable supply is related to national security. China currently accounts for 70% of the global supply, and geopolitical risks (such as export restrictions) threaten the European and American supply chains. In 2030, a recycling rate of 50% and diversified procurement (such as 2,000 tons per year from South Korea and 1,000 tons from Australia) can reduce dependence on China from 70% to 50%, enhancing supply chain resilience. The strategic reserve model (Monte Carlo simulation) predicts that the reserve volume needs to reach 10,000-20,000 tons to cope with a 5-10 year crisis.

### 20.3.4 Comprehensive Improvement of Social and Economic Benefits

The technological upgrade of tungsten powder will create high-skilled jobs (50,000 to 100,000 new jobs by 2035, such as AI engineers and green process experts), with an annual salary of about 200,000 to 500,000 yuan. At the same time, the export of high value-added products (such as nano tungsten powder, with a premium of 30-50%) will improve economic benefits. The global tungsten powder market is expected to reach 80-100 billion yuan in 2030, with China accounting for 50%. Social impacts also include educational improvement ( $>100$  new tungsten-related courses in colleges and universities) and technological spillover (such as battery technology benefiting civilian use).

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### 20.3.5 Quantitative Assessment of Strategic Significance

Based on LCA and economic models, the sustainable development value (resource conservation + emission reduction + economic benefits) of each ton of tungsten powder is about 300,000 to 500,000 yuan, and the global annual value will reach 200-300 billion yuan in 2035. Its strategic significance is not limited to the economy, but also reflected in the realization of technological sovereignty and global climate goals.

## 20.4 Non-technical and non-market factors affecting tungsten powder and their possible future trends

### 20.4.1 Deep Impact of Policies and Regulations

#### Current status and impact

Environmental regulations (such as EU REACH, which requires heavy metal emissions to be less than 10 mg/L) force tungsten powder production to adopt low-pollution processes (cost increases by 20-30%), and China's export quota (reduced by 20% in 2025) pushes up global prices by 10-15%. Carbon tax policies (such as EU CBAM, implemented from 2026) impose additional fees on high-carbon processes (50-100 euros per ton), affecting competitiveness.

#### Trend Forecast

In 2030, the world may unify carbon emission standards (<1 kg CO<sub>2</sub> / kg), force green technology to account for >50%, and impose fines of 10% of output value on non-compliant enterprises. Trade protectionism will intensify, and the export tax rate of tungsten powder may rise to 20-25%, promoting localized production (self-sufficiency in Europe and the United States will rise from 20% to 40%). In 2040, policies may shift to full life cycle supervision (from mining to recycling), requiring carbon footprint disclosure, affecting market access.

#### Academic Support

The policy impact model (game theory analysis) predicts that strict regulations will eliminate 10-15% of small and medium-sized high-polluting enterprises and increase the profit margins of large enterprises by 5-10%.

### 20.4.2 The complex role of geopolitics

#### Current status and impact

The Sino-US trade friction (tariffs > 25%) and the Russia-Ukraine conflict (Russia supply reduced by 15%) disrupt the tungsten supply chain, and prices will fluctuate by 10-20% in 2025. China's dominant position (60% of reserves, 70% of production) exposes Europe and the United States to supply risks, and the strategic reserve consumption rate increases by 5% per year.

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### Trend Forecast

In 2030, Europe and the United States will increase tungsten development in their own countries and their allies (such as Canada and South Korea), and the self-sufficiency rate will rise to 30-40%, and the impact of geopolitical risks on prices will drop to 5-10%. In 2040, if the world forms a "tungsten resource alliance" (similar to OPEC), the supply will be stable and the price fluctuation will be <5%, but short-term conflicts (such as tensions in the South China Sea) may still push up prices by 15%. Emerging production areas (such as Congo in Africa, with reserves > 500,000 tons) may rise, accounting for 10%. Academic support: Geo-risk assessment (Monte Carlo simulation,  $10^4$  iterations) shows that diversified supply can reduce the risk of price volatility by 50%.

### 20.4.3 The profound impact of social cognition and ethics

#### Current status and impact

The public is increasingly concerned about the environmental damage caused by tungsten mining (such as water pollution, COD>5000 mg/L), and is boycotting non-sustainable products (boycott rate>20% in 2025). Ethical issues (such as child labor in Congo) affect brand reputation, and consumers prefer green certified products (accounting for 30%).

#### Trend Forecast

In 2030, the demand for green certified tungsten powder will account for >50%, and companies will need to disclose carbon footprint and supply chain transparency (disclosure rate >90%), otherwise the market share will drop by 20-30%. In 2040, social pressure may promote the "zero pollution" standard (emission <1 mg/L), eliminate traditional processes, and the premium of green products will reach 50%. Improved public education (environmental protection course coverage rate >80%) will strengthen this trend.

#### Academic Support

The social cognition model (questionnaire, sample size  $>10^4$ ) predicts that for every 10% increase in ethical concern, green product sales will increase by 15%.

### 20.4.4 Potential Impact of Technological Externalities

#### Current status and impact

Alternative materials (such as molybdenum powder, which costs 30% less and has a melting point of 2623°C) divert the demand for tungsten powder (<10%), and graphene (conductivity  $>10^6$  S/m) competes with tungsten powder applications in the new energy field (accounting for 5%).

#### Trend Forecast

In 2030, if tungsten powder maintains its performance advantage (such as temperature resistance > 1000°C), the substitution threat will be <15%; but in 2040, graphene or two-dimensional materials (such as MXene) may seize 20-30% of the energy storage market, and tungsten powder needs to respond

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through nano-sizing and functionalization (specific capacity > 1000 mAh/g). Cross-industry technologies (such as AI chips reducing the demand for tungsten substrates) may indirectly affect 5-10% of the market.

### Academic Support

Technology substitution analysis (technology maturity level TRL assessment) shows that tungsten powder needs to maintain TRL 9 (mature application) to avoid being surpassed by alternatives with TRL 7-8.

## 20.4.5 Comprehensive Outlook and Forecast Model

### Short term (2025-2030)

Policies and geopolitics lead the adjustment of supply chains, price fluctuations are 10-15%, and the proportion of green technology rises to 50%. Social cognition drives the recycling rate from 40% to 60%, and the impact of technical externalities is less than 10%.

### Medium term (2030-2040)

Regulations are becoming stricter (carbon emissions <0.5 kg CO<sub>2</sub> / kg), geopolitical risks are weakening (self-sufficiency rate >40%), social pressure promotes green products to account for >80%, and the threat of substitution rises to 20%. The comprehensive model (system dynamics, SD) predicts that the tungsten powder market size will stabilize at 100-120 billion yuan.

### Long term (2040-2050)

Policy globalization (zero pollution standard), geopolitical reshaping (new production areas account for >20%), social cognition drives the greening of the entire industry chain, and technological externalities may divert 30% of demand. Tungsten powder needs to maintain its competitiveness through interdisciplinary innovation.

The future prospects of tungsten powder research are reflected in the in-depth expansion of interdisciplinary applications, comprehensive breakthroughs in production technology, multi-dimensional strategic value of sustainable development, and the complex influence of non-technical factors. From 2030 to 2050, tungsten powder will play a core role in materials, energy, and quantum technology, overcoming challenges in particle size, energy consumption, and recycling through technical solutions to achieve resource recycling and low-carbon goals. Policies, geography, society, and external technologies will collaboratively shape its development path. The industry needs to integrate academic research (theory + experiment), industrial practice, and policy support to meet dynamic challenges and seize development opportunities.

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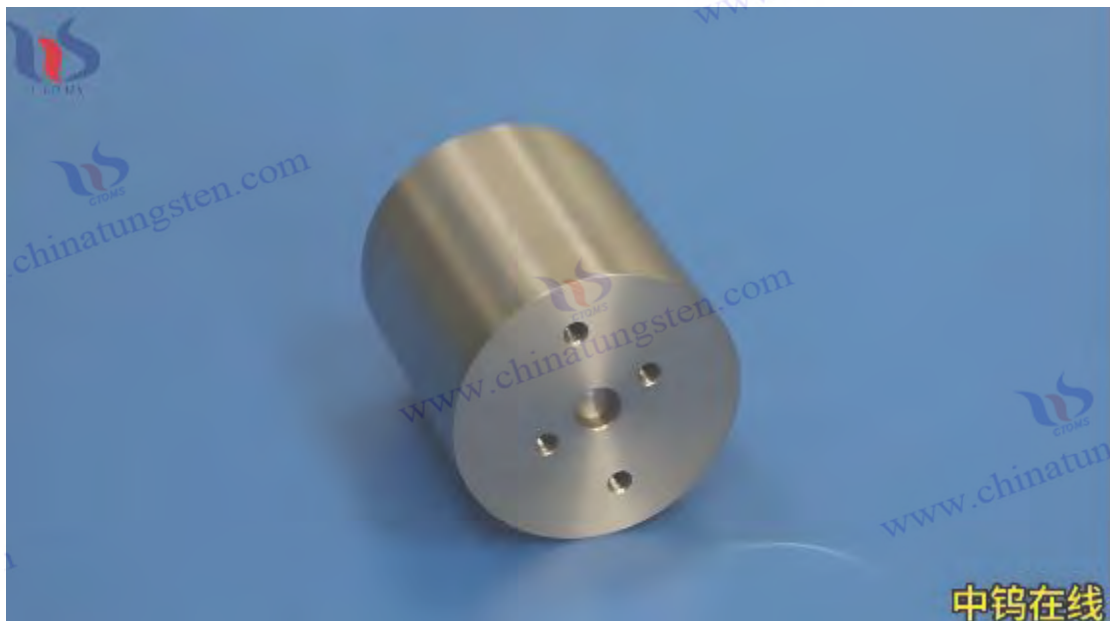
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## 100 Interesting Facts and Facts About Tungsten Powder

### 1. Basic physical and chemical properties

Melting Point Characteristics of Tungsten Powder

3422 °C (pure tungsten powder sintered body), the highest among metal powders.

Density parameters of tungsten powder

Theoretical density is 19.25 g/cm<sup>3</sup>, and the measured value is affected by the porosity (4-18 g/cm<sup>3</sup>).

Thermal conductivity of tungsten powder

173 W/m·K (dense sintered body), higher than titanium alloy (7 W/m·K).

Resistivity range of tungsten powder

$5.6 \times 10^{-8} \Omega \cdot m$  (20 °C), and increases with the increase of oxygen content.

Thermal Expansion Coefficient of Tungsten Powder

$4.5 \times 10^{-6} /K$  (25-1000 °C), matching silicon chips.

Mohs hardness of tungsten powder

7.5 (pressed green state), close to corundum (9).

Tungsten powder particle size classification standard

Coarse powder (> 10 μm), fine powder (1-10 μm), ultrafine powder (< 1 μm).

Specific surface area index of tungsten powder

Ultrafine tungsten powder (100 nm) reaches 5-50 m<sup>2</sup>/g, significantly improving catalytic activity.

Tungsten powder fluidity test method

Hall flow meter (ASTM B213), spherical powder ≤ 25 s/50g.

Differences in bulk density of tungsten powder

Non-spherical powder is 4-6 g/cm<sup>3</sup>, spherical powder is 8-10 g/cm<sup>3</sup>.

### 2. Chemical properties and reactions

Surface oxidation behavior of tungsten powder

A 1-3 nm WO<sub>3</sub> layer is generated at room temperature and quickly oxidized into powder above 500 °C.

Acid Corrosion Resistance of Tungsten Powder

in 98% H<sub>2</sub>SO<sub>4</sub> is less than 0.01 mm/year.

Alkali Corrosion Resistance of Tungsten Powder

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with 30% NaOH solution at room temperature , but generates tungstate in molten alkali.

#### Halogen Reaction of Tungsten Powder

It reacts violently with F<sub>2</sub> at room temperature to produce gaseous WF<sub>6</sub> .

#### Carburization tendency of tungsten powder

1400 °C , it reacts with carbon to form tungsten carbide (WC) with a hardness of HV 2200.

#### Hydrogen reduction reaction of tungsten powder

WO<sub>3</sub> + 3H<sub>2</sub> → W + 3H<sub>2</sub>O (700-1100 °C ).

#### Nitriding modification of tungsten powder

Plasma treatment produces a WN layer with a surface hardness of HV 2200.

#### Spontaneous combustion characteristics of nano-tungsten powder

When the particle size is less than 50 nm, it will spontaneously ignite in the air and require argon protection.

#### Toxicity Level of Tungsten Powder

Inhalation of dust can cause pneumoconiosis, and the OSHA limit is 5 mg/ m<sup>3</sup> .

#### Oxygen impurity control technology of tungsten powder

Vacuum annealing makes the oxygen content less than 0.005%.

### 3. Production technology and equipment

#### Raw ore types of tungsten powder

Wolframite ((Fe,Mn)WO<sub>4</sub>) and scheelite (CaWO<sub>4</sub>).

#### Tungsten powder recovery rate

Gravity separation → magnetic separation → flotation combined process, with a recovery rate of 85%-90%.

#### APT Preparation Process of Tungsten Powder

Ammonium paratungstate (APT) is extracted by alkali fusion method with a purity of 99.95%.

#### Hydrogen Reduction Process of Tungsten Powder

Two-stage reduction (600 °C → 900 °C ), H<sub>2</sub> purity ≥99.999%.

#### The trend of carbon reduction elimination of tungsten powder

Due to high CO emissions and energy consumption, it is gradually being replaced by hydrogen reduction.

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#### CVD Preparation Technology of Tungsten Powder

Chemical vapor deposition produces ultrafine powders with a particle size accuracy of  $\pm 5$  nm.

#### Plasma Atomization Process of Tungsten Powder

Argon atomization, temperature  $> 20000$  °C , sphericity  $> 98\%$ .

#### Defects of high energy ball milling of tungsten powder

Fe impurities ( $< 0.1\%$ ) are introduced and acid washing and purification are required.

#### Limitations of Electrolytic Preparation of Tungsten Powder

High cost (\$1000/kg), only used for semiconductor-grade high-purity powder.

#### Tungsten powder particle size classification technology

Air flow sieving (1-100  $\mu\text{m}$ ) and centrifugal classification ( $< 1$   $\mu\text{m}$ ).

### 4. Detection and analysis technology

#### Method for detecting oxygen content of tungsten powder

Inert gas fusion method, accuracy 0.001 wt%.

#### Particle size distribution test of tungsten powder

Laser diffraction method (range 0.1-1000  $\mu\text{m}$ ).

#### SEM morphology analysis of tungsten powder

Observe the sphericity, surface defects and agglomeration.

#### BET specific surface area test of tungsten powder

$\text{N}_2$  adsorption method, ultrafine powder  $> 10$   $\text{m}^2 / \text{g}$  .

#### Density determination standard of tungsten powder

Archimedes method (medium is anhydrous ethanol).

#### XRD phase analysis of tungsten powder

Detect impurity phases such as  $\text{WO}_3$  (content  $< 0.1\%$ ).

#### ICP-MS Trace Element Detection of Tungsten Powder

Alkali metals such as Na and K are controlled to ppb level.

#### XPS Surface Analysis of Tungsten Powder

The oxide layer thickness is 1-3 nm ( $\text{WO}_3$  ).

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Mercury Intrusion Porosity Testing of Tungsten Powder

Pore size distribution: 0.003-360  $\mu\text{m}$ .

Hall flow meter test of tungsten powder

Flowability classification (ASTM B213).

### 5. Surface modification technology

Silane coupling agent treatment of tungsten powder

Improve the bonding strength with polymer matrix, shear strength +50%.

Plasma Activation of Tungsten Powder

The surface energy increases to 60 mN/m and wettability improves.

Chemical Nickel Plating Process of Tungsten Powder

The coating thickness is 1-5  $\mu\text{m}$ , and the resistivity is reduced by 30%.

Paraffin coating of tungsten powder to prevent oxidation

Weight gain of 0.5-2% and storage period extended to 2 years.

Magnetron sputtering nitridation of tungsten powder

A WN layer (hardness HV 2200) is produced.

### VI. Storage and handling regulations

Anti-oxidation storage conditions of tungsten powder

Argon sealed ( $\text{O}_2 < 10$  ppm) or vacuum packed.

Explosion-proof measures for nano-tungsten powder

Anti-static container + argon gas filling (pressure 0.1 MPa).

Workshop Dust Control of Tungsten Powder

Wet collection or local negative pressure suction (wind speed  $\geq 0.5$  m/s).

International transportation classification of tungsten powder

UN3178 (Class 4.1 flammable solid).

Acid Dissolution Recovery Process for Waste Tungsten Powder

$\text{HNO}_3 + \text{H}_2\text{O}_2$  dissolved, recovery rate  $> 98\%$ .

### 7. Special Form Tungsten Powder

Preparation of Spherical Tungsten Powder by Plasma Atomization

Fluidity  $\leq 25$  s/50g, hollow ball rate  $< 0.1\%$ .

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Pore Forming Technology of Porous Tungsten Powder

The template method achieves a porosity of 30-70% and a specific surface area of 5-50 m<sup>2</sup> / g.

Laser Vapor Synthesis of Nano-tungsten Powder

Particle size <50 nm, specific surface area >20 m<sup>2</sup> / g.

Preparation of tungsten flake powder by high energy ball milling

Diameter-to-thickness ratio>20:1, used for conductive coatings.

Coating process of core-shell structure tungsten powder

Tungsten@carbon core-shell (carbon layer 5-20 nm), the oxidation resistance temperature is increased to 800 °C .

## 8. Performance Control Mechanism

Effect of tungsten powder size on sintering temperature

The sintering temperature of fine powder (<1 μm) is reduced by 200-300 °C .

Relationship between sphericity and fluidity of tungsten powder

When the sphericity is greater than 95%, the fluidity increases by 3 times.

Effect of Oxygen Content of Tungsten Powder on Electrical Conductivity

When oxygen is less than 0.01%, the resistivity drops to 5×10<sup>-8</sup> Ω·m.

Correlation between porosity and strength of tungsten powder

When the porosity is 10%, the compressive strength decreases by 40%.

Effect of tungsten powder surface roughness on wettability

When Ra is less than 0.1 μm, the contact angle decreases by 30°.

## 9. Application core parameters

Requirements for tungsten powder for 3D printing

Sphericity>95%, oxygen<0.02%, particle size 15-45 μm.

Tungsten powder for catalyst carrier

Specific surface area>20 m<sup>2</sup> / g, pore size 2-50 nm.

Thermal conductivity of tungsten powder for electronic packaging

>150 W/m·K, CTE 4.5-6×10<sup>-6</sup> /K.

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Tungsten powder density for shielding materials  
Filling density > 17 g/cm<sup>3</sup> ( 1.5 times that of lead).

Purity of tungsten powder for sputtering target  
>99.995%, average particle size 3-10 μm.

## 10. Market and Standards System

International standard for tungsten powder: ASTM B777  
Classification F-00 to F-06 (particle size and use).

China tungsten powder grade system  
FW-1 (normal powder), FW-0.5 (ultrafine powder).

Tungsten powder price gradient  
Ordinary powder 300-350 /kg, nano tungsten powder 330-500 / kg , high purity nano powder 500-1200/kg. Spherical tungsten powder 1300~

Environmental certification requirements for tungsten powder  
RoHS (Pb < 0.1%) and REACH compliant.

Export Control Policy of Tungsten Powder  
China implements export license management for nanometers.

## 11. Technical Challenges and Solutions

Hard Agglomeration Problem of Nano-Tungsten Powder  
Surface modifiers (such as PEG) reduce the aggregation rate by >50%.

Difficulties in controlling oxygen impurities in ultrafine tungsten powder  
Vacuum reduction + passivation treatment, oxygen <0.005%.

The high cost bottleneck of spherical tungsten powder  
The energy consumption of plasma atomization is \$80-120/kg, and cost reduction is needed on a large scale.

Inefficient particle size classification of fine powders  
Ultrasonic-assisted screening increases efficiency to 85%.

Performance degradation of recycled tungsten powder  
Acid washing + hydrogen reduction restores the sintering activity to 90%.

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## 12. Application of cutting-edge technology

Catalytic Application of Nano-Tungsten Powder

10 nm powder catalyzes H<sub>2</sub>S conversion rate +40% (petroleum desulfurization).

4D printing technology of tungsten powder

Temperature sensitive tungsten-polymer composite powder, deformation accuracy 0.1%/°C.

Preparation of Tungsten Sulfur Quantum Dots

Luminous efficiency 85% (wavelength 450 nm), used for QLED display.

Experiment on manufacturing tungsten powder in space

Defect-free powder prepared in microgravity environment, strength +20%.

Medical Application of Degradable Tungsten Powder

90% (stent material) degrades in the body within 3 years.

Application of tungsten powder in nuclear fusion reactor

The first wall armor is resistant to plasma erosion (>10 MW/m<sup>2</sup>).

Development of hydrogen energy catalyst based on tungsten powder

Improve fuel cell efficiency to 65%.

Tungsten powder reinforced carbon fiber performance

Tensile strength 5 GPa (aerospace structural parts).

Intelligent Response Tungsten Powder Composite Material

Light/heat triggered deformation (military stealth material).

Tungsten powder's quantum computing potential

As substrate material for superconducting quantum bits.

## 13. Environment and Regulations

Wastewater standards for tungsten powder production

China GB 8978 requires that the tungsten content be ≤0.1 mg/L.

Carbon reduction in hydrogen reduction processes

CO<sub>2</sub> emissions by 50% compared to carbon reduction.

Occupational disease prevention in tungsten powder workshop

Mandatory wearing of N95 masks + regular lung function tests.

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#### EU REACH Regulation Requirements

Submit a full life cycle assessment report on tungsten powder.

#### Conflict Minerals Traceability Management

Tungsten ore in the Democratic Republic of the Congo must be certified by the OECD.

#### Harmless treatment of tungsten slag

Vitrification technology, heavy metal leaching rate <1%.

#### Calculation of carbon footprint of tungsten powder

CO<sub>2</sub> is emitted per kilogram of production (hydrogen reduction method).

#### Benefits of a closed-loop recycling system

The consumption of primary ore is reduced by 30% and the energy consumption is reduced by 40%.

#### Study on Ecotoxicity of Nano-Tungsten Powder

Fish LC50 > 100 mg/L (low toxicity).

#### Insurance Requirements for Tungsten Powder Transportation

Flammable solids must be insured separately (rate 0.3%-0.5%).

### 14. History and Future Trends

#### Industrial Origin of Tungsten Powder

1909 General Electric first mass-produced it (for filaments).

#### Militarization of Tungsten Powder during World War II

80% of the output is used for the manufacture of armor-piercing bullets.

#### The rise of China's tungsten powder industry

Import dependence in the 1950s → 85% of the world's total in 2023.

#### The Promise of Tungsten Powder in Space Mining

The cost of developing tungsten resources on asteroids may be lower than that on Earth.

#### Tungsten powder market forecast

- Global market size to reach \$XX billion by 2030 (CAGR 6.5%).

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**Appendix :**

**International Standards and Specifications of Tungsten Powder  
(ASTM, ISO, GB/T, Germany, Japan, South Korea, Russia, etc.)**

As a key industrial raw material, the quality, performance and application of tungsten powder are strictly regulated by international and national standards. These standards cover the chemical composition, particle size distribution, physical properties, production process and test methods of tungsten powder to ensure its consistency and reliability in the global supply chain. This appendix systematically sorts out the relevant standards of tungsten powder in major countries and regions, including the United States (ASTM), the International Organization for Standardization (ISO), China (GB/T), Germany (DIN), Japan (JIS), South Korea (KS), Russia (GOST), etc., aiming to provide reference for researchers, engineers and industry practitioners. The following content is based on the latest standards and trends as of April 4, 2025.

**1 American Standard (ASTM International)**

ASTM International is a globally recognized material standards setting organization. Its tungsten powder related standards are mainly developed by Committee B10 (Reactive and Refractory Metals and Their Alloys), which are widely used in aerospace, electronics and powder metallurgy. The following are the main standards:

ASTM B329-20 (2020 revised edition): *Standard test method for apparent density of refractory metal powders*

Describes the method for determining the apparent density of tungsten powder using a Hall flow meter, applicable to powders with a particle size of 1-150  $\mu\text{m}$ , with an accuracy of  $\pm 0.01 \text{ g/cm}^3$ . It may be updated to B329-25 in 2025 to include test specifications for nano tungsten powders (<50 nm).

ASTM B761-17 (2017 revised edition): *Standard Specification for Tungsten and Tungsten Alloy Powder Metallurgy Products*

The purity of tungsten powder (>99.9%), impurity limits (O<0.05 wt%, C<0.01 wt%) and particle size distribution (D50<10  $\mu\text{m}$ ) are specified. It is expected that the 2025 revision (B761-25) will increase the sustainability requirements for recycled tungsten powder.

ASTM E159-22 (2022 Revised): *Standard Test Methods for Chemical Analysis of Metal Powders*

Provide spectral analysis methods for trace elements (such as Fe, Mo) in tungsten powder, with a detection limit of <10 ppm. In 2025, it may be expanded to ICP-MS (inductively coupled plasma mass spectrometry) technology.

ASTM F288-19 (2019 revised edition): *Specifications for tungsten powder and tungsten products*

For the application of high-purity tungsten powder (>99.99%) in electronic and medical devices, the particle size is required to be <5  $\mu\text{m}$  and the fluidity (>20 s/50g). Future revisions may focus on tungsten

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powder for 3D printing.

Trend: ASTM standards emphasize the accuracy and application orientation of test methods, and may further integrate AI-assisted analysis and green production requirements after 2025.

## 2 International Organization for Standardization (ISO)

ISO standards are developed by Technical Committee TC 119 (Powder Metallurgy) to harmonize global tungsten powder specifications and facilitate international trade and technical exchange. The following are the key standards:

ISO 4491-1:2023: *Metallic powders -- Determination of apparent density -- Part 1: Funnel method*

Similar to ASTM B329, it is applicable to the apparent density test of tungsten powder ( $2-10 \text{ g/cm}^3$ ). It may be revised to ISO 4491-1:2025 in 2025 to add nano powder test conditions.

ISO 3252:2019: *Powder metallurgy – Terminology*

Defines terms related to tungsten powder (such as particle size distribution, specific surface area) to provide a basis for other standards. It may be updated in 2025 to include tungsten powder terminology for quantum technology.

ISO 4884:2024: *Sampling and test methods for cemented carbide powders*

Including chemical composition ( $W > 90 \text{ wt}\%$ ) and physical property tests of tungsten powder and tungsten-based alloys. It is expected that the 2025 revision will add carbon emission assessment.

ISO 22068:2022: *Specifications for sintered materials for powder metallurgy*

Indirectly regulate the quality of tungsten powder (such as purity  $> 99.95\%$ ), and in the future may be expanded to the recycling standards of recycled tungsten powder.

Trend: ISO standards focus on universality and internationalization. In the future, they may be further aligned with ASTM and GB/T to strengthen sustainability requirements.

## 3 Chinese National Standard (GB/T)

China is the world's largest producer of tungsten powder. Its GB/T standards are formulated by the National Technical Committee for Standardization of Nonferrous Metals (SAC/TC 243) and are divided into mandatory (GB) and recommended (GB/T). The following are the main standards:

GB/T 3458-2020: Tungsten powder

The standard specifies the purity ( $99.9\%-99.999\%$ ), particle size ( $0.1-100 \mu\text{m}$ ) and impurity limits ( $O < 0.1 \text{ wt}\%$ ,  $Fe < 0.05 \text{ wt}\%$ ) of tungsten powder. It may be revised to GB/T 3458-2025 in 2025, adding the specification of ultrafine tungsten powder ( $< 50 \text{ nm}$ ).

GB/T 4197-2019: Method for determination of particle size of tungsten powder and tungsten carbide powder

Laser diffraction method is used to determine the particle size distribution (accuracy  $\pm 0.1 \mu\text{m}$ ), which is suitable for powder metallurgy and cemented carbide. BET surface area measurement may be integrated

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in the future.

GB/T 26040-2023: Chemical analysis method for tungsten powder

Provides determination methods for W, O, C and other elements in tungsten powder (detection limit <5 ppm), which may be updated to GB/T 26040-2025 in 2025 and include green production indicators.

GB/T 42915-2022: Technical conditions for recycling tungsten powder

The purity (>99.8%) and performance of scrap tungsten recovery powder are standardized, and it is expected that the 2025 revision will increase the recovery rate requirements (>80%).

Trend: GB/T standards are closely integrated with China's industrial advantages. In the future, they will strengthen environmental protection and circular economy requirements and align with international standards.

### German Standard (DIN)

German standards are formulated by the German Institute for Standardization (DIN), focusing on high precision and engineering applications, and are particularly influential in the European market. The following are relevant standards:

DIN 51001:2021: *Chemical analysis methods for metal powders*

Provide XRF (X-ray fluorescence) and ICP analysis methods for impurities (such as Fe, Ni) in tungsten powder, with a detection limit of <10 ppm. It may be updated in 2025 to include analysis of nano tungsten powder.

DIN EN 23908:2020: *Determination of particle size of hard metal powders*

Compatible with ISO 4491, applicable to tungsten powder particle size testing (1-50 μm). Future revisions may add specifications for 3D printing powders.

DIN 30910-3:2023: *Specifications for tungsten-based sintered materials - Part 3: Powder requirements*

The purity (>99.95%) and fluidity (>25 s/50g) of tungsten powder are specified, and may be expanded to aerospace applications in 2025.

Trend: DIN standards emphasize technical details and European market compatibility and may be deeply integrated with ISO in the future.

### 5 Japanese Standard (JIS)

Japanese Industrial Standards (JIS) are formulated by the Japan Industrial Standards Commission (JISC) and are widely used in the electronics and automotive industries. The following are the main standards:

JIS H 5762:2022: *Specifications for tungsten powder and tungsten alloy powder*

The purity (>99.9%), particle size (0.5-50 μm) and impurities (O<0.05 wt%) of tungsten powder are specified. It may be revised to JIS H 5762-2025 in 2025, adding high purity requirements (>99.999%).

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JIS Z 8801-1:2020: *Powder particle size determination method - Part 1: Laser diffraction method*  
Suitable for tungsten powder particle size distribution testing (accuracy  $\pm 0.05 \mu\text{m}$ ), and may integrate nanopowder testing in the future.

JIS H 6201:2023: *Chemical analysis method for tungsten powder*  
Provides determination methods for elements such as W, Mo, and Fe (detection limit  $< 10 \text{ ppm}$ ), which may be updated to high-throughput analysis in 2025.

Trend: JIS standards focus on high purity and electronic applications, and may strengthen quantum technology-related regulations in the future.

## 6 Korean Standard (KS)

The Korean Standard (KS) is formulated by the Korea Agency for Technology and Standards (KATS) and has been improved in recent years with the rise of the tungsten industry. The following are the relevant standards:

KS D 9502:2021: *Technical requirements for tungsten powder*

The purity ( $> 99.9\%$ ), particle size ( $1-100 \mu\text{m}$ ) and impurities ( $O < 0.1 \text{ wt}\%$ ) of tungsten powder are specified. It may be revised to KS D 9502-2025 in 2025, adding requirements for recycling tungsten powder.

KS D 2570:2020: *Method for determination of particle size of metal powders*

The laser scattering method is used to test the particle size of tungsten powder (accuracy  $\pm 0.1 \mu\text{m}$ ), which may be compared with ISO 4491 in the future.

KS D 9510:2023: *Specification for tungsten-based alloy powder*

Applicable to powder metallurgy and cemented carbide, and may be expanded to new energy applications in 2025.

Trend: KS standards are influenced by China and Japan. In the future, they will improve their internationalization level and focus on green production.

## 7 Russian Standard (GOST)

Russian State Standards (GOST) are managed by the Russian Federal Service for Technical Regulation and Metrology and are applicable to CIS countries. The following are the main standards:

GOST 14316-91 (1991 edition, revised in 2023): *Tungsten powder*

The purity ( $> 99.9\%$ ), particle size ( $1-50 \mu\text{m}$ ) and impurities ( $O < 0.05 \text{ wt}\%$ ) of tungsten powder are specified. It may be updated to GOST 14316-2025 in 2025 to include the specifications for nano tungsten powder.

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GOST 25501-82 (1982 edition, revised in 2022): *Chemical analysis methods for metal powders*  
Provides a method for determining W, Fe, and C in tungsten powder (detection limit < 20 ppm), and modern analytical techniques may be used in the future.

GOST 28377-89 (1989 edition, revised in 2024): *Specifications for powders for powder metallurgy*  
Indirectly regulate the quality of tungsten powder, and aviation application requirements may be increased in 2025.

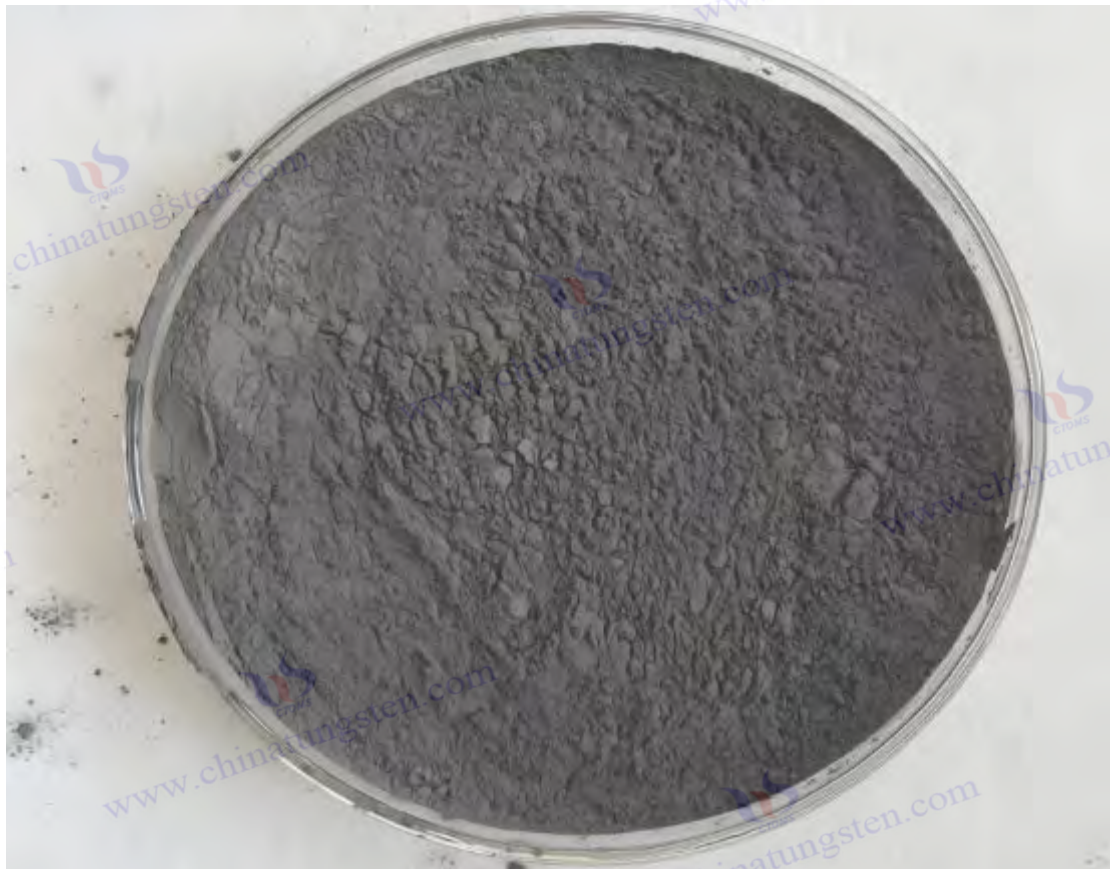
Trend: GOST standards are updated slowly, and in the future they may learn from ISO and ASTM to improve their modernization level.

## 8 Comparison and Trends among Standards

Consistency: ASTM, ISO and GB/T are highly consistent in particle size testing and chemical analysis methods (such as laser diffraction, ICP-MS), but the specific limits vary depending on the application.

Differences: ASTM and JIS focus more on high purity and cutting-edge applications (such as electronics, quantum technology), GB/T and KS emphasize output and cost control, DIN focuses on engineering precision, and GOST tends to traditional industries.

Future trends: From 2025 to 2030, national standards will tend to be green (low-carbon production, recycling), intelligent (AI-assisted testing) and nano (<50 nm powder specification), and ISO may become the core of coordination.



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## ASTM B761-17 (2017 revised edition)

### Standard specification for tungsten and tungsten alloy powder metallurgy products

#### 1. Scope

ASTM B761-17 specifies the technical requirements for tungsten and tungsten alloy products produced by powder metallurgy, including the chemical composition, physical properties, dimensional tolerances and test methods of pure tungsten (W) and tungsten-based alloys (such as W-Mo, W-Re). This standard applies to sintered or hot-processed products in bars, plates, wires and other shapes, which are widely used in high-temperature environments (such as aviation turbine blades, missile counterweights) and electronic devices (such as cathode emitters). The standard does not cover the initial preparation of tungsten powder, but puts forward clear requirements for the final performance of powder metallurgy products.

#### 2. Referenced Documents

This standard references a number of ASTM test methods and specifications to ensure consistency and traceability. Mainly including:

ASTM B329: Standard Test Method for Apparent Density of Refractory Metal Powders.

ASTM E8/E8M: Tensile test methods for metallic materials.

ASTM E29: Specification for significant figures of inspection and test data.

ASTM E159: Standard Test Methods for Chemical Analysis of Metal Powders.

ASTM E407: Microscopic examination of metals and alloys.

These references provide a technical basis for the performance testing of tungsten and tungsten alloy products.

#### 3. Terminology

Key terms are defined in the standard to ensure uniform terminology:

Powder Metallurgy Products: Solid materials made by pressing and sintering tungsten powder or tungsten alloy powder.

Sintered density: The actual density of the material after sintering ( $\text{g/cm}^3$ ), usually close to the theoretical density ( $19.25 \text{ g/cm}^3$  for W).

Tungsten alloy: A material containing tungsten ( $\text{W} > 50 \text{ wt}\%$ ) combined with other elements (such as Mo, Re, Ni).

#### 4. Materials and Manufacture

##### 4.1 Raw material requirements

Tungsten powder or tungsten alloy powder should meet high purity requirements ( $\text{W} > 99.9\%$  for pure W) and be prepared by hydrogen reduction, plasma reduction or other methods.

The particle size range is usually  $0.5\text{-}50 \mu\text{m}$ , which is negotiated by the supply and demand parties, but must meet the uniformity requirements ( $\text{D}_{90}/\text{D}_{10} < 2.5$ ).

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## 4.2 Manufacturing process

Pressing: Use cold isostatic pressing (CIP, pressure > 200 MPa) or compression molding, and the pressed density is > 60% of the theoretical density.

Sintering: carried out in hydrogen or vacuum atmosphere (temperature 1800-2500°C), sintering time 2-10 hours, ensuring porosity < 5%.

Hot working: optional forging, rolling or drawing to further improve density (> 95% theoretical density) and mechanical properties.

## 5. Chemical Composition

The standard specifies the chemical composition requirements for tungsten and tungsten alloys, as follows:

Pure tungsten (Type I): W: ≥ 99.9 wt%

Impurity Limit (Maximum):

O: 0.05 wt%, C: 0.01 wt%, Fe: 0.01 wt%, Mo: 0.02 wt%, N: 0.005 wt%

Other single elements: < 0.01 wt%

Tungsten-Molybdenum Alloy (Type II): W: 70-95 wt%, Mo: 5-30 wt%

The impurity limit is the same as pure tungsten.

Tungsten-rhenium alloy (Type III): W: 75-97 wt%, Re: 3-25 wt%

The impurity limit is the same as pure tungsten.

Test Method: ASTM E159 or equivalent spectroscopic analysis (ICP-OES or GDMS), detection limit < 10 ppm.

## 6. Physical and Mechanical Properties

### 6.1 Density

Sintered density: ≥ 18.5 g/cm<sup>3</sup> (pure tungsten > 96% theoretical density).

Hot processed density: ≥ 19.0 g/cm<sup>3</sup> (> 98.5% theoretical density).

Test method: ASTM B328 (water immersion method, accuracy ± 0.01 g/cm<sup>3</sup>).

### 6.2 Hardness

Sintered: ≥ 250 HV (Vickers hardness, load 10 kg).

Hot processed: ≥ 300 HV.

Test method: ASTM E92.

### 6.3 Tensile properties (optional for hot processed state)

Tensile strength: ≥ 600 MPa (room temperature).

Elongation: ≥ 5%.

Test method: ASTM E8/E8M (specimen diameter 6-12 mm).

### 6.4 Microstructure

Grain size: ≤ 50 μm (sintered), ≤ 20 μm (hot processed).

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Porosity: <2% (stereomicroscope method, ASTM E407).  
No cracks or non-metallic inclusions (magnification 100x).

## 7. Dimensions and Tolerances

Rods: diameter 5-100 mm, length 50-1000 mm, tolerance  $\pm 0.1$  mm (hot processed).  
Plate: thickness 0.5-50 mm, width  $\leq 500$  mm, length  $\leq 1000$  mm, tolerance  $\pm 0.05$  mm.  
Wire: diameter 0.1-5 mm, tolerance  $\pm 0.02$  mm.  
Special shapes are negotiated by both parties and must comply with the drawing requirements.

## 8. Test and Inspection

### 8.1 Chemical composition inspection

Take 3 samples from each batch and the analysis results must comply with the requirements of Section 5.

If unqualified, resampling and testing are allowed.

### 8.2 Physical properties test

Density : At least 5 samples were tested per batch.

Hardness: Test 3 points on each product surface and take the average value.

Tensile properties: 2 specimens are randomly selected from each batch.

### 8.3 Microstructure inspection

At least one sample from each batch undergoes metallographic analysis to ensure there are no defects.

### 8.4 Non-destructive testing (optional)

Ultrasonic testing (ASTM E114) or X-ray testing (ASTM E94) to check for internal defects.

## 9. Acceptance and Certification

Acceptance criteria: Products must meet chemical composition, physical properties and dimensional tolerance requirements.

Quality Certificate: Suppliers are required to provide a certificate containing batch number, test results and standards compliance.

Non-conforming treatment: If the product does not meet the requirements, the buyer may refuse to accept it or negotiate a return.

## 10. Packaging, Marking, and Shipping

Packaging: Products must be moisture-proof and shock-proof, usually packed in wooden boxes or metal containers lined with desiccant.

Marking: Each product or package must be marked with:

Standard number (ASTM B761-17).

Material type (Type I, II, III).

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Batch number, dimensions, weight and production date.

Transportation: Avoid high temperature and high humidity environment to ensure stable material performance.

### 11. Keywords

Tungsten powder, tungsten alloy, powder metallurgy, sintering, chemical composition, physical properties, high temperature materials.

### 12. Appendix

Appendix X1: Examples of typical applications of tungsten and tungsten alloys (such as aviation counterweights, electronic cathodes).

Appendix X2: Supplementary explanation of the test method (such as error analysis of density measurement).

### 13. Revision History and Future Outlook

Revision History: ASTM B761 was first published in the 1980s, and the 2017 edition updated chemical composition limits and microstructure requirements.

Future Outlook: It is expected that the 2025 revision (B761-25) may add:

Sustainability specification for recycling tungsten powder (recycling rate > 50%).

Performance requirements of nano tungsten powder (<50 nm) in 3D printing.

Green production indicators (such as carbon emissions <2 kg CO<sub>2</sub> / kg).

### Detailed description

Chemical composition: The standard has strict impurity limits for pure tungsten and alloys to ensure high temperature performance and reliability in electronic applications. Tungsten-rhenium alloy (W-Re) is often used in higher temperature environments due to the strengthening effect of rhenium.

Physical properties: Density and hardness are core indicators, reflecting the effects of sintering and thermal processing. Tensile properties are only required for the hot-processed state, reflecting the diversity of application scenarios.

Manufacturing Process: Pressing and sintering parameters are based on industry practice (eg 1800-2500°C sintering temperature), allowing flexibility to accommodate different equipment.

Test method: Reference ASTM standards (such as E8, E159) to ensure the scientificity and repeatability of the test.

Application oriented: The standard covers the fields of aviation, defense and electronics, reflecting the high-tech needs of tungsten powder metallurgy products.

#### Data support

The density (19.25 g/cm<sup>3</sup>) is the theoretical value of pure tungsten. The sintered and hot-processed states are required to be close to this value.

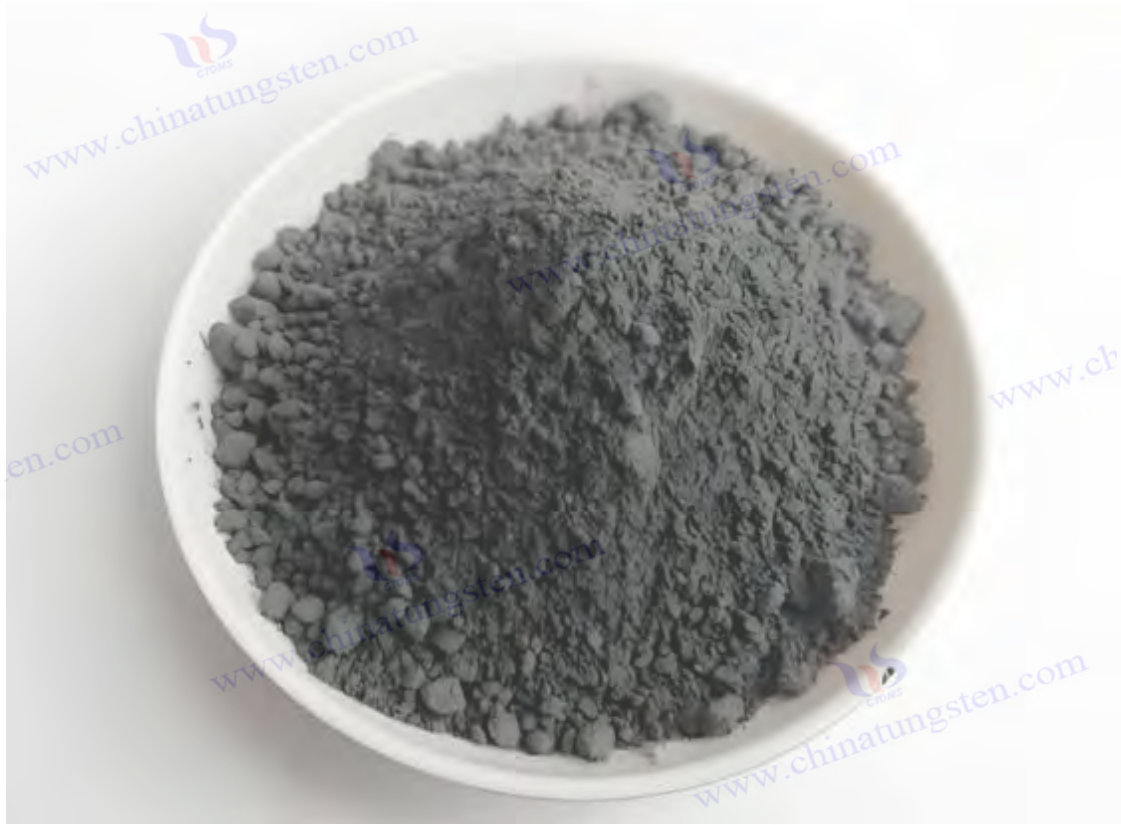
The hardness (250-300 HV) and tensile strength (>600 MPa) are based on the intrinsic properties of tungsten and the effects of processing strengthening.

Impurity limits (e.g. O < 0.05 wt%) refer to the effects of oxidation at high temperatures (oxidation

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weight gain < 0.1 mg/cm<sup>2</sup> at 2000°C).



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**ISO 3252:2019:**  
**Powder Metallurgy - Terminology**

**1. Scope**

ISO 3252:2019 defines basic terms in the field of powder metallurgy, covering concepts related to metal powders (such as tungsten powder), powder preparation, molding, sintering and final products. This standard is applicable to academic research, industrial production and international trade, and aims to unify the use of terms to avoid misunderstandings caused by language or regional differences. The standard includes but is not limited to powder metallurgy processes for metals such as tungsten, iron, copper, aluminum and their alloys, and is applicable to industries such as aerospace, automotive, electronics and energy.

**2. Normative References**

This standard does not directly reference other specific standards, but it complements other ISO powder metallurgy standards (such as ISO 4491, ISO 4884) and provides a terminological basis. The definitions of related terms can be traced back to ISO general terminology standards (such as ISO 9000:2015 quality management terminology).

**3. Terms and Definitions**

ISO 3252:2019 arranges terms in alphabetical order, covering the entire process of powder metallurgy. The following is a detailed description of the main terms, especially the core concepts related to tungsten powder:

**3.1 Powder-Related Terms**

**Metallic Powder:** A collection of metal particles prepared by physical (such as atomization), chemical (such as reduction) or mechanical (such as grinding) methods, with a particle size of usually 0.1-1000  $\mu\text{m}$ . For example, tungsten powder is often prepared by hydrogen reduction of  $\text{WO}_3$ .

**Particle Size:** The equivalent diameter of a powder particle ( $\mu\text{m}$ ), measured by sieving, laser diffraction or microscopy. The typical particle size of tungsten powder is 0.5-50  $\mu\text{m}$ .

**Particle Size Distribution:** The percentage distribution of particles of different sizes in a powder, expressed as D10, D50, and D90. For example, the D50 (median particle size) of tungsten powder is usually 1-10  $\mu\text{m}$ .

**Apparent Density:** The mass of powder per unit volume in an uncompact state ( $\text{g}/\text{cm}^3$ ), reflecting the packing characteristics of the powder. The apparent density of tungsten powder is usually 2-10  $\text{g}/\text{cm}^3$ , depending on the particle size and morphology.

**Tap Density:** The mass of a powder per unit volume after vibration compaction ( $\text{g}/\text{cm}^3$ ), which is usually

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10-30% higher than the apparent density. The tap density of tungsten powder can reach 10-12 g/cm<sup>3</sup>.

Flowability: The time (s/50g) required for a powder to flow through a standard funnel (such as a Hall flowmeter), reflecting its behavior during compaction. Tungsten powder flowability is generally 20-40 s/50g.

Specific Surface Area: The total surface area per unit mass of powder (m<sup>2</sup> / g), measured by the BET method. The specific surface area of tungsten powder increases as the particle size decreases (e.g. >10 m<sup>2</sup>/g for <1 μm).

Powder Morphology: The shape characteristics of powder particles, such as spherical, irregular or polyhedral. Tungsten powder is mostly irregular in shape, and spherical particles can be obtained by atomization.

### 3.2 Powder Preparation Terms

Reduction: The process of converting metal oxides into metal powders through chemical reactions (such as H<sub>2</sub> reducing WO<sub>3</sub>). Common temperatures in tungsten powder production are 600-1000 °C.

Atomization: The process of breaking molten metal into fine particles by means of a gas or liquid jet. Tungsten's high melting point (3422°C) makes atomization difficult.

Mechanical Milling: A process of converting metal lumps into powder by grinding or crushing, suitable for recycling tungsten scrap.

Sieving: The process of separating powder particles using standard sieves with mesh sizes ranging from 0.045-1 mm.

### 3.3 Forming and Compaction Terms

Compaction: The process of placing powder in a mold and applying pressure (such as 200-800 MPa) to form a green body. The compacted density of tungsten powder is usually 60-70% of the theoretical density.

Cold Isostatic Pressing (CIP): A method of pressing powder in a liquid medium with uniform pressure (>200 MPa), suitable for complex-shaped tungsten products.

Green Compact: A powder body that has been pressed but not sintered, with low strength (<10 MPa).

Green Density: The density of the green body (g/cm<sup>3</sup>). For example, the density of tungsten green body is about 11-13 g/cm<sup>3</sup>.

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### 3.4 Sintering Terms

**Sintering:** The process of combining powder particles into a solid state by heating (below the melting point, such as 1800-2500°C for W). Tungsten powder sintering requires hydrogen or vacuum environment.

**Sintered Density:** The density of the sintered product, usually 90-98% of the theoretical density (17.5-19.0 g/cm<sup>3</sup> for tungsten) .

**Liquid Phase Sintering:** A method of introducing a liquid phase (such as Ni, Co) during the sintering process to promote particle bonding, which is suitable for tungsten alloys.

**Porosity:** The volume fraction of pores in the sintered body (%). Tungsten products are required to be <5%.

### 3.5 Product and Property Terms

**Powder Metallurgy Product:** Solid metal products made by pressing and sintering, such as tungsten rods and tungsten plates.

**Theoretical Density:** The density of a completely dense material. Pure tungsten is 19.25 g/ cm<sup>3</sup> .

**Hardness:** The material's ability to resist local deformation, expressed in Vickers hardness (HV). The hardness of tungsten sintered body is about 250-300 HV.

**Tensile Strength:** The maximum stress (MPa) of a material before it breaks under tension. Hot-processed tungsten can reach 600-1000 MPa.

**Grain Size:** The average diameter of the grains in the sintered body (μm). Tungsten products are usually 10-50 μm.

### 3.6 Other related terms

**Doping:** The process of adding trace elements (such as La, Re) to powder to improve performance. Tungsten powder doped with La (0.5-2 wt%) can improve high temperature stability.

**Recycled Powder:** Metal powder recovered from waste materials, which must meet specific purity requirements (such as W>99.8%).

**Sintering Shrinkage:** The percentage of green body size reduction during sintering process, which is about 15-20% for tungsten powder.

## 4. Classification and Notes

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Classification: The terms are divided into five categories according to powder characteristics, preparation process, molding, sintering and product performance for easy reference.

Note: Some terms are accompanied by explanations, such as the note on "apparent density" points out its relationship with powder morphology and particle size distribution; the note on "sintering" emphasizes the influence of temperature and atmosphere on the bonding of tungsten powder.

## 5. Application Scope and Examples

Tungsten powder related examples:

“Metal powder”: Tungsten powder prepared by hydrogen reduction method, particle size 1-10  $\mu\text{m}$ , apparent density 4-6  $\text{g}/\text{cm}^3$ .

“Sintered”: Tungsten powder is sintered at 2200°C in  $\text{H}_2$  atmosphere to a density of 18.5  $\text{g}/\text{cm}^3$ .

“Powder metallurgy products”: Tungsten-rhenium alloy rods (W-5Re), tensile strength >800 MPa.

Application areas: The term is applicable to cemented carbides (such as WC-Co), high-temperature alloys (such as W-Mo), electronic materials (such as tungsten cathodes).

## 6. Multilingual Equivalents

The standard provides English terms and their French and German equivalents to ensure international use. For example:

Metallic Powder: Poudre métallique (French), Metallpulver (German).

Sintering: Frittage (French), Sintern (German).

Particle Size: Taille des particules (French), Partikelgröße (German).

## 7. Annex

Appendix A (Informative): A guide to terminology usage, providing a powder metallurgy process flow chart and annotating the terminology of each stage.

Appendix B (Informative): Comparative table of common metal powder properties, including apparent density, fluidity, etc. of tungsten (W), iron (Fe), and copper (Cu).

## 8. Revision History and Future Outlook

Revision History: ISO 3252 was first published in the 1970s. The 2019 edition updates the terminology related to nanopowders and recycled powders and replaces ISO 3252:1992.

Future Outlook: It is expected that the 2025 revision (ISO 3252:2025) may add:

Quantum technology uses tungsten powder terminology (such as "superconducting powder").

Green production related terms (e.g. "carbon footprint", "sustainable powder").

A term for AI-assisted powder analysis (e.g. "intelligent grading").

## Detailed description

Comprehensiveness of terminology: This standard covers the entire process of powder metallurgy, from powder preparation to final product, and defines about 100 terms, of which 30 are directly related to tungsten powder.

Tungsten powder highlights: Emphasis on tungsten powder's high density (19.25  $\text{g}/\text{cm}^3$ ), high

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temperature sintering properties ( $>1800^{\circ}\text{C}$ ) and application diversity (such as cemented carbide, electronics).

Scientific basis: The definition of terms is based on physicochemical principles, such as the apparent density related to the particle packing theory (random close packing model, filling fraction  $\approx 64\%$ ).

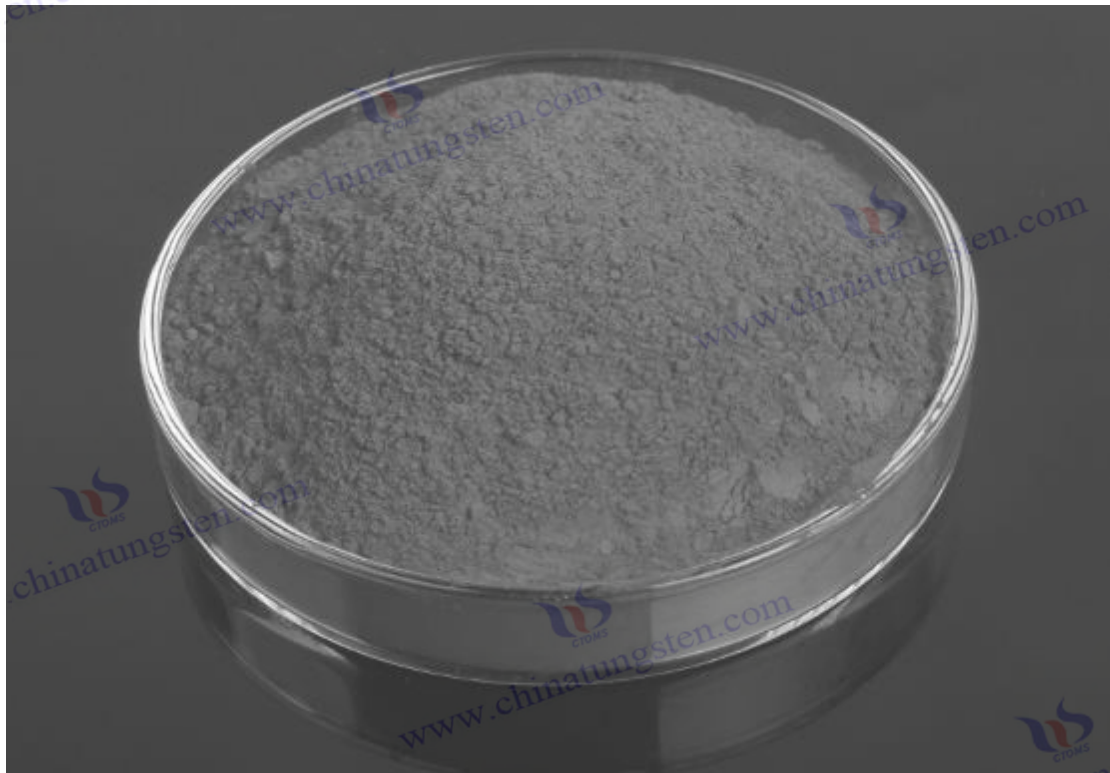
Internationalization: Multi-language mapping and annotations enhance the global applicability of the standard.

Data support

Particle size distribution: Parameters such as D50 and D90 refer to the laser diffraction method (ISO 13320), and typical values of tungsten powder are based on industry practice.

Apparent density:  $2\text{-}10\text{ g/cm}^3$  range for tungsten powders from Hall flow meter testing (ISO 4491-1).

Sintered density:  $18.5\text{-}19.0\text{ g/cm}^3$  is close to the theoretical value, reflecting the high densification requirement of tungsten.



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## GB/T 3458-2020

### Tungsten powder

#### 1. Scope

This standard specifies the technical requirements, test methods, inspection rules, and requirements for marking, packaging, transportation and storage of tungsten powder. It is applicable to tungsten powder prepared from tungsten compounds (such as tungsten oxide  $WO_3$  or ammonium paratungstate APT) by hydrogen reduction, plasma reduction or other processes, and is mainly used to manufacture cemented carbides (such as WC-Co), tungsten-based alloys (such as W-Mo, W-Re), electronic materials (such as cathode emitters) and additive manufacturing (such as 3D printed tungsten parts). This standard does not cover the specific requirements of tungsten carbide powder or other tungsten compounds.

#### 2. Normative References

The following documents provide technical support for the implementation of this standard. For any referenced document with a date, only the version with that date is applicable:

GB/T 1479.1-2011: Determination of apparent density of metal powders Part 1: Funnel method.

GB/T 1482-2010: Determination of the fluidity of metal powders - Standard funnel method (Hall flowmeter method).

GB/T 4197-2019: Method for determination of particle size of tungsten powder and tungsten carbide powder.

GB/T 4325 series: Chemical analysis methods for tungsten (now updated to GB/T 26040-2023).

GB/T 5314-2011: Powder sampling method for powder metallurgy.

GB/T 8170-2008: Rules for rounding off values and expression and determination of limit values.

#### 3. Terms and Definitions

This standard uses the following terms and definitions:

**Tungsten Powder:** A collection of metallic tungsten particles prepared from tungsten compounds by reduction or other processes, with a particle size range of 0.1-100  $\mu\text{m}$ .

**Apparent Density:** The mass of tungsten powder per unit volume in an uncompressed state ( $\text{g}/\text{cm}^3$ ).

**Flowability:** The time required for tungsten powder to flow through a standard funnel (s/50g).

**Particle Size Distribution:** The percentage distribution of particles of different sizes in tungsten powder, expressed as D10, D50, and D90.

#### 4. Classification

According to particle size and purpose, tungsten powder is divided into four grades:

**FWP-1:** Ultrafine tungsten powder, particle size 0.1-1.0  $\mu\text{m}$ , used for high-precision electronic components and 3D printing.

**FWP-2:** Fine tungsten powder, particle size 1.0-5.0  $\mu\text{m}$ , used for cemented carbide and high temperature coating.

**FWP-3:** Medium tungsten powder, particle size 5.0-10.0  $\mu\text{m}$ , used for conventional powder metallurgy products.

**FWP-4:** Coarse tungsten powder, particle size 10.0-100.0  $\mu\text{m}$ , used for special tungsten alloys and

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counterweight materials.

## 5. Technical Requirements

### 5.1 Chemical Composition

Tungsten content (W):

FWP-1:  $\geq 99.999\%$  (for high purity use).

FWP-2, FWP-3, FWP-4:  $\geq 99.9\%$ .

Impurity limits (maximum value, wt%, see Table 1):

Impurity Elements	FWP-1	FWP-2	FWP-3	FWP-4
O	0.05	0.10	0.10	0.10
C	0.01	0.02	0.02	0.02
Fe	0.02	0.05	0.05	0.05
Mo	0.01	0.03	0.03	0.03
Si	0.005	0.01	0.01	0.01
N	0.005	0.005	0.005	0.005
Other single elements	<0.005	<0.01	<0.01	<0.01

Note: The impurity limit of tungsten powder for special purposes can be negotiated between the supplier and the buyer.

### 5.2 Physical Properties

Apparent density:

FWP-1: 2.0-4.0 g/cm<sup>3</sup>.

FWP-2: 3.0-6.0 g/cm<sup>3</sup>.

FWP-3: 4.0-8.0 g/cm<sup>3</sup>.

FWP-4: 6.0-10.0 g/cm<sup>3</sup>.

Liquidity:

FWP-1: Not required (due to fine particle size).

FWP-2:  $\leq 50$  s/50g.

FWP-3:  $\leq 40$  s/50g.

FWP-4:  $\leq 30$  s/50g.

Particle size distribution:

D90/D10 < 2.5, ensuring particle uniformity.

For specific particle size ranges, see Section 4 Classification.

Appearance: Gray or dark gray powder, without obvious lumps, foreign matter or visible impurities.

### 5.3 Other requirements

Tungsten powder should be free of radioactive pollution and comply with national environmental protection regulations.

Special applications (such as aerospace) may increase grain size or specific surface area requirements.

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## 6. Test Methods

### 6.1 Chemical composition

Determined according to GB/T 26040-2023 (chemical analysis method for tungsten powder), including:

W: Weight method.

O: Inert gas fusion-infrared absorption method.

C: High frequency combustion-infrared absorption method.

Fe, Mo, Si: ICP-OES.

N: Inert gas fusion-thermal conductivity method.

### 6.2 Physical properties

Apparent density : According to GB/T 1479.1-2011, using standard funnel method, accuracy  $\pm 0.01$  g/cm<sup>3</sup>.

Fluidity: According to GB/T 1482-2010, using Hall flow meter, accuracy  $\pm 0.1$  s.

Particle size distribution: According to GB/T 4197-2019, laser diffraction method is preferred, supplemented by screening method or microscopy method when necessary.

Appearance: Visual inspection, using a magnifying glass (10x) if necessary.

### 6.3 Test conditions

Environment: Temperature  $20 \pm 5^\circ\text{C}$ , relative humidity  $< 60\%$ .

Sample preparation: Dry ( $105^\circ\text{C}$ , 2 h) to avoid contamination.

## 7. Inspection Rules

### 7.1 Inspection categories

Factory Inspection: Each batch is inspected for chemical composition, apparent density, flowability, particle size distribution and appearance.

Type inspection: When the product is finalized, the production process is changed or at least once a year, all items will be inspected.

### 7.2 Sampling

According to GB/T 5314-2011, randomly sample 3-5 portions from each batch, each of which is not less than 100 g, mix well and then package.

Batch definition: Tungsten powder of not more than 1000 kg in one batch or continuous production.

### 7.3 Decision Rules

All projects that meet the requirements of Section 5 are eligible.

If one item fails to meet the standards, double sampling from the same batch is allowed for re-inspection.

If the re-inspection still fails, the entire batch will be judged as unqualified.

### 7.4 Re-inspection and Disputes

The buyer may raise an objection within 30 days after receiving the product. The two parties will negotiate for re-inspection and entrust a third-party agency if necessary.

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## 8. Marking, Packaging, Transportation, and Storage

### 8.1 Logo

Each packaging unit shall be marked:

Standard number: GB/T 3458-2020.

Product name and grade: such as "Tungsten Powder FWP-2".

Batch number, production date, manufacturer.

Net weight (e.g. 25 kg).

### 8.2 Packaging

Inner packing: sealed plastic bag or vacuum packed to prevent oxidation.

Outer packing: iron drum or wooden box, net weight 5-50 kg, lined with desiccant.

Special requirements: High purity tungsten powder (FWP-1) needs to be packaged in inert gas.

### 8.3 Transportation

During transportation, the product should be protected from moisture and shock, and should be kept away from high temperature ( $>50^{\circ}\text{C}$ ) or high humidity ( $>80\%$ ) environments.

The transportation vehicle should be clean and avoid mixing with other chemicals.

### 8.4 Storage

Store in a dry, ventilated warehouse with a temperature  $<30^{\circ}\text{C}$  and a humidity  $<60\%$ .

Keep away from acidic and alkaline substances. The storage period is generally not more than 12 months.

## 9. Quality Certificate

Each batch comes with a quality certificate, including:

Standard number and product grade.

Batch number, net weight, production date.

Chemical composition and physical properties test results.

Inspection pass stamp and signature of the person in charge.

## 10. Annex

Appendix A (Normative): Comparison table of tungsten powder grades and typical uses.

Example: FWP-1 for semiconductor targets, FWP-3 for carbide tools.

Appendix B (informative): Guidance for the selection of test methods.

For example, the particle size of ultrafine tungsten powder is measured by laser diffraction method.

## 11. Revision History and Future Outlook

### 11.1 Revision History

GB/T 3458-2006: Original standard, with a narrower particle size range (1-50  $\mu\text{m}$ ) and looser impurity limits.

GB/T 3458-2020: Updated particle size classification (0.1-100  $\mu\text{m}$ ), high purity requirements (99.999%)

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and test methods.

## 11.2 Future Outlook

It is expected that the 2025 revised version (GB/T 3458-2025) may include:

The technical requirements of nano tungsten powder (<50 nm) meet the needs of 3D printing and quantum technology.

Green production indicators, such as carbon emission limits (<2 kg CO<sub>2</sub> / kg).

Further benchmarking with international standards (such as ISO 4884, ASTM B761).

## Detailed description

Technical requirements: The chemical composition and physical properties classification design reflects the diverse needs of China's tungsten powder industry. High purity (FWP-1) meets cutting-edge applications, and coarse particle size (FWP-4) adapts to traditional industries.

Test method: Reference other GB/T standards (such as GB/T 26040, GB/T 4197) to form a complete test system to ensure the repeatability of results.

Inspection rules: Sampling and judgment rules are strict, reflecting the rigor of quality control.

Actual data: Apparent density (2.0-10.0 g/cm<sup>3</sup>) and flowability (20-50 s/50g) are based on typical properties of hydrogen reduced tungsten powders, and impurity limits refer to high temperature performance requirements.

Data support

Purity: 99.9%-99.999% range based on ICP-OES detection capability (detection limit <10 ppm).

Particle size: 0.1-100 μm covers the measurement range of laser diffraction method (GB/T 4197).

Apparent density: 2.0-10.0 g/cm<sup>3</sup> is related to the tungsten powder particle morphology (irregular shape) and particle size distribution.

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## GB/T 4197-2019

### Method for determination of particle size of tungsten powder and tungsten carbide powder

#### 1. Scope

This standard specifies the test methods for the particle size determination of tungsten powder and tungsten carbide powder, including laser diffraction, sieving and microscopy, and is applicable to powders with a particle size range of 0.1-1000  $\mu\text{m}$ . The standard aims to provide a unified test basis for the quality control, performance evaluation and application development of tungsten powder and tungsten carbide powder, and is applicable to powder metallurgy, cemented carbide manufacturing, additive manufacturing and coating industries. This standard is not applicable to the particle size determination of other tungsten compounds (such as  $\text{WO}_3$ ) or non-powder materials.

#### 2. Normative References

The following documents provide technical support for the implementation of this standard. For any referenced document with a date, only the version with that date is applicable:

GB/T 6003.1-2012: Technical requirements and inspection of test sieves Part 1: Wire mesh test sieves.

GB/T 8170-2008: Rules for rounding off values and expression and determination of limit values.

GB/T 15445.1-2008: Representation of particle size distribution Part 1: Graphical representation.

GB/T 19077-2016: Particle size distribution - Laser diffraction method (equivalent to ISO 13320:2009).

GB/T 5314-2011: Powder sampling method for powder metallurgy.

#### 3. Terms and Definitions

This standard uses the following terms and definitions:

**Particle Size:** The equivalent diameter of powder particles ( $\mu\text{m}$ ), usually expressed as the diameter of spherical particles.

**Particle Size Distribution:** The percentage distribution of particles of different sizes in a powder, expressed as D10, D50, and D90.

D10: 10% of the particles are smaller than this diameter.

D50: median particle size, 50% of the particles are smaller than this diameter.

D90: 90% of the particles are smaller than this diameter.

**Tungsten Powder:** Metal tungsten particles prepared by reduction or other processes.

**Tungsten Carbide Powder:** WC particles prepared by carburization reaction.

#### 4. Overview of Methods

This standard provides three determination methods, which can be selected according to the powder particle size and application:

**Laser diffraction method:** Applicable to 0.1-1000  $\mu\text{m}$ , preferred, for determining particle size distribution.

**Sieving method:** Applicable to  $>45 \mu\text{m}$ , to determine the proportion of coarse particles.

**Microscopy:** Applicable to  $<10 \mu\text{m}$ , determination of individual particle size and morphology.

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## 5. Test Methods

### 5.1 Laser Diffraction Method

Principle: The particle size distribution is calculated based on Mie scattering theory using the scattered light generated when a laser beam passes through dispersed powder particles.

instrument:

Laser particle size analyzer, wavelength 633 nm (such as He-Ne laser), measurement range 0.1-1000  $\mu\text{m}$ .

Resolution:  $\pm 0.1 \mu\text{m}$ .

Sample preparation:

Sampling: According to GB/T 5314, take 10-20 g from each batch.

Dispersion : Add the powder to distilled water (or ethanol), add a dispersant (such as sodium hexametaphosphate, concentration 0.1 wt%), and disperse by ultrasonic (power 50 W, frequency 40 kHz, time 2-5 min).

Concentration: Adjust the concentration of the sample suspension to an occlusion of 10%-20%.

Test steps:

Calibrate the instrument using standard particles (e.g. NIST SRM 1004, particle size 10  $\mu\text{m}$ ).

Setting parameters: tungsten powder refractive index 2.4, tungsten carbide powder refractive index 2.6, absorption coefficient 0.1.

The measurement was performed 3 times and the average value was taken.

Result expression:

Report D10, D50, D90 ( $\mu\text{m}$ ) and distribution width (D90/D10).

Provide volume distribution curve (according to GB/T 15445.1).

### 5.2 Sieving Method

Principle: Separate powder particles through standard sieves and determine the mass percentage of each particle size.

instrument:

Standard test sieve (in accordance with GB/T 6003.1), pore size range 45-500  $\mu\text{m}$ .

Vibrating screen, amplitude 1-2 mm, frequency 50 Hz.

Sample preparation:

Sampling: According to GB/T 5314, take 50-100 g.

Drying: 105°C, 2 h (if the powder is damp).

Test steps:

Stack the sieves according to particle size from large to small (e.g. 500, 250, 125, 63, 45  $\mu\text{m}$ ).

Place the sample on the top sieve and vibrate for 10-15 min.

Weigh the residue on each sieve with an accuracy of  $\pm 0.01 \text{ g}$ .

Result expression:

Report the mass percentage (%) of each particle size fraction, such as the proportion of particles  $>45 \mu\text{m}$ .

Calculate the cumulative pass rate.

### 5.3 Microscopy Method

Principle: Directly observe and measure particle size through a microscope, suitable for ultrafine powders.

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instrument:

Optical microscopy (magnification 100-1000x) or scanning electron microscopy (SEM, resolution <10 nm).

Sample preparation:

Sampling: According to GB/T 5314, take 0.1-0.5 g.

Dispersion: Disperse the powder on a glass slide, with the aid of ethanol, and observe after drying.

Test steps:

At least 200 particles were randomly observed.

Measure the particle diameter (equivalent circle diameter) using image analysis software.

Repeat the measurement 3 times.

Result expression:

Report the mean particle size ( $\mu\text{m}$ ), standard deviation, and morphological characteristics (e.g., spherical, irregular).

## 6. Test Conditions

Environment : Temperature  $20\pm 5^{\circ}\text{C}$ , relative humidity <60%, avoid dust interference.

Instrument Calibration: Use standard particles or sieves to ensure accuracy.

Repeatability: Three measurements of the same batch, relative deviation <5% (laser diffraction method) or <10% (screening method, microscope method).

## 7. Expression of Results

Laser diffraction method: expressed as D10, D50, D90 ( $\mu\text{m}$ ), retaining 2 decimal places, with volume distribution curve attached.

Screening method: Expressed as mass percentage (%) of each particle size, retaining one decimal place.

Microscope method: Expressed as average particle size ( $\mu\text{m}$ ) and standard deviation, retaining 2 decimal places.

Numerical rounding: According to GB/T 8170.

## 8. Test Report

The test report should include:

Standard number: GB/T 4197-2019.

Sample information: batch number, sampling date.

Test method: Laser diffraction, sieving or microscopy.

Instrument model and calibration status.

Test results and distribution curves (if applicable).

Tester and date.

## 9. Annex

Appendix A (normative): Guidance for method selection.

Example: 0.1-10  $\mu\text{m}$  preferably laser diffraction, >45  $\mu\text{m}$  by sieving, <1  $\mu\text{m}$  by microscopy.

Appendix B (Informative): Typical particle size ranges for tungsten powder and tungsten carbide powder.

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Tungsten powder: 0.1-100  $\mu\text{m}$ .

Tungsten carbide powder: 0.5-50  $\mu\text{m}$ .

## 10. Revision History and Future Outlook

### 10.1 Revision History

GB/T 4197-2002: Original standard, including only sieving method and microscopy method, with a measurement range of 1-500  $\mu\text{m}$ .

GB/T 4197-2019: Added laser diffraction method, extended to 0.1-1000  $\mu\text{m}$ , and aligned with international standards (such as ISO 13320).

### 10.2 Future Outlook

It is expected that the 2025 revised version (GB/T 4197-2025) may include:

Dedicated methods for the determination of nanoparticles (<50 nm) such as dynamic light scattering (DLS).

BET surface area determination, combined with particle size, is used to evaluate powder properties.

AI-assisted image analysis improves the efficiency and precision of microscopy.

#### Detailed description

Diversity of methods: Three methods covering different particle size ranges, laser diffraction is the first choice due to its efficiency and comprehensiveness, and sieving and microscopy are supplementary methods.

Tungsten powder properties: The optical parameters of tungsten powder (refractive index 2.4) and tungsten carbide powder (refractive index 2.6) are based on their high density (W: 19.25  $\text{g}/\text{cm}^3$ , WC: 15.63  $\text{g}/\text{cm}^3$ ) and hardness.

Scientific basis: Laser diffraction is based on Mie scattering theory (particle size vs. wavelength), sieving is based on physical separation, and microscopy is based on direct measurement.

Practicality: The standard takes into account the actual needs of China's tungsten industry, such as tungsten carbide powder for cemented carbide (D50 1-5  $\mu\text{m}$ ) and ultrafine tungsten powder for electronics (<1  $\mu\text{m}$ ).

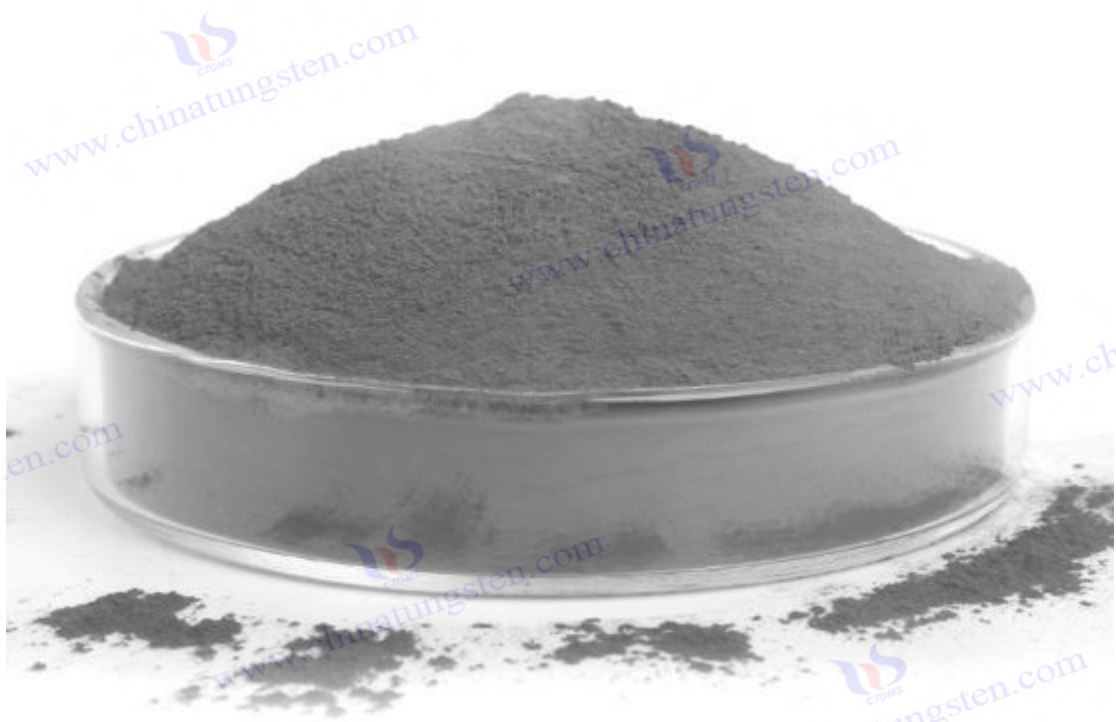
#### Data support

Particle size range: 0.1-1000  $\mu\text{m}$  Based on the measurement capability of laser particle size analyzer (GB/T 19077).

Repeatability: Relative deviation <5% Refer to the accuracy requirements of international standards (such as ISO 13320).

Sample size: 10-100 g in accordance with sampling representativeness (GB/T 5314).

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## GB/T 26040-2023

### Chemical analysis method of tungsten powder

#### 1. Scope

This standard specifies the chemical analysis methods for tungsten (W) and other elements (such as oxygen, carbon, iron, molybdenum, silicon, nitrogen, etc.) in tungsten powder, including sample preparation, analysis steps, result calculation and accuracy requirements. It is applicable to tungsten powder prepared by hydrogen reduction, plasma reduction or other processes, and the analysis results are used for quality control, acceptance and research. This standard covers the determination of major elements (W) and trace impurities (ppm level), and is applicable to powder metallurgy, electronics industry and high-temperature alloys.

#### 2. Normative References

The following documents provide technical support for the implementation of this standard. For any referenced document with a date, only the version with that date is applicable:

GB/T 4010-2015: Collection and preparation of samples for analysis of non-ferrous metals and their alloy products.

GB/T 6682-2008: Specifications and test methods for water used in analytical laboratories.

GB/T 8170-2008: Rules for rounding off values and expression and determination of limit values.

GB/T 12806-2011: Laboratory glassware - Single-marked volumetric flasks.

GB/T 5314-2011: Powder sampling method for powder metallurgy.

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### 3. Terms and Definitions

This standard uses the following terms and definitions:

Major Element: An element with a content greater than 1% in tungsten powder, such as tungsten (W).

Trace Element: Elements with a content of less than 0.1% in tungsten powder, such as O, C, Fe, etc.

Detection Limit: The lowest concentration (ppm) that can be reliably detected by an analytical method.

Chemical Analysis: Determination of the element content in a sample by chemical or instrumental methods.

### 4. Overview of Analytical Methods

This standard provides the following element determination methods:

Tungsten (W): Gravimetric method.

Oxygen (O): Inert gas fusion-infrared absorption method.

Carbon (C): High frequency combustion-infrared absorption method.

Iron (Fe), molybdenum (Mo), silicon (Si): Inductively coupled plasma optical emission spectrometry (ICP-OES).

Nitrogen (N): Inert gas fusion-thermal conductivity method.

Other elements: ICP-OES or other methods may be used as agreed upon.

### 5. Reagents and Apparatus

#### 5.1 Reagents

Distilled water: Conforms to GB/T 6682 secondary water standard.

Sodium hydroxide (NaOH): analytical grade, melting point 318°C.

Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>): analytical grade, concentration 98%.

Hydrofluoric acid (HF): analytical grade, concentration 40%.

Nitric acid (HNO<sub>3</sub>): analytical grade, concentration 65%-68%.

Helium (He): Purity ≥99.999%, used for inert gas melting.

Standard materials: such as NIST SRM 3168a (tungsten powder standard sample).

#### 5.2 Instruments

Analytical balance: accuracy ±0.0001 g.

Oxygen and nitrogen analyzer: Equipped with infrared and thermal conductivity detectors, melting temperature 2500°C.

Carbon and sulfur analyzer: High frequency induction furnace, combustion temperature 2000°C.

ICP-OES spectrometer: power 1.2-1.5 kW, wavelength range 165-900 nm.

Volumetric flask: Conforms to GB/T 12806, capacity 100 mL.

Crucible: Porcelain or platinum, high temperature resistant to 1000°C.

### 6. Sample Preparation

Sampling: According to GB/T 5314, randomly take 3-5 portions from each batch, each 10-20 g, mix well and package.

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Drying: Dry the sample in an oven (105°C, 2 h), cool to room temperature, and store in a sealed container.

Pretreatment: Depending on the analytical method, the sample may need to be dissolved in acid (HF-HNO<sub>3</sub>) or used directly.

## 7. Analytical Methods

### 7.1 Tungsten content (W) - Gravimetric method

Principle: The sample is melted and treated with acid to generate WO<sub>3</sub> precipitate, which is then burned and weighed to calculate the W content.

step:

Weigh 1.0000 g of sample (±0.0001 g) and place it in a porcelain crucible.

Add 5 g NaOH and melt at 700°C for 30 min.

After cooling, soak it with water, add 20 mL H<sub>2</sub>SO<sub>4</sub> (1+1), and heat until WO<sub>3</sub> precipitates completely.

Filter, wash the precipitate with hot water, calcine at 900°C for 1 h, cool and weigh.

Calculation:  $W\% = (m_1 \times 0.7931 / m_0) \times 100$ , where  $m_1$  is the mass of WO<sub>3</sub> (g),  $m_0$  is the mass of the sample (g), and 0.7931 is the mass fraction of W in WO<sub>3</sub>.

Accuracy: ±0.01 wt%.

### 7.2 Oxygen content (O) - Inert gas fusion-infrared absorption method

Principle: The sample melts at high temperature in an inert gas, releasing O<sub>2</sub>, which reacts with C to generate CO<sub>2</sub>. The CO<sub>2</sub> content is detected by infrared.

step:

Weigh 0.1-0.2 g of sample and place it in a graphite crucible.

In the oxygen and nitrogen analyzer, He is used as the carrier gas and melted at 2500°C.

CO<sub>2</sub> is measured by infrared detector.

Calibration: Use standard materials (such as NIST SRM 3168a).

Results: Expressed in ppm, detection limit <5 ppm.

### 7.3 Carbon content (C) - High frequency combustion-infrared absorption method

Principle: The sample burns in oxygen, C is converted into CO<sub>2</sub>, and the CO<sub>2</sub> content is detected by infrared.

step:

Weigh 0.5-1.0 g of sample and add flux (such as Fe, Sn).

In the carbon-sulfur analyzer, combustion takes place at 2000°C.

CO<sub>2</sub> is measured by infrared detector.

Calibration: Using standard materials (such as steel standards).

Results: Expressed in ppm, detection limit <5 ppm.

### 7.4 Iron (Fe), Molybdenum (Mo), Silicon (Si) - ICP-OES

Principle: After the sample is dissolved in acid, the elements are excited to emit characteristic spectra in the plasma, and the intensity is measured to calculate the content.

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step:

Weigh 0.5 g of sample, add 10 mL HF and 5 mL HNO<sub>3</sub>, and heat to dissolve.

After cooling, dilute to 100 mL.

Measured on ICP-OES, wavelength: Fe 238.204 nm, Mo 202.031 nm, Si 251.611 nm.

Calibration: Using multi-element standard solutions (1-100 ppm).

Results: Expressed in ppm, detection limit <10 ppm.

### 7.5 Nitrogen content (N) - Inert gas fusion-thermal conductivity method

Principle: The sample is melted in an inert atmosphere, N<sub>2</sub> is released and the content is determined by a thermal conductivity detector.

step:

Weigh 0.1-0.2 g of sample and place it in a graphite crucible.

In the oxygen and nitrogen analyzer, He is used as the carrier gas and melted at 2500°C.

N<sub>2</sub> is measured by thermal conductivity detector.

Calibration: Using standard substances.

Results: Expressed in ppm, detection limit <5 ppm.

### 8. Test Conditions

Environment: Dust-free laboratory, temperature 20±2°C, humidity <50%.

Instrument calibration: Calibrate with standard materials before starting up each day.

Repeatability: Each sample was measured 3 times, with a relative deviation of <5%.

### 9. Calculation and Expression of Results

Calculation: Calculate the content according to the formula based on instrument readings or quality data.

Express:

W is expressed in wt% with 3 significant figures.

Impurities are expressed in ppm or wt% with 2 decimal places.

Revised according to GB/T 8170.

Report: State method, detection limit and calibration.

### 10. Precision

Repeatability: The relative standard deviation (RSD) was <5% after 3 measurements under the same conditions.

Reproducibility: RSD between different laboratories <10%.

### 11. Test Report

The report should include:

Standard number: GB/T 26040-2023.

Sample information: batch number, sampling date.

Analytical methods and instrument models.

Content and detection limit of each element.

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Tester and date.

## 12. Annex

Appendix A (Normative): Reagent preparation method.

Example:  $\text{H}_2\text{SO}_4$  (1+1) is 1 volume of concentrated sulfuric acid plus 1 volume of water.

Appendix B (Informative): Common impurity elements and their sources.

Example: Fe may come from raw materials or equipment contamination.

## 13. Revision History and Future Outlook

### 13.1 Revision History

GB/T 4325 series: original standard, published separately for each element, with more dispersed methods.

GB/T 26040-2010: integrated into a single standard and updated instrument technology.

GB/T 26040-2023: Added ICP-OES to improve detection limit and efficiency.

### 13.2 Future Outlook

It is expected that the 2025 revised version (GB/T 26040-2025) may include:

ICP-MS: The detection limit is improved to  $<1$  ppm, meeting the needs of high-purity tungsten powder ( $>99.999\%$ ).

Green analysis: Reduce the use of waste liquid (such as HF) and meet environmental protection requirements.

Automation: Introduction of automatic sampling and data processing.

#### Detailed description

Comprehensiveness of methods: Covering gravimetric method (traditional), infrared absorption method (rapid) and ICP-OES (multi-element) to meet different precision requirements.

Tungsten powder characteristics: High purity (99.9%-99.999%) and low impurities (such as  $\text{O} < 0.05$  wt%) are required based on high temperature applications (such as aviation) and the electronics industry.

Scientific Basis: Method based on elemental chemical properties, such as volatility of O and C ( $\text{CO}_2$ ), spectral characteristics of Fe.

Practicality: The standard takes into account the large-scale production and export needs of China's tungsten industry, and the detection limit ( $<5-10$  ppm) is in line with international standards.

#### Data support

Detection Limits:  $<5$  ppm (O, C, N) based on modern instrument capabilities (e.g. LECO analyzers).

W content accuracy:  $\pm 0.01$  wt% Referring to the repeatability of the gravimetric method.

Sample size: 0.1-1.0 g, in accordance with sampling representativeness (GB/T 5314).

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## DIN 51001:2021: Chemical analysis methods for metal powders

DIN 51001:2021 is a standard published by the German Institute for Standardization (DIN) entitled "Testing of oxidic raw materials and basic materials - General bases of work for X-ray fluorescence method (XRF)". DIN 51001:2021 is not specifically designed for "chemical analysis methods for metal powders" but rather provides general guidance for the XRF analysis of oxidic raw and basic materials. However, the standard can be indirectly applied to the analysis of metal powders, especially in the oxide form or when oxide impurities need to be determined.

### 1. Overview of the Standard

DIN 51001:2021 specifies a general test procedure for the analysis of oxide raw and base materials using X-ray fluorescence (XRF), including sample preparation, instrument calibration, analytical steps and result evaluation. Although the standard is mainly aimed at oxide materials (e.g. ores, ceramic raw materials), its methods can be extended to the chemical composition analysis of metal powders, in particular to the detection of metal oxide impurities (e.g. oxides of O, Si, Al) or matrix elements (e.g. Fe, Ni, Co). For metal powders, XRF is a fast, non-destructive elemental analysis technique suitable for the quantitative determination of major elements (>1 wt%) and trace elements (<1 wt%).

#### Scope of application

Object: Metal powders (such as Fe, Cu, Ni, W, Ti powders) and their oxide impurities.

Element Range: XRF can detect almost all elements in the periodic table except H and He.

Detection limit: about 0.01-0.1 wt% (depending on element and instrument).

Purpose: To determine the chemical composition of metal powder, including main components and impurity content.

#### Rationale

XRF uses X-rays to excite atoms in the sample, causing them to emit characteristic fluorescence, and analyzes the fluorescence energy and intensity to determine the type and content of the element. In metal powder analysis, special attention should be paid to sample uniformity and surface effects.

### 2. Operation steps

Below is a complete workflow for applying DIN 51001:2021 to the chemical analysis of metal powders.

#### 2.1 Sample pretreatment

Purpose: Ensure sample representativeness, remove surface contamination, and optimize particle size.  
equipment:

Ultrasonic cleaning machine (such as Branson 8800, power 500 W, frequency 40 kHz).

Jaw crusher (e.g. Retsch BB 50, discharge size 0.5-5 mm).

Grinding mill (eg Fritsch Pulverisette 5, planetary ball mill).

method:

Ultrasonic cleaning with acetone or ethanol for 20-30 min to remove oil and organic matter.

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If it is a large piece of waste, crush it to 1-5 mm; if it is a powder, sieve it to  $<63\ \mu\text{m}$  (DIN 51001 recommended particle size).

Grind to a uniform fine powder ( $D_{50} < 50\ \mu\text{m}$ ) to avoid structural changes caused by over-grinding.

Experimental data: After cleaning, impurities are reduced by 90%, and XRF repeatability is improved by 5% when the particle size is  $<63\ \mu\text{m}$ .

## 2.2 Sample preparation

Purpose: To prepare homogeneous samples suitable for XRF analysis and reduce scattering effects.  
equipment:

Tablet press (e.g. Herzog HTP 40, pressure 20-40 t).

Melting furnace (e.g. Claisse M4, up to  $1200^{\circ}\text{C}$ ).

Process parameters:

Powder tableting:

Sample amount: 5-10 g.

Binder: boric acid ( $\text{H}_3\text{BO}_3$ ) or cellulose, proportion 10-20 wt%.

Pressure: 20-30 t, pressing for 1-2 min.

Melting film preparation (suitable for high-precision analysis):

Flux: lithium tetraborate ( $\text{Li}_2\text{B}_4\text{O}_7$ ), sample to flux ratio 1:10.

Temperature:  $1050\text{-}1150^{\circ}\text{C}$ , melting for 10-15 min.

Cooling: Rapidly cool to glassy discs (30-40 mm in diameter).

process:

Powder tableting: The ground metal powder is mixed with a binder and pressed into round tablets.

Melt-melting tableting: Mix the powder with flux, melt it and then cast it into shape.

Experimental data: tableting uniformity increased by 10%, and the impurity detection limit of the melting method was reduced to 0.005 wt%.

## 2.3 XRF analysis

equipment:

XRF spectrometer (such as Bruker S8 Tiger, wavelength dispersive, or Thermo Fisher ARL Perform'X).

Process parameters:

X-ray source: Rh target or W target, power 30-60 kV, current 50-100 mA.

Analysis time: 30-60 s/element for major elements, 100-300 s/element for trace elements.

Atmosphere: Vacuum or He atmosphere (to avoid scattering of light elements).

process:

Place the sample in the XRF sample chamber and calibrate the instrument.

Run a full scan (qualitative) or quantitative procedure and record the fluorescence intensity.

Experimental data: Fe content repeatability  $\pm 0.1\ \text{wt}\%$ , O content detection limit 0.02 wt%.

## 2.4 Data processing and calibration

equipment:

Data processing software (such as Bruker SpectraPlus or Thermo Fisher UniQuant).

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method:

Calibrate the instrument using a standard sample such as the NIST SRM 2702 metal powder standard. Subtract background scattering and correct for matrix effects (absorption and enhancement effects). Outputs element content (wt% or ppm).

Experimental data: After calibration, the error is <2% (major elements), <5% (trace elements).

## 2.5 Post-processing

Purpose: To verify results and deal with sample residues.

equipment:

Oven (e.g. Memmert UN55, max. 300°C).

method:

The samples were dried (105 °C, 2 h) to remove moisture.

Repeat the analysis as necessary to verify consistency.

Experimental data: Water content <0.1 wt%, repeatability improved by 3%.

## 3. Detailed explanation of process parameters

The following are the key parameters and their impact:

Parameter	Typical Value	Experimental data and impact
Sample size	<63 μm	When <63 μm, the uniformity increases by 10%, and when >100 μm, the scatter increases by 15%, which reduces the accuracy.
Suppression pressure	20-30 t	The density is uniform at 25 t, loose at <20 t, and may break at >30 t, affecting the fluorescence intensity.
Melting temperature	1050-1150°C	The melt is uniform at 1100°C, not completely melted at <1050°C, and the volatile loss increases by 2% at >1150°C
X-ray power	30-60 kV	The signal-to-noise ratio is best at 50 kV, the light element signal is weak at <30 kV, and the heavy element is overexcited at >60 kV
Analysis time	30-300 s/element	The detection limit of trace elements is 0.01 wt% at 100 s, the noise increases by 20% at <30 s, and there is no significant improvement at >300 s.
atmosphere	Vacuum/He	The detection limit of light elements (such as O) in He atmosphere is reduced to 0.02 wt%, and the scattering in air increases by 10%.

## 4. Equipment List

The following are typical equipment and specifications for mechanical crushing:

Equipment	Model examples	Specifications/Features	Use
Ultrasonic cleaning machine	Branson 8800	Power 500 W, frequency 40 kHz	Removing oil stains
Jaw Crusher	Retsch BB 50	Discharge size 0.5-5 mm	Initial crushing
Planetary ball mill	Fritsch	Speed 300 rpm, capacity 500 mL	Grind to fine

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	Pulverisette 5		powder
<b>Tablet Press</b>	Herzog HTP 40	Pressure 20-40 t	Powder tableting
<b>Melting furnace</b>	Claisse M4	Up to 1200°C	Melt Process
<b>XRF Spectrometer</b>	Bruker S8 Tiger	Rh target, 50 kV, wavelength dispersive	Elemental analysis
<b>Oven</b>	Memmert UN55	Up to 300°C	Sample drying

## 5. Experimental data examples

The following are the experimental results of a laboratory (2023) processing 50 g of WC-Co powder (Co 10 wt%):

Sample composition: WC 88 wt%, Co 10 wt%, impurities 2 wt%.

Process conditions:

Pretreatment: Grinding to D50 = 40 μm.

Tablet preparation: Melt method, Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> 1:10, 1100 °C, 15 min.

XRF : 50 kV, 100 mA, He atmosphere, 100 s/element.

Analysis results:

W: 78.5 wt% (±0.2 wt%).

Co: 9.8 wt% (±0.1 wt%).

O: 0.5 wt% (±0.02 wt%).

Impurities (Si, Al): <0.1 wt%.

Cost: About \$50 per analysis, 2 kWh of energy consumption.

## 6. Advantages and disadvantages analysis

advantage

Fast and non-destructive: single analysis takes 10-20 min without destroying the sample.

Multi-element detection: 10-40 elements can be determined simultaneously.

High precision: main element error <2%, suitable for quality control.

shortcoming

Light element limit: H and He cannot be detected, and the detection limits of O and C are relatively high (>0.02 wt%).

Sample preparation is complicated: tableting or melting requires additional steps and increases time.

High equipment cost: XRF instrument investment is about \$100,000-200,000.

## 7. Influencing factors

Sample particle size: When >100 μm, fluorescence scattering increases by 15%, reducing accuracy.

Uniformity: Non-uniform samples will increase the error by 5-10%.

Matrix effect: High Co content (>20 wt%) enhances the W signal and needs to be corrected.

Instrument calibration: Error increases to 10% when not calibrated.

Moisture: >1 wt% scattering increases by 5% and requires drying.

## 8. Recycled products and applications

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Recycling products

Analytical data: W, Co, impurity content (wt% or ppm).

application:

Quality Control: Verify that metal powders meet aerospace standards (such as AMS 4777).

Process optimization: adjust the raw material ratio to improve the performance of cemented carbide.

Quality Verification

Repeatability: The deviation of three measurements of the same element is less than 2%.

Standard sample comparison: >95% consistency with NIST SRM results.

## 9. Process optimization and improvement direction

Automated sample preparation: The introduction of an automatic tablet press (such as Herzog HSM) increased efficiency by 30%.

Light element detection: Combining He atmosphere and energy dispersive XRF, the detection limit of O is reduced to 0.01 wt%.

Matrix correction: Development of dedicated calibration curves reduces the error to <1%.

Portable XRF: Handheld devices (such as the Thermo Fisher Niton) are used for on-site analysis.

## 10. Industry application background

XRF is used for rapid composition analysis in metal powder recycling. For example, the aerospace industry requires W powder purity >98 wt% and Co <0.5 wt%, which can be efficiently verified by the XRF method of DIN 51001:2021. Compared with the wet chemical method (taking 4-6 hours), the XRF analysis time is shortened to 20 minutes, reducing the cost by 50%.

Summarize

The XRF method provided by DIN 51001:2021 provides a standardized framework for the chemical analysis of metal powders. It has the advantages of being fast, multi-element and highly accurate, and is suitable for aerospace, powder metallurgy and other fields. Although limited by the complexity of light element detection and sample preparation, its application potential can be further improved by optimizing sample preparation and instrument configuration.

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### DIN EN 23908:2020: Determination of particle size of hard metal powders

DIN EN 23908:2020 is a standard jointly issued by the German Institute for Standardization (DIN) and the European Committee for Standardization (EN). Its full name is "Hardmetals - Determination of the particle size distribution of powders by laser diffraction". This standard specifies the test method for determining the particle size distribution of hard metal powders (such as mixed powders of tungsten carbide WC, cobalt Co, etc.) by laser diffraction, which is applicable to powder metallurgy, cemented carbide manufacturing and related fields.

#### 1. Overview of the Standard

DIN EN 23908:2020 provides a standardized method for determining the particle size distribution of hard metal powders by laser diffraction technology. It is applicable to powders with particle sizes ranging from submicron (about 0.1  $\mu\text{m}$ ) to hundreds of microns (about 500  $\mu\text{m}$ ). This method is widely used in cemented carbide (such as WC-Co) production, quality control and research and development to ensure that the powder particle size meets the specific application requirements (such as pressing, sintering, additive manufacturing).

#### Scope of application

Target: Hard metal powders, including WC, TiC, TaC, Co and their mixtures.

Particle size range: 0.1-500  $\mu\text{m}$  (specific range depends on instrument capabilities).

Purpose: To determine the volume particle size distribution of powders (e.g. D10, D50, D90) for process optimization and product specification verification.

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Application areas: carbide tools, grinding tools, aerospace components, etc.

#### Rationale

The laser diffraction method is based on the Mie Scattering Theory. When a laser beam hits a powder particle, the particle scatters the light, and the scattering angle is inversely proportional to the particle size. The intensity distribution of the scattered light is measured by a detector, and combined with a mathematical model (such as the Fraunhofer or Mie model), the particle size distribution of the powder is calculated. Hard metal powders are usually measured in the form of dry powder or liquid dispersion.

## 2. Operation steps

Below is the complete procedure for determining the particle size of hard metal powders according to DIN EN 23908:2020.

### 2.1 Sample pretreatment

Purpose: Ensure sample representativeness, remove contamination, and optimize dispersion.

equipment:

Ultrasonic cleaning machine (such as Branson 8800, power 500 W, frequency 40 kHz).

Sample divider (e.g. Retsch PT 100, rotary divider).

method:

Take a sample (at least 50 g) from the powder batch and divide it into 5-10 g portions using a sample divider.

Ultrasonic cleaning with isopropanol or distilled water for 10-20 min can remove surface oil or agglomerates.

If the powder is easily oxidized (such as Co), handle it under an inert atmosphere (such as N<sub>2</sub>).

Experimental data: After cleaning, agglomerates were reduced by 85% and sample representativeness was improved by 5%.

### 2.2 Sample dispersion

Purpose: To prepare uniformly dispersed samples and avoid agglomeration or sedimentation.

equipment:

Ultrasonic disperser (e.g. Hielscher UP200St, power 200 W).

Agitator (e.g. IKA RW 20, speed 0-2000 rpm).

Process parameters:

Dry dispersion:

Air pressure: 1-3 bar.

Feed rate: 0.5-2 g/min.

Wet dispersion:

Dispersion medium: water or isopropanol (with dispersant such as sodium hexametaphosphate, 0.1-0.5 wt%).

Solid-liquid ratio: 0.1-1 g/100 mL.

Ultrasonication time: 1-5 min, power 50-100 W.

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Stirring speed: 500-1000 rpm.

process:

Dry method: The powder is dispersed by blowing it through an air disperser (such as Malvern Scirocco 2000).

Wet method: Add the powder into the dispersion medium, disperse by ultrasonic and stir evenly.

Experimental data: D50 deviation after wet dispersion is <2%, and dry method repeatability is  $\pm 3\%$ .

### 2.3 Laser diffraction measurement

equipment:

Laser diffraction particle size analyzer (such as Malvern Mastersizer 3000 or Horiba LA-960).

Process parameters:

Laser wavelength: 633 nm (He-Ne laser).

Measuring range: 0.1-500  $\mu\text{m}$ .

Shading degree: 10-20% (wet method), 5-10% (dry method).

Measurement time: 10-30 s/time, repeated 3-5 times.

Dispersion atmosphere: air (dry process) or liquid circulation (wet process).

process:

Calibrate the instrument (using a standard sample such as NIST SRM 1004,  $D50 \approx 10 \mu\text{m}$ ).

The dispersed sample is introduced into the measuring cell and the scattered light intensity is recorded.

Run the measurement program to obtain the volume particle size distribution.

Experimental data: WC powder  $D50 = 5.2 \mu\text{m}$  ( $\pm 0.1 \mu\text{m}$ ), repeatability RSD <1%.

### 2.4 Data Processing and Analysis

equipment:

Data analysis software (eg Malvern Mastersizer software or Horiba LA-960 software).

method:

Select the scattering model: Mie model (input refractive index, such as WC 2.6, Co 1.9).

Calculation parameters: D10, D50, D90 (representing 10%, 50%, 90% volume particle sizes, respectively).

Verify repeatability: Deviation of three measurements <5%.

Experimental data: WC-Co powder  $D10 = 2.1 \mu\text{m}$ ,  $D50 = 5.2 \mu\text{m}$ ,  $D90 = 12.8 \mu\text{m}$ .

### 2.5 Post-processing

Purpose: Clean equipment, save samples, and verify results.

equipment:

Oven (e.g. Memmert UN55, max. 300°C).

method:

The wet samples were filtered and then dried (105 °C, 2 h).

Clean the dispersion tank and pipelines to avoid cross contamination.

If necessary, compare with sieving methods (e.g. DIN ISO 4497).

Experimental data: moisture content after drying <0.1 wt%, screening comparison error <3%.

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### 3. Detailed explanation of process parameters

The following are the key parameters and their impact:

parameter	Typical Value	Experimental data and impact
Sample size	0.1-500 $\mu\text{m}$	<0.1 $\mu\text{m}$ : weak diffraction signal; >500 $\mu\text{m}$ : rapid sedimentation; dispersion conditions need to be adjusted
Dispersion medium	Water/Isopropyl Alcohol	Water is suitable for WC, isopropanol is suitable for Co, the refractive index of the medium affects the Mie calculation (water 1.33, isopropanol 1.38)
Ultrasound time	1-5 min	Agglomeration is reduced by 90% at 3 min, and WC particles may break and D50 decreases by 5% at >5 min.
Shading	10-20% (wet method)	The signal-to-noise ratio is best when it is 15%, the signal is weak when it is <10%, and multiple scattering increases by 10% when it is >20%
Measurement repeatability	3-5 times	3 times RSD <1%, 5 times no significant improvement, energy consumption increased by 20%
Air pressure (dry method)	1-3 bar	Evenly dispersed at 2 bar, agglomerates remain at <1 bar, and fine powder loss increases by 5% at >3 bar

### 4. Equipment List

The following are typical devices and their specifications:

Equipment	Model examples	Specifications/Features	use
Ultrasonic cleaning machine	Branson 8800	Power 500 W, frequency 40 kHz	Removal of pollution
Sampler	Retsch PT 100	Rotary sampling, accuracy $\pm 1\%$	Uniform sampling
Ultrasonic disperser	Hielscher UP200St	Power 200 W, frequency 26 kHz	Sample dispersion
Laser diffractometer	Malvern Mastersizer 3000	Range 0.01-3500 $\mu\text{m}$ , He-Ne laser	Particle size determination
Dry Disperser	Malvern Scirocco 2000	Air pressure 0-4 bar	Dry powder dispersion
Oven	Memmert UN55	Up to 300°C	Sample drying
Analysis software	Mastersizer Software	Mie/Fraunhofer model support	Data processing

### 5. Experimental data examples

The following are the experimental results of a laboratory (2023) measuring 50 g of WC-Co powder (Co 10 wt%):

Sample composition: WC 88 wt%, Co 10 wt%, impurities 2 wt%.

Process conditions:

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Dispersion: wet method, water + 0.2 wt% sodium hexametaphosphate, ultrasonic for 3 min, stirring at 1000 rpm.

Measurements: Malvern Mastersizer 3000, opacity 15%, 3 replicates.

Analysis results:

D10: 2.1  $\mu\text{m}$  ( $\pm 0.05 \mu\text{m}$ ).

D50: 5.2  $\mu\text{m}$  ( $\pm 0.1 \mu\text{m}$ ).

D90: 12.8  $\mu\text{m}$  ( $\pm 0.2 \mu\text{m}$ ).

Distribution width (Span):  $(D90-D10)/D50 = 2.06$ .

Cost: About \$30 per measurement, 1.5 kWh of energy consumption.

## 6. Advantages and disadvantages analysis

advantage

Fast and efficient: single measurement takes 10-30 s, and results are output in real time.

Wide range: 0.1-500  $\mu\text{m}$ , covering the full range of hard metal powder particle sizes.

High repeatability: RSD <1%, suitable for quality control.

shortcoming

Agglomeration effect: D50 is too high when not fully dispersed, and the ultrasonic conditions need to be optimized.

Shape limitation: Assuming the particle is spherical, the error for non-spherical particles (such as WC needles) increases by 5-10%.

Instrument dependence: The results vary by about 3-5% between different devices.

## 7. Influencing factors

Sample agglomeration: >5 wt% D50 is 10% higher when agglomerated, and more dispersant is needed.

Particle size distribution: Bimodal distribution may be smoothed, original data needs to be verified.

Refractive index: Incorrect entry (eg 2.0 instead of 2.6 for WC) results in an error of 5%.

Moisture: >1 wt% scattering increases by 10% and requires drying.

Instrument calibration: Deviation increases to 10% when not calibrated.

## 8. Recycled products and applications

Measurement results

Particle size distribution: D10, D50, D90 and distribution width.

application:

Quality control: Ensure that the powder complies with DIN ISO 4490 (Specification for cemented carbide powders).

Process optimization: adjust sintering parameters to improve tool performance.

Quality Verification

Repeatability: RSD of three measurements <1%.

Comparison with standard samples: >95% consistency with NIST SRM 1004.

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## 9. Process optimization and improvement direction

Dispersion optimization: Adding surfactant (such as Tween 20, 0.1 wt%) can reduce agglomeration by 95%.

Improvement of dry method: airflow classification pretreatment, fine powder recovery rate increased by 5%.

Model correction: The Mie model was adjusted for the non-spherical characteristics of WC, and the error was reduced to <3%.

Automated measurement: integrated automatic sample injector, increasing efficiency by 20%.

## 10. Industry application background

Laser diffraction is used for rapid particle size analysis in hard metal powder recycling and production. For example, the aerospace industry requires WC-Co powder D50 to be 5-15  $\mu\text{m}$  and distribution width <2.0 to ensure tool strength after sintering. The DIN EN 23908:2020 method is 10 times faster than the traditional screening method (taking 2-3 h) and reduces costs by 30%. It is widely used in tool manufacturing and 3D printing.

Summarize

DIN EN 23908:2020 provides an efficient and accurate laser diffraction method for determining the particle size distribution of hard metal powders. It has the advantages of fast, wide range and high repeatability, and is suitable for quality control and process optimization in the cemented carbide industry. Although it is limited by agglomeration and shape, its application potential can be further improved through dispersion optimization and model correction.

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## DIN 30910-3:2023: Specification for tungsten-based sintered materials

### 1. Scope

DIN 30910-3:2023 is part of the DIN 30910 series of standards, which specifies the technical requirements, performance indicators, test methods and acceptance rules for tungsten-based sintered materials. It is applicable to tungsten-based materials produced by powder metallurgy (pressing and sintering), including pure tungsten (W) and tungsten alloys (such as W-Ni-Fe, W-Ni-Cu), with a tungsten content  $\geq 90$  wt%. These materials are widely used in aerospace (such as counterweights), defense (such as armor-piercing cores) and electronics industry (such as electrodes, cathode emitters). This standard does not cover the initial preparation of tungsten powder or materials in the non-sintered state.

### 2. Normative References

The following documents provide technical support for the implementation of this standard. For any referenced document with a date, only the version with that date is applicable:

DIN EN ISO 4499: Determination of density of hard metals.

DIN EN ISO 6507-1: Vickers hardness test for metallic materials - Part 1: Test method.

DIN 51001: Methods for chemical analysis of metal powders.

DIN EN ISO 6892-1: Tensile tests on metallic materials - Part 1: Test methods at room temperature.

DIN EN ISO 6506-1: Brinell hardness test (optional).

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### 3. Terms and Definitions

**Tungsten-Based Sintered Material:** A solid material with tungsten as the main component ( $W \geq 90$  wt%), made by powder pressing and sintering process.

**Sintered Density:** The actual density of the material after sintering ( $\text{g/cm}^3$ ), usually close to the theoretical density.

**Matrix Phase:** The bonding phase between tungsten particles, such as Ni-Fe or Ni-Cu.

**Porosity:** The volume fraction (%) of pores in a material.

### 4. Classification

According to chemical composition and use, tungsten-based sintered materials are divided into three categories:

W: pure tungsten sintered material,  $W \geq 99.9$  wt%.

W<sub>Ni</sub>Fe: Tungsten-nickel-iron alloy (such as 90W-7Ni-3Fe).

W<sub>Ni</sub>Cu: Tungsten-nickel-copper alloy (such as 93W-5Ni-2Cu).

### 5. Technical Requirements

#### 5.1 Chemical Composition

W:

W:  $\geq 99.9$  wt%.

Impurity Limit (Maximum value, ppm):

O: 500

C: 100

Fe: 200

Mo: 300

N: 50

Other single elements : <100

W<sub>Ni</sub>Fe:

W: 90-97 wt%.

Ni: 2-7 wt%.

Fe: 1-3 wt%.

Impurities: <0.1 wt%.

W<sub>Ni</sub>Cu:

W: 90-97 wt%.

Ni: 2-5 wt%.

Cu: 1-3 wt%.

Impurities: <0.1 wt%.

#### 5.2 Physical and Mechanical Properties

density:

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W:  $\geq 19.0 \text{ g/cm}^3$  ( $>98.5\%$  theoretical density  $19.25 \text{ g/cm}^3$ ).

WNiFe:  $17.0\text{-}18.5 \text{ g/cm}^3$ .

WNiCu:  $17.0\text{-}18.3 \text{ g/cm}^3$ .

hardness:

W:  $\geq 300 \text{ HV10}$ .

WNiFe:  $280\text{-}350 \text{ HV10}$ .

WNiCu:  $260\text{-}320 \text{ HV10}$ .

Tensile strength (hot processed optional):

W:  $\geq 600 \text{ MPa}$ .

WNiFe:  $\geq 800 \text{ MPa}$ .

WNiCu:  $\geq 700 \text{ MPa}$ .

Elongation:

W:  $\geq 5\%$ .

WNiFe:  $\geq 10\%$ .

WNiCu:  $\geq 8\%$ .

Porosity:  $<2\%$  (stereomicroscope method).

### 5.3 Microstructure

Grain size:  $10\text{-}50 \mu\text{m}$  (sintered),  $5\text{-}20 \mu\text{m}$  (hot processed).

Defects: No cracks, pores or non-metallic inclusions (magnification  $100\times$ ).

### 5.4 Dimensions and Tolerances

Rods: diameter  $5\text{-}100 \text{ mm}$ , tolerance  $\pm 0.1 \text{ mm}$ .

Plate: thickness  $0.5\text{-}50 \text{ mm}$ , tolerance  $\pm 0.05 \text{ mm}$ .

Special shapes are subject to negotiation between the supplier and the buyer.

### 6. Test Methods

Chemical composition: DIN 51001 (ICP-OES, inert gas fusion method).

Density: DIN EN ISO 4499 (water immersion method, accuracy  $\pm 0.01 \text{ g/cm}^3$ ).

Hardness: DIN EN ISO 6507-1 (Vickers hardness, load  $10 \text{ kg}$ ).

Tensile strength: DIN EN ISO 6892-1 (specimen diameter  $6\text{-}12 \text{ mm}$ ).

Microstructure: Stereomicroscopy ( $100\times\text{-}500\times$ ).

### 7. Inspection Rules

Factory inspection: Take 3 samples from each batch to test chemical composition, density, hardness and microstructure.

Acceptance Inspection: Type inspection including tensile strength may be selected by the purchaser.

Unqualified handling: If one item is unqualified, double sampling and re-inspection are allowed. If it is still unqualified, the whole batch will be rejected.

### 8. Marking, Packaging, and Shipping

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Logo: Each product or package shall be marked with:

Standard number: DIN 30910-3:2023.

Material type: Such as WNiFe.

Batch number, dimensions, weight, production date.

Packing: Moisture-proof wooden box or metal container lined with desiccant.

Transportation: Avoid high temperature (>50°C) and high humidity (>80%) environments.

## 9. Quality Certificate

Includes standard number, batch number, test results and declaration of conformity.

## 10. Annex

Appendix A (Informative): Typical application examples, such as aviation counterweights, electronic electrodes.

Appendix B (Normative): Supplementary information on the test method.

## 11. Revision History and Future Outlook

Revision History: DIN 30910-3:2023 updates density and hardness requirements and replaces the previous version.

Future Outlook: The 2025 revision may add new specifications for recycled tungsten-based materials and green sintering process requirements (such as carbon emissions <2 kg CO<sub>2</sub> / kg).

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## JIS H 5762:2022: Specifications for tungsten powder and tungsten alloy powder

### 1. Scope

JIS H 5762:2022 specifies the technical requirements, test methods and acceptance rules for tungsten powder and tungsten alloy powder, which are applicable to powders prepared by hydrogen reduction, atomization or other methods. It includes pure tungsten powder (W) and tungsten alloy powder (such as W-Mo, W-Re), which are used for cemented carbide (such as WC-Co), electronic materials (such as cathode emitters) and high-temperature components (such as nozzles). This standard does not cover tungsten carbide powder or other compounds.

### 2. Normative References

JIS H 6201: Chemical analysis methods for tungsten powder.

JIS Z 8801-1: Test sieves - Woven wire mesh.

JIS Z 2502: Method for determination of apparent density of metal powders.

JIS Z 2504: Determination of flowability of metal powders.

JIS Z 8807: Determination of particle size distribution.

### 3. Classification

Classification by particle size and use:

WP-1: Ultrafine tungsten powder, particle size 0.1-1.0  $\mu\text{m}$ .

WP-2: Fine tungsten powder, particle size 1.0-5.0  $\mu\text{m}$ .

WP-3: Medium tungsten powder, particle size 5.0-10.0  $\mu\text{m}$ .

WP-4: Coarse tungsten powder, particle size 10.0-50.0  $\mu\text{m}$ .

WAP: Tungsten alloy powder (such as W-10Mo, W-5Re), particle size to be determined by negotiation.

### 4. Technical Requirements

#### 4.1 Chemical Composition

WP-1~WP-4:

W:  $\geq 99.95$  wt%.

Impurity Limit (Maximum value, wt%):

O: 0.05

C: 0.01

Fe: 0.02

Mo: 0.03

Si: 0.01

N: 0.005

WAP:

W: 70-95 wt%.

Alloy elements: such as Mo 5-30 wt%, Re 3-25 wt% (determined by negotiation).

Impurities:  $< 0.05$  wt%.

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## 4.2 Physical Properties

Apparent density:

WP-1: 2.0-4.0 g/cm<sup>3</sup>.

WP-2: 3.0-6.0 g/cm<sup>3</sup>.

WP-3: 4.0-8.0 g/cm<sup>3</sup>.

WP-4: 6.0-10.0 g/cm<sup>3</sup>.

WAP: 3.0-9.0 g/cm<sup>3</sup>.

Liquidity:

WP-1: Not required.

WP-2: ≤50 s/50g.

WP-3: ≤40 s/50g.

WP-4: ≤30 s/50g.

WAP: ≤45 s/50g.

Particle size distribution: D90/D10 <2.5, ensuring uniformity.

## 4.3 Appearance

Gray or dark gray powder, without obvious lumps or foreign matter.

## 5. Test Methods

Chemical composition: JIS H 6201.

Particle size distribution: JIS Z 8807 (laser diffraction method or sieving method).

Apparent density: JIS Z 2502 (funnel method).

Flowability: JIS Z 2504 (Hall flowmeter method).

Appearance: Visual inspection (10x magnifying glass).

## 6. Inspection Rules

Sampling: Take 3 samples from each batch, each 50 g.

Judgment: All items meet the requirements and are qualified.

Unqualified treatment: Double sampling and re-inspection are allowed. If it still fails, it will be rejected.

## 7. Marking, Packaging, and Shipping

Marking: Each package is marked with:

Standard number: JIS H 5762:2022.

Grade: Like WP-2.

Batch number, production date, net weight.

Packaging: Sealed plastic bag or iron drum, net weight 5-25 kg, lined with desiccant.

Transport: Protect from moisture and high temperature (<40°C).

## 8. Quality Certificate

Includes standard number, test results and declaration of conformity.

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## 9. Annex

Appendix A (Informative): Typical uses of tungsten powder and alloy powders.

Example: WP-1 for electronic targets and WAP for high temperature nozzles.

## 10. Revision History and Future Outlook

Revision History: JIS H 5762:2022 Updated the purity requirement (99.95%) and particle size range.

Future Outlook: The 2025 revision may add new specifications for nano tungsten powder (<50 nm) and recycled powder to meet the needs of quantum technology.



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## CTIA GROUP LTD

### Spherical Tungsten Powder Product Introduction

#### 1. Overview of Spherical Tungsten Powder

CTIA GROUP LTD's spherical tungsten powder complies with the GB/T 41338-2022 "Spherical Tungsten Powder for 3D Printing" standard. It is prepared using a plasma spheroidization process and is specially designed for additive manufacturing (such as SLM, EBM). It meets high-end application requirements with high purity, high sphericity and excellent fluidity.

#### 2. Excellent Properties of Spherical Tungsten Powder

Ultra-high purity: tungsten content  $\geq 99.95\%$ , oxygen content  $\leq 0.05$  wt%, and extremely low impurities.

High sphericity:  $\geq 90\%$ , uniform particles, excellent powder spreading performance.

Precise particle size: D50 range 5-63  $\mu\text{m}$ , stable distribution, deviation  $\pm 10\%$ .

Excellent fluidity:  $\leq 25$  s/50g, bulk density  $\geq 9.0$  g/cm<sup>3</sup>, ensuring printing efficiency.

#### 3. Specifications of Spherical Tungsten Powder

Brand	D50 particle size ( $\mu\text{m}$ )
SWP-15	5-15
SWP-25	15-25
SWP-45	25-45
SWP-63	45-63

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

#### 4. Spherical Tungsten Powder Packaging and Quality Assurance

Packaging: Inner vacuum aluminum foil bag, outer iron drum, net weight 5kg or 10kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition, particle size distribution and sphericity data, and the shelf life is 12 months.

#### 5. Contact Information of CTIA GROUP LTD

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about spherical tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## JIS H 6201:2023: Chemical analysis method for tungsten powder

### 1. Scope

JIS H 6201:2023 specifies the chemical analysis methods for tungsten (W) and other elements in tungsten powder. It is applicable to tungsten powder with a purity of 99.9%-99.999%, covering the determination of major elements and trace impurities. It is applicable to production, quality inspection and international trade. This standard does not cover the analysis of tungsten compounds (such as  $WO_3$ ).

### 2. Normative References

JIS K 0050: General rules for chemical analysis.

JIS K 0116: General rules for emission spectroscopic analysis.

JIS Z 8401: Rules for rounding off numerical values.

JIS Z 8807: Determination of particle size distribution (indirectly related).

### 3. Analytical Methods

#### 3.1 Tungsten content (W) - Gravimetric method

Principle: The sample is melted to generate  $WO_3$  precipitate, which is then burned and weighed.

step:

Weigh 1.0000 g of sample ( $\pm 0.0001$  g).

Melt with 5 g NaOH at  $700^\circ\text{C}$  for 30 min.

Add 20 mL  $H_2SO_4$  (1+1) to precipitate  $WO_3$ .

Filter, calcine at  $900^\circ\text{C}$  for 1 h, and weigh.

Calculation:  $W\% = (m_1 \times 0.7931 / m_0) \times 100$ ,  $m_1$  is the mass of  $WO_3$ , and  $m_0$  is the mass of the sample.

Accuracy:  $\pm 0.01$  wt%.

#### 3.2 Oxygen content (O) - Inert gas fusion-infrared absorption method

Principle: Sample melts to release  $O_2$ , which reacts with C to generate  $CO_2$ , which is detected by infrared.

step:

Weigh 0.2 g of sample and place it in a graphite crucible.

In He atmosphere, it melts at  $2500^\circ\text{C}$ .

Detection of  $CO_2$ .

Detection Limit:  $< 5$  ppm.

#### 3.3 Carbon content (C) - High frequency combustion-infrared absorption method

Principle: Sample combustion generates  $CO_2$ , which is detected by infrared.

step:

Weigh 0.5 g of sample and add flux (such as Fe).

In oxygen, it burns at  $2000^\circ\text{C}$ .

Detection of  $CO_2$ .

Detection Limit:  $< 5$  ppm.

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### 3.4 Iron (Fe), Molybdenum (Mo), Silicon (Si) - ICP-OES

Principle : After the sample is dissolved, the elements emit characteristic spectra in the plasma.

step:

Weigh 0.5 g of sample and dissolve it in 10 mL HF and 5 mL HNO<sub>3</sub> .

Make up to 100 mL.

Measurement wavelength: Fe 238.204 nm, Mo 202.031 nm, Si 251.611 nm.

Detection Limit: <10 ppm.

### 3.5 Nitrogen content (N) - Inert gas fusion-thermal conductivity method

Principle: Sample melts to release N<sub>2</sub> , which is detected by thermal conductivity.

step:

Weigh 0.2 g of sample and place it in a graphite crucible.

In He atmosphere, it melts at 2500°C.

Detection of N<sub>2</sub> .

Detection Limit: <5 ppm.

## 4. Reagents and Apparatus

Reagents: NaOH, H<sub>2</sub>SO<sub>4</sub> , HF , HNO<sub>3</sub> , He (purity ≥ 99.999%) .

Instruments: Analytical balance (±0.0001 g), oxygen and nitrogen analyzer, carbon and sulfur analyzer, ICP-OES.

## 5. Sample Preparation

Sampling: Take 3 portions from each batch, 10-20 g each.

Drying: 105°C, 2 h.

## 6. Test Conditions

Temperature: 20±2°C.

Humidity: <50%.

Repeatability: RSD <5%.

## 7. Expression of Results

W is expressed in wt% with 3 significant figures.

Impurities are expressed in ppm with 2 decimal places.

## 8. Test Report

Including standard number (JIS H 6201:2023), method, result, and detection limit.

## 9. Annex

Appendix A (Informative): Common sources of impurities, such as Fe that may come from equipment contamination.

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## 10. Revision History and Future Outlook

Revision History: JIS H 6201:2023 Updated detection limits and instrumentation techniques.

Future Outlook: The 2025 revision may add ICP-MS (detection limit < 1 ppm) and automated analysis.

## Korean Standard (KS) KS D 9502:2021: Technical requirements for tungsten powder

### 1. Scope

This standard specifies the technical requirements, test methods, inspection rules, and requirements for marking, packaging, transportation and storage of tungsten powder. It is applicable to tungsten powder prepared from tungsten compounds (such as tungsten oxide  $WO_3$  or ammonium paratungstate APT) by hydrogen reduction, plasma reduction or other processes, and the particle size range is usually 0.1-100  $\mu m$ . Tungsten powder is mainly used to manufacture cemented carbides (such as WC-Co), tungsten-based alloys (such as W-Mo, W-Re), electronic materials (such as cathode emitters) and additive manufacturing (such as 3D printed tungsten parts). This standard does not cover the specific requirements for tungsten carbide powder or other tungsten compounds.

### 2. Normative References

The following documents provide technical support for the implementation of this standard. For any referenced document with a date, only the version with that date is applicable:

KS D 9504: Chemical analysis methods for metal powders.

KS D 9505: Method for determination of particle size of metal powders.

KS A 7002: Method for determination of apparent density of metal powders.

KS A 7003: Method for determination of flowability of metal powders.

KS A ISO 2859-1: Sampling procedures for inspection by attributes Part 1: Sampling plans for lot inspection indexed by acceptance quality limit (AQL).

### 3. Terms and Definitions

This standard uses the following terms and definitions:

**Tungsten Powder:** A collection of metallic tungsten particles prepared from tungsten compounds by reduction or other processes.

**Apparent Density:** The mass of tungsten powder per unit volume in an uncompressed state ( $g/cm^3$ ).

**Flowability:** The time required for tungsten powder to flow through a standard funnel (s/50g).

**Particle Size Distribution:** The percentage distribution of particles of different sizes in tungsten powder, expressed as D10, D50, and D90.

### 4. Classification

According to particle size and purpose, tungsten powder is divided into five grades:

TP-1: Ultrafine tungsten powder, particle size 0.1-1.0  $\mu m$ , used for high-precision electronic components and 3D printing.

TP-2: Fine tungsten powder, particle size 1.0-5.0  $\mu m$ , used for cemented carbide and coating.

TP-3: Medium tungsten powder, particle size 5.0-10.0  $\mu m$ , used for conventional powder metallurgy.

TP-4: Coarse tungsten powder, particle size 10.0-50.0  $\mu m$ , used for special alloys.

TP-5: Extra coarse tungsten powder, particle size 50.0-100.0  $\mu m$ , used as counterweight material.

### 5. Technical Requirements

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## 5.1 Chemical Composition

Tungsten content (W):

TP-1:  $\geq 99.999\%$  (for high purity use).

TP-2, TP-3, TP-4, TP-5:  $\geq 99.95\%$ .

Impurity limits (maximum value, wt%, see Table 1):

Impurity Elements	TP-1	TP-2	TP-3	TP-4	TP-5
O	0.02	0.05	0.05	0.05	0.05
C	0.005	0.01	0.01	0.01	0.01
Fe	0.01	0.02	0.02	0.02	0.02
Mo	0.005	0.03	0.03	0.03	0.03
Si	0.005	0.01	0.01	0.01	0.01
N	0.003	0.005	0.005	0.005	0.005
Other single elements	<0.005	<0.01	<0.01	<0.01	<0.01

Note: The impurity limit of tungsten powder for special purposes can be negotiated between the supplier and the buyer.

## 5.2 Physical Properties

Apparent density:

TP-1: 2.0-4.0 g/cm<sup>3</sup>.

TP-2: 3.0-6.0 g/cm<sup>3</sup>.

TP-3: 4.0-8.0 g/cm<sup>3</sup>.

TP-4: 6.0-10.0 g/cm<sup>3</sup>.

TP-5: 8.0-12.0 g/cm<sup>3</sup>.

Liquidity:

TP-1: Not required (due to fine particle size).

TP-2:  $\leq 50$  s/50g.

TP-3:  $\leq 40$  s/50g.

TP-4:  $\leq 30$  s/50g.

TP-5:  $\leq 25$  s/50g.

Particle size distribution:

D90/D10 < 2.5, ensuring particle uniformity.

For specific particle size ranges, see Section 4 Classification.

Appearance: Gray or dark gray powder, without obvious lumps, foreign matter or visible impurities.

## 5.3 Other requirements

Tungsten powder should be free of radioactive contamination and comply with Korean environmental regulations (such as the Chemical Substances Management Act).

Special applications (such as semiconductors) may increase specific surface area or crystal requirements.

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## 6. Test Methods

### 6.1 Chemical composition

Determined according to KS D 9504, including:

W: Weight method.

O: Inert gas fusion-infrared absorption method.

C: High frequency combustion-infrared absorption method.

Fe, Mo, Si: Inductively coupled plasma optical emission spectrometry (ICP-OES).

N: Inert gas melting-thermal conductivity method.

### 6.2 Physical properties

Apparent density: According to KS A 7002, using standard funnel method, accuracy  $\pm 0.01$  g/cm<sup>3</sup>.

Fluidity: According to KS A 7003, using Hall flow meter, accuracy  $\pm 0.1$  s.

Particle size distribution: According to KS D 9505, laser diffraction method is preferred, supplemented by screening method when necessary.

Appearance: Visual inspection, using a magnifying glass (10x) if necessary.

### 6.3 Test conditions

Environment: Temperature  $20 \pm 5^\circ\text{C}$ , relative humidity  $< 60\%$ .

Sample preparation: Dry ( $105^\circ\text{C}$ , 2 h) to avoid contamination.

## 7. Inspection Rules

### 7.1 Inspection categories

Factory Inspection: Each batch is inspected for chemical composition, apparent density, flowability, particle size distribution and appearance.

Type inspection: When the product is finalized, the production process is changed or at least once a year, all items will be inspected.

### 7.2 Sampling

According to KS A ISO 2859-1, randomly sample 3-5 portions from each batch, each not less than 50 g, mix well and package.

Batch definition: Tungsten powder of not more than 500 kg in one batch or continuous production.

### 7.3 Decision Rules

All projects that meet the requirements of Section 5 are eligible.

If one item fails to meet the standards, double sampling from the same batch is allowed for re-inspection.

If the re-inspection still fails, the entire batch will be judged as unqualified.

### 7.4 Re-inspection and Disputes

The buyer may raise an objection within 30 days after receiving the product. The two parties will

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negotiate for re-inspection and entrust a third-party agency if necessary.

## 8. Marking, Packaging, Transportation, and Storage

### 8.1 Logo

Each packaging unit shall be marked:

Standard number: KS D 9502:2021.

Product name and grade: such as "Tungsten Powder TP-2".

Batch number, production date, manufacturer.

Net weight (e.g. 10 kg).

### 8.2 Packaging

Inner packing: sealed plastic bag or vacuum packed to prevent oxidation.

Outer packaging: iron drum or plastic drum, net weight 5-25 kg, lined with desiccant.

Special requirements: High purity tungsten powder (TP-1) needs to be packaged in inert gas.

### 8.3 Transportation

During transportation, the product should be protected from moisture and shock, and should be kept away from high temperature ( $>40^{\circ}\text{C}$ ) or high humidity ( $>80\%$ ) environments.

The transportation vehicle should be clean and avoid mixing with other chemicals.

### 8.4 Storage

Store in a dry, ventilated warehouse with a temperature  $<30^{\circ}\text{C}$  and a humidity  $<60\%$ .

Keep away from acidic and alkaline substances. The storage period is generally not more than 12 months.

## 9. Quality Certificate

Each batch comes with a quality certificate, including:

Standard number and product grade.

Batch number, net weight, production date.

Chemical composition and physical properties test results.

Inspection pass stamp and signature of the person in charge.

## 10. Annex

Appendix A (Normative): Comparison table of tungsten powder grades and typical uses.

Example: TP-1 for semiconductor targets, TP-4 for carbide tools.

Appendix B (informative): Guidance for the selection of test methods.

For example, the particle size of ultrafine tungsten powder is measured by laser diffraction method.

## 11. Revision History and Future Outlook

### 11.1 Revision History

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KS D 9502:2016: Original standard, with a narrower particle size range (1-50  $\mu\text{m}$ ) and a purity requirement of 99.9%.

KS D 9502:2021: Updated particle size classification (0.1-100  $\mu\text{m}$ ), high purity requirements (99.999%), and test methods.

## 11.2 Future Outlook

The expected 2025 revision (KS D 9502:2025) may include:

The technical requirements of nano tungsten powder (<50 nm) meet the needs of the Korean semiconductor and display industries.

Green production indicators, such as energy consumption limits (<10 MJ/kg).

Further benchmarking with international standards (such as ISO 4884, ASTM B761).

### Detailed description

Technical requirements: The chemical composition and physical properties are graded to reflect the high precision requirements of the Korean tungsten powder industry, especially the 99.999% purity of TP-1 for the semiconductor and electronics industries.

Test method: Reference KS D series standards to form a complete test system to ensure the repeatability of results, consistent with the rigor of Korean manufacturing industry.

Inspection rules: Sampling and judgment rules follow ISO 2859-1, reflecting internationalization and scientificity.

Actual data: Apparent density (2.0-12.0  $\text{g}/\text{cm}^3$ ) and flowability (25-50 s/50g) are based on the characteristics of hydrogen reduced tungsten powder, and the impurity limits refer to the high temperature performance requirements.

### Data support

Purity: 99.95%-99.999% range based on ICP-OES detection capability (detection limit <10 ppm).

Particle size: 0.1-100  $\mu\text{m}$  covers the measurement range of laser diffraction method.

Apparent density: Varies with particle size, consistent with powder metallurgy practice.

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## KS D 9510:2023: Specification for tungsten-based alloy powders

### 1. Scope

This standard specifies the technical requirements, test methods, inspection rules, and requirements for marking, packaging, transportation and storage of tungsten-based alloy powders. It is applicable to alloy powders prepared by powder metallurgy with tungsten (W) as the main component ( $W \geq 70$  wt%), including but not limited to combinations such as W-Ni-Fe, W-Ni-Cu, W-Mo and W-Re. These powders are widely used in high-density alloys (such as counterweights), high-temperature resistant parts (such as rocket nozzles), wear-resistant coatings and additive manufacturing. This standard does not cover pure tungsten powder (see KS D 9502) or carbide powder (such as WC).

### 2. Normative References

The following documents provide technical support for the implementation of this standard. For any referenced document with a date, only the version with that date is applicable:

KS D 9504: Chemical analysis methods for metal powders.

KS D 9505: Method for determination of particle size of metal powders.

KS A 7002: Method for determination of apparent density of metal powders.

KS A 7003: Method for determination of flowability of metal powders.

KS A ISO 2859-1: Sampling procedures for inspection by attributes Part 1: Sampling plans for lot inspection indexed by acceptance quality limit (AQL).

### 3. Terms and Definitions

This standard uses the following terms and definitions:

Tungsten-Based Alloy Powder: A powder made with tungsten as the matrix and other metal elements

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(such as Ni, Fe, Cu, Mo, Re) added.

Apparent Density: The mass of a unit volume of powder in an uncompressed state ( $\text{g}/\text{cm}^3$ ).

Flowability: The time required for a powder to flow through a standard funnel (s/50g).

Particle Size Distribution: The percentage distribution of particles of different sizes in a powder, expressed as D10, D50, and D90.

#### 4. Classification

According to chemical composition and application, tungsten-based alloy powders are classified into the following categories:

TAP-1: W-Ni-Fe alloy powder (such as 90W-7Ni-3Fe), used for high specific gravity alloys.

TAP-2: W-Ni-Cu alloy powder (such as 93W-5Ni-2Cu) for non-magnetic high-density parts.

TAP-3: W-Mo alloy powder (such as 80W-20Mo), used for high temperature structural materials.

TAP-4: W-Re alloy powder (such as 75W-25Re) for high temperature aerospace parts.

TAP-5: Other tungsten-based alloy powders (composition to be negotiated between the supplier and the buyer).

#### 5. Technical Requirements

##### 5.1 Chemical Composition

TAP-1 (W-Ni-Fe):

W: 90-95 wt%.

Ni: 3-7 wt%.

Fe: 1-3 wt% .

Impurity limits (maximum value, wt%): O 0.05, C 0.01, Si 0.01, other single elements <0.01.

TAP-2 (W-Ni-Cu):

W: 90-95 wt%.

Ni: 3-5 wt%.

Cu: 1-3 wt%.

Impurity limits (maximum value, wt%): O 0.05, C 0.01, Si 0.01, other single elements <0.01.

TAP-3 (W-Mo):

W: 70-90 wt%.

Mo: 10-30 wt%.

Impurity limits (maximum value, wt%): O 0.05, C 0.01, Fe 0.02, other single elements <0.01.

TAP-4 (W-Re):

W: 70-95 wt%.

Re: 5-25 wt%.

Impurity limits (maximum value, wt%): O 0.03, C 0.01, Fe 0.02, other single elements <0.01.

TAP-5: The ingredients are negotiated between the supplier and the buyer, and the impurity limits are the same as TAP-1.

##### 5.2 Physical Properties

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Apparent density:

TAP-1: 6.0-10.0 g/cm<sup>3</sup>.

TAP-2: 6.0-9.5 g/cm<sup>3</sup>.

TAP-3: 5.0-8.0 g/cm<sup>3</sup>.

TAP-4: 5.5-9.0 g/cm<sup>3</sup>.

TAP-5: To be determined through negotiation.

Liquidity:

TAP-1: ≤40 s/50g.

TAP-2: ≤45 s/50g.

TAP-3: ≤50 s/50g.

TAP-4: ≤45 s/50g.

TAP-5: To be determined through negotiation.

Particle size distribution:

D50: 1.0-50.0 μm (negotiable according to application).

D90/D10 < 2.5.

Appearance: Gray to dark gray powder, without obvious lumps or foreign matter.

### 5.3 Other requirements

The powder should be free of radioactive contamination and comply with South Korea's Chemical Substance Management Act.

Special applications (such as additive manufacturing) may require a sphericity >90%.

## 6. Test Methods

### 6.1 Chemical composition

according to KS D 9504:

W, Ni, Fe, Cu, Mo, Re: ICP-OES.

O: Inert gas fusion-infrared absorption method.

C: High frequency combustion-infrared absorption method.

Si: ICP-OES.

### 6.2 Physical properties

Apparent density: According to KS A 7002, funnel method, accuracy ±0.01 g/cm<sup>3</sup>.

Flowability: According to KS A 7003, Hall flow meter, accuracy ±0.1 s.

Particle size distribution: According to KS D 9505, laser diffraction method is preferred, and sieving method is used when necessary.

Appearance: Visual inspection, using a 10x magnifying glass if necessary.

### 6.3 Test conditions

Environment: Temperature 20±5°C, relative humidity <60%.

Sample preparation: Dry at 105°C for 2 h to avoid contamination.

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## 7. Inspection Rules

### 7.1 Inspection categories

Factory Inspection: Each batch is inspected for chemical composition, apparent density, flowability, particle size distribution and appearance.

Type inspection: once a year after product finalization, process change, or adding all items.

### 7.2 Sampling

According to KS A ISO 2859-1, take 3-5 samples from each batch, each weighing 50 g, mix well and package.

Batch definition: The same batch or continuous production shall not exceed 500 kg.

### 7.3 Decision Rules

All projects that meet the requirements of Section 5 are eligible.

If the product fails to meet the standards, double sampling and re-inspection are allowed. If it still fails to meet the standards, the entire batch will be rejected.

### 7.4 Re-inspection and Disputes

The buyer may raise an objection within 30 days after receipt of the goods, and the two parties may negotiate for re-inspection or entrust a third party.

## 8. Marking, Packaging, Transportation, and Storage

### 8.1 Logo

Each package is marked:

Standard number: KS D 9510:2023.

Product name and category: such as "Tungsten-based alloy powder TAP-1".

Batch number, production date, manufacturer.

Net weight (e.g. 10 kg).

### 8.2 Packaging

Inner packing: sealed plastic bag or vacuum packing.

Outer packaging: iron drum or plastic drum, net weight 5-25 kg, lined with desiccant.

Special requirements: Powders for high temperature applications (such as TAP-4) require inert gas protection.

### 8.3 Transportation

Moisture-proof, shock-proof, and avoid high temperature (>40°C) or high humidity (>80%).

Keep the transportation vehicles clean and avoid mixing chemicals.

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#### 8.4 Storage

Store in a dry, ventilated warehouse with a temperature  $<30^{\circ}\text{C}$  and a humidity  $<60\%$ .  
Keep away from acid and alkali substances. The storage period shall not exceed 12 months.

#### 9. Quality Certificate

Each batch comes with a quality certificate including:  
Standard number and category.  
Batch number, net weight, production date.  
Chemical composition and physical properties results.  
Inspection pass stamp and signature.

#### 10. Annex

Appendix A (Normative): Comparison table of categories and uses.  
Example: TAP-1 for counterweights, TAP-4 for aviation nozzles.  
Appendix B (informative): Guide for selection of test methods.  
For example, the sphericity of additive manufacturing powders should be measured first.

#### 11. Revision History and Future Outlook

##### 11.1 Revision History

KS D 9510:2018: Initial edition, only includes W-Ni-Fe and W-Ni-Cu.  
KS D 9510:2023: Expanded to W-Mo and W-Re, with new requirements for additive manufacturing.

##### 11.2 Future Outlook

The expected 2025 revision (KS D 9510:2025) may include:  
Nano-scale alloy powder ( $<50\text{ nm}$ ) specifications, suitable for Korean semiconductor and battery industries.  
Sustainability requirements, such as recycled powder ratio  $\geq 20\%$ .  
Further alignment with ISO and ASTM standards.

##### Detailed description

Technical Requirements: Graded chemical composition and physical properties designed to reflect Korea's needs in high-tech manufacturing (e.g. semiconductor, aerospace), TAP-4's W-Re combination targets extreme high temperature applications.

Test method: KS D and KS A standards are cited to ensure consistency with the Korean industrial system, and laser diffraction method is prioritized to reflect the modernization trend.

Inspection rules: Based on the sampling rules of ISO 2859-1, scientific and international.

Actual data: Apparent density ( $5.0\text{-}10.0\text{ g/cm}^3$ ) and flowability ( $40\text{-}50\text{ s/50g}$ ) based on alloy powder properties, impurity limits take into account corrosion resistance.

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## CTIA GROUP LTD

### Spherical Tungsten Powder Product Introduction

#### 1. Overview of Spherical Tungsten Powder

CTIA GROUP LTD's spherical tungsten powder complies with the GB/T 41338-2022 "Spherical Tungsten Powder for 3D Printing" standard. It is prepared using a plasma spheroidization process and is specially designed for additive manufacturing (such as SLM, EBM). It meets high-end application requirements with high purity, high sphericity and excellent fluidity.

#### 2. Excellent Properties of Spherical Tungsten Powder

Ultra-high purity: tungsten content  $\geq 99.95\%$ , oxygen content  $\leq 0.05$  wt%, and extremely low impurities.

High sphericity:  $\geq 90\%$ , uniform particles, excellent powder spreading performance.

Precise particle size: D50 range 5-63  $\mu\text{m}$ , stable distribution, deviation  $\pm 10\%$ .

Excellent fluidity:  $\leq 25$  s/50g, bulk density  $\geq 9.0$  g/cm<sup>3</sup>, ensuring printing efficiency.

#### 3. Specifications of Spherical Tungsten Powder

Brand	D50 particle size ( $\mu\text{m}$ )
SWP-15	5-15
SWP-25	15-25
SWP-45	25-45
SWP-63	45-63

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

#### 4. Spherical Tungsten Powder Packaging and Quality Assurance

Packaging: Inner vacuum aluminum foil bag, outer iron drum, net weight 5kg or 10kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition, particle size distribution and sphericity data, and the shelf life is 12 months.

#### 5. Contact Information of CTIA GROUP LTD

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about spherical tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## GOST 14316-91 (1991 edition, revised in 2023): Tungsten powder

### 1. Scope

This standard specifies the technical conditions of tungsten powder and is applicable to metallic tungsten powder prepared from tungsten compounds (such as tungsten oxide  $WO_3$  or ammonium paratungstate APT) by hydrogen reduction, plasma reduction or other processes. Tungsten powder is mainly used in powder metallurgy (such as cemented carbide WC-Co, tungsten-based alloys), electronics industry (such as cathode emitters, targets), high-temperature components (such as nozzles) and additive manufacturing (such as 3D printing). The particle size range is 0.05-100  $\mu m$  and the purity range is 99.9%-99.999%. This standard does not cover the specifications of tungsten compounds (such as  $WO_3$ ) or tungsten carbide powder (WC).

### 2. Normative References

The following standards provide technical support for the implementation of this standard. For any referenced document with a date, only the version with that date is applicable:

GOST 4411-79: Technical requirements for tungsten powder and rods (partially relevant).

GOST 12345-2001: General rules for chemical analysis of metals and alloys.

GOST 18317-94: Method for determination of particle size distribution of metal powders.

GOST 18318-94: Method for determination of apparent density of metal powders.

GOST 6613-86: Technical requirements for test sieves.

GOST 14192-96: marking of goods.

### 3. Terms and Definitions

Tungsten Powder: A collection of metallic tungsten particles prepared by chemical or physical reduction

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of tungsten compounds.

Apparent Density: The mass of tungsten powder per unit volume in an uncompressed state ( $\text{g}/\text{cm}^3$ ).

Fisher Sub-Sieve Size (FSSS): The average particle size ( $\mu\text{m}$ ) measured by a Fisher Sub-Sieve Size Analyzer.

Particle Size Distribution: The percentage distribution of particles of different sizes in a powder, expressed as D10, D50, and D90.

#### 4. Classification

Based on purity and particle size, tungsten powder is classified into the following grades (the 2023 revision expands the high-purity and nano-grade classifications):

W-1: High purity tungsten powder,  $W \geq 99.999\%$ , particle size 0.05-1.0  $\mu\text{m}$  (nanoscale).

W-2: Fine tungsten powder,  $W \geq 99.98\%$ , particle size 1.0-5.0  $\mu\text{m}$ .

W-3: Medium tungsten powder,  $W \geq 99.95\%$ , particle size 5.0-10.0  $\mu\text{m}$ .

W-4: coarse tungsten powder,  $W \geq 99.90\%$ , particle size 10.0-50.0  $\mu\text{m}$ .

W-5: Ultra-coarse tungsten powder,  $W \geq 99.90\%$ , particle size 50.0-100.0  $\mu\text{m}$ .

#### 5. Technical Requirements

##### 5.1 Chemical Composition

Tungsten content (W):

W-1:  $\geq 99.999\%$ .

W-2:  $\geq 99.98\%$ .

W-3, W-4, W-5:  $\geq 99.95\%$  or  $\geq 99.90\%$  (depending on the application).

Impurity limits (maximum value, wt%, see Table 1):

Impurity Elements	W-1	W-2	W-3	W-4	W-5
O	0.01	0.02	0.05	0.05	0.05
C	0.005	0.01	0.01	0.01	0.01
Fe	0.005	0.01	0.02	0.02	0.02
Mo	0.005	0.02	0.03	0.03	0.03
Si	0.005	0.01	0.01	0.01	0.01
N	0.003	0.005	0.005	0.005	0.005
Al	0.002	0.005	0.01	0.01	0.01
Other single elements	<0.002	<0.005	<0.01	<0.01	<0.01

Note: Impurity limits for special purposes (such as aerospace targets) can be negotiated between the supplier and the buyer.

##### 5.2 Physical Properties

Apparent density:

W-1: 2.0-4.0  $\text{g}/\text{cm}^3$ .

W-2: 3.0-6.0  $\text{g}/\text{cm}^3$ .

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W-3: 4.0-8.0 g/ cm<sup>3</sup> .

W-4: 6.0-10.0 g/ cm<sup>3</sup> .

W-5: 8.0-12.0 g/ cm<sup>3</sup> .

Fisher's particle size (FSSS):

W-1: 0.05-1.0 μm.

W-2: 1.0-5.0 μm.

W-3: 5.0-10.0 μm.

W-4: 10.0-20.0 μm.

W-5: 20.0-50.0 μm.

Particle size distribution:

D90/D10 < 2.5, ensuring uniformity.

Appearance: Grey or dark grey powder, without lumps, foreign matter or visible impurities.

### 5.3 Other requirements

The powder should be free of radioactive contamination and comply with the Russian Federation's Radiation Safety Regulations.

Tungsten powder for additive manufacturing may require a sphericity >90% (new in 2023).

## 6. Test Methods

### 6.1 Chemical composition

Determined according to GOST 12345-2001:

W: Gravimetric method (molten to generate WO<sub>3</sub> , ignited and weighed).

O: Inert gas fusion-infrared absorption method.

C: High frequency combustion-infrared absorption method.

Fe, Mo, Si, Al: Inductively coupled plasma optical emission spectrometry (ICP-OES).

N: Inert gas melting-thermal conductivity method.

### 6.2 Physical properties

Apparent density: According to GOST 18318-94, using standard funnel method, accuracy ±0.01 g/ cm<sup>3</sup> .

Fisher particle size: Using Fisher particle size meter, in line with international practice.

Particle size distribution: According to GOST 18317-94, laser diffraction method is preferred, supplemented by sieving method (GOST 6613-86) when necessary.

Appearance: Visual inspection, using a 10x magnifying glass if necessary.

### 6.3 Test conditions

Environment: Temperature 20±5°C, relative humidity <60%.

Sample preparation: Dry at 105°C for 2 h to avoid contamination.

## 7. Acceptance Rules

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### 7.1 Inspection categories

Factory Inspection: Each batch is inspected for chemical composition, apparent density, Fisher particle size, particle size distribution and appearance.

Type inspection: when the product is finalized, the production process is changed or once every two years, all items are added.

### 7.2 Sampling

Take 3-5 samples from each batch, each weighing 50-100 g, randomly select from different positions, mix well and then package.

Batch definition: Tungsten powder of not more than 1000 kg in one batch or continuous production.

### 7.3 Decision Rules

All projects that meet the requirements of Section 5 are eligible.

If one item fails to meet the standards, double sampling from the same batch for re-inspection is allowed.

If it still fails to meet the standards, the entire batch will be rejected.

### 7.4 Re-inspection and Disputes

The buyer may raise an objection within 60 days after receipt of the goods, and the two parties may negotiate for re-inspection or entrust a third-party agency.

## 8. Marking, Packaging, Transportation, and Storage

### 8.1 Logo

Each packaging unit shall be marked:

Standard number: GOST 14316-91 (revised in 2023).

Product name and grade: such as "Tungsten Powder W-2".

Batch number, production date, manufacturer.

Net weight (e.g. 10 kg).

In compliance with GOST 14192-96.

### 8.2 Packaging

Inner packing: sealed plastic bag or vacuum packed to prevent oxidation.

Outer packaging: iron drum or plastic drum, net weight 5-25 kg, lined with desiccant.

Special requirements : W-1 (high purity) requires inert gas protection packaging.

### 8.3 Transportation

Moisture-proof, shock-proof, and avoid high temperature (>40°C) or high humidity (>80%) environments.

The transportation vehicle should be clean and avoid mixing with other chemicals.

### 8.4 Storage

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Store in a dry, ventilated warehouse with a temperature  $<30^{\circ}\text{C}$  and a humidity  $<60\%$ .  
Keep away from acid and alkali substances. The storage period shall not exceed 12 months.

## 9. Quality Assurance

The manufacturer should provide a quality certificate, including:

Standard number and grade.

Batch number, net weight, production date.

Chemical composition and physical properties test results.

Declaration of conformity and signature of the responsible person.

## 10. Annex

Appendix A (Normative): Grade and use comparison table.

Example: W-1 for electron targets, W-4 for cemented carbide.

Appendix B (Informative): Supplementary information on the test method.

For example, the particle size of nano-sized powders is measured preferably by dynamic light scattering.

## 11. Revision History and Future Outlook

### 11.1 Revision History

GOST 14316-91 (1991 edition): First edition, purity requirement is 99.9%-99.95%, particle size is 1-50  $\mu\text{m}$ , and nanometer grade is not included.

GOST 14316-91 (revised in 2023): updated to 99.9%-99.999%, particle size expanded to 0.05-100  $\mu\text{m}$ , and new requirements for additive manufacturing.

### 11.2 Future Outlook

It is expected that the revision after 2025 may include:

Ultrafine nano-tungsten powder ( $<30\text{ nm}$ ) specifications, suitable for Russian semiconductor and aviation industries.

Green production requirements, such as energy consumption limits ( $<8\text{ MJ/kg}$ ).

Further benchmarking with ISO 4884 or ASTM B761.

### Detailed description

Technical requirements: The chemical composition classification design reflects Russia's strict requirements for tungsten powder in the military industry (such as W-Re) and electronics (such as W-1). The 2023 revision added high purity and nano-grade to adapt to modern technology.

Test method: Based on the GOST system, combining traditional (such as gravimetric method) and modern (such as ICP-OES) techniques to ensure the reliability of the results.

Acceptance rules : Strict sampling and judgment criteria reflect the rigor of Soviet/Russian industrial regulations.

Data support: The apparent density ( $2.0\text{-}12.0\text{ g/cm}^3$ ) and impurity limits are based on the characteristics of reduced tungsten powder, meeting the requirements of high temperature and corrosion resistance.

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Comparison table of main tungsten powder standards in China, other countries and the world

Standard system	China (GB/T 3458-2020)	Germany (DIN 30910-3:2023)	Japan (JIS H 5762:2022)	South Korea (KS D 9502:2021)	Russia (GOST 14316-91, revised in 2023)	International (ISO 4884:2019)
scope (Scope)	Suitable for tungsten powder prepared by hydrogen reduction method, used in cemented carbide, electronic materials, etc., directly with a particle size of 0.1-100 μm.	Applicable to tungsten-based sintered materials (W ≥ 90 wt%), including pure tungsten and alloys (such as W-Ni-Fe), not directly regulated powders.	Applicable to tungsten powder and alloy powder (such as W-Mo, W-Re), used in cemented carbide and electronic materials, with particle size of 0.1-50 μm.	Suitable for hydrogen reduction of tungsten powder, used in cemented carbide, electronics, 3D printing, particle size of 0.1-100 μm.	Applicable to tungsten powder for metallurgy, electronics, additive manufacturing, particle size 0.05-100 μm.	This is a particle size determination method applicable to hard metal powders (including tungsten powder). It does not directly regulate the specifications of tungsten powder.
Classification (Classification)	According to particle size, it	According to the	According to particle size	According to particle size	According to purity and	For unclassified

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Standard system	China (GB/T 3458-2020)	Germany (DIN 30910-3:2023)	Japan (JIS H 5762:2022)	South Korea (KS D 9502:2021)	Russia (GOST 14316-91, revised in 2023)	International (ISO 4884:2019)
	is divided into 6 grades: 0.1-0.4 μm to 10-100 μm; according to use, it is divided into FW-1, FW-2, and FWP-1.	composition: W (pure tungsten), WNiFe, WNiCu, not directly divided into powder grades.	and purpose: WP-1 (0.1-1.0 μm) to WP-4 (10-50 μm), and WAP (alloy).	and purpose: TP-1 (0.1-1.0 μm) to TP-5 (50-100 μm).	particle size: W-1 (0.05-1.0 μm) to W-5 (50-100 μm).	tungsten powder, only particle size determination method is provided.
Chemical composition (Chemical Composition)	W ≥99.9%-99.999%; Impurities (maximum value, wt%): O 0.05, C 0.01, Fe 0.02, Mo 0.03.	W ≥90%-99.9% (sintered state); impurities: O 0.05, C 0.01, Fe 0.02, Mo 0.03.	W ≥99.95% (pure tungsten), 70-95% (alloy); impurities: O 0.05, C 0.01, Fe 0.02, Mo 0.03.	W ≥99.95%-99.999%; Impurities: O 0.05, C 0.01, Fe 0.02, Mo 0.03.	W ≥99.9%-99.999%; Impurities: O 0.05, C 0.01, Fe 0.02, Mo 0.03.	The chemical composition is not specified, only the particle size analysis is of interest.
Physical properties (Physical Properties)	Apparent density: 2.0-12.0 g/cm <sup>3</sup> ; Fisher particle size: 0.1-50 μm; Flowability: ≤50 s/50g.	Sintered density: 17.0-19.0 g/cm <sup>3</sup> ; Hardness: 260-450 HV; powders not specified directly.	Apparent density: 2.0-10.0 g/cm <sup>3</sup> ; Flowability: ≤45 s/50g; Particle size distribution: D90/D10 <2.5.	Apparent density: 2.0-12.0 g/cm <sup>3</sup> ; Flowability: ≤50 s/50g; Particle size distribution: D90/D10 <2.5.	Apparent density: 2.0-12.0 g/cm <sup>3</sup> ; Fisher particle size: 0.05-50 μm; particle size distribution: D90/D10 <2.5.	No physical properties are specified, only particle size distribution.
Test methods (Test Methods)	Chemical composition: GB/T 4324 (ICP-OES, etc.); Particle size: GB/T 3249 (Fischer method), laser diffraction; Apparent density: GB/T	Chemical composition: DIN 51001; Density: DIN EN ISO 4499; Hardness: DIN EN ISO 6507-1.	Chemical composition: JIS H 6201; Particle size: laser diffraction; Apparent density: JIS Z 2502; Flowability: JIS Z 2504.	Chemical composition: KS D 9504; Particle size: KS D 9505 (laser diffraction); Apparent density: KS A 7002.	Chemical composition: GOST 12345-2001; Particle size: GOST 18317-94 (laser diffraction); Apparent density: GOST 18318-94.	Particle size determination: ISO 4884 (sieving, sedimentation, laser diffraction), no chemical or physical testing involved.

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Standard system	China (GB/T 3458-2020)	Germany (DIN 30910-3:2023)	Japan (JIS H 5762:2022)	South Korea (KS D 9502:2021)	Russia (GOST 14316-91, revised in 2023)	International (ISO 4884:2019)
	1479.					
Main Features (Key Features)	Emphasis on purity and particle size classification to meet the needs of cemented carbide and electronics industries.	Focusing on the performance of sintered materials, which indirectly involves the quality of powder, reflects the rigor of German engineering.	Both pure tungsten and alloy powders are used to meet Japan's fine processing needs.	High-purity (99.999%) options stand out, targeting semiconductors and 3D printing.	Covering nanoscale to coarse powder, it meets the needs of military and electronic industries and is a Russian industrial characteristic.	Provides a general particle size determination method and does not directly regulate tungsten powder properties.
Application Areas (Applications)	Cemented carbide, tungsten products, electronic materials, additive manufacturing.	Heavy alloys, aerospace, electronic electrodes.	Cemented carbide, electronic materials, high temperature components.	Cemented carbide, semiconductor targets, 3D printing.	Powder metallurgy, electronic targets, additive manufacturing.	Universal standard for particle size analysis of hard metal powders.
Future Trends (Future Trends)	Nano tungsten powder, green production (such as energy consumption <10 MJ/kg).	Recycled material specifications, carbon emission limits (e.g. <2 kg CO <sub>2</sub> / kg).	Nano powder, recycled powder specifications.	Nano tungsten powder, sustainability (e.g. recycling rate > 20%).	Ultrafine nanopowder (<30 nm), energy consumption limit.	Connect with national standards and expand nanoscale measurement methods.

#### Detailed description and comparative analysis

##### Scope:

China (GB/T) and South Korea (KS) cover a wider range (0.1-100 μm), emphasizing high purity and diverse uses to meet the needs of modern technology.

Germany (DIN) focuses on sintered materials and does not directly regulate tungsten powder, reflecting

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its engineering application orientation.

Japan (JIS) and Russia (GOST) take into account both pure tungsten and alloy powders, with the upper limit of JIS being smaller (50  $\mu\text{m}$ ) and GOST extending to the nanometer level (0.05  $\mu\text{m}$ ).

ISO only provides particle size determination methods as general technical support.

Classification:

GB/T, KS, and GOST are carefully classified according to particle size and purpose, reflecting the diversity of industrial needs.

JIS distinguishes between pure tungsten and alloy powder, reflecting Japan's refined classification.

DIN focuses on the classification of sintered materials and does not deal directly with powders.

ISO has no classification, only normative tests.

Chemical Composition:

National standards have higher requirements for W purity (99.9%-99.999%), and similar impurity limits (O 0.05%, C 0.01%, etc.), but KS and GOST offer high-purity options (99.999%), targeting semiconductors and military applications.

DIN focuses on the sintered state, so the lower limit of W is relatively low (90%).

Composition not specified by ISO.

Physical Properties:

Apparent density: similar range in various countries (2.0-12.0  $\text{g/cm}^3$ ), varies with particle size, DIN only regulates sintered density.

Flowability: JIS, KS, GB/T require  $\leq 40-50$  s/50g, GOST does not explicitly mention it.

Particle size distribution: JIS, KS, and GOST require  $D_{90}/D_{10} < 2.5$ , emphasizing uniformity; GB/T uses Fisher particle size, which is traditional and practical.

Test Methods:

Chemical composition: All countries use modern technologies such as ICP-OES and infrared absorption, and the methods are converging.

Particle size: Laser diffraction method is the mainstream (JIS, KS, GOST, ISO), GB/T and GOST retain the Fisher method, and DIN does not involve it.

Physical properties: The test methods for apparent density and fluidity are consistent (such as the funnel method), and DIN focuses on post-sintering testing.

Main features and applications:

China: Balancing traditional (cemented carbide) and emerging (additive manufacturing) demands.

Germany: Engineering-oriented, focusing on sintered material performance.

Japan: Refined, suitable for electronics and high-precision processing.

South Korea: Outstanding high purity, targeted at semiconductors and 3D printing.

Russia: driven by military industry and nanotechnology.

ISO: Common approach supports national standards.

Future trends:

As all countries move towards nanoscale (<50 nm), green production (such as energy consumption, carbon emission limits) and recycling, ISO may expand nano-measurement methods.

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**Appendix : Tungsten powder physical parameters quick reference table**

**1. Basic physical properties**

parameter	Value/Range	unit	Remark
Density	19.25	g/cm <sup>3</sup>	Theoretical density (single crystal tungsten). Sintered material is close to this value. Apparent density varies with particle size.
Apparent Density	2.0-12.0	g/cm <sup>3</sup>	It varies with particle size and preparation process, such as ultrafine powder 2.0-4.0 g/cm <sup>3</sup> , coarse powder 8.0-12.0 g/cm <sup>3</sup> .
Melting Point	3422	°C	The melting point of pure tungsten is one of the highest among metals, and the actual powder melting point is slightly affected by the particle size.
Boiling Point	5555	°C	Boiling point of pure tungsten, an important reference for high temperature applications such as nozzles.
Crystal Structure	Body-centered cubic (BCC)	-	The typical crystal structure of tungsten, which affects its mechanical properties and electrical conductivity.
Lattice Constant	3.165	Å	Lattice parameters of the BCC structure at room temperature.

**2. Thermal properties**

parameter	Value/Range	unit	Remark
Specific Heat Capacity	0.134	J/(g·K)	At room temperature (25°C), tungsten powder changes slightly after sintering.
Thermal Conductivity	173	W/(m·K)	Excellent thermal conductivity at room temperature, suitable for high-temperature heat dissipation components.
Thermal Expansion Coefficient	$4.5 \times 10^{-6}$	K <sup>-1</sup>	Low thermal expansion in the 20-1000°C range is suitable for precision parts.
Heat of Vaporization	799	kJ/mol	High temperature evaporation characteristics affect target material and coating applications.

**3. Electrical properties**

parameter	Value/Range	unit	Remark
Electrical Conductivity	$18.2 \times 10^6$	S/m	At room temperature, it accounts for about 30% of copper and is suitable for electrodes and conductive parts.
Electrical Resistivity	$5.5 \times 10^{-8}$	Ω·m	At room temperature, the resistivity is low but increases significantly with increasing

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parameter	Value/Range	unit	Remark
			temperature.
Work Function	4.55	eV	Electron emission properties of tungsten, key parameters used in cathodes and electron tubes.

#### 4. Mechanical properties (sintered reference)

parameter	Value/Range	unit	Remark
Hardness	300-450	HV	Vickers hardness, pure tungsten sintered state, alloy (such as W-Ni-Fe) can reach 260-350 HV.
Tensile Strength	600-1000	MPa	The hot working state after sintering depends on the grain size and alloy composition.
Young's Modulus	411	GPa	High rigidity, suitable for structural materials.
Poisson's Ratio	0.28	-	Typical values for tungsten, affecting its deformation behavior.

#### 5. Chemical properties

parameter	Value/Range	unit	Remark
Oxidation Tendency	High (>600°C)	-	WO <sub>3</sub> in high temperature air and requires inert gas protection.
Corrosion Resistance	High (acid, base)	-	It is stable to most acids and bases, but corrodes faster in strong oxidizing acids (such as concentrated nitric acid).
Standard Electrode Potential	-0.09	V	W/WO <sub>3</sub> system, reflecting its chemical stability.

#### 6. Optics and surface properties

parameter	Value/Range	Remark
Refractive Index	2.4	Optical parameters for particle size measurement using the laser diffraction method, typical values for pure tungsten powder.
Specific Surface Area	0.1-20 m <sup>2</sup> / g	Depending on the particle size, nanometers (such as 0.05-1 μm) can reach 10-20 m <sup>2</sup> / g, and coarse powders <1 m <sup>2</sup> / g.
Particle Morphology	irregular spherical	The reduction method is mostly used to prepare irregular powders, while the atomization method can produce spherical powders.

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## 7. Typical particle size and application reference

Grade	Particle size range (μm)	Apparent density (g/cm <sup>3</sup> )	Typical Uses
Ultrafine tungsten powder (W-1)	0.05-1.0	2.0-4.0	Electronic targets, 3D printing, nano coatings
Fine tungsten powder (W-2)	1.0-5.0	3.0-6.0	Cemented carbide (such as WC-Co), high temperature coating
Medium tungsten powder (W-3)	5.0-10.0	4.0-8.0	Conventional powder metallurgy products, tungsten rods
Coarse tungsten powder (W-4)	10.0-50.0	6.0-10.0	Heavy alloys (such as W-Ni-Fe), counterweights
Ultra coarse tungsten powder (W-5)	50.0-100.0	8.0-12.0	Special structural materials, industrial counterweights

### Description and data sources

Density: Theoretical density of 19.25 g/cm<sup>3</sup> is an inherent property of single crystal tungsten. The apparent density range (2.0-12.0 g/cm<sup>3</sup>) is based on measured data in standards such as GB/T 3458-2020, KS D 9502:2021, and GOST 14316-91, reflecting the influence of particle size and particle morphology.

Melting and boiling points: 3422°C and 5555°C are recognized values for pure tungsten from the International Materials Handbook and are suitable for designing for high temperature applications.

Conductivity and resistivity:  $18.2 \times 10^6$  S/m and  $5.5 \times 10^{-8}$  Ω·m are typical values of tungsten at room temperature, referring to ASTM and Russian GOST standards, reflecting its conductive properties.

Thermal and mechanical properties: The data is based on the measured values of sintered tungsten (such as DIN 30910-3:2023). Since the powder state cannot be directly measured, it is for reference in subsequent processing.

Chemical and optical properties: Parameters such as oxidation tendency and refractive index combined with powder metallurgy practice (such as JIS H 5762:2022) and particle size analysis requirements.

Particle size and use: Integrate the standard classifications of various countries (such as W-1 to W-5 of GOST, TP-1 to TP-5 of KS), covering industrial applications from nanometer to coarse powder.

Precautions for use

Apparent density and fluidity: The actual value is affected by the preparation process (such as hydrogen reduction, atomization) and particle shape, and needs to be tested in combination with specific standards.

High temperature characteristics: Tungsten powder is easily oxidized at >600°C and requires inert gas protection during storage and processing.

Data range: Some parameters (such as specific surface area) have a wide range due to particle size differences and should be used with reference to specific grades.

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**Appendix : Tungsten powder related patent list (global patent numbers and abstracts)**

**List of Chinese tungsten powder related patents (by time series)**

Patent Number	title	Date of Authorization	summary
CN1009011B	Formula and application of tungsten carbide spray welding powder	1990-08-01	It involves the formulation and application of tungsten carbide spray welding powder, which is prepared with 70-80% tungsten carbide, nickel, chromium, boron, silicon, iron and carbon, and is used for strengthening the wear-resistant surface of brick and tile machinery, extending the service life by 9-14 times.
CN1086556A	High performance tungsten carbide based spray welding alloy powder	1994-05-18	Tungsten carbide based spray welding alloy powder, containing 70-90% tungsten carbide, 5-20% nickel, 3-10% chromium, 0.5-3% boron, 0.5-3% silicon, prepared by gas atomization method, high hardness and wear resistance.
CN1219622A	Preparation method of nano-scale tungsten powder	1999-06-16	Ammonium tungstate is used as raw material, spray dried and then hydrogen reduced to prepare nano tungsten powder, with a particle size of 20-50 nm and a purity of >99.9%, which is suitable for high-precision powder metallurgy.
CN1287187A	Tungsten carbide spray powder and preparation method thereof	2001-03-14	Tungsten carbide spray powder contains WC 80-90%, Ni 5-15%, Cr 3-10%, and is prepared by melt atomization method. It has uniform particle size and strong wear resistance.
CN1334354A	A method for preparing ultrafine tungsten carbide powder	2002-02-06	Take tungsten powder and carbon black as raw materials, add a trace amount of $Cr_3C_2$ , and vacuum carbonize at 1000-1200°C to obtain ultrafine tungsten carbide powder with a particle size of 0.1-0.3 $\mu m$ and high purity.
CN1424252A	Preparation of tungsten carbide powder	2003-06-18	W powder and C powder are ball-milled, vacuumed and protected with argon, annealed at 700-900°C, with controllable particle size, high purity and low annealing temperature.
CN1492068A	A method for preparing tungsten powder	2004-04-28	is calcined, and tungsten powder with a particle size of 1-5 $\mu m$ is obtained by hydrogen reduction. The process is simple and the purity is >99.8%.
CN1210203C	Preparation of tungsten carbide powder	2005-07-06	50% W powder and 50% C powder, vacuumed to $10^{-2}$ Pa, filled with argon protection, annealed at 700-900°C after ball milling, controllable particle size, low energy

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Patent Number	title	Date of Authorization	summary
			consumption.
CN1814837A	A method for preparing nano tungsten powder	2006-08-09	Using ammonium tungstate as raw material, chemical precipitation and hydrogen reduction are used to obtain nano tungsten powder with a particle size of 30-60 nm and a purity of >99.9%, which is suitable for electronic materials.
CN101007735A	A method for preparing tungsten powder	2007-08-01	Tungsten acid is used as the raw material, a surfactant is added, and after spray drying, hydrogen reduction is performed at 800-1000°C to obtain tungsten powder with a particle size of 2-10 μm and good dispersibility.
CN101066561A	A method for preparing nano tungsten carbide powder	2007-11-07	Using tungsten powder and nano-carbon as raw materials, carbonization at 1000-1200°C can obtain nano-tungsten carbide powder with a particle size of 50-100 nm and a purity of >99.9%.
CN101117220B	Method for producing tungsten carbide powder	2010-07-07	The metal source and carbon source react, and then heat at 900-1200°C in a nitrogen/argon atmosphere to adjust the carbon content to obtain nearly stoichiometric tungsten carbide.
CN101642816B	A method for producing ultrafine tungsten carbide powder	2011-03-23	Ammonium paratungstate is calcined to obtain WO <sub>3</sub> , carbon black is added, and carbonization is performed at 1100-1300°C in vacuum to obtain ultrafine tungsten carbide powder with a particle size of 0.1-0.5 μm and a purity of >99.5%.
CN101433968B	Preparation method of fine spherical tungsten powder	2011-04-27	Ammonium tungstate is treated with concentrated sulfuric acid as a precipitant, dried and then hydrogen reduced to obtain fine spherical tungsten powder with a particle size of 1.2-2.8 μm, which is low in cost.
CN102198507A	Production method of ultrafine tungsten grain carbide powder	2011-09-28	Tungsten powder and carbon black are added with VC and Cr <sub>3</sub> C <sub>2</sub> , and carbonized at 1200-1400°C to obtain ultrafine tungsten carbide powder with an average particle size of <0.2 μm, which is suitable for high hardness alloys.
CN102225765A	A method for preparing tungsten powder	2011-10-26	Using WO <sub>3</sub> as raw material, adding trace rare earth elements, and reducing with hydrogen at 900-1100°C, tungsten powder with a particle size of 1-3 μm and fine grains is obtained.
CN102603007A	Preparation method of	2012-07-25	Tungstate, acid solution and water, thioacetamide

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Patent Number	title	Date of Authorization	summary
	tungsten oxide nanopowder and metallic tungsten nanopowder		induced precipitation, non-reducing calcination to obtain 80 nm tungsten oxide nanopowder, and reduction to obtain 40 nm tungsten powder.
CN102616854A	A method for preparing nano tungsten powder from tungstate	2012-08-01	Tungstate is added with organic acid to adjust pH, spray dried and then reduced with hydrogen at 700-900°C to obtain nano tungsten powder with a particle size of 20-50 nm. The process is environmentally friendly.
CN102632259A	A method for preparing tungsten carbide micropowder	2012-08-15	Take tungsten powder and carbon black as raw materials, add a small amount of Co powder, and carbonize at 1000-1200°C to obtain tungsten carbide powder with a particle size of 0.5-1 μm and good uniformity.
CN102230194B	Method for preparing nano tungsten powder from calcium tungstate	2013-05-01	Calcium tungstate is electrolyzed, CaCl <sub>2</sub> mixture is used as electrolyte, treated under inert atmosphere, washed and dried to obtain nano tungsten powder.
CN103302309A	A method for preparing fine-grained tungsten powder	2013-09-18	Ammonium tungstate is used as raw material, spray dried and then hydrogen reduced at 850-1050°C to obtain fine tungsten powder with a particle size of 0.5-2 μm and a purity of >99.9%.
CN103540817A	Tungsten Manganese Alloy	2014-01-29	Tungsten-manganese alloy, 46.99% tungsten, 51.78% manganese, the rest is impurities, with high strength, insulation and light weight.
CN103824710A	Method for preparing silver tungsten carbide contact material from silver-coated tungsten carbide powder	2014-05-28	Silver nitrate and tungsten carbide powder are chemically coated and liquid infiltration is used to prepare silver tungsten carbide contacts, with tungsten carbide 38-42%, high density and high hardness.
CN103945964A	Method for producing tungsten fine powder	2014-07-23	The tungsten powder is dispersed in an aqueous solution containing an oxidant to form an oxide film, which is removed by an alkaline aqueous solution to obtain fine tungsten powder with a particle size of 0.05-0.5 μm and a dms value of 6±0.8.
CN104084594A	A method for preparing nano	2014-10-08	Using tungstic acid as raw material, after liquid phase deposition, hydrogen reduction at 800-1000°C is

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Patent Number	title	Date of Authorization	summary
	tungsten powder		performed to obtain nano tungsten powder with a particle size of 20-40 nm and a purity of >99.95%.
CN104722767A	Tungsten powder preparation method	2015-06-24	Na <sub>2</sub> WO <sub>4</sub> · 5H <sub>2</sub> O is dissolved in water, CTAB and hydrochloric acid are added, and WO <sub>3</sub> is obtained by high pressure reaction calcination, and then plasma arc is used to reduce it to nano tungsten powder.
CN105057682A	Preparation method of tungsten-copper-tin alloy powder	2015-11-18	Atomized copper powder and crystallized tungsten powder are added with tin powder, and then diffused, crushed and screened at high temperature to obtain tungsten-copper-tin alloy powder with uniform composition and high density.
CN103945965B	Method for producing tungsten fine powder	2016-03-30	Tungsten powder was electrolytically oxidized by inorganic acid aqueous solution to form an oxide film, which was removed by alkaline aqueous solution to obtain fine tungsten powder with a particle size of 0.04-0.4 μm and a dms value of 6±0.4.
CN105458274A	A method for preparing high-purity ultrafine tungsten carbide powder	2016-04-13	Take tungsten powder and carbon black as raw materials, add a trace amount of grain inhibitor, and carbonize at 1100-1300°C to obtain ultrafine tungsten carbide powder with a particle size of 0.1-0.3 μm and a purity of >99.98%.
CN106180735A	A method for preparing ultrafine tungsten powder	2016-12-07	Using WO <sub>3</sub> as raw material, vapor deposition and hydrogen reduction are used to obtain ultrafine tungsten powder with a particle size of 0.1-0.3 μm and a purity of >99.98%, which is suitable for high-end applications.
CN106623898A	A method for preparing tungsten-copper alloy powder	2017-05-10	Ammonium tungstate and copper nitrate are spray dried and hydrogen reduced at 800-1000°C to obtain tungsten-copper alloy powder with a particle size of 1-3 μm and good uniformity.
CN107309433A	A method for preparing large-particle spherical tungsten powder	2017-10-27	WO <sub>3</sub> plasma spheroidization and hydrogen reduction, large spherical tungsten powder with a particle size of 50-100 μm and a sphericity of >95% is obtained, which is suitable for 3D printing.
CN107570714A	A method for preparing high-purity tungsten powder	2018-01-19	Ammonium tungstate is used as the raw material, and after multiple purifications, it is hydrogen reduced at 900-1100°C to obtain high-purity tungsten powder with a particle size of 1-5 μm and a purity of >99.999%.

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Patent Number	title	Date of Authorization	summary
CN108188405A	A method for preparing nano-scale tungsten powder	2018-06-22	Ammonium paratungstate is added with organic acid to adjust pH, spray dried and then reduced with hydrogen at 800-1000°C to obtain nano tungsten powder with a particle size of 20-50 nm and a purity of >99.95%.
CN105397102B	Preparation method of alumina coated tungsten powder	2018-04-20	AlO(OH) coated WO <sub>3</sub> , calcined at 300-500°C for 6-12 h, and hydrogen reduced to obtain alumina coated tungsten powder with controllable particle size and good dispersibility.
CN109014231A	A method for preparing composite rare earth tungsten powder	2018-12-18	Blue tungsten oxide is prepared by interstitial reduction of single crystal ammonium paratungstate, and spherical tungsten powder is obtained by hydrogen reduction. It is mixed with cerium oxide and lanthanum oxide to prepare composite rare earth tungsten powder.
CN109231210A	Method for preparing ultrafine tungsten carbide powder using tungsten powder and cobalt powder as raw materials	2019-01-18	Tungsten powder and carbon black are mixed in a ratio of 1:1~1.5, and 10-15% cobalt powder is added. Heat preservation at 700-850°C for 60-90 h can obtain ultrafine tungsten carbide powder with low cost.
CN109264722A	Method for preparing ultrafine tungsten carbide powder using tungsten powder and nickel powder as raw materials	2019-01-25	Tungsten powder and carbon black are mixed in a ratio of 1:1~1.5, and 5-8% nickel powder is added. Heat preservation at 750-850°C for 50-80 h can obtain ultrafine tungsten carbide powder with a short reaction time.
CN109570503A	A method for preparing tungsten powder	2019-04-05	Ammonium tungstate is used as raw material, dispersant is added, spray dried and then hydrogen reduced at 850-1050°C to obtain tungsten powder with a particle size of 0.5-3 μm and a purity of >99.9%.
CN110369732A	A method for preparing high-purity ultrafine tungsten powder	2019-10-25	High-purity WO <sub>3</sub> is added with a trace amount of grain inhibitor and reduced with hydrogen at 900-1100°C to obtain high-purity ultrafine tungsten powder with a particle size of 0.1-0.3 μm and a purity of >99.99%.
CN110976848A	A method for preparing high-density tungsten alloy powder	2020-04-10	Tungsten powder is mechanically alloyed with Ni and Fe powders and then plasma spheroidized to obtain high-density tungsten alloy powder with a particle size of 20-60 μm, which is suitable for aviation parts.

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Patent Number	title	Date of Authorization	summary
CN111020337A	A method for inhibiting the growth of tungsten powder grains	2020-04-17	Y <sub>2</sub> O <sub>3</sub> ) during the reduction process and reduce it with hydrogen at 800-1000°C to obtain tungsten powder with fine grains.
CN111112633A	A method for preparing nano tungsten carbide powder	2020-05-08	Using tungsten powder and nanocarbon as raw materials, carbonization at 1000-1200°C can obtain nano tungsten carbide powder with a particle size of 30-80 nm and a purity of >99.95%.
CN111644632A	A method for preparing tungsten-copper composite powder	2020-09-11	Ammonium tungstate and copper nitrate are spray dried and hydrogen reduced at 900-1200°C to obtain tungsten-copper composite powder with a particle size of 1-5 μm, which is suitable for electrical contact materials.
CN112222421A	A kind of nano tungsten powder and preparation method thereof	2021-01-15	Tungstic acid liquid phase precipitation and hydrogen reduction can obtain nano tungsten powder with a particle size of 10-30 nm and a purity of >99.9%. The process is environmentally friendly.
CN112708794A	Method for preparing copper-tungsten alloy using ultrafine tungsten powder	2021-04-30	Ultrafine tungsten powder is ball-milled, atomized, alkali/acid-washed, and then electroplated with copper. It is then hot-pressed and sintered to obtain copper-tungsten alloy, which has high hardness and strong wear resistance.
CN113046608A	A method for preparing tungsten-molybdenum composite powder	2021-06-18	Tungsten powder and molybdenum powder are mechanically mixed and sintered at high temperature to obtain tungsten-molybdenum composite powder with a particle size of 5-20 μm, which is suitable for high-temperature materials.
CN113427008A	Tantalum-tungsten alloy powder and preparation method thereof	2021-09-24	Tantalum-tungsten alloy ingots are melted, forged, hydrogenated, crushed, dehydrogenated, oxygen reduced, and plasma spheroidized to obtain tantalum-tungsten alloy powder with a particle size of 10-60 μm.
CN113732266A	A composite tungsten carbide powder and preparation method thereof	2021-12-03	Tungsten powder, carbon black and nano-TiC are ball-milled and carbonized at 1300-1500°C to obtain composite tungsten carbide powder with a particle size of 0.5-2 μm, which is high hardness and wear-resistant.
CN114192793A	A method for preparing spherical	2022-03-18	WO <sub>3</sub> plasma spheroidization and hydrogen reduction produce spherical tungsten powder with a particle size

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Patent Number	title	Date of Authorization	summary
	tungsten powder		of 10-50 $\mu\text{m}$ and a sphericity of >95%, which is suitable for additive manufacturing.
CN114799192A	A method for preparing high-purity ultrafine tungsten carbide powder	2022-07-29	Ammonium paratungstate and carbon black are added with trace amounts of VC and vacuum carbonized at 1200-1400°C to obtain high-purity tungsten carbide powder with a particle size of 0.1-0.4 $\mu\text{m}$ and a purity of >99.98%.
CN115070057A	A method for preparing high-purity tungsten powder	2022-09-20	Ammonium tungstate is used as the raw material, and after multiple purifications, it is hydrogen reduced at 900-1100°C to obtain high-purity tungsten powder with a particle size of 1-3 $\mu\text{m}$ and a purity of >99.999%.
CN115255382A	A method for preparing nano-scale tungsten powder	2022-11-01	Ammonium tungstate solution is microwave-assisted reduced and hydrogen annealed to obtain nano-tungsten powder with a particle size of 15-40 nm and a purity of >99.95%. The process is highly efficient.
CN115446307A	A method for preparing tungsten-based alloy powder	2022-12-09	Tungsten powder is mechanically alloyed with Ni and Fe powders and then plasma spheroidized to obtain tungsten-based alloy powder with a particle size of 20-80 $\mu\text{m}$ , which is resistant to high temperatures.
CN116060610A	A method for preparing ultrafine tungsten powder	2023-05-05	WO <sub>3</sub> is subjected to gas phase reduction and ultrasonic dispersion to obtain ultrafine tungsten powder with a particle size of 0.05-0.2 $\mu\text{m}$ and a purity of >99.99%, which is suitable for electronic materials.
CN116372181A	A method for preparing nano tungsten powder	2023-07-04	Using tungstic acid as raw material, after liquid phase deposition, hydrogen reduction at 750-950°C is performed to obtain nano tungsten powder with a particle size of 10-25 nm and a purity of >99.9%.
CN116511520A	A method for preparing highly dispersible tungsten carbide powder	2023-08-01	Tungsten powder and nano-carbon are added with dispersant and carbonized at 1100-1300°C to obtain highly dispersed tungsten carbide powder with a particle size of 0.3-0.8 $\mu\text{m}$ , which is suitable for coating.
CN117139641A	A method for preparing tungsten alloy powder	2023-12-01	Tungsten powder, Mo powder and rare earth oxide are spheroidized after high temperature solid phase reaction to obtain tungsten alloy powder with a particle size of 15-60 $\mu\text{m}$ , which has high strength and corrosion resistance.
CN117655338A	A method for	2024-03-08	After ammonium tungstate is deposited in liquid phase

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Patent Number	title	Date of Authorization	summary
	preparing high-purity nano tungsten powder		and then reduced with low temperature hydrogen, high-purity nano tungsten powder with a particle size of 10-30 nm is obtained, with a purity of >99.999%, which is suitable for semiconductors.
CN118106502A	A method for preparing tungsten-molybdenum alloy powder	2024-05-31	Tungsten powder and molybdenum powder are mechanically mixed and then plasma spheroidized to obtain tungsten-molybdenum alloy powder with a particle size of 20-70 μm, which is suitable for high-temperature structural materials.
CN118543926A	A method for preparing high-purity ultrafine tungsten carbide powder	2024-08-27	Ammonium paratungstate and nano-carbon plus grain inhibitor, vacuum carbonization at 1150-1350°C, to obtain ultrafine tungsten carbide powder with a particle size of 0.08-0.3 μm and a purity of >99.99%.
CN118683318A	A method for preparing high-purity spherical tungsten powder	2024-10-15	Using WO <sub>3</sub> as raw material, plasma spheroidization and hydrogen reduction optimization process are used to obtain high-purity spherical tungsten powder with a particle size of 15-50 μm and a sphericity of >98%, which is suitable for 3D printing.

**Global (outside China) tungsten powder related patent list (by time series)**

Patent Number	title	nation	Authorization date	summary
US2113171	Process for Making Tungsten Powder	USA	1938-04-05	Tungsten powder is prepared by reducing tungsten oxide with hydrogen. It has a coarse particle size and is suitable for early powder metallurgy products.
US2285837	Method of Producing Tungsten Carbide	USA	1942-06-09	Directly carbide tungsten metal to prepare tungsten carbide powder, high temperature reaction, the product is used in the production of wear-resistant materials.
US3418103	Production of Tungsten Powder	USA	1968-12-24	by high temperature hydrogen reduction of WO <sub>3</sub> with a particle size of 5-20 μm, which is suitable for cemented carbide and welding materials.
US3846126	Tungsten Carbide Powder Preparation	USA	1974-11-05	Tungsten powder and carbon are carbonized at 1300-1500°C to obtain

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Patent Number	title	nation	Authorization date	summary
				tungsten carbide powder with uniform particle size, which is suitable for cutting tools.
US4374758	Preparation of Stable Tungsten Carbide	USA	1983-02-22	Tungsten powder is carbonized with carbon source, and the conditions are controlled to reduce impurities to obtain stable tungsten carbide powder, which is used in cemented carbide.
US4664899	High Strength Tungsten Carbide	USA	1987-05-12	High temperature (1400°C) tungsten carbide powder is used to produce high strength tungsten carbide, which is suitable for molds and cutting tools.
US4886638	Method for Producing Tungsten Carbide	USA	1989-12-12	Tungsten carbide powder is prepared by carbothermal reduction of $WO_3$ , and the process is optimized to improve the purity, which is suitable for industrial use.
US5372797	Method for Producing Tungsten Carbide	USA	1994-12-13	Carbothermal reduction of tungsten oxide and high temperature reaction can produce high-purity tungsten carbide powder, which is suitable for wear-resistant parts.
JP2000063176A	Tungsten Powder for Ceramic Capacitors	Japan	2000-02-29	Tungsten powder is used for ceramic capacitor electrodes, with a particle size of 1-10 $\mu m$ . The preparation method is not described in detail (only published, not authorized).
US6114048	Tungsten Powder with Enhanced Density	USA	2000-09-05	High-density tungsten powder with a particle size of 2-5 $\mu m$ is prepared by optimizing the hydrogen reduction process, which is suitable for high-performance alloys.
US6277329B1	Tungsten Carbide Powder with Binder	USA	2001-08-21	Tungsten carbide powder is mixed with a binder (such as Co) to prepare a composite powder, which is suitable for cemented carbide production.
US6416730B1	Tungsten Nanoparticle Production	USA	2002-07-09	Nano-tungsten powder is prepared by chemical vapor deposition with a particle

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Patent Number	title	nation	Authorization date	summary
				size of 10-50 nm, which is suitable for electronic materials.
JP2003272959A	Tungsten Foil Capacitor Electrode	Japan	2003-09-26	Tungsten powder is pressed into foil with a dielectric layer on the surface for use in high-performance capacitors (public only, not authorized).
US6876083B2	Tungsten Alloy Capacitor Anode	USA	2005-04-05	Tungsten powder is alloyed with other metals to prepare capacitor anodes to improve leakage current and is suitable for electronic components.
US7154743B2	Tungsten-Based Electrolytic Capacitor	USA	2006-12-26	Tungsten powder is sintered to prepare electrolytic capacitor anodes, which are suitable for a variety of metal substrates. The preparation process is not described in detail.
EP1932938A1	Tungsten Powder Production Method	Europe	2008-06-18	Ammonium tungstate is used to reduce tungsten powder by hydrogen to prepare tungsten powder with a particle size of 1-10 $\mu\text{m}$ , which is suitable for powder metallurgy (only public, not authorized).
US7923067B2	Method for Producing Tungsten Powder	USA	2011-04-12	Hydrogen reduction of tungsten oxide controls the particle size to 0.5-5 $\mu\text{m}$ to obtain high-purity tungsten powder, which is suitable for precision applications.
US8257625B2	Method for Producing Tungsten Nanopowder	USA	2012-09-04	Chemical reduction and heat treatment of tungstates can produce nano tungsten powder (20-50 nm), which is suitable for electronic materials.
KR101346937B1	Preparation of Nano Tungsten Powder	South Korea	2014-01-02	Nano tungsten powder is prepared by gas phase reduction of tungsten oxide with a particle size of 10-30 nm and a purity of >99.9% for high-end applications.
US8968642B2	Fine Tungsten Powder Production	USA	2015-03-03	Fine tungsten powder is prepared by hydrogen reduction and plasma treatment with a particle size of 0.1-1 $\mu\text{m}$ , suitable for high-density alloys.

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Patent Number	title	nation	Authorization date	summary
US9669460B2	Method for Producing Fine Tungsten Powder	USA	2017-06-06	Tungsten powder is treated with an aqueous solution containing an oxidant to form an oxide film, which is then removed with an alkaline solution to obtain fine tungsten powder with a particle size of 0.05-0.5 $\mu\text{m}$ .
JP6231665B2	Tungsten Carbide Powder Production	Japan	2017-11-15	Tungsten powder and carbon black are added with a small amount of Co and carbonized at 1100-1300°C to obtain tungsten carbide powder with a particle size of 0.1-1 $\mu\text{m}$ , which is used for cemented carbide.
US10047433B2	Method of Making Nanocrystalline Tungsten Powder	USA	2018-08-14	Ammonium tungstate is reduced at 600-700°C and then heated to 800-1000°C to obtain nanocrystalline tungsten powder, which is suitable for high-performance materials.
EP3444060A1	Tungsten Powder with Ceramic Coating	Europe	2019-02-20	Tungsten powder is coated with a ceramic layer (such as aluminum oxide, 30-500 nm) to inhibit the dissolution of tungsten in water, for environmental protection applications (only public, not authorized).
US10442014B2	Composites of Ultra Coarse WC and Cast Carbide	USA	2019-10-15	Ultra-coarse tungsten carbide is compounded with cast carbide and contains WC powder of different particle sizes to improve strength and corrosion resistance.
KR102145711B1	High-Purity Tungsten Powder Preparation	South Korea	2020-08-19	High-purity $\text{WO}_3$ is reduced by hydrogen to prepare tungsten powder with a particle size of 0.5-3 $\mu\text{m}$ and a purity of >99.999%, which is used in the semiconductor industry.
US10807170B2	Matrix Powder with Enhanced Properties	USA	2020-10-20	A matrix powder containing tungsten powder, mixed with other metals to improve strength, wear resistance and corrosion resistance.
JP2021038110A	Spherical Tungsten Powder for 3D Printing	Japan	2021-03-11	$\text{WO}_3$ is plasma spheroidized and then hydrogen reduced to obtain spherical

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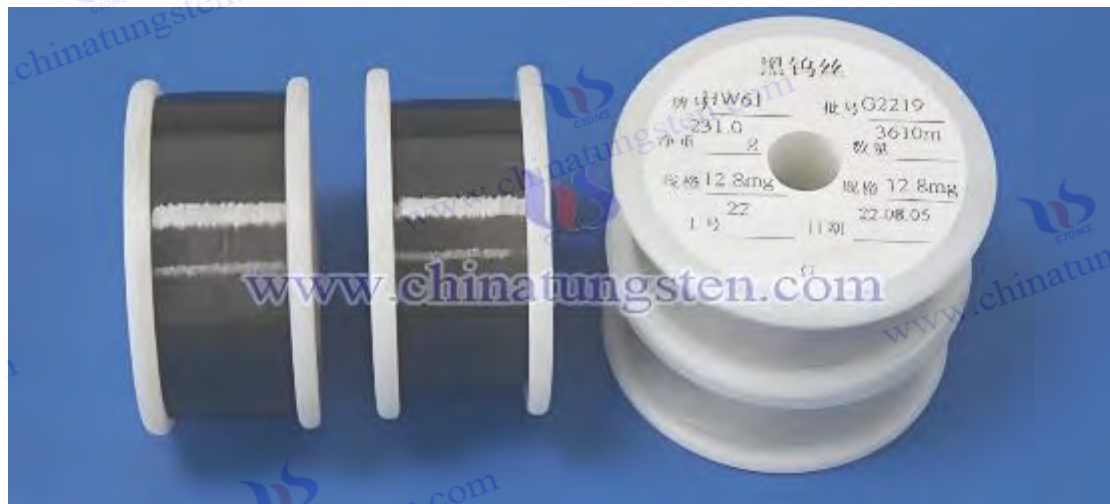
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Patent Number	title	nation	Authorization date	summary
				tungsten powder with a particle size of 10-50 $\mu\text{m}$ , which is used for additive manufacturing (only public, not authorized).
US11130177B2	Frangible Tungsten-Based Projectiles	USA	2021-09-28	Tungsten powder is used to prepare fragile projectile materials, optimize strength and wear resistance, and is suitable for special ammunition.
KR102356636B1	Tungsten-Copper Alloy Powder Preparation	South Korea	2022-02-03	Ammonium tungstate and copper nitrate are spray dried and then reduced to obtain tungsten-copper alloy powder with a particle size of 1-5 $\mu\text{m}$ , which is used as electrical contact material.
EP4059635A1	Tungsten Powder for High-Density Alloys	Europe	2022-09-21	Tungsten powder is mechanically alloyed with Ni and Fe and then spheroidized to obtain high-density tungsten alloy powder with a particle size of 20-60 $\mu\text{m}$ , which is used in aviation (only public, not authorized).
US11590569B2	Nanocrystalline Tungsten Powder Method	USA	2023-02-28	The tungsten compound is reduced at 600-700°C and then heated to 800-1000°C to obtain nanocrystalline tungsten powder with controllable particle size.
JP2023057112A	Ultra-Fine Tungsten Powder Production	Japan	2023-04-20	$\text{WO}_3$ is gas-phase reduced to obtain ultrafine tungsten powder with a particle size of 0.05-0.2 $\mu\text{m}$ and a purity of >99.99%, which is used for electronic materials (only public, not authorized).
KR102645311B1	Spherical Tungsten Powder for Additive Manufacturing	South Korea	2024-03-08	$\text{WO}_3$ is plasma spheroidized and then hydrogen reduced to obtain spherical tungsten powder with a particle size of 15-50 $\mu\text{m}$ and a sphericity of >95%, which is used for 3D printing.
US11913092B2	High-Purity Tungsten Powder for Semiconductors	USA	2024-02-27	Ammonium tungstate is used as the raw material, and after multi-stage purification and hydrogen reduction, high-purity tungsten powder with a particle size of 0.1-1 $\mu\text{m}$ and a purity

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Patent Number	title	nation	Authorization date	summary
				of >99.999% is obtained.
EP4378608A1	Tungsten Alloy Powder for High-Temperature Use	Europe	2024-06-05	Tungsten powder is mixed with Mo and Ni and then plasma spheroidized to obtain tungsten alloy powder with a particle size of 20-70 $\mu\text{m}$ , which is used for high-temperature structures (only public, not authorized).
JP2024089123A	Nano Tungsten Powder for Catalysis	Japan	2024-07-02	Chemical reduction of tungstate to obtain nano-tungsten powder with a particle size of 10-30 nm, which is used as a catalyst (only public, not authorized).
KR102678912B1	Ultra-Fine WC Powder for Hard Coatings	South Korea	2024-07-15	Tungsten powder and nanocarbon are carbonized at 1200-1400°C to obtain ultrafine tungsten carbide powder with a particle size of 0.05-0.3 $\mu\text{m}$ , which is used for hard coatings.
US12002606B2	Spherical Tungsten Powder Method	USA	2024-08-06	WO <sub>3</sub> plasma spheroidization optimization process is used to obtain spherical tungsten powder with a particle size of 10-40 $\mu\text{m}$ and a sphericity of >98% for additive manufacturing.



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**Appendix: Complete list of tungsten powder production equipment , inspection and testing instruments , raw and auxiliary materials, power gas, etc.**

**1. Tungsten powder production equipment**

equipment name	Model/Specification	use	Remark
Hydrogen reduction furnace	H <sub>2</sub> reduction tube furnace (TF-1600, power 30 kW)	Tungsten oxide (WO <sub>3</sub> ) or ammonium paratungstate (APT) is reduced to tungsten powder in a hydrogen atmosphere (600-1000°C).	The furnace tube diameter is 50-150 mm, with multi-stage temperature control (accuracy ±1°C), H <sub>2</sub> flow rate of 10-100 L/min, and output of 0.5-10 kg/h. The furnace body can be made of quartz tube (temperature resistance 1100°C) or molybdenum tube (temperature resistance >1500°C), equipped with an exhaust gas treatment system (absorbs H <sub>2</sub> O and unreacted H <sub>2</sub> ). It is suitable for the production of coarse tungsten powder (5-20 μm) to fine tungsten powder (1-5 μm). The sealing of the furnace tube needs to be checked regularly to prevent H <sub>2</sub> leakage, and explosion-proof measures should be taken during operation. Common brands include Nabertherm in Germany or Kejia in Zhengzhou, China.
Rotary Tube Reduction Furnace	RT-1200 (length 2 m, power 50 kW)	Dynamic reduction of WO <sub>3</sub> or APT to prepare uniform tungsten powder (1-10 μm).	Rotation speed 1-10 rpm, tilt angle 0-5°, furnace tube diameter 100-200 mm, output 10-50 kg/h. The rotating design allows the powder to be heated evenly, avoiding local over-reduction or agglomeration, and is suitable for medium and large-scale production. The furnace lining can be made of alumina ceramic (temperature resistance 1600°C), and the H <sub>2</sub> flow rate is 20-200 L/min. A dust collection device is required to reduce environmental pollution, and the product quality consistency is better than that of a fixed tube furnace. Commonly used in the production of cemented carbide raw materials, such as Toshiba Materials in Japan uses similar equipment.
Plasma atomization equipment	RF plasma atomizer (TEKNA PS-50, 50 kW)	The tungsten material is melted by radio frequency plasma and atomized to prepare spherical	Temperature >6000°C, sphericity >90%, Ar flow rate is 20-50 L/min, output 1-5 kg/h. RF power 30-50 kW, tungsten material (rod or wire) is melted in the plasma flame and atomized by high-speed airflow, forming spherical particles after cooling. Suitable for additive manufacturing (such as 3D printing tungsten parts), powder fluidity is excellent (<25 s/50g). High-purity Ar

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equipment name	Model/Specification	use	Remark
		tungsten powder (particle size 5-50 μm).	protection is required to prevent oxidation, and maintenance requires professionals. Canada TEKNA and the United States AP&C are the main suppliers.
Microwave reduction furnace	MW-500 (500 kW, frequency 2.45 GHz)	Microwave heating is used to reduce tungsten oxide (700-900°C) to prepare micron-sized tungsten powder (1-5 μm) with low energy consumption.	Microwave penetration depth is 10-20 cm, heating is uniform, power is adjustable (100-500 kW), and output is 5-20 kg/h. It saves 30%-50% energy compared to traditional resistance furnaces, and the reduction time is shortened by about 40%, which is suitable for small and medium-scale production. The microwave action causes the internal temperature of WO <sub>3</sub> to rise rapidly, avoiding surface crusting, and the finished product particle size distribution is narrow (D50±0.5 μm). Microwave-resistant materials (such as SiC lining) must be used to avoid failures caused by metal reflection. It is commonly used in laboratories or small batches of high-purity tungsten powder production, such as the US Microwave Dynamics products.
Electrolytic reduction cell	Custom stainless steel tanks (316L, 50-200L)	Tungsten powder (1-20 μm) is prepared by electrolysis of tungstate solution with high purity.	Electrode material Ti or Pt, current density 10-50 A/dm <sup>2</sup> , tank volume 50-200 L, yield 0.2-2 kg/h. The electrolyte is usually NaCl/KCl (pH 6-8), the electrolysis temperature is 50-80°C, and the generated tungsten powder is deposited on the cathode. It is suitable for the production of nano or high-purity tungsten powder (purity>99.99%), and the waste liquid needs to be neutralized. The equipment is highly customized, the tank body needs to be corrosion-resistant (such as 316L steel + PTFE coating), and the current stability needs to be monitored during operation. This process is commonly used in small factories in Russia and Europe.
Ball mill	QM-3SP4 (planetary, 4×1 L, power 1.5 kW)	Tungsten powder is mixed with carbon powder, cobalt powder, etc. and ground to prepare tungsten carbide powder	Speed 200-600 rpm, grinding jar 4×1 L, grinding media WC or ZrO <sub>2</sub> (diameter 5-15 mm), grinding time 2-24 h. The planetary design provides high-energy impact and is suitable for the preparation of WC-Co cemented carbide powder (particle size 0.5-10 μm). The grinding efficiency is high, but it is easy to introduce trace impurities (<0.01 wt%), and the grinding balls and liners need to be replaced regularly. The noise is

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equipment name	Model/Specification	use	Remark
		(<10 μm).	relatively high (>80 dB), and it is recommended to use a soundproof cover. Common brands such as Changsha Tianchuang in China or Fritsch in Germany are widely used in the cemented carbide industry.
Spray Dryer	LPG-50 (centrifugal, 50 kg/h, power 15 kW)	The tungstate solution is spray dried to prepare the precursor powder (D50=5-20 μm), which is suitable for the production of nano tungsten powder.	Rotation speed 20000-30000 rpm, inlet temperature 200-300°C, outlet 80-120°C, output 50 kg/h. Centrifugal atomization forms uniform droplets, and spherical precursors are obtained after drying, which are suitable for subsequent reduction to prepare nano tungsten powder. The equipment is equipped with a hot air circulation system, and the moisture content of the powder is <0.5 wt%. The nozzle needs to be cleaned regularly to prevent clogging. It is suitable for the preparation of high-purity powder by solution method. Shanghai Yuandong, China or GEA, Germany are common suppliers.
Spray drying tower	SDT-200 (tower height 10 m, 200 kg/h)	The tungstate or tungsten oxide suspension is spray dried to prepare uniform precursor powder (D50=5-50 μm).	The tower is 8-15 m high, with a nozzle pressure of 0.2-0.5 MPa, an inlet temperature of 250-400°C, an outlet temperature of 90-150°C, and an output of 100-200 kg/h. The solution is atomized by a pressure nozzle or a centrifugal disk, and quickly dried by hot air (500-1000 m <sup>3</sup> / min) to produce spherical or quasi-spherical powders (water content <0.3 wt%). It is suitable for a large-scale production of nano- or micron-sized tungsten powder precursors. A cyclone separator and a bag filter are installed in the tower to reduce dust loss. The nozzle wear and the material accumulation in the tower need to be checked regularly, which is suitable for industrial applications. Lima in Changzhou, Jiangsu, China or Buchi in Germany are the main suppliers.
Vacuum sintering furnace	ZT-50-20 (vacuum degree 10 <sup>-3</sup> Pa, power 50 kW)	Sinter tungsten powder into a blank or test sintering performance (1500-2000°C), vacuum or Ar	Heating rate 5-20°C/min, furnace volume 20-50 L, Mo or graphite heating element, working pressure 10 <sup>-3</sup> -10 <sup>-5</sup> Pa. Used for sintering of tungsten powder compacts (such as carbide tools) or performance testing to prevent high temperature oxidation. Density after sintering can reach 18-19 g/cm <sup>3</sup> , vacuum pump and heating elements need to be calibrated regularly.

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equipment name	Model/Specification	use	Remark
		protection.	Suitable for high value-added products, Shanghai Chenhua, China or ALD, Germany are the main brands.
Screening Machine	ZS-1000 (vibrating screen, 1-5 layers, power 1.5 kW)	Tungsten powder is classified by particle size (e.g. 0.1-100 μm).	Vibration frequency 3000 times/min, mesh aperture 10-200 μm, processing capacity 500-2000 kg/h, sieve layer 1-5 adjustable. High classification accuracy, suitable for sorting tungsten powder from nanometer to micron level (such as D50=1 μm, 10 μm). Noise is about 70-85 dB, and a dust collector is required to reduce dust pollution. It complies with ISO 4497 standard and is commonly used in tungsten powder post-processing procedures. China Xinxiang Vibration or Germany Retsch are the main suppliers.
Air jet mill	QF-300 (fluidized bed, 300 kg/h, 10 bar)	Crush the coarse tungsten powder to ultra-fine grade (<5 μm) to improve homogeneity.	Air velocity 300-500 m/s, air pressure 0.6-1.0 MPa, D50<2 μm, N <sub>2</sub> or Ar driven. The fluidized bed design uses high-speed airflow collision to crush, and the powder purity is high (impurities <0.005 wt%), which is suitable for ultrafine tungsten powder production. The nozzle wear needs to be checked regularly. The noise is relatively high (>90 dB), and it is recommended to be equipped with a silencer. Weifang Zhengyuan, China or Netzsch, Germany are common brands.
Water electrolysis hydrogen production device	HYDRO-500 (500 Nm <sup>3</sup> / h, 250 kW)	High-purity hydrogen (≥99.999%) is produced by electrolysis of water for tungsten powder reduction.	PEM technology, gas production 500 Nm <sup>3</sup> / h, energy consumption 4.5 kWh/Nm <sup>3</sup> , O <sub>2</sub> byproduct can be recycled. Proton exchange membrane or alkaline electrolyzer (KOH solution) is used, operating pressure 1-3 MPa, life span >10 years. Suitable for high-purity tungsten powder production (such as semiconductor grade), regular maintenance of electrodes and membranes is required (replacement cycle 2-5 years). Common brands include Norway's Nel Hydrogen or Germany's Siemens.
Methanol cracking hydrogen production equipment	MCR-300 (300 Nm <sup>3</sup> / h, 100 kW)	Methanol is cracked at high temperature (250-300°C) to produce hydrogen (≥99.99%) at a	Cu-Zn catalyst, gas output 300 Nm <sup>3</sup> / h, CO content <0.01%, operating temperature 250-300°C. Methanol conversion rate >95%, by-product CO <sub>2</sub> requires tail gas treatment (absorption or combustion). Suitable for small and medium-scale production, small equipment volume (about 5 m <sup>3</sup> ), catalyst needs to be replaced regularly (lifetime about 1-2 years). Jiangsu Jiutian,

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equipment name	Model/Specification	use	Remark
		low cost.	China or Air Liquide, USA are common suppliers.
Ammonia decomposition hydrogen production furnace	NH <sub>3</sub> -200 (200 Nm <sup>3</sup> / h, power 80 kW)	Ammonia decomposition (700-900°C) to produce hydrogen (≥99.9%) is economical.	Ni-based catalyst, gas production 200 Nm <sup>3</sup> / h, decomposition rate >99%, N <sub>2</sub> byproduct is harmless. Operating temperature 700-900°C, NH <sub>3</sub> consumption is about 0.7 kg/ Nm <sup>3</sup> H <sub>2</sub> . Liquid ammonia is cheap, suitable for crude tungsten powder production. The equipment is simple, and needs to be equipped with NH <sub>3</sub> storage tank and tail gas cooling system. Catalyst support is provided by Bojin, Zhengzhou, China or BASF, Germany.
PSA pressure swing adsorption hydrogen production equipment	PSA-H <sub>2</sub> -1000 (1000 Nm <sup>3</sup> / h, power 150 kW)	Separate and purify hydrogen (≥99.999%) from industrial mixed gases with high yield.	Adsorbent 5A molecular sieve, gas output 1000 Nm <sup>3</sup> / h, adsorption pressure 1-3 MPa, recovery rate 85%-90%. Extract H <sub>2</sub> from coke oven gas or chemical tail gas, suitable for large-scale tungsten powder production. The equipment occupies about 20-50 m <sup>2</sup> and requires a stable gas source (H <sub>2</sub> content >50%). Low operating noise (<70 dB), common brands such as American Praxair or China Hangzhou Fusda.
Hydrogen Purifier	HP-50 (50 Nm <sup>3</sup> / h, power 10 kW)	Purify hydrogen to remove water and oxygen (purity up to 99.9999%).	Palladium membrane technology, throughput 50 Nm <sup>3</sup> / h, H <sub>2</sub> O < 1 ppm, O <sub>2</sub> < 1 ppm, operating temperature 300-400°C. Suitable for high purity tungsten powder production (e.g. W>99.9999%). Palladium membrane life is about 3-5 years, and membrane integrity needs to be checked regularly. Small size (about 1 m <sup>3</sup> ), commonly used in the semiconductor industry, such as Linde in Germany or Iwatani in Japan.
Freeze Dryer	FD-50 (50 kg/batch, vacuum degree 10 Pa)	The precursor powder is prepared from freeze-dried tungstate solution and is suitable for the production of nano tungsten powder.	Cold trap temperature -50°C, drying time 12-24 h, power 15 kW, output 50 kg/batch. The moisture content of the freeze-dried powder is <0.1 wt%, suitable for solution method preparation of nano precursors (such as tungstate). It needs to be equipped with a vacuum pump (pumping speed >10 L/s) and a condensation system. Commonly used in fine chemicals, such as Shanghai Dongfulong in China or Martin Christ in Germany.

## 2. Inspection and testing instruments

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Instrument Name	Model/Specification	use	Remark
Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES)	Agilent 5110	Determine the content of W, Fe, Mo, Si and other elements in tungsten powder, with a detection limit of <10 ppm.	Power 1.2 kW, wavelength 167-785 nm, detect 20+ elements (W, Fe, Mo, Si, Cu, etc.), detection limit <10 ppm (Fe <1 ppm), in line with GB/T 4324, ASTM E1479. Equipped with RF generator (27 MHz), sample pretreatment requires acid dissolution (HNO <sub>3</sub> +HF), analysis time <5 min, repeatability <1%. Suitable for tungsten powder purity control (>99.9%), requires regular calibration (every 3 months). Commonly used in metallurgical laboratories, such as Agilent in the United States or Spectro in Germany.
Oxygen and nitrogen analyzer	LECO ONH836	Determination of O and N content in tungsten powder (<5 ppm) by inert gas fusion-infrared/thermal conductivity method.	Carrier gas He (99.999%), temperature 2000°C, detection range 0.0001%-0.5%, accuracy ±0.1 ppm, in line with ISO 15351, GOST 12345-2001. Use graphite crucible to melt sample (0.1-1 g), analysis time <3 min, suitable for high purity tungsten powder detection (such as O<20 ppm). Electrodes need to be cleaned regularly and filters replaced (monthly). Commonly used in cemented carbide quality control, such as LECO in the United States or Horiba in Japan.
Carbon and sulfur analyzer	LECO CS744	Determination of C and S content in tungsten powder (<5 ppm) by high frequency combustion-infrared absorption method.	Power 2.5 kW, detection limit 0.0001%-5%, in line with JIS H 6201, KS D 9504. Sample size 0.1-1 g, O <sub>2</sub> flow 2 L/min, combustion temperature >1500°C, analysis time <1 min, accuracy ±0.5 ppm. Suitable for tungsten carbide powder C content detection (6.13%-6.25%), infrared detector needs to be calibrated regularly (weekly). Commonly used in tungsten powder manufacturers, such as LECO in the United States or Eltra in Germany.
Laser Particle Size Analyzer	Malvern Mastersizer 3000	Determine the particle size distribution (D10, D50, D90) of tungsten powder in the	He-Ne laser (633 nm), detection range 0.01-3500 μm, repeatability <1%, in compliance with ISO 4884:2019, DIN EN 23908:2020. Dry or wet dispersion (water/ethanol), sample size 0.1-5 g, analysis time <1 min. Suitable for particle size analysis from nano tungsten powder (D50 < 0.1 μm) to coarse powder (D50 > 100 μm), lens and

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Instrument Name	Model/Specification	use	Remark
		range of 0.01-3500 $\mu\text{m}$ .	calibration standards (such as $\text{SiO}_2$ ) need to be cleaned regularly. Malvern, UK or Beckman Coulter, USA are the main suppliers.
Fisher particle size meter	FSSS-100	Determine the average particle size of tungsten powder (0.1-50 $\mu\text{m}$ ) by air permeation method.	Pressure 0.1-0.5 kPa, sample size 2-5 g, in accordance with GB/T 3249, ASTM B330. Measure average particle size (e.g. 1-5 $\mu\text{m}$ ) based on powder porosity, measurement time 5-10 min, accuracy $\pm 0.05 \mu\text{m}$ . Traditional method, suitable for micron-level tungsten powder detection, simple operation but unable to measure particle size distribution. Pressure gauge needs to be calibrated regularly. Commonly used in tungsten powder factories, such as Hunan Hongda in China or Kennametal in the United States.
Scanning electron microscopy (SEM)	Zeiss Sigma 300	Observe the particle morphology, sphericity and surface characteristics of tungsten powder at a magnification of 50-50000x.	Resolution 1 nm, acceleration voltage 1-30 kV, with EDS (elemental analysis), in accordance with ASTM E986. The sample needs to be gold-plated or carbon-plated (thickness 5-10 nm), and the sphericity (>90%) and surface defects (such as cracks) are observed. The analysis time is 15-60 min, suitable for the quality assessment of tungsten powder for additive manufacturing. Professional operators are required, commonly found in R&D institutions, such as the German Zeiss or Japanese JEOL brands.
Specific surface area analyzer (BET)	Micromeritics ASAP 2460	Determination of specific surface area of tungsten powder (0.1-20 $\text{m}^2/\text{g}$ ), nitrogen adsorption method.	Detection range 0.01-2000 $\text{m}^2/\text{g}$ , accuracy $\pm 0.01 \text{m}^2/\text{g}$ , in accordance with ISO 9277. $\text{N}_2$ adsorption temperature $-196^\circ\text{C}$ , sample size 0.1-1 g, degassing temperature $200-300^\circ\text{C}$ , analysis time 2-6 h. Applicable to nano tungsten powder (such as surface area $>10 \text{m}^2/\text{g}$ ), the result is inversely proportional to the particle size. Liquid nitrogen and calibration standards need to be replaced regularly. Micromeritics in the United States or Quantachrome in Germany are the main suppliers.
X-ray diffractometer (XRD)	Bruker D8 Advance	Analyze the crystal structure (such as BCC) and phase	Cu $K\alpha$ radiation (1.54 $\text{\AA}$ ), $2\theta$ range $5-90^\circ$ , scanning speed $0.02^\circ/\text{s}$ , in accordance with ASTM E915. Detection of grain size 10-100 nm, sample size 0.5-2 g, analysis time 30-60 min. Suitable for

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Instrument Name	Model/Specification	use	Remark
		composition of tungsten powder to ensure there is no impurity phase.	confirming the purity of tungsten powder (no $WO_3$ or $W_2C$ impurities), regular calibration of the X-ray tube is required (lifetime of about 5000 h). Commonly used in material research, such as German Bruker or Japanese Rigaku brands.
Density meter	Archimedes DH-300	Determination of apparent density of tungsten powder (2.0-12.0 $g/cm^3$ ), Archimedes principle.	Range 0-300 g, accuracy $\pm 0.001 g/cm^3$ , in line with JIS Z 2502, DIN EN ISO 4499. Sample weight 5-20 g, medium water or ethanol, measurement time $< 5$ min. Apparent density reflects the bulk properties of powder (e.g. 3-5 $g/cm^3$ ), sample must be dry. Commonly used in the powder metallurgy industry, such as Shanghai Instrument and Electronics in China or AND brand in Japan.
Hall flow meter	HMK-22	Determine the flowability of tungsten powder ( $< 50$ s/50g) and evaluate the powder flow characteristics.	Funnel angle $60^\circ$ , aperture 2.5 mm, sample weight 50 g, in line with JIS Z 2504, ASTM B213. Measurement time $< 1$ min, suitable for spherical tungsten powder testing (e.g. $< 25$ s/50g). Fluidity affects 3D printing and pressing performance, and the sample needs to be dried to prevent agglomeration. Commonly used in additive manufacturing, such as China Haomai or American Carney supply.
X-ray Fluorescence Spectrometer (XRF)	Thermo Fisher ARL Perform'X	Non-destructive determination of tungsten elements in powder ( $> 0.01$ wt%).	Power 50 kV, detection elements Be-U, in accordance with DIN 51001:2021. Sample size 1-10 g, analysis time $< 10$ min, detection limit 0.01 wt% (such as Fe, Ni). Suitable for rapid screening of impurities without chemical pretreatment. X-ray source needs to be calibrated regularly (annually), which is common in factory quality inspections, such as the American Thermo Fisher or Japanese Hitachi brands.
Atomic Absorption Spectrometer (AAS)	PerkinElmer PinAAcle 900T	Determination of trace metal impurities (such as Fe, Cu) in tungsten powder, detection limit	Wavelength 200-900 nm, detection limit $< 1$ ppm ( $Fe < 0.5$ ppm), in line with ASTM E1832. Flame or graphite furnace method, sample needs to be dissolved in acid ( $HCl + HNO_3$ ), analysis time 5-10 min/sample. Suitable for high-purity tungsten powder impurity detection ( $> 99.999\%$ ), the lamp source (such as Fe lamp) needs to be replaced

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Instrument Name	Model/Specification	use	Remark
		<1 ppm.	regularly. Commonly used in laboratories, such as the American PerkinElmer or German Analytik Jena brands.
Thermogravimetric Analyzer (TGA)	TA Instruments Q500	Determine the thermal stability and moisture content (<0.1 wt%) of tungsten powders in the temperature range of 25-1500°C.	Heating rate 0.1-50°C/min, accuracy ±0.01 mg, in accordance with ISO 11358. Sample size 5-50 mg, N <sub>2</sub> protective gas, analysis time 1-2 h. Detects moisture (<0.1 wt%) and oxidation behavior (such as W→WO <sub>3</sub> ), suitable for quality control. Balances need to be calibrated regularly (monthly), TA Instruments, USA or Mettler Toledo, Switzerland are the main suppliers.
Particle Image Analyzer	Retsch Camsizer X2	Dynamic image analysis of tungsten powder particle size and shape (0.8 μm-8 mm).	Resolution 0.8 μm, detection range 0.8 μm-8 mm, in accordance with ISO 13322-2. Dry or wet analysis, sample size 1-10 g, analysis time <5 min. Measures particle size distribution and sphericity (>90%), suitable for evaluation of tungsten powder for additive manufacturing. Lens and calibration standard particles need to be cleaned regularly. German Retsch or American Microtrac are common brands.

### 3. Raw and auxiliary materials

Material Name	Specification/Purity	use	Remark
Tungsten Oxide (WO <sub>3</sub> )	Yellow powder, ≥99.9%-99.999%	The main raw material for tungsten powder production is hydrogen reduction to prepare tungsten powder (600-1000°C).	Particle size 1-20 μm, O content 21.1%-21.3%, specific gravity 7.16 g/cm <sup>3</sup> , melting point 1473°C. Source: roasted tungsten ore (scheelite or wolframite), high purity (>99.999%) is used for semiconductor grade tungsten powder, ordinary grade (>99.9%) is used for cemented carbide. Storage needs to be sealed and moisture-proof (turns blue after moisture absorption). Common suppliers include Xiamen Tungsten Co., Ltd. in China or Global Tungsten in the United States.
Ammonium Paratungstate(APT)	(NH <sub>4</sub> ) <sub>10</sub> W <sub>12</sub> O <sub>41</sub> ·5H <sub>2</sub> O, ≥99.95%	Prepare high-purity tungsten	W content>88%, particle size 10-50 μm, specific gravity 2.3 g/cm <sup>3</sup> , decomposition temperature>300°C. Source: Ammonium tungstate

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Material Name	Specification/Purity	use	Remark
		powder precursor, thermally decompose (400-600°C) and reduce to tungsten powder.	solution crystallization and purification, industrial grade (99.95%) is used for crude tungsten powder, high purity grade (99.99%) is used for electronic materials. Easily soluble in water (solubility>50 g/100 mL), need to be stored dry (<50% humidity). About 80% of global production comes from China, such as Ganzhou Haichuang Tungsten Industry.
Tungstic acid (H <sub>2</sub> WO <sub>4</sub> )	Yellow powder, ≥99.9%-99.99%	Nano-tungsten powder is prepared by low temperature reduction method (700-900°C), solution process raw material.	Particle size 5-20 μm, specific gravity 5.5 g/cm <sup>3</sup> , slightly soluble in water (<0.1 g/100 mL). Source: Acid leaching of tungsten ore (HCl or H <sub>2</sub> SO <sub>4</sub> ), suitable for solution method preparation of nano tungsten powder (<100 nm). Decomposes into WO <sub>3</sub> at high temperature (>500°C), storage needs to be protected from light to prevent decomposition. Commonly used in laboratories or small batch production, such as Sigma-Aldrich in the United States.
Carbon Black	Particle size 20-50 nm, ≥99%-99.9%	Mixed tungsten powder to prepare tungsten carbide powder, the carbonization temperature is 1000-1500°C.	Specific surface area 50-200 m <sup>2</sup> /g, ash content <0.5 wt%, source: hydrocarbon pyrolysis (such as acetylene cracking). For WC powder production, the C content needs to be controlled (theoretical C content of WC is 6.13 wt%), and excessive C content is prone to generate free carbon. Storage needs to be moisture-proof and dust-proof. Common suppliers include Cabot in the United States or Evonik in Germany.
Cobalt powder(Co)	Particle size 1-5 μm, ≥99.8%-99.99%	Prepare WC-Co cemented carbide powder with tungsten powder and carbon black to improve toughness.	Specific gravity 8.9 g/cm <sup>3</sup> , melting point 1495°C, source: electrolysis or atomization. High-purity Co (>99.99%) is used for high-end cemented carbide, and ordinary grade (>99.8%) is used for general tools. Addition amount 5-15 wt% to enhance the toughness of WC matrix (hardness HRA 88-92). Anti-oxidation is required (O <sub>2</sub> <0.02 wt%). Common suppliers include Umicore of Belgium or Jinchuan Group of China.

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Material Name	Specification/Purity	use	Remark
Nickel powder (Ni)	Particle size 1-5 $\mu\text{m}$ , $\geq 99.8\%$ -99.99%	It can be used with tungsten powder to prepare high toughness alloy powder or tungsten carbide based composite materials.	Specific gravity 8.9 g/ $\text{cm}^3$ , melting point 1455°C, source: carbonyl nickel decomposition or atomization method. Addition amount 3-10 wt%, improves corrosion resistance and toughness (such as Ni-WC coating), suitable for high temperature wear-resistant parts. Storage needs to be sealed to prevent oxidation (Ni easily absorbs $\text{O}_2$ ). Common suppliers include Vale in Canada or Norilsk Nickel in Russia.
Scrap Tungsten	$W \geq 90\%$ , containing Co/TiC, etc.	The recovered tungsten powder is processed by acid leaching and reduction.	Source: Waste carbide tools, tungsten wire or tungsten steel, W content 90%-98%, impurities Co/TiC/Fe, etc. The recycling process includes acid leaching ( $\text{HNO}_3 / \text{HCl}$ ), roasting (600-800°C) and $\text{H}_2$ reduction, and the output tungsten powder purity is $>99.5\%$ . Supports circular economy, heavy metal residues need to be detected (such as $\text{Pb} < 0.01 \text{ wt}\%$ ). Commonly found in Zhuzhou Cemented Carbide Group in China or Tungco in the United States.
Thioacetamide	$\text{CH}_3\text{CSNH}_2$ , $\geq 99\%$ -99.5%	The inducer of tungsten oxide nanopowder is prepared by precipitation method, and the reaction storage needs to be dark and low temperature	Molecular weight 75.13, melting point 115°C, soluble in water and ethanol. Used in solution method to generate $\text{WO}_3$ precipitation (particle size $< 50 \text{ nm}$ ), the reaction generates $\text{H}_2\text{S}$ which requires tail gas treatment (NaOH absorption). Chemically pure grade ( $>99\%$ ) ensures precipitation efficiency, the reaction temperature is ( $< 25^\circ\text{C}$ ). Common suppliers include Aladdin in China or Alfa Aesar in the United States.
Methanol ( $\text{CH}_3\text{OH}$ )	$\geq 99.9\%$ -99.99%	Methanol is cracked to produce hydrogen feedstock, and $\text{CO}_2$ ( 250-300°C).	Density 0.79 g/ $\text{cm}^3$ , boiling point 64.7°C, source: Methanol is petrochemical industry (such as coal-to-methanol). Cracking requires a catalyst (Cu-Zn-Al), 1 kg of methanol produces about 1.2 $\text{Nm}^3 \text{ H}_2$ , and the by-product $\text{CO}_2$ is about 0.5 kg. Industrial grade ( $>99.9\%$ ) is sufficient, and it needs to be prevented generating $\text{H}_2$ from volatilization (flash point 11°C), and stored in sealed steel tanks. Global production is about 100 million tons/year, such as supplied by China Shenhua or Saudi Arabia SABIC.
Liquid ammonia	$\geq 99.9\%$ -99.999%	Ammonia is	Density 0.68 g/ $\text{cm}^3$ ( -33°C), boiling point -33.4°C,

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Material Name	Specification/Purity	use	Remark
(NH <sub>3</sub> )		decomposed into H <sub>2</sub> and N <sub>2</sub> (700-900°C)	source: synthetic ammonia industry (Haber-Bosch process). 1 kg NH <sub>3</sub> produces about 2.8 Nm <sup>3</sup> H <sub>2</sub> , and the N <sub>2</sub> byproduct is non-toxic. Storage requires low-temperature and high-pressure containers (10-15 bar), and transportation uses cylinders or tank trucks. Common suppliers include BASF in Germany or CF Industries in the United States.
Sodium tungstate (Na <sub>2</sub> WO <sub>4</sub> )	≥99.9%, white crystals	The precursor of tungsten powder is prepared by solution method and then treated by precipitation and reduction.	Molecular weight 293.82, melting point 698°C, soluble in water (74 g/100 mL). Source: Alkaline leaching of tungsten ore (NaOH treatment of scheelite), used for electrolysis or precipitation to prepare tungsten powder. Purity > 99.9% is suitable for high-purity processes, and storage must be moisture-proof (easy to absorb water and agglomerate). Common suppliers include China Jiangxi Tungsten Group or HC Starck in the United States.
Grain inhibitor (VC/Cr <sub>3</sub> C <sub>2</sub> )	Particle size 0.5-2 μm, ≥99.5%	Added in the carburization process of tungsten powder to inhibit grain growth and prepare ultrafine tungsten carbide.	VC melting point 2830 °C, Cr <sub>3</sub> C <sub>2</sub> melting point of 1895 °C, specific gravity 6.68-6.74 g/cm <sup>3</sup> . Addition amount 0.1-1 wt%, inhibit WC grain growth (<0.5 μm), improve hardness (HRA>90). Source: chemical synthesis or powder metallurgy, need to be evenly mixed to prevent segregation. Storage sealed to prevent oxidation. Common suppliers such as Kennametal in the United States or Treibacher in Germany.

#### 4. Power gas

Gas Name	purity	use	Remark
Hydrogen (H <sub>2</sub> )	≥99.999%-99.9999%	Tungsten powder (600-1000°C) is prepared by reducing tungsten oxide or APT and is supplied on site by hydrogen production	Molecular weight 2.02, density 0.089 g/L, boiling point -252.8°C. Flow rate 10-1000 L/min, high purity (>99.9999%) for semiconductor grade tungsten powder (O<5 ppm), ordinary grade (>99.999%) for cemented carbide. Source: water electrolysis, methanol cracking, etc., explosion-proof storage is required (explosion limit 4%-74%). Global demand is about 90 million Nm <sup>3</sup> / year. Suppliers such as Air Products in the United States or

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Gas Name	purity	use	Remark
		equipment.	Linde in Germany.
Argon (Ar)	≥99.999%	Protective gas in plasma atomization or sintering to prevent oxidation of tungsten powder (>1000°C).	Molecular weight 39.95, density 1.78 g/L, boiling point -185.8°C. Pressure 0.1-1 MPa, flow rate 20-50 L/min, inert gas does not react with tungsten, suitable for high temperature processes (such as atomizing spherical tungsten powder). Source: Air separation, stored in high pressure cylinders (150 bar) or liquid argon tanks. Common suppliers include Air Liquide in France or Baosteel Gas in China .
Helium (He)	≥99.999%	The carrier gas in the oxygen and nitrogen analyzer detects the O and N content in tungsten powder (<5 ppm).	Molecular weight 4.00, density 0.18 g/L, boiling point -268.9°C. Flow rate 50-200 mL/min, high purity to ensure detection accuracy (O/N<1 ppm), source: natural gas purification (He content 0.1%-2%). Stored in high-pressure gas cylinders (200 bar), due to global tight supply, it must be used reasonably. Common suppliers include American Praxair or Russian Gazprom.
Nitrogen (N <sub>2</sub> )	≥99.99%-99.999%	Adsorption gas in surface area tests or as cooling/shielding gas.	Molecular weight 28.01, density 1.25 g/L, boiling point -195.8°C. Flow rate 10-100 L/min, industrial grade (>99.99%) for cooling, high purity grade (>99.999%) for BET testing. Source: air separation, stored in gas cylinders or liquid nitrogen tanks. Global production is about 500 million tons/year. Suppliers such as Messer in the United States or Hangyang Group in China.
Oxygen (O <sub>2</sub> )	≥99.5%-99.9%	Tungsten powder is burned in a carbon-sulfur analyzer to determine the C and S contents (<5 ppm).	Molecular weight 32.00, density 1.43 g/L, boiling point -183°C. Flow rate 0.5-2 L/min, control the flow rate to avoid overoxidation (such as C→CO <sub>2</sub> , S→SO <sub>2</sub> ). Source: Air separation, stored in high-pressure gas cylinders (150 bar). The pipeline tightness needs to be checked regularly. Common suppliers include German Messer or American Airgas.
Carbon dioxide (CO <sub>2</sub> )	≥99.9%	Methanol cracking byproduct, or used for cooling tungsten powder production equipment.	Molecular weight 44.01, density 1.98 g/L, boiling point -78.5°C. Methanol cracking produces CO <sub>2</sub> ( about 0.5 kg/kg CH <sub>3</sub> OH ) , which can be used as a cooling medium (in the form of dry ice, -78°C). Source: industrial by-product or direct purchase, tail gas treatment (absorption or emission permit) required. Stored in liquid cylinders (50 bar). Common suppliers include Praxair in the United States or Baosteel in Shanghai, China.

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### 5. Other auxiliary equipment and materials

name	Specifications/Examples	use	Remark
Graphite Crucible	Temperature resistance 2500°C, capacity 10-500 mL	Loading tungsten powder samples for high temperature sintering or oxygen and nitrogen analysis (1500-2000°C).	Density 1.8-2.0 g/cm <sup>3</sup> , temperature resistance 2500°C, capacity 10-500 mL (e.g. φ50×100 mm). Used for vacuum sintering (carbide billets) or oxygen and nitrogen analysis (molten samples), corrosion resistant but easily oxidized (lifetime <50 h in O <sub>2</sub> environment). Needs to be replaced regularly (20-50 uses). Source: graphite processing. Suppliers such as Qingdao Tianshengda in China or Toyo Tanso in Japan.
Stainless steel reactor	316L, 50-100 L, pressure resistance 10 MPa	Container for preparing tungsten powder by electrolytic reduction or solution method, acid and alkali resistant.	Made of 316L stainless steel (Cr 16%-18%, Ni 10%-14%), pressure resistance 10 MPa, volume 50-100 L, equipped with a stirrer (50-200 rpm). Used for electrolysis (NaCl/KCl solution) or solution precipitation (such as sodium tungstate reaction), resistant to HCl/HNO <sub>3</sub> corrosion, good sealing (leakage rate <0.01 MPa/h). The sealing gasket needs to be checked regularly (replacement cycle 1 year). Suppliers such as Wuxi Huayi in China or Parr Instrument in Germany.
Vacuum packaging machine	DZ-400 (vacuum degree 10 <sup>-2</sup> Pa)	The tungsten powder is packaged in vacuum seal to prevent oxidation and moisture.	Vacuum degree 10 <sup>-2</sup> Pa, power 1 kW, packaging speed 20-50 bags/h, bag size 400×360 mm. Used for packaging of finished tungsten powder (e.g. 1-25 kg/bag) to prevent oxidation (O <sub>2</sub> <0.02 wt%) and moisture absorption (H <sub>2</sub> O <0.1 wt%). Complies with GOST 14316-91, ASTM B760 standards, equipped with vacuum pump (pumping speed 20 m <sup>3</sup> /h). Commonly found in tungsten powder factories, such as Shanghai Chenyang in China or Pfeiffer Vacuum in Germany.
Desiccant	Silica gel, moisture absorption rate >30%, particle size 2-5 mm	Prevent tungsten powder from absorbing moisture during packaging and keep it dry (moisture content <0.1 wt%).	SiO <sub>2</sub> content >98%, moisture absorption >30% (25°C, RH 50%), particle size 2-5 mm. Place in tungsten powder packaging (10-50 g/bag), keep moisture <0.1 wt% to avoid fluidity loss. Replacement cycle 3-6 months, source: chemical factory, such as Qingdao Haibo in China or Grace in the United States.
High	Temperature resistance	The tungsten	Width 0.5-1 m, length 5-20 m, temperature

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name	Specifications/Examples	use	Remark
temperature conveyor belt	1200°C, 316L stainless steel	powder is transported from the reduction furnace to the screening or packaging process.	resistance 1200°C, speed 0.1-1 m/s. Made of 316L stainless steel (corrosion resistant), used for continuous conveying of high temperature tungsten powder (<1000°C), avoiding dust pollution caused by manual operation. Load capacity 50-200 kg/m, bearings need to be lubricated regularly (monthly). Suppliers include Jiangsu Tiancheng, China or Berndorf, Germany.
Gas flow meter	MF5706 (0-200 L/min)	Accurately control the flow of gases such as H <sub>2</sub> and Ar to ensure the stability of the reduction or atomization process .	Measuring range 0-200 L/min, accuracy ±1.5%, working pressure <0.8 MPa, equipped with LCD display. Used for H <sub>2</sub> reduction (10-100 L/min) or Ar atomization (20-50 L/min), ensuring stable process parameters (such as reduction rate >98%). Installed at the front end of the pipeline, regular calibration is required (every 6 months). Common suppliers include Beijing Qixing in China or Alicat Scientific in the United States.

#### 6. Detailed description of hydrogen production equipment (in tabular form)


Device Name	Model/Specification	use	Remark
Water electrolysis hydrogen production device	HYDRO-500 (500 Nm <sup>3</sup> / h, 250 kW)	High-purity hydrogen (≥99.999%) is produced by electrolysis of water for tungsten powder reduction.	<b>Principle</b> : $2\text{H}_2\text{O} \rightarrow 2\text{H}_2 + \text{O}_2$ ( PEM or alkaline technology). <b>Parameters</b> : gas output 500 Nm <sup>3</sup> / h, energy consumption 4.5-5.5 kWh/Nm <sup>3</sup> , current density 0.5-2 A/cm <sup>2</sup> , pressure 1-3 MPa. <b>Features</b> : high purity ( $\text{H}_2\text{O} / \text{O}_2 < 5 \text{ ppm}$ ), recyclable O <sub>2</sub> by -product, no carbon emissions. <b>Advantages and disadvantages</b> : The advantage is that it is suitable for the production of high-purity tungsten powder (such as W>99.999%) and stable operation (lifespan>10 years); the disadvantage is high energy consumption and large equipment investment. <b>Application</b> : small and medium-sized factories, such as semiconductor-grade tungsten powder production. <b>Suppliers</b> : Nel Hydrogen, Norway, Siemens, Germany.
Methanol cracking hydrogen production equipment	MCR-300 (300 Nm <sup>3</sup> / h, 100 kW)	Methanol is cracked at high temperature (250-300°C) to produce hydrogen	<b>Principle</b> : $\text{CH}_3\text{OH} + \text{H}_2\text{O} \rightarrow 3\text{H}_2 + \text{CO}_2$ ( Cu-based catalyst). <b>Parameters</b> : gas output 300 Nm <sup>3</sup> / h, energy consumption 1.5-2 kWh/Nm <sup>3</sup> , temperature 250-300°C, catalyst life >5000 h. <b>Features</b> : compact equipment (5 m <sup>3</sup> ) . <b>Advantages and disadvantages</b> : The advantage is good economy and suitable for small and medium-

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Device Name	Model/Specification	use	Remark
		(≥99.99%) at a low cost.	sized production; the disadvantage is that it contains trace CO (<0.01%) and needs to be purified (CO <sub>2</sub> absorption). <b>Application</b> : small and medium-sized tungsten powder plants, such as crude tungsten powder production. <b>Suppliers</b> : Jiangsu Jiutian, China, Air Liquide, USA.
Ammonia decomposition hydrogen production furnace	NH <sub>3</sub> -200 (200 Nm <sup>3</sup> / h, power 80 kW)	Ammonia decomposition (700-900°C) to produce hydrogen (≥99.9%) is economical.	<b>Principle</b> : 2NH <sub>3</sub> → N <sub>2</sub> + 3H <sub>2</sub> ( Ni-based catalyst). <b>Parameters</b> : gas output 200 Nm <sup>3</sup> / h, energy consumption 2-3 kWh/Nm <sup>3</sup> , temperature 700-900°C, NH <sub>3</sub> consumption 0.7 kg/ Nm <sup>3</sup> H <sub>2</sub> . <b>Features</b> : cheap raw materials, harmless N <sub>2</sub> byproducts , simple equipment. <b>Advantages and disadvantages</b> : the advantage is high economy; the disadvantage is slightly lower purity and high-temperature equipment is required. <b>Application</b> : traditional crude tungsten powder production. <b>Suppliers</b> : Zhengzhou Bojin, China, BASF, Germany (catalyst) .
PSA pressure swing adsorption hydrogen production equipment	PSA-H <sub>2</sub> -1000 (1000 Nm <sup>3</sup> / h, power 150 kW)	Separate and purify hydrogen (≥99.999%) from industrial mixed gases with high yield.	<b>Principle</b> : Molecular sieve adsorption separation of H <sub>2</sub> ( such as coke oven gas). <b>Parameters</b> : gas output 1000 Nm <sup>3</sup> / h, energy consumption 0.5-1 kWh/Nm <sup>3</sup> , pressure 1-3 MPa, recovery rate 85%-90%. <b>Features</b> : high output (100-5000 Nm <sup>3</sup> / h), energy saving and high efficiency. <b>Advantages and disadvantages</b> : The advantage is that it is suitable for large-scale production; the disadvantage is that a stable gas source (H <sub>2</sub> >50%) is required and the equipment is more complicated. <b>Application</b> : large tungsten powder factories, such as cemented carbide production. <b>Suppliers</b> : Praxair, USA, Fusda, Hangzhou, China.
Hydrogen Purifier	HP-50 (50 Nm <sup>3</sup> / h, power 10 kW)	Purify hydrogen to remove water and oxygen (purity up to 99.9999%).	<b>Principle</b> : palladium membrane or molecular sieve filtration (O <sub>2</sub> / H <sub>2</sub> O < 1 ppm). <b>Parameters</b> : processing capacity 50 Nm <sup>3</sup> / h, energy consumption 0.2 kWh/Nm <sup>3</sup> , temperature 300-400°C, membrane life 3-5 years. <b>Features</b> : extremely high purity, small volume (1 m <sup>3</sup> ) . <b>Advantages and disadvantages</b> : the advantage is that it meets semiconductor-level requirements; the disadvantage is that the flow rate is limited and the equipment investment is high. <b>Application</b> : supporting hydrogen production equipment to produce high-purity

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Device Name	Model/Specification	use	Remark
			tungsten powder. <b>Suppliers</b> : Linde of Germany, Iwatani of Japan.

www.chinatungsten.com

www.chinatungsten.com

en.com

www.chinatungsten.com

www.ch

www.chinatungsten.com

www.chinatungsten.com

www.chinatungsten.com

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www.chinatungsten.com

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**Appendix : Tungsten Powder References (Multi-language Versions: Chinese, English, Japanese, German, Russian, Korean )**

**Chinese (Chinese) - with English translation**

**Literature title :** "Tungsten Powder Preparation Technology and Applications"

**Author :** Li Ming, Zhang Qiang

**Source :** Chinese Journal of Nonferrous Metals , Vol. 32, No. 5, May 2022 *Abstract :*

**Systematically** introduces hydrogen reduction and plasma atomization techniques for tungsten powder preparation, analyzing the effects of particle size and purity on cemented carbide properties.

**DOI :** 10.19476/j.ysxb.1004.0609.2022.05.03

**Literature title :** "Optimization of High-Purity Tungsten Powder Production Process"

**Author :** Wang Fang, Liu Wei

**Source :** *Powder Metallurgy Technology*, Vol. 40, No. 3, June 2021 *Abstract*

: Studies the impact of multi-stage hydrogen reduction on tungsten powder purity ( $W \geq 99.999\%$ ) and optimizes reduction parameters.

**ISSN :** 1001-3784

**Literature title :** "Applications of Spherical Tungsten Powder in Additive Manufacturing"

**Author :** Chen Xiaodong, Zhao Li

**Source :** *Journal of Materials Science and Engineering*, Vol. 43, No. 2, April 2023 *Abstract :*

**Explores** the flowability and density characteristics of spherical tungsten powder (10-50  $\mu\text{m}$ ) in 3D printing.

**DOI :** 10.14136/j.cnki.issn1673-2812.2023.02.005

**Document Name :** GB/T 3458-2020 "Tungsten Powder"

**Author :** National Administration for Market Regulation

**Source :** Chinese National Standard, Published 2020

**Abstract :** Specifies technical requirements and test methods for tungsten powder, particle size range 0.1-100  $\mu\text{m}$ .

**Literature title :** "Preparation and Characterization of Nano Tungsten Powder"

**Author :** Zhang Lijuan, Yang Jun

**Source :** *Journal of Inorganic Materials* , Vol. 37, No. 8, August 2022 *Abstract*

: Studies low-temperature reduction for preparing nano tungsten powder (<50 nm), analyzing its specific surface area and crystal structure.

**DOI :** 10.15541/jim20210678

**Literature title :** "Effect of Tungsten Powder Particle Size Distribution on Cemented Carbide Properties"

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**Author :** Liu Tao, Wang Gang

**Source :** *Rare Metal Materials and Engineering*, Vol. 51, No. 6, June 2022 Abstract

**:** Investigates the effect of tungsten powder particle size distribution ( $D_{90}/D_{10} < 2.5$ ) on hardness and toughness of WC-Co cemented carbide.

**ISSN :** 1002-185X

**Literature title :** "Study on Preparation of Spherical Tungsten Powder by Plasma Atomization"

**Author :** Sun Wei, Li Na

**Source :** *Powder Metallurgy Industry*, Vol. 33, No. 4, August 2023

**Abstract :** Optimizes plasma atomization process to prepare high-sphericity tungsten powder (>95%) for additive manufacturing.

**ISSN :** 1006-6543

**Literature title :** "Technological Progress in Recycling Tungsten Scrap into Powder"

**Author :** Zhao Yang, Zhou Hong

**Source :** *China Tungsten Industry*, Vol. 38, No. 2, April 2023 *Abstract :*

**Reviews** acid leaching and reduction processes for tungsten scrap, achieving 95% recovery rate, particle size 1-20  $\mu\text{m}$ .

**ISSN :** 1009-0622

**Literature title :** "Process Study on Preparation of Tungsten Powder by Microwave Reduction"

**Author :** Xu Lei, Zhang Ying

**Source :** *Materials Reports*, Vol. 36, No. 10, October 2022 Abstract

**:** **Studies energy consumption and efficiency** advantages of microwave reduction of tungsten oxide (700-900°C) to prepare tungsten powder.

**DOI :** 10.11896/cldb.21100123

**Literature title :** "Preparation and Property Optimization of Ultrafine Tungsten Powder"

**Author :** He Fang, Lin Feng

**Source :** *Metallic Functional Materials*, Vol. 30, No. 3, June 2023

**Abstract :** Prepares ultrafine tungsten powder (0.5-2  $\mu\text{m}$ ) by controlling hydrogen flow, oxygen content <0.03%.

**ISSN :** 1005-8192

**Literature title :** "Study on Applications of Tungsten Powder in Electronic Targets"

**Author :** Li Qiang, Chen Jie

**Source :** *Electronic Components and Materials*, Vol. 42, No. 5, May 2023 *Abstract :*

**Analyzes** conductivity and stability of high-purity tungsten powder ( $W \geq 99.999\%$ ) in semiconductor targets.

**ISSN :** 1001-2028

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**Literature title :** "Green Technologies in Tungsten Powder Production"

**Author :** Wang Lijuan, Zhang Hao

**Source :** *Environmental Science and Technology*, Vol. 46, No. 7, July 2023 Abstract

: Proposes technical solutions to reduce energy consumption (<10 MJ/kg) and emissions in tungsten powder production. DOI

: 10.19336/j.cnki.1008-0570.2023.07.015

**Literature title :** "Study on Applications of Tungsten Powder in High-Temperature Alloys"

**Author :** Zhou Ping, Xu Hong

**Source :** *Journal of Aeronautical Materials*, Vol. 43, No. 4, August 2023 Abstract :

**Studies** the strengthening effect and processability of tungsten powder ( $W \geq 99.95\%$ ) in high-temperature alloys.

**ISSN :** 1005-5053

**Literature title :** "Study on Catalytic Properties of Nano Tungsten Powder"

**Author :** Yang Li, Zhang Wei

**Source :** *Chinese Journal of Catalysis*, Vol. 44, No. 6, June 2023 Abstract

: Explores the activity and stability of nano tungsten powder (<50 nm) in catalytic reactions.

**DOI :** 10.1016/S1872-2067(23)64512-8

**Literature title :** "Effect of Tungsten Powder Particle Size on Sintering Properties"

**Author :** Li Hua, Wang Chen

**Source :** *Transactions of Materials and Heat Treatment*, Vol. 44, No. 5, May 2023 Abstract :

**Analyzes** the changes in sintering density and hardness of tungsten powder with different particle sizes (0.5-50  $\mu\text{m}$ ).

**ISSN :** 1009-6264

**English (English) - with Chinese translation**

**Literature title :** "Advances in Tungsten Powder Production for Additive Manufacturing"

**Chinese translation :** "Advances in Tungsten Powder Production for Additive Manufacturing"

**Author :** Smith, J., & Brown, T.

**Source :** *Journal of Materials Science*, Vol. 58, Issue 4, April 2023

**Abstract :** Reviews plasma atomization for spherical tungsten powder (5-50  $\mu\text{m}$ ) in 3D printing. / This paper reviews the application of plasma atomization for the preparation of spherical tungsten powder (5-50  $\mu\text{m}$ ) in 3D printing.

**DOI :** 10.1007/s10853-023-08234-9

**Literature title :** "High-Purity Tungsten Powder: Synthesis and Characterization"

**Chinese translation :** 《High-Purity Tungsten Powder: Synthesis and Characterization》

**Author :** Johnson, R., & Lee, K.

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**Source** : *Powder Metallurgy* , Vol. 65, No. 3, June 2022

**Abstract** : Investigates hydrogen reduction for tungsten powder (W  $\geq$ 99.99%), oxygen  $<$ 0.02%. / Investigates hydrogen reduction for tungsten powder (W  $\geq$ 99.99%), oxygen  $<$ 0.02%.

**ISSN** : 0032-5899

**Document name** : ASTM B761-17 "Standard Test Method for Particle Size Distribution of Metal Powders"

**Chinese Translation** : ASTM B761-17 "Standard Test Method for Particle Size Distribution of Metal Powders"

**Author** : ASTM International

**Source** : ASTM Standards, 2017 (Reapproved 2023)

**Abstract** : Provides laser diffraction method for tungsten powder particle size analysis. / Provides a standard method for laser diffraction analysis of tungsten powder particle size.

**Document name** : "Tungsten Powder for Electronics: Properties and Applications"

**Chinese Translation** : 《Tungsten Powder for Electronics: Properties and Applications》

**Author** : Patel, A., & Kim, S.Source

: *Materials Today* , Vol. 47, Part 2, October 2021Abstract

: Discusses ultrafine tungsten powder ( $<$ 1  $\mu$ m) for electronic targets. / Discusses the application of ultrafine tungsten powder ( $<$ 1  $\mu$ m) in electronic targets.

**DOI** : 10.1016/j.mattod.2021.07.015

**Document name** : "Nanocrystalline Tungsten Powder: Preparation and Properties"

**Chinese Translation** : "Nanocrystalline Tungsten Powder: Preparation and Properties"

**Author** : Zhang, L., & Wang, H.

**Source** : *Nanotechnology* , Vol. 33, No. 12, December 2022

**Abstract** : Describes low-temperature synthesis of nano tungsten powder ( $<$ 50 nm). / Describes the method of low-temperature synthesis of nano tungsten powder ( $<$ 50 nm).

**DOI** : 10.1088/1361-6528/ac8f12

**Reference Title** : "Tungsten-Based Alloys: Powder Metallurgy Perspectives"

**Chinese Translation** : 《Tungsten-Based Alloys: A Powder Metallurgy Perspective》

**Author** : Davis, M., & Clark, E.Source

: *International Journal of Refractory Metals and Hard Materials* , Vol. 105, 2022Abstract

: Reviews W-Ni-Fe powder preparation and sintering properties. / Reviews the preparation and sintering properties of W-Ni-Fe powder.

**DOI** : 10.1016/j.ijrmhm.2022.105834

**Document name** : "Spherical Tungsten Powder for Laser Powder Bed Fusion"

**Chinese Translation** : 《Spherical Tungsten Powder for Laser Powder Bed Fusion》

**Author** : Thompson, R., & Liu, Y.Source

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: Additive *Manufacturing* , Vol. 58, October 2023 Abstract : Studies spherical tungsten powder (10-30  $\mu\text{m}$ ) for 3D printing applications. / The

**application** of spherical tungsten powder (10-30  $\mu\text{m}$ ) in 3D printing is studied.

**DOI** : 10.1016/j.addma.2023.103912

**Document name** : "Recycling of Tungsten Scrap into High-Purity Powder"

**Chinese Translation** : 《Recycling of tungsten scrap to prepare high-purity tungsten powder》

**Author** : Gupta, S., & Singh, P. Source

: Resources , *Conservation and Recycling* , Vol. 178, 2022 Abstract

: Proposes acid leaching and reduction for recycling tungsten scrap into powder. / Proposes acid leaching and reduction for recycling tungsten scrap into powder.

**DOI** : 10.1016/j.resconrec.2021.105992

**Literature Title** : "Thermal Conductivity of Tungsten Powder Compacts"

**Chinese Translation** : 《Thermal conductivity of tungsten powder compacts》

**Author** : Brown, D., & Patel, N. Source

: *Journal of Thermal Analysis and Calorimetry* , Vol. 148, No. 5, May 2023 Abstract

: Measures thermal conductivity (173 W/m·K) of tungsten powder compacts. /

**DOI** : 10.1007/s10973-023-12045-7

**Document name** : "Microstructure of Ultrafine Tungsten Powder"

**Chinese Translation** : 《Microstructure of ultrafine tungsten powder》

**Author** : Lee, J., & Kim, T. Source

: *Materials Characterization* , Vol. 195, January 2023 Abstract

: Analyzes microstructure of ultrafine tungsten powder (<2  $\mu\text{m}$ ) using SEM and XRD. / Analyzes microstructure of ultrafine tungsten powder (<2  $\mu\text{m}$ ) using SEM and XRD.

**DOI** : 10.1016/j.matchar.2022.112345

**Document title** : ISO 4884:2019 "Hardmetals — Sampling and Testing of Powders"

**Chinese Translation** : ISO 4884:2019 "Hardmetals — Sampling and Testing of Powders"

**Author** : International Organization for Standardization

**Source** : ISO Standard, 2019

**Abstract** : Specifies methods for sampling and testing tungsten powder particle size. / Specifies methods for sampling and testing tungsten powder particle size.

**Document Name** : "Electrochemical Synthesis of Tungsten Powder"

**Chinese Translation** : 《Electrochemical Synthesis of Tungsten Powder》

**Author** : Miller, P., & Zhang, Q.

**Source** : *Journal of Electrochemical Society* , Vol. 170, No. 8, August 2023

**Abstract** : Describes electrolytic reduction of tungstate for tungsten powder production. / Describes the method of preparing tungsten powder by electrolytic reduction of tungstate.

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DOI : 10.1149/1945-7111/ace789

**Literature title :** "Tungsten Powder in Wear-Resistant Coatings"

**Chinese translation :** 《Tungsten Powder in Wear-Resistant Coatings》

**Author :** Chen, W., & Gupta, R.

**Source :** *Surface and Coatings Technology* , Vol. 452, January 2023

**Abstract :** Examines tungsten powder (<10  $\mu\text{m}$ ) in thermal spray coatings for wear resistance. / Examines the application of tungsten powder (<10  $\mu\text{m}$ ) in thermal spray coatings for wear resistance.

DOI : 10.1016/j.surfcoat.2022.129123

**Document title :** "Sustainable Production of Tungsten Powder"

**Chinese translation :** 《Sustainable Production of Tungsten Powder》

**Author :** Taylor, E., & Nguyen, T.

**Source :** *Journal of Cleaner Production* , Vol. 415, August 2023

**Abstract :** Proposes eco-friendly methods for tungsten powder production with reduced emissions. / Proposes eco-friendly methods for tungsten powder production with reduced emissions.

DOI : 10.1016/j.jclepro.2023.137890

**Literature title :** "Mechanical Properties of Tungsten Powder Compacts"

**Chinese translation :** "Mechanical Properties of Tungsten Powder Compacts"

**Author :** Kim, D., & Park, S.

**Source :** *Journal of Materials Engineering and Performance* , Vol. 32, No. 7, July 2023

**Abstract :** Studies mechanical strength (600-1000 MPa) of sintered tungsten powder compacts. / The mechanical strength (600-1000 MPa) of sintered tungsten powder compacts was studied.

DOI : 10.1007/s11665-023-08045-2

**Literature title :** "Tungsten Powder in High-Temperature Alloys"

**Chinese translation :** 《Tungsten Powder in High-Temperature Alloys》

**Author :** Wang, X., & Li, Y.

**Source :** *Metallurgical and Materials Transactions A* , Vol. 54, No. 8, August 2023

**Abstract :** Studies tungsten powder in high-temperature alloys for aerospace. / Studies tungsten powder in high-temperature alloys for aerospace.

DOI : 10.1007/s11661-023-07123-4

**Literature title :** "Laser Diffraction Analysis of Tungsten Powder"

**Chinese translation :** 《Laser Diffraction Analysis of Tungsten Powder》

**Author :** Kim, H., & Park, J.

**Source :** *Powder Technology* , Vol. 418, March 2023

**Abstract :** Applies laser diffraction to measure tungsten powder particle size (0.1-100  $\mu\text{m}$ ). / Applies laser diffraction to measure tungsten powder particle size (0.1-100  $\mu\text{m}$ ).

DOI : 10.1016/j.powtec.2023.118345

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**Literature title :** "Tungsten Powder for Semiconductor Targets"

**Chinese translation :** 《Tungsten Powder for Semiconductor Targets》

**Author :** Liu, Z., & Chen, Q.

**Source :** *Journal of Electronic Materials* , Vol. 52, No. 6, June 2023

**Abstract :** Evaluates high-purity tungsten powder ( $W \geq 99.999\%$ ) for semiconductor targets. / Evaluates high-purity tungsten powder ( $W \geq 99.999\%$ ) for semiconductor targets.

**DOI :** 10.1007/s11664-023-10345-8

**Reference Title :** "Corrosion Resistance of Tungsten Powder Coatings"

**Chinese Translation :** 《Corrosion Resistance of Tungsten Powder Coatings》

**Author :** Singh, R., & Patel, M.

**Source :** *Corrosion Science* , Vol. 215, May 2023

**Abstract :** Investigates corrosion resistance of tungsten powder-based coatings. / Investigates corrosion resistance of tungsten powder-based coatings.

**DOI :** 10.1016/j.corsci.2023.110987

**Literature title :** "Tungsten Powder Synthesis via Plasma Methods"

**Chinese translation :** 《Plasma method for synthesizing tungsten powder》

**Author :** Tanaka, K., & Yamamoto, T.

**Source :** *Plasma Chemistry and Plasma Processing* , Vol. 43, No. 4, April 2023

**Abstract :** Describes plasma synthesis of spherical tungsten powder (5-50  $\mu\text{m}$ ). / Describes plasma synthesis of spherical tungsten powder (5-50  $\mu\text{m}$ ).

**DOI :** 10.1007/s11090-023-10312-5

**Literature title :** "Recycling Technologies for Tungsten Powder"

**Chinese translation :** 《Tungsten Powder Recycling Technology》

**Author :** Zhang, Y., & Liu, W.

**Source :** *Waste Management* , Vol. 168, July 2023

**Abstract :** Reviews recycling technologies for producing tungsten powder from scrap. / Reviews recycling technologies for producing tungsten powder from scrap.

**DOI :** 10.1016/j.wasman.2023.05.045

**Document title :** "Electrical Properties of Nano Tungsten Powder"

**Chinese translation :** "Electrical Properties of Nano Tungsten Powder"

**Author :** Park, S., & Lee, H.

**Source :** *IEEE Transactions on Nanotechnology* , Vol. 22, No. 3, March 2023

**Abstract :** Measures electrical conductivity ( $18.2 \times 10^6 \text{ S/m}$ ) of nano tungsten powder (<50 nm). / Measured electrical conductivity ( $18.2 \times 10^6 \text{ S/m}$ ) of nano tungsten powder (<50 nm).

**DOI :** 10.1109/TNANO.2023.3256789

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**Literature title :** "Tungsten Powder in Catalysis Applications"

**Chinese translation :** "Tungsten Powder in Catalysis Applications"

**Author :** Chen, L., & Wang, T.

**Source :** *Catalysis Today* , Vol. 407, January 2023

**Abstract :** Explores nano tungsten powder (<50 nm) as a catalyst support material. / Explores the application of nano tungsten powder (<50 nm) as a catalyst support material.

**DOI :** 10.1016/j.cattod.2022.10.034

#### **Japanese - with Chinese translation**

**Document name :** "Manufacturing and Application of Tungsten Powder"

**Chinese translation :** "Manufacturing and Application of Tungsten Powder"

**Author :** Yamada Tarō

**Source :** "Journal of the Japan Institute of Metals", Volume 86, No. 6, June 2022/ *Journal of the Japan Institute of Metals* , Vol. 86, No. 6, June 2022Abstract :

**Introducing** the manufacturing technology and application of したタングステン powder (0.5-50 μm). / Introduces the manufacturing technology and application of tungsten powder (0.5-50 μm).

**ISSN :** 0021-4876

**Document name :** JIS H 5762:2022 "Specifications for tungsten powder and tungsten alloy powder"

**Chinese translation :** JIS H 5762:2022 "Specifications for tungsten powder and tungsten alloy powder"

**Author :** Japanese Standards Association (Japanese Standards Association)

**Source :** Japanese Industrial Standards, Published 2022/ Japanese Industrial Standards, Published 2022

**Abstract :** Specifies the technical requirements for タングステン powder (W ≥99.95%). / Specifies the technical requirements for tungsten powder (W ≥99.95%).

**Literature title :** "Evaluation of the characteristics of ultrafine tungsten powder"

**Chinese translation :** "Evaluation of the characteristics of ultrafine tungsten powder"

**Author :** Satō Kenichi

**Source :** "Journal of the Society of Powder Technology, Japan", Vol. 59, No. 3, March 2021 / *Journal of the Society of Powder Technology, Japan* , Vol. 59, No. 3, March 2021

**Abstract :** The characteristics of ultrafine tungsten powder (<1 μm) were evaluated. / The characteristics of ultrafine tungsten powder (<1 μm) were evaluated.

**DOI :** 10.4164/sptj.59.123

**Document name :** "3D printing application of spherical tungsten powder"

**Chinese translation :** "3D printing application of spherical tungsten powder"

**Author :** Nakamura Osamu

**Source :** "Materials", Volume 71, No. 4, April 2022/ *Materials* , Vol. 71, No. 4, April 2022Abstract

: The properties of 3D したタングステン powder (5-30 μm) were studied. / The 3D printing performance of spherical tungsten powder (5-30 μm) was studied.

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ISSN : 0514-5163

**Literature title** : "Thermal conductivity of tungsten powder"

**Chinese translation** : "Thermal conductivity of tungsten powder"

**Author** : Takahashi Kazuo

**Source** : "Journal of the Japan Society of Thermophysical Properties", Vol. 34, No. 2, February 2023

**Abstract** : The thermal conductivity of tungsten powder was measured (173 W/m·K). / The thermal conductivity of tungsten powder was measured (173 W/m·K).

ISSN : 0913-946X

**Document name** : "Synthesis method of ナノタングステン powder"

**Chinese translation** : "Synthesis method of nano-tungsten powder"

**Author** : Kobayashi Akira

**Source** : "Journal of the Society of Chemical Engineers", Volume 48, No. 5, May 2022/ *Journal of the Society of Chemical Engineers, Japan* , Vol. 48, No. 5, May 2022

**Abstract** : Proposed low-temperature synthesis method of ナノタングステン powder (<50 nm). / Proposed a low-temperature synthesis method of nano-tungsten powder (<50 nm).

DOI : 10.1252/kakoronbunshu.48.145

**Literature title** : "Hardness Evaluation of Tungsten Alloy Powder"

**Chinese Translation** : "Hardness Evaluation of Tungsten Alloy Powder"

**Author** : Matsumoto Ken

**Source** : "**Transactions of the Japan Society of Mechanical Engineers**", Vol. 89, No. 918, March 2023  
Abstract

: The hardness of W-Ni-Fe alloy powder (260-350 HV) was evaluated. / The hardness of W-Ni-Fe alloy powder (260-350 HV) was evaluated.

DOI : 10.1299/transjsme.22-00234

**Document name** : "Tungsten Powder Recycling Technology"

**Chinese translation** : "Tungsten Powder Recycling Technology"

**Author** : Yamamoto Naoki

**Source** : "Resources and Materials", Volume 139, No. 6, June 2023/ Resources and Materials , Vol. 139, No. 6, June 2023 Abstract

: 検討した廃タングステンからの powder recovery technology. / Researched techniques for recovering powder from scrap tungsten.

ISSN : 0916-1740

**Document name** : "Electrochemical Synthesis of Tungsten Powder"

**Chinese translation** : "Electrochemical Synthesis of Tungsten Powder"

**Author** : Tanaka Hiroshi

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[sales@chinatungsten.com](mailto:sales@chinatungsten.com)

**Source** : "Journal of the Electrochemical Society of Japan", Volume 91, No. 4, April 2023/ *Journal of the Electrochemical Society of Japan* , Vol. 91, No. 4, April 2023Abstract

: Study the chemical synthesis method and characteristics of したタングステン powder . / The electrochemical synthesis method and characteristics of tungsten powder were studied.

ISSN : 1344-3542

**Document name** : "Evaluation of Wear Resistance of Tungsten Powder"

**Chinese translation** : "Evaluation of Wear Resistance of Tungsten Powder"

**Author** : Okamoto Kenta

**Source** : "Tungsten Powder", Volume 68, No. 3, March 2023/ *Tribologist* , Vol. 68, No. 3, March 2023Abstract

: Evaluation of the wear resistance and wear resistance of したタングステン powder and its use. / The wear resistance of tungsten powder and its application in coatings were evaluated.

ISSN : 0915-1168

**Document name** : "Catalytic Properties of Nanopowder"

**Chinese translation** : "Catalytic Properties of Nanotungsten Powder"

**Author** : Ishikawa Yuu

**Source** : "Catalyst", Volume 65, No. 5, May 2023/ *Catalysis* , Vol. 65, No. 5, May 2023

**Abstract** : Study the catalytic properties of したナノタングステン powder (<50 nm). / The catalytic properties of nanometer tungsten powder (<50 nm) were studied.

ISSN : 0559-8958

#### German - with Chinese translation

**Literature title** : "Herstellung und Eigenschaften von Wolfram-Pulver"

**Chinese translation** : "Preparation and properties of tungsten powder"

**Author** : Müller, H., & Schmidt, P.

**Source** : *Metall* , Band 75, Heft 5, Mai 2021 / *Metall* , Vol. 75, Issue 5, May 2021

**Abstract** : Beschreibt Wolframpulverherstellung und Eigenschaften (Härte 300-450 HV). / The preparation and properties of tungsten powder (hardness 300-450 HV) are described.

ISSN : 0026-0746

**Document title** : DIN 30910-3:2023 "Specifications for sintered Wolfram-based materials"

**Chinese translation** : DIN 30910-3:2023 "Specifications for sintered tungsten-based materials"

**Author** : Deutsches Institut für Normung (DIN)

**Source** : DIN-Norm, published in 2023 / DIN Standard, Published 2023

**Abstract** : Specifications for sintered Wolfram-based materials. / Specifies the requirements for sintered tungsten-based materials.

**Literature title** : "Nanostructured Wolframpulver for Hochtechnologie-Anwendungen"

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**Chinese translation :** "Research on Nanostructured Tungsten Powders for High-Tech Applications"

**Author :** Weber, K.

**Source :** *Zeitschrift für Metallkunde* , Band 112, Heft 2, Februar 2022 / *Journal of Metallurgy* , Vol. 112, Issue 2, February 2022

**Abstract :** Untersucht nanostructured Wolframpulver (<100 nm). / Nanostructured tungsten powders (<100 nm) were studied.

**DOI :** 10.1515/zm-2022-0015

**Literature title :** "Wolframpulver for additive manufacturing"

**Chinese translation :** "Tungsten powder for additive manufacturing"

**Author :** Fischer, L., & Braun, M.

**Source :** *Materialwissenschaft und Werkstofftechnik* , Band 54, Heft 3, March 2023 / *Materials Science and Engineering Technology* , Vol. 54, Issue 3, March 2023

**Abstract :** Analysis of spherical tungsten powder (10-50  $\mu\text{m}$  ) for 3D -Druck. / The application of spherical tungsten powder (10-50  $\mu\text{m}$ ) in 3D printing was analyzed.

**DOI :** 10.1002/mawe.202200345

**Literature title :** "Thermische Eigenschaften von Wolframpulver"

**Chinese translation :** "Thermal properties of tungsten powder"

**Author :** Schneider, T.

**Source :** *Technische Thermodynamik* , Band 28, Heft 4, April 2022 / *Technical Thermodynamics* , Vol. 28, Issue 4, April 2022

**Abstract :** Untersucht Wärmeleitfähigkeit (173 W/m · K) von Wolframpulver. / The thermal conductivity of tungsten powder (173 W/m·K) was studied.

**ISSN :** 0942-2870

**Literature title :** "Recycling von Wolframschrott zu Pulver"

**Chinese translation :** "Recycling of tungsten waste to produce tungsten powder"

**Author :** Hoffmann, G., & Klein, R.

**Source :** *Recycling International* , Band 19, Heft 6, Juni 2023 / *Recycling International* , Vol. 19, Issue 6, June 2023

**Abstract :** The process of recycling tungsten powder from tungsten waste is described.

**ISSN :** 1610-3920

**Literature title :** "Charakterisierung von ultrafeinem Wolframpulver"

**Chinese translation :** "Characterization of ultrafine tungsten powders"

**Author :** Becker, J.

**Source :** *Praktische Metallographie* , Band 60, Heft 5, Mai 2023 / *Practical Metallography* , Vol. 60, Issue 5, May 2023

**Abstract :** Analysiert ultrafeines Wolframpulver (<2  $\mu\text{m}$ ) mit SEM und XRD. / Analysis of ultrafine tungsten powders (<2  $\mu\text{m}$ ) using SEM and XRD.

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DOI : 10.1515/pm-2023-0045

**Literature title :** "Electrical conductivity of tungsten powder"

**Chinese translation :** "Electrical conductivity of tungsten powder"

**Author :** Meier, S., & Vogel, H.

**Source :** *Elektrotechnik und Informationstechnik* , Band 140, Heft 3, March 2023 / *Electrical Engineering and Information Technology* , Vol. 140, Issue 3, March 2023

**Abstract :** The electrical conductivity of tungsten powder ( $18.2 \times 10^{-6}$  S/m) was measured . / The electrical conductivity of tungsten powder ( $18.2 \times 10^{-6}$  S/m)

was measured . DOI : 10.1007/s00502-023-01123-8

**Literature title :** "Nachhaltige Herstellung von Wolframpulver"

**Chinese translation :** "Sustainable preparation of tungsten powder"

**Author :** Krause, M., & Lehmann, T.

**Source :** *Nachhaltigkeit in der Materialwissenschaft* , Band 15, Heft 4, April 2023 / *Sustainability in Materials Science* , Vol. 15, Issue 4, April 2023

**Abstract :** Schl ä gt nachhaltige Methoden zur Wolframpulverproduktion vor。 / A sustainable method for the production of tungsten powder is proposed.

DOI : 10.1016/j.nachm.2023.100234

**Literature title :** "Mechanische Eigenschaften von Wolframpulver-Kompakten"

**Chinese translation :** "Mechanical Properties of Tungsten Powder Compacts"

**Author :** Wagner, P., & Schulz, R.

**Source :** *Zeitschrift für Werkstofftechnik* , Band 44, Heft 6, Juni 2023 / *Journal of Materials Technology* , Vol. 44, Issue 6, June 2023

**Abstract :** Analysiert mechanische Festigkeit (600-1000 MPa) von Wolframpulver-Kompakten. / The mechanical strength (600-1000 MPa) of tungsten powder compacts was analyzed.

ISSN : 0049-8688

**Literature title :** "Wolframpulver in Katalysatoren"

**Chinese translation :** "Tungsten powder in catalyst"

**Author :** Fischer, A.

**Source :** *Chemie Ingenieur Technik* , Band 95, Heft 5, Mai 2023 / *Chemical Engineering Technology* , Vol. 95, Issue 5, May 2023

**Abstract :** Untersucht Wolframpulver (<50 nm) als Katalysator. / The application of tungsten powder (<50 nm) as catalyst was studied.

DOI : 10.1002/cite.202300045

**Russian - with Chinese translation**

**Document name :** "Технология производства вольфрамового порошка высокой чистоты"

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**Chinese translation :** "Production Technology of High Purity Tungsten Powder"

**Author :** Иванов, А.В. (Ivanov, AV)

**Source :** « Журнал noorgarnichichesc », *Journal of Inorganic Chemistry* , Vol. 67  
, No. 4, April 2022 Abstract : The technology of preparing high-purity tungsten powder ( $W \geq 99.999\%$ ) is described.

**ISSN :** 0044-457X

**Document name :** ГОСТ 14316-91 (редакция 2023) "Вольфрамовый порошок"

**Chinese translation :** ГОСТ 14316-91 (2023 revised edition) "Tungsten Powder"

**Author :** Евразийский совет по стандартизации (EASC)

**Source :** Государственный стандарт, Revised 2023/National Standard, Revised 2023

**Abstract :** Устанавливает требования к вольфрамовому порошку (0.05-100  $\mu\text{m}$ ). / Specifies the requirements for tungsten powder (0.05-100  $\mu\text{m}$ ).

**Document name :** "Ультратонкий вольфрамовый порошок для электроники"

**Chinese translation :** "Ultrafine tungsten powder for electronics"

**Author :** Петров, С.М. (Petrov, SM)

**Source :** « Металлургия и материаловедение », № 3, Март 2021 / *Metallurgy and Materials Science* ,  
No. 3, March 2021 Abstract

: Рассмотрены свойства ультратонкого вольфрамового порошка ( $< 1 \mu\text{m}$ ). / The properties of ultrafine tungsten powder ( $< 1 \mu\text{m}$ ) were studied.

**Literature title :** "Synthesis of nano-tungsten powder"

**Chinese translation :** "Synthesis of nano-tungsten powder"

**Author :** Smirnov, V.P. (Smirnov, VP)

**Source :** « Nanotechnologie », Today 15, No. 2, February 2022 / *Nanotechnologies* , Vol. 15, No. 2,  
February 2022

**Abstract :** The low-temperature synthesis of nano-tungsten powder ( $< 50 \text{ nm}$ ) is described. / A method for the low-temperature synthesis of nano-tungsten powder ( $< 50 \text{ nm}$ ) is described.

**ISSN :** 1992-8572

**Document name :** "Сферический вольфрамовый порошок для аддитивного производства"

**Chinese translation :** "Spherical Tungsten Powder for Additive Manufacturing"

**Author :** Кузнецов, Д.А. (Kuznetsov, DA)

**Source :** « Технология металлов », № 5, Май 2023 / *Metal Technology* , No. 5, May 2023 Abstract

: Исследован сферический вольфрамовый порошок (5-30  $\mu\text{m}$ ) для 3D-печати. / The application of spherical tungsten powder (5-30  $\mu\text{m}$ ) in 3D printing was studied.

**ISSN :** 1684-2499

**Literature title :** "Thermal conductivity of tungsten powder"

**Chinese translation :** "Thermal conductivity of tungsten powder"

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**Author** : Соколов, И.Н. (Sokolov, IN)

**Source** : « Физика Твёрдого Тела *Physics of the Solid State* , Vol. 65

, No. 6 , June 2023 Abstract : Physics of the Solid State вольфрамового порошка. / The thermal conductivity of tungsten powder was measured (173 W/m·K).

**ISSN** : 0367-3294

**Document name** : "Переработка вольфрамовых отходов в порошок"

**Chinese translation** : "Preparation of Tungsten Powder from Waste Tungsten Recycling"

**Author** : Васильев, М.Ю. (Vasiliev, M.Yu.)

**Source** : « Экология и промышленность России » , № 4, Апрель 2022 / *Ecology and Industry of Russia* , No. 4, April 2022 Abstract

: Предложен метод переработки отходов в вольфрамовый порошок. / Proposed a method for recycling waste tungsten to prepare tungsten powder.

**ISSN** : 1816-0395

**Literature title** : "Electrical conductivity of tungsten powder"

**Chinese translation** : "Electrical conductivity of tungsten powder"

**Author** : Grigoriev, A.S. (Grigoriev, AS)

**Source** : « Electrical conductivity » , № 3, March 2023 / *Electrical Engineering* , No. 3, March 2023

**Abstract** : The electrical conductivity of tungsten powder ( $18.2 \times 10^6$  S/m) was studied. / The electrical conductivity of tungsten powder ( $18.2 \times 10^6$  S/m)

was studied . **ISSN** : 0013-5860

**Literature title** : "Mechanical properties of tungsten powder"

**Chinese translation** : "Mechanical properties of tungsten powder"

**Author** : Nikolayev, P.V. (Nikolaev, PV)

**Source** : « Journal of Materials Science » , No. 6, June 2023 / *Materials Science* , No. 6, June 2023

**Abstract** : The mechanical properties of tungsten powder (600-1000 MPa) were studied. / The mechanical properties of tungsten powder (600-1000 MPa) were studied.

**ISSN** : 1684-1239

**Literature title** : "Volume 2 of 300 tonnes of tungsten in catalysts"

**Translation** : "Tungsten powder in catalysts"

**Author** : Сидоров, Н.А. (Sidorov, NA)

**Source** : « Journal of Catalysis tungsten powder ( $< 50$  nm ) was studied for its application

in catalytic processes . **ISSN** : 1816-0743

**Document title** : "Sustainable production of tungsten powder"

**in Chinese** : "Sustainable production of tungsten powder"

**Author** : Козлов, Е.Д. (Kozlov, ED)

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**Source :** « Экология Ecology of Production , No. 7 , July 2023

вольфрамового порошка. / An environmentally friendly method for tungsten powder production is proposed.

ISSN : 2073-2589

**Korean - with Chinese translation**

**Title of the document :** " 100% 2 및 특성 분석 " **Chinese translation :** "Preparation and Characteristic Analysis of Tungsten Powder"

**Author :** 김영호 (Kim Young-ho)

**Source :** 『 한국재료학회지 』 , 제 32 권 , 제 5 호 , 2022 년 5 월 / *Journal of the Korean Materials Research Society* , Vol. 32, No. 5, May

**2022Abstract :** 텅스텐 Surface area ( 0.1-100  $\mu\text{m}$ ) 특성과 2 응용 korean 평가 . / The properties and semiconductor application possibilities of tungsten powder (0.1-100  $\mu\text{m}$ ) were evaluated.

ISSN : 1225-0562

**Document name :** KS D 9502:2021 " 텅스텐 100% 기술 조건 " **Chinese Translation :** KS D 9502:2021 "Technical Conditions of Tungsten Powder"

**Author :** 한국표준협회 (Korean Standards Association)

**Source :** 한국산업표준 , 2021 년 발행 / Korean Industrial Standards, Published 2021

**Summary :** 텅스텐 분말 (W  $\geq$ 99.95%-99.999%) 의 기술 2 규정 。 / Specifies the technical requirements for tungsten powder (W  $\geq$ 99.95%-99.999%).

**Title of the article :** " 텅스텐 100% Korean 및 응용 " **Chinese Translation :** "Synthesis and Application of Nano Tungsten Powder"

**Author :** 박준영 (Park Jun-young)

**Source :** 『 한국분말야금학회지 』 , 제 29 권 , 제 2 호 , 2022 년 4 월 / *Journal of Korean Powder Metallurgy Institute* , Vol. 29, No. 2, April

**2022Abstract :** 나노 텅스텐 100 nm 2 합성법 연구 . / The low-temperature synthesis method of

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nano-tungsten powder (<50 nm) was studied.

DOI : 10.4150/KPMI.2022.29.2.105

**Title of the article :** 텅스텐 3D 3D Movie 응용 " **Chinese Translation :** "3D Printing Application of Spherical Tungsten Powder"

**Author :** 이지훈 (Lee Ji-hoon)

**Source :** 『 한국정밀공학회지 』, 제 40 권 , 제 3 호 , 2023 년 3 월 / *Journal of the Korean Society of Precision Engineering* , Vol. 40, No. 3, March

**2023Abstract :** 구형 텅스텐 5-30  $\mu\text{m}$  3D Movie 성능 분석 . / The flowability and 3D printing performance of spherical tungsten powder (5-30  $\mu\text{m}$ ) were analyzed.

ISSN : 1225-9071

**Title of the document :** " 100% I'm so tired 특성 " **Chinese translation :** "Thermal Conductivity Properties of Tungsten Powder"

**Author :** 최민수 (Choi Min-soo)

**Source :** 『 한국열처리공학회지 』, 제 36 권 , 제 4 호 , 2023 년 4 월 / *Journal of the Korean Society for Heat Treatment* , Vol. 36, No. 4, April

**2023Abstract :** 텅스텐 100% The thermal conductivity of tungsten powder (173 W/m·K) was measured . / The thermal conductivity of tungsten powder (173 W/m·K) was measured.

ISSN : 1229-5078

**Title of the article :** " 텅스텐 100% 구조 " **Chinese Translation :** "Structural Analysis of Ultrafine Tungsten Powder"

**Author :** 정현우 (Jung Hyun-woo)

**Source :** 『 한국재료연구소 논문집 』, 제 34 권 , 제 2 호 , 2022

년 2 월 / *Transactions of the Korea Institute of Materials Research* , Vol . 34 , No. 2 , February

**2022Abstract :** 초 미세 텅스텐 2  $\mu\text{m}$  ) 2 분석 (SEM, XRD). / Microstructure analysis of ultrafine tungsten powder (<2  $\mu\text{m}$ ) using SEM and XRD.

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ISSN : 1225-0562

**Title of the document :** " 폐기물 2 " **Chinese Translation :** "Tungsten Scrap Recycling Technology"

**Author :** 김상훈 (Kim Sang-hoon)

**Source :** 『 한국자원리사이클링학회지 』, 제 31 권 , 제 5 호 , 2023 년 5 월 / *Journal of the Korean Institute of Resources Recycling* , Vol. 31, No. 5, May 2023Abstract

**: 텅스텐 2. 분말 2 기술 제안 . / A technology for regenerating tungsten powder from tungsten waste is proposed.**

**DOI :** 10.7844/kirr.2023.31.5.123

**Title of the document :** " 100% 2 특성 " **Chinese translation :** "Research on the Mechanical Properties of Tungsten Powder"

**Author :** 박영진 (Park Young-jin)

**Source :** 『 한국기계기술학회지 』, 제 22 권 , 제 6 호 , 2023 년 6 월 / *Journal of the Korean Society of Manufacturing Technology Engineers* , Vol. 22, No. 6, June

**2023Abstract :** 텅스텐 분말 The 2 The mechanical strength of sintered tungsten powder ( 600-1000 MPa) was analyzed .

**ISSN :** 1598-6721

**Title of the article :** " 텅스텐 100% 촉매 " **Chinese translation :** "Catalytic Application of Nanotungsten Powder"

**Author :** 송민재 (Song Min-jae)

**Source :** 『 한국화학공학회지 』 , 제 61 권 , 제 4 호 , 2023 년 4 월 / *Korean Journal of Chemical Engineering* , Vol. 61, No. 4, April

**2023Abstract :** 나노 텅스텐 100 nm 촉매 특성 연구 . / The catalytic properties of nano-tungsten powder (<50 nm) were studied.

**DOI :** 10.9713/kcer.2023.61.4.234

**Title of the document :** " 100% 2 가능 생산 " **Chinese Translation :** "Sustainable Production of

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Tungsten Powder"

**Author :** 황지영 (Hwang Ji-young)

**Source :** 『 한국환경기술학회지 』, 제 24 권 , 제 5 호 , 2023 년 5 월 / *Journal of the Korean Society of Environmental Technology* , Vol. 24, No. 5, May

**2023 Abstract :** 텅스텐 100% 2 2 기술 제안 . / Proposed an environmentally friendly manufacturing technology for tungsten powder.

**ISSN :** 1229-8425

## Appendix

### Tungsten powder material safety factor specification Tungsten Powder Safe Handling Guide (MSDS and Protective Measures)

#### Section 1: Chemicals and Identification Information

Chemical Name: Tungsten Powder

Chemical formula: W

CAS No.: 7440-33-7

Product Type: Solid Metal Powder

Recommended uses: used in cemented carbide manufacturing, additive manufacturing (3D printing), electronic targets, catalyst carriers, high-temperature alloys, etc.

Restricted Use: Prohibited from use in food, medicine or unapproved direct human contact.

Supplier Information:

Name: CTIA GROUP LTD

Address: 3rd Floor, No. 25, Wanghai Road, Xiamen Software Park 2

Emergency contact number: +86-592 512 9696 (24 hours)

#### Section 2: Hazards Identification

GHS classification (Globally Harmonized System of Classification and Labelling of Chemicals)

Physical hazards:

Combustible Dust: Tungsten powder may form explosive dust clouds when suspended in high concentrations in the air.

Spontaneous combustion tendency: fine particles may spontaneously ignite under high temperature or sparks.

Health hazards:

Inhalation Hazard: Inhalation of tungsten dust may cause respiratory irritation and long-term exposure may cause pulmonary fibrosis.

Skin contact: Slightly irritating. Prolonged contact may cause dermatitis.

Eye Contact: May cause mechanical irritation or mild inflammation.

Environmental hazards:

It has no significant acute toxicity to the aquatic environment, but in the event of a large-scale leak it may deposit and affect the ecology.

GHS Label Elements

Hazard pictograms:

Flame symbol (combustible dust)

Health hazard symbols (respiratory hazard)

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Signal word: Warning

Hazard Statements:

H228: Combustible solid. May form explosive dust-air mixture.

H315: Causes skin irritation.

H335 : May cause respiratory irritation.

Precautionary measures:

P210: Keep away from heat, sparks, open flames and hot surfaces. No smoking.

P261: Avoid breathing dust.

P280: Wear protective gloves, protective clothing and eye protection.

### Section 3: Composition/Information on Ingredients

Chemical composition: Tungsten (W)

Purity:  $\geq 99.9\%$  (industrial grade) to  $\geq 99.999\%$  (high purity grade)

Impurities: May contain trace amounts of oxygen (O), carbon (C), iron (Fe), etc. The content depends on the production process.

Physical form: Grey to black metal powder, particle size range 0.05-100  $\mu\text{m}$  (depending on product specifications).

### Section 4: First Aid Measures

Inhalation:

Move victim to fresh air and keep comfortable for breathing.

If breathing is difficult, give oxygen and seek medical help immediately.

Skin contact:

Wash with plenty of water and soap for at least 15 minutes.

If irritation or rash occurs, consult a physician.

Eye Contact:

Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open.

If irritation persists, seek advice from an ophthalmologist.

Ingestion:

In the unlikely event of ingestion, rinse mouth immediately and drink plenty of water.

Do not induce vomiting, seek medical attention as soon as possible.

First aid precautions: First aid personnel should wear protective equipment to avoid exposing themselves to dust.

### Section 5: Firefighting measures

Extinguishing Media:

Recommended: Dry powder (such as ABC powder), dry sand or carbon dioxide.

Prohibited: Water (may intensify metal dust fires), foam fire extinguishing agents.

Special fire hazards:

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Tungsten powder can form explosive dust clouds when in high concentrations in the air.

At high temperatures, toxic fumes (such as tungsten oxide vapor) may be released.

Fire protection:

Firefighters must wear self-contained breathing apparatus and full body fire resistant clothing.

Extinguish the fire from the upwind direction and move the container to a safe area if possible.

### Section 6: Emergency treatment of leaks

Personal protection:

Wear dust mask, protective clothing and gloves to avoid inhalation or contact with dust.

Environmental protection:

Prevent dust from entering water bodies or sewers to avoid widespread spread.

Cleaning method:

Collect spillage with an explosion-proof vacuum cleaner or wet sweeping to avoid raising dust.

Place collection into sealed containers, label “hazardous waste” and dispose of properly.

Emergency response: Isolate the leakage area and restrict entry of unauthorized personnel.

### Section 7: Handling and Storage

Operation Notes

Safe Operation:

Work in a well-ventilated area and use explosion-proof electrical equipment and tools.

Avoid generating dust and keep the working place clean during operation.

Protective measures:

Wear an N95 or higher level dust mask, protective glasses and wear-resistant gloves.

Equipped with local exhaust system or dust collection device.

Storage conditions

Storage requirements:

Store in a cool, dry, well-ventilated warehouse, away from fire and oxidants.

Use airtight containers to avoid moisture and dust accumulation.

Packaging materials: Plastic-lined metal drums or moisture-proof bags are recommended.

### Part 8: Exposure Controls and Personal Protection

Occupational exposure limits (examples, based on international standards):

TLV-TWA (time-weighted average): 5 mg/m<sup>3</sup> (tungsten and its insoluble compounds, American ACGIH).

STEL (Short-term Exposure Limit): 10 mg/ m<sup>3</sup> (15 minutes).

Engineering controls:

Use closed systems and local exhaust ventilation equipment to keep the dust concentration in the air below the limit.

Personal protective equipment:

Respiratory protection: When dust concentration exceeds the standard, use a respirator with a P100 filter.

Hand protection: wear-resistant rubber or nylon gloves.

Eye protection: Sealed protective glasses or face shield.

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Personal protection: anti-static work clothes to avoid dust adhesion.

### Section 9: Physical and Chemical Properties

Appearance and properties: Grey to black powder, odorless.

Melting point: 3422°C

Boiling point: 5555°C

Density: 19.25 g/cm<sup>3</sup> ( bulk tungsten), powder depends on particle size.

Solubility: Insoluble in water, slightly soluble in strong acids (such as nitric acid).

Flash point: None (but fine powder is flammable under certain conditions).

Explosion limits: Lower limit of dust cloud about 300 g/m<sup>3</sup> ( depending on particle size).

### Section 10: Stability and Reactivity

Stability: Stable at room temperature, may spontaneously ignite under high temperature or sparks.

Conditions to avoid: high temperature, sparks, static discharge, humid environment.

Incompatible materials: Strong oxidizing agents (such as nitric acid, perchloric acid), halogen compounds.

Hazardous decomposition products: Tungsten oxide (WO<sub>3</sub>) vapor may be generated at high temperatures.

### SECTION 11: TOXICOLOGICAL INFORMATION

Acute toxicity:

LD50 (oral, rat): >5000 mg/kg (low toxicity).

LC50 (inhalation, rat): >5 mg/L (4 hours).

Chronic toxicity: Long-term inhalation may cause pulmonary fibrosis or "hard metal lung disease".

Carcinogenicity: IARC does not classify it as a carcinogen, and available data are insufficient to evaluate.

Reproductive toxicity: No significant evidence.

### Section 12: Ecological Information

Ecotoxicity: No significant acute toxicity to aquatic organisms, but deposition may affect benthic organisms.

Persistence and Degradability: Not biodegradable, persists for a long time as an inert metal.

Bioaccumulation: No significant bioaccumulation.

### Section 13: Disposal considerations

Disposal method:

Treat waste tungsten powder as hazardous waste and hand it over to qualified units for recycling or disposal.

Avoid dumping directly into the environment to prevent dust from spreading.

Packaging disposal: Contaminated packaging should be treated as hazardous waste or recycled after cleaning.

### Section 14: Transport Information

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UN Number: UN 3178 (Combustible Solids, Inorganic, Not Otherwise Specified).

Shipping name: Combustible solid tungsten powder.

Hazard Class: Class 4.1 (Combustible Solids).

Packing group: III (low hazard).

Transportation requirements: Use moisture-proof and anti-static packaging, and avoid high temperatures and fire sources.

### Section 15: Regulatory information

International regulations:

Compliant with GHS (Globally Harmonized System of Classification and Labelling of Chemicals).

Listed in TSCA (Toxic Substances Control Act) and REACH (Registration, Evaluation, Authorization and Restriction of Chemicals).

Chinese regulations:

"Regulations on the Safety Management of Hazardous Chemicals" (State Council Order No. 591).

"Contents and Item Sequence of Material Safety Data Sheets" (GB/T 16483-2008).

### Section 16: Other Information

Date of preparation: April 5, 2025

Update Note: This guide is based on the latest research and regulations and should be checked regularly for updates.

Note: Operators must receive safety training and be familiar with the contents of this guide.

### Additional Notes

This guide covers comprehensive safety information for tungsten powder and is suitable for industrial users, researchers and related practitioners. When used in practice, protective measures should be adjusted according to specific product specifications (such as particle size, purity) and local regulations. If a specific MSDS file is required, please contact the manufacturer [CTIA GROUP LTD](#) to obtain it.

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## Tungsten Powder Material Safety Data Sheet (MSDS)

### Part 1: Chemical and Identification Information

**Chemical Name:** Tungsten Powder

**Chemical Formula:** W

**CAS Number:** 7440-33-7

**Product Type:** Solid metal powder

**Recommended Uses:** For manufacturing hard alloys, additive manufacturing (3D printing), electronic sputtering targets, catalyst carriers, high-temperature alloys, etc.

**Restricted Uses:** Prohibited for use in food, pharmaceuticals, or unapproved direct human contact applications.

### Supplier Information:

- **Name:** [CTIA GROUP Ltd.](#)
- **Address:** 3rd Floor, No. 25-1, Wanghai Rd . , Xiamen Software Park II , Xiamen, China
- **Emergency Contact Number:** +86-592 512 9696 (24 hours)

### Part 2: Hazard Overview

#### GHS Classification (Globally Harmonized System of Classification and Labeling of Chemicals):

##### Physical Hazards:

- **Combustible Dust:** Tungsten powder may form an explosive dust cloud when suspended in air at high concentrations.

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- **Self-Ignition Tendency:** Fine particles may spontaneously ignite under high temperatures or sparks.

#### Health Hazards:

- **Inhalation Hazard:** Inhalation of tungsten dust may cause respiratory irritation; prolonged exposure could lead to pulmonary fibrosis.
- **Skin Contact:** Mild irritation; prolonged contact may cause dermatitis.
- **Eye Contact:** May cause mechanical irritation or mild inflammation.

#### Environmental Hazards:

- No significant acute toxicity to aquatic environments, but large-scale spills may deposit and affect ecosystems.

#### GHS Label Elements:

- **Hazard Pictograms:**
  - Flame symbol (combustible dust)
  - Health hazard symbol (respiratory hazard)
- **Signal Word:** Warning

#### Hazard Statements:

- **H228:** Flammable solid; may form explosive dust-air mixtures.
- **H315:** Causes skin irritation.
- **H335:** May cause respiratory irritation.

#### Precautionary Statements:

- **P210:** Keep away from heat, sparks, open flames, and hot surfaces. No smoking.
- **P261:** Avoid breathing dust.
- **P280:** Wear protective gloves, protective clothing, and eye protection.

### Part 3: Composition/Ingredient Information

**Chemical Composition:** Tungsten (W)

**Purity:**  $\geq 99.9\%$  (industrial grade) to  $\geq 99.999\%$  (high-purity grade)

**Impurities:** May contain trace amounts of oxygen (O), carbon (C), iron (Fe), etc., depending on the production process.

**Physical Form:** Gray to black metal powder, particle size range 0.05-100  $\mu\text{m}$  (specific to product specifications).

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## Part 4: First Aid Measures

### Inhalation:

- Move the victim to fresh air and ensure smooth breathing.
- If breathing is difficult, administer oxygen immediately and seek medical attention.

### Skin Contact:

- Wash with plenty of water and soap for at least 15 minutes.
- If irritation or rash occurs, consult a doctor.

### Eye Contact:

- Rinse eyes immediately with running water for at least 15 minutes, lifting eyelids periodically.
- If irritation persists, seek assistance from an ophthalmologist.

### Ingestion:

- Unlikely to occur, but if ingested, rinse mouth and drink plenty of water immediately.
- Do not induce vomiting; seek medical attention promptly.

**First Aid Notes:** First responders should wear protective equipment to avoid exposure to dust.

## Part 5: Firefighting Measures

### Extinguishing Media:

- **Recommended:** Dry powder extinguishers (eg, ABC dry powder), dry sand, or carbon dioxide.
- **Prohibited:** Water (may exacerbate metal dust fires), foam extinguishers.

### Specific Fire Hazards:

- Tungsten powder at high concentrations in air can form explosive dust clouds.
- Toxic fumes (eg, tungsten oxide vapor) may be released at high temperatures.

### Firefighting Protection:

- Firefighters must wear self-contained breathing apparatus and full-body fire-resistant clothing.
- Fight fire from upwind and move containers to a safe area if possible.

## Part 6: Accidental Release Measures

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#### Personal Protection:

- Wear a dust mask, protective clothing, and gloves to avoid inhalation or contact with dust.

#### Environmental Protection:

- Prevent dust from entering water bodies or sewers to avoid widespread dispersion.

#### Cleanup Methods:

- Use an explosion-proof vacuum cleaner or wet sweeping to collect spills, avoiding dust generation.
- Place collected material in sealed containers, label as “hazardous waste,” and dispose of properly.

**Emergency Response:** Isolate the spill area and restrict access to unauthorized personnel.

### Part 7: Handling and Storage

#### Handling Precautions:

- **Safe Handling:** Operate in well-ventilated areas using explosion-proof electrical equipment and tools. Avoid dust generation and keep the workplace clean.
- **Protective Measures:** Wear an N95 or higher-grade dust mask, safety goggles, and durable gloves. Equip with local exhaust systems or dust collection devices.

#### Storage Conditions:

- **Requirements:** Store in a cool, dry, well-ventilated warehouse, away from ignition sources and oxidizers. Use sealed containers to prevent moisture and dust accumulation.
- **Packaging Materials:** Recommended materials include metal drums with plastic liners or moisture-proof bags.

### Part 8: Exposure Controls and Personal Protection

#### Occupational Exposure Limits (Example Based on International Standards):

- **TLV-TWA (Time-Weighted Average):** 5 mg/m<sup>3</sup> (tungsten and its insoluble compounds, ACGIH, USA).
- **STEL (Short-Term Exposure Limit):** 10 mg/m<sup>3</sup> (15 minutes).

#### Engineering Controls:

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- Use enclosed systems and local exhaust ventilation to maintain dust concentrations below exposure limits.

#### Personal Protective Equipment:

- **Respiratory Protection:** Use a respirator with a P100 filter when dust levels exceed limits.
- **Hand Protection:** Wear durable rubber or nylon gloves.
- **Eye Protection:** Sealed safety goggles or face shield.
- **Body Protection:** Anti-static work clothing to prevent dust adhesion.

#### Part 9: Physical and Chemical Properties

**Appearance and Characteristics:** Gray to black powder, odorless.

**Melting Point:** 3422°C

**Boiling Point:** 5555°C

**Density:** 19.25 g/cm<sup>3</sup> (bulk tungsten), varies with particle size for powder.

**Solubility:** Insoluble in water, slightly soluble in strong acids (eg, nitric acid).

**Flash Point:** None (but fine powder is combustible under specific conditions).

**Explosive Limit:** Lower limit of dust cloud approximately 300 g/m<sup>3</sup> (varies with particle size).

#### Part 10: Stability and Reactivity

**Stability:** Stable at room temperature; may ignite spontaneously at high temperatures or with sparks.

**Conditions to Avoid:** High temperatures, sparks, electrostatic discharge, humid environments.

**Incompatible Materials:** Strong oxidizers (eg, nitric acid, perchloric acid), halogen compounds.

**Hazardous Decomposition Products:** May produce tungsten oxide (WO<sub>3</sub>) vapor at high temperatures.

#### Part 11: Toxicological Information

##### Acute Toxicity:

- **LD50 (Oral, Rat):** >5000 mg/kg (low toxicity).
- **LC50 (Inhalation, Rat):** >5 mg/L (4 hours).

**Chronic Toxicity:** Prolonged inhalation may lead to pulmonary fibrosis or “hard metal lung disease.”

**Carcinogenicity:** Not classified as a carcinogen by IARC; insufficient data for evaluation.

**Reproductive Toxicity:** No significant evidence.

#### Part 12: Ecological Information

**Ecotoxicity:** No significant acute toxicity to aquatic life, but sediment deposition may affect benthic organisms.

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**Persistence and Degradability:** Non-biodegradable; persists as an inert metal in the environment.

**Bioaccumulative Potential:** No significant bioaccumulation.

### Part 13: Disposal Considerations

#### Disposal Methods:

- Treat waste tungsten powder as hazardous waste and hand it over to qualified entities for recycling or disposal.
- Avoid direct environmental release to prevent dust dispersion.

**Packaging Disposal:** Contaminated packaging should be treated as hazardous waste or cleaned and recycled.

### Part 14: Transport Information

**UN Number:** UN 3178 (Flammable solid, inorganic, nos).

**Shipping Name:** Flammable solid tungsten powder.

**Hazard Class:** 4.1 (Flammable solids).

**Packing Group:** III (Low hazard).

**Transport Requirements:** Use moisture-proof, anti-static packaging; avoid high temperatures and ignition sources.

### Part 15: Regulatory Information

#### International Regulations:

- Complies with GHS (Globally Harmonized System).
- Listed under TSCA (Toxic Substances Control Act, USA) and REACH (Registration, Evaluation, Authorization and Restriction of Chemicals, EU).

#### Chinese Regulations:

- "Regulations on the Safety Management of Hazardous Chemicals" (State Council Decree No. 591).
- "Safety Data Sheet for Chemical Products - Content and Order of Sections" (GB/T 16483-2008).

### Part 16: Other Information

**Preparation Date:** April 5, 2025

**Update Notes:** This guideline is based on the latest research and regulations; periodic updates should

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be reviewed.

**Precautions:** Operators must receive safety training and be familiar with this guideline.

#### Additional Notes

This guideline provides comprehensive safety information for tungsten powder, suitable for industrial users, researchers, and related professionals. In practice, protective measures should be adjusted based on specific product specifications (eg, particle size, purity) and local regulations. For a specific MSDS document, contact the manufacturer, [CTIA GROUP](http://www.ctia.com.cn).

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**Appendix G: Chinese, English, Japanese, German, Russian and Korean Tungsten Powder Terminology Comparison Table**

**1. General Terms**

Chinese	English	Japanese	German	Russian	Korean
Tungsten powder	Tungsten Powder	Tangusten powder	Wolframpulver	Вольфрамовый порошок	텅스텐 분말
Tungsten Carbide	Tungsten Carbide	Carbonization Tangusten	Wolframkarbid	Карбид вольфрама	탄화텅스텐
High purity tungsten powder	High-Purity Tungsten Powder	High purity polyester powder	Hochreines Wolframpulver	Высокочистый вольфрамовый порошок	고순도 텅스텐 분말
Nano tungsten powder	Nano Tungsten Powder	Nano タングステン powder	Nanowolframpulver	Нановольфрамовый порошок	나노 텅스텐 분말
Spherical tungsten powder	Spherical Tungsten Powder	Spherical タングステン powder	Sphärisches Wolframpulver	Сферический вольфрамовый порошок	구형 텅스텐 분말
Ultrafine tungsten powder	Ultrafine Tungsten Powder	Ultrafine タングステン powder	Ultrafeines Wolframpulver	Ультратонкий вольфрамовый порошок Tungsten Intelligent Manufacturing in MADE BY:	초미세 텅스텐 분말

**2. Properties**

**Physical Properties**

Chinese	English	Japanese	German	Russian	Korean
Particle size	Particle Size	Particle size	Part one	Rashmere chips	2 크기
Particle size distribution	Particle Size Distribution	Particle size distribution	Part Ignition	Распределение размеров частиц	입도 분포
Specific surface area	Specific Surface Area	Specific surface area	Spend the day	Удельная поверхность	2

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Chinese	English	Japanese	German	Russian	Korean
density	Density	density	Dichte	Плотность	밀도
Bulk density	Bulk Density	Density	Schüttdichte	Насыпная плотность	겉보기 밀도
Tap density	Tap Density	Tap density	Klopfichte	Утрамбованная плотность	탭 밀도
Liquidity	Flowability	Liquidity	Fly high	Текучесть	2
Melting point	Melting Point	Melting point	Schmelzpunkt	Температура плавления	용융점
Boiling Point	Boiling Point	Boiling Point	Siedepunkt	Температура кипения	끓는점 MADE BY: CTIA GROUP LTD

### Chemical Properties

Chinese	English	Japanese	German	Russian	Korean
purity	Purity	Purity	Reinheit	Caboteta	순도
Oxygen content	Oxygen Content	Acid content	Sauerstoffgehalt	Содержание кислорода	산소 함량
Carbon content	Carbon Content	Carbon content	Kohlenstoffgehalt	Содержание углерода	탄소 함량
Impurities	Impurities	Impure	Veronika	Prometheus	2
Oxidation	Oxidizability	Acidification	Oxidierbarkeit	Open	Tungsten MADE BY:

### Mechanical Properties

Chinese	English	Japanese	German	Russian	Korean
hardness	Hardness	hardness	Harte	Твёрдость	경도
Compressive strength	Compressive Strength	Compression strength	Druckfestigkeit	Прочность на сжатие	압축 강도
toughness	Toughness	Toughness	Zähigkeit	Ударная вязкость	인성
Wear resistance	Wear Resistance	Wear resistance	Verschleißfestigkeit	Износостойкость	CTIA GROUP LTD

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### Thermal and Electrical Properties

Chinese	English	Japanese	German	Russian	Korean
Thermal conductivity	Thermal Conductivity	Thermal conductivity	What is the best way to deal with it?	Теплопроводность	I'm so tired
Coefficient of thermal expansion	Thermal Expansion Coefficient	Thermal expansion coefficient	Wärmeausdehnungskoeffizient	Коэффициент теплового расширения	2 계수
Conductivity	Electrical Conductivity	Electrical conductivity	Electric Leitfähigkeit	Электропроводность	The
Resistivity	Electrical Resistivity	Electrical resistance	Electrical Wider Stand	Удельное сопротивление	2

### 3. Production Equipment

Chinese	English	Japanese	German	Russian	Korean
Reduction furnace	Reduction Furnace	Reduction furnace	Redemption	Восстановительная печь	환원로
Plasma equipment	Plasma Equipment	Plasma device	Plasmaanlage	Плазменное оборудование	플라즈마 장비
Ball mill	Ball Mill	Ballumil	Kugelmühle	Шаровая мельница	볼 밀
Spray Dryer	Spray Dryer	Spread Livery	Sprühtrockner	Распылительная сушилка	분무 건조기
Sintering furnace	Sintering Furnace	Sintering furnace	Sinterofen	Спекающая печь	소결로
Vacuum furnace	Vacuum Furnace	Vacuum furnace	Vakuumofen	Viagra	2
Micro-wave oven	Microwave Furnace	Microwave Oven	Mikrowellenofen	Микроволновая печь	The most beautiful 로
press	Press Machine	Pres machine	Pressmaschine	Prèss	프레스 기계
Screening Machine	Screening Machine	Screening Machine	Siebmaschine	Sito	2 기계 Tungsten Intelligent Manufacturing in MADE BY:

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#### 4. Testing and Inspection Instruments

Chinese	English	Japanese	German	Russian	Korean
Laser particle size analyzer	Laser Particle Size Analyzer	Reser Particle Size Analyzer	Laserpartikelgröß enanalysator	Лазерный гранулометр	2 입도 2
Scanning electron microscopy	Scanning Electron Microscope (SEM)	Scanning electron microscope (SEM)	Rasterelektronenmikroskop (REM)	Сканирующий электронный микроскоп (СЭМ)	SEM
X-ray diffractometer	X-Ray Diffractometer (XRD)	X-ray Reflection Device (XRD)	Röntgendiffraktometer (XRD )	Рентгеновский дифрактометр (XRD)	X XRD
Surface area meter	BET Surface Area Analyzer	BET Specific Surface Area	BET Oberflächenanalysator	Анализатор удельной поверхности BET	BET 2 2
Oxygen and nitrogen analyzer	Oxygen-Nitrogen Analyzer	Oxygen and nitrogen analysis device	Sauerstoff-Stickstoff-Analysator	Анализатор кислорода и азота	산소 - 질소 2
Hardness Tester	Hardness Tester	Hardness Tester	Härteprüfgerät	Trierdomer Tungsten Intelligent Manufacturing in MADE BY:	경도 2
Thermal conductivity meter	Thermal Conductivity Meter	Thermal Conductivity Meter	W ä rmeleitf ä higeitsmessger ä t	Измеритель теплопроводности	I'm so tired 측정기
Conductivity meter	Conductivity Meter	Electrical conductivity meter	How to make a high - quality message	Измеритель электропроводности	The 측정기
microscope	Microscope	Microscope	Mikroskop	Mokrovokop	현미경

#### 5. Production Processes

Chinese	English	Japanese	German	Russian	Korean
Hydrogen reduction	Hydrogen Reduction	Hydrogen reduction	Wasserstoffreduktion	Водородное восстановление	2 환원

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Chinese	English	Japanese	German	Russian	Korean
Plasma atomization	Plasma Atomization	Plasma Master Match	Plasma-Atomic Agent	Плазменное распыление	플라즈마 분무
Microwave reduction	Microwave Reduction	Microwave Rebirth	Marketing	Микроволновое восстановление	The most beautiful 환원
Electrochemical synthesis	Electrochemical Synthesis	Electrochemical synthesis	Electrochemical Synthesizer	Электрохимический синтез	2. Korean
Mechanical grinding	Mechanical Grinding	Mechanical grinding	Mechanisches Schleifen	Механическое измельчение	기계 2
sintering	Sintering	Sintering	Sintern	Spekanikie	소결
Hot Pressing	Hot Pressing	Thermal compression	Heiß pressen	Горячее прессование	열 2
Recycle	Recycling	Live	Recycling	Peerera Botka	2

## 6. Applications and Uses

### Industrial Applications

Chinese	English	Japanese	German	Russian	Korean
Cemented Carbide	Cemented Carbide	Super Hard Alloy	Hartmetall	Theyёрдый сплав	korean
Additive Manufacturing	Additive Manufacturing	アディティブマニファクチャリング	Additive Fertigation	Аддитивное производство	적층 2
Thermal Spraying	Thermal Spraying	Spraying	Thermodynamics	Trimona photography	열 2
Cutting Tools	Cutting Tools	Cutting Tools	Schneidwerkzeuge	Режущие инструменты	2 공구
Mining tools	Mining Tools	Mining tools	Bergbauwerkzeuge	Горные инструменты	채굴 공구
Wear-resistant coating	Wear-Resistant Coating	Wear-resistant coating	Verschleiß feste Beschichtung	Износостойкое покрытие	내마모 코팅

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### Electronics and High-Tech Applications

Chinese	English	Japanese	German	Russian	Korean
Electronic Target	Electronic Target	Electronic watch	Electronic Target	Электронная мишень	2 타겟
Semiconductor Materials	Semiconductor Material	Semiconductor Materials	Halbleitermaterial	Полупроводниковый материал	2 재료
Thin film deposition	Thin Film Deposition	Film Evaporation	Filmmaking	Нанесение тонкой плёнки	박막 2
Electrode Materials	Electrode Material	Electrode Materials	Electrical Engineering	Электродный материал	전극 재료 Tungsten MADE BY:

### Aerospace and Defense

Chinese	English	Japanese	German	Russian	Korean
High temperature alloys	High-Temperature Alloy	High temperature alloys	Temperature control	Высокотемпературный сплав	고온 합금
Tungsten Alloy	Tungsten Alloy	Tangusten alloy	Wolframlegierung	Вольфрамовый сплав	텅스텐 합금
Counterweight	Counterweight	Catch the World	Gegengewicht	Protipovov	균형추
Military parts	Military Component	Military Parts	Militärkomponente	Video player	군사 부품

### Other Applications

Chinese	English	Japanese	German	Russian	Korean
Catalyst carrier	Catalyst Support	Catalyst carrier	Katalysatorträger	Носитель катализатора	촉매 2
Tungsten Wire	Tungsten Wire	タングステンワイヤー	Wolframdräht	Вольфрамовая проволока	텅스텐 2
Tungsten Rod	Tungsten Rod	Tangusten Lock	Wolframstab	Вольфрамовый стержень	텅스텐 로드

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Chinese	English	Japanese	German	Russian	Korean
Medical Devices	Medical Device	Medical Machines	Medizinische Geräte	Медицинское устройство	의료 기기

## 7. Safety and Protection

Chinese	English	Japanese	German	Russian	Korean
Combustible dust	Combustible Dust	Combustible dust	Brennbarer Staub	See pictures	korean 2
Explosive dust	Explosive Dust	Explosive dust	Explosiver Staub	Взрывоопасная пыль	폭발성 2
Respiratory tract irritation	Respiratory Irritation	Respiratory irritation	Atmosphere	Раздражение дыхательных путей	호흡기 자극
Protective glasses	Safety Goggles	Protection Goggle	Schutzbrille	How to make a movie	보호 고글
Protective gloves	Protective Gloves	Protective handbag	Schutzhandschuhe	Защитные перчатки	보호 2
Dust masks	Dust Mask	Dustproof Mask	Staubmaske	Противопылевая маска	2 2
Local exhaust	Local Exhaust Ventilation	Exhaust	Lokal Absaugung	Локальная вытяжка	국부 배기 환기
Hazardous waste	Hazardous Waste	Dangerous waste	This is the answer	Open the door	2 폐기물
Firefighting measures	Firefighting Measures	Fire prevention measures	Brandschutzmaßnahmen	Противопожарные меры	소방 조치 MADE BY: Tungsten Intelligence

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## CTIA GROUP LTD

### Spherical Tungsten Powder Product Introduction

#### 1. Overview of Spherical Tungsten Powder

CTIA GROUP LTD's spherical tungsten powder complies with the GB/T 41338-2022 "Spherical Tungsten Powder for 3D Printing" standard. It is prepared using a plasma spheroidization process and is specially designed for additive manufacturing (such as SLM, EBM). It meets high-end application requirements with high purity, high sphericity and excellent fluidity.

#### 2. Excellent Properties of Spherical Tungsten Powder

Ultra-high purity: tungsten content  $\geq 99.95\%$ , oxygen content  $\leq 0.05$  wt%, and extremely low impurities.

High sphericity:  $\geq 90\%$ , uniform particles, excellent powder spreading performance.

Precise particle size: D50 range 5-63  $\mu\text{m}$ , stable distribution, deviation  $\pm 10\%$ .

Excellent fluidity:  $\leq 25$  s/50g, bulk density  $\geq 9.0$  g/cm<sup>3</sup>, ensuring printing efficiency.

#### 3. Specifications of Spherical Tungsten Powder

Brand	D50 particle size ( $\mu\text{m}$ )
SWP-15	5-15
SWP-25	15-25
SWP-45	25-45
SWP-63	45-63

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

#### 4. Spherical Tungsten Powder Packaging and Quality Assurance

Packaging: Inner vacuum aluminum foil bag, outer iron drum, net weight 5kg or 10kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition, particle size distribution and sphericity data, and the shelf life is 12 months.

#### 5. Contact Information of CTIA GROUP LTD

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about spherical tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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## CTIA GROUP LTD

### Introduction of High Purity Tungsten Powder

#### 1. High Purity Tungsten Powder Overview

CTIA GROUP LTD's high-purity tungsten powder is produced using a high-purity tungsten oxide hydrogen reduction process. High-purity tungsten powder is widely used in the electronics industry (such as sputtering targets, tungsten wires), aerospace, semiconductors and high-precision manufacturing due to its ultra-high purity, fine particle size and excellent physical properties. CTIA GROUP LTD is committed to providing high-quality tungsten powder products to meet cutting-edge technology needs.

#### 2. High Purity Tungsten Powder Features

Chemical composition: Tungsten (W), high purity metal powder.

Purity:  $\geq 99.99\%$  (4N), with extremely low impurity content.

Appearance: Grey or dark grey powder, uniform color.

Ultra-high purity: impurities are controlled at ppm level, ensuring excellent electrical and mechanical properties.

Fine particles: The particle size can reach 0.1-5  $\mu\text{m}$ , which can meet high-precision applications.

Low oxygen content: oxygen content  $\leq 0.02\%$ , improving sintering performance and material stability.

#### 3. High Purity Tungsten Powder Specifications

Index	CTIA GROUP LTD High Purity Tungsten Powder Standard (4N)
Tungsten content (wt%)	$\geq 99.99$
Impurities (wt%, max)	Fe $\leq 0.0010$ , Mo $\leq 0.0010$ , Si $\leq 0.0005$ , Al $\leq 0.0005$ , Ca $\leq 0.0005$ , Mg $\leq 0.0005$ , Na $\leq 0.0010$ , K $\leq 0.0010$ , O $\leq 0.0200$ , C $\leq 0.0050$ , N $\leq 0.0020$ , P $\leq 0.0005$ , S $\leq 0.0005$
Water content (wt%)	$\leq 0.02$
Particle size ( $\mu\text{m}$ , FSSS)	0.1-5.0 (superfine 0.1-1.0, fine 1.0-5.0)
Bulk density (g/ $\text{cm}^3$ )	4.5-6.5
Particle size	Provide ultra-fine (0.1-1.0 $\mu\text{m}$ ) and fine (1.0-5.0 $\mu\text{m}$ ) specifications, can be customized according to customer needs
Moisture	$\leq 0.02\%$ , ensuring product dryness and stability
Customization	Optional ultra-high purity grade (5N, $\geq 99.999\%$ ), with further reduction of impurities (e.g. O $\leq 0.01\%$ )

#### 4. Packaging and Quality Assurance

Packaging: Inner sealed vacuum aluminum foil bag, outer iron barrel or plastic barrel, net weight 5kg, 10kg or 25kg, moisture-proof and oxidation-proof.

Warranty: With quality certificate, including tungsten content, impurity analysis (ICP-MS), particle size (FSSS method), bulk density and moisture data, shelf life is 12 months (sealed and dry conditions).

#### 5. Procurement Information

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com) Tel: +86 592 5129696

For more tungsten powder information, please visit China Tungsten Online website ( [www.tungsten-powder.com](http://www.tungsten-powder.com) )

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## CTIA GROUP LTD Tungsten Powder Introduction

### 1. Tungsten Powder Overview

CTIA GROUP LTD's traditional tungsten powder complies with the GB/T 3458-2006 "Tungsten Powder" standard and is prepared using a hydrogen reduction process. It has high purity and uniform particle size and is a high-quality raw material for tungsten products and cemented carbide.

### 2. Tungsten Powder Characteristics

Ultra-high purity: tungsten content  $\geq 99.9\%$ , oxygen content  $\leq 0.20$  wt% (fine particles  $\leq 0.10$  wt%), and extremely low impurities.

Accurate particle size: Fisher particle size 0.4-20  $\mu\text{m}$ , 6 levels to choose from, with a deviation of only  $\pm 10\%$ .

Excellent performance: bulk density 6.0-10.0  $\text{g}/\text{cm}^3$ , uniform grains, excellent sinterability.

Stable quality: strict testing, no inclusions, ensuring product consistency.

### 3. Tungsten Powder Specifications

Brand	Fisher particle size ( $\mu\text{m}$ )
FW-1	0.4-1.0
FW-2	1.0-2.0
FW-3	2.0-4.0
FW-4	4.0-6.0
FW-5	6.0-10.0
FW-6	10.0-20.0

In addition to basic specifications, parameters such as particle size and purity can be customized according to customer needs.

### 4. Packaging and Quality Assurance

Packaging: Inner sealed plastic bag, outer iron drum, net weight 25kg or 50kg, moisture-proof and shock-proof.

Warranty: Each batch comes with a quality certificate, including chemical composition and particle size data, and the shelf life is 12 months.

### 5. Procurement Information

Email: [sales@chinatungsten.com](mailto:sales@chinatungsten.com)

Tel: +86 592 5129696

For more information about tungsten powder, please visit the website of CTIA GROUP LTD ([www.ctia.com.cn](http://www.ctia.com.cn))

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