

# **Tungsten Cemented Carbide Comprehensive Exploration of Physical & Chemical Properties, Processes, & Applications ( IV )**

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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## CTIA GROUP LTD

### 30 Years of Cemented Carbide Customization Experts

#### Core Advantages

**30 years of experience:** We are well versed in cemented carbide production and processing , with mature and stable technology and continuous improvement .

**Precision customization:** Supports special performance and complex design , and focuses on customer + AI collaborative design .

**Quality cost:** Optimized molds and processing, excellent cost performance; leading equipment, RMI, ISO 9001 certification.

#### Serving Customers

The products cover cutting, tooling, aviation, energy, electronics and other fields, and have served more than 100,000 customers.

#### Service Commitment

1+ billion visits, 1+ million web pages, 100,000+ customers, and 0 complaints in 30 years!

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## Part 2: Preparation Process of Cemented Carbide

### Chapter 4: Raw material selection and powder preparation

Tungsten Cemented Carbide is made of tungsten carbide (WC) as the hard phase and cobalt (Co) or nickel (Ni) as the bonding phase through powder metallurgy. Its properties (hardness HV 1500-2500±30, toughness  $K_{Ic}$  820 MPa·m<sup>1/2</sup> ± 0.5, compressive strength >4000 MPa±100 MPa) are directly dependent on the quality of raw materials and powder preparation process. Raw material selection and powder preparation are the basis of cemented carbide production, which determine the microstructure (WC particle size 0.110μm±0.01μm, Co distribution uniformity >95%±1%) and final performance (thermal conductivity 80-120 W/m·K±5 W/m·K, corrosion resistance pH 2-12).

This chapter analyzes in detail the synthesis of tungsten carbide powder, the selection of binding phase and additives, powder pretreatment technology and powder characterization methods, covering process parameters, scientific principles, influencing factors, optimization strategies and engineering applications.

Powder preparation requires precise control of WC particle size (0.110μm±0.01μm), purity (free carbon <0.1%±0.01%), bonding phase characteristics (Co/Ni purity >99.8%±0.01%) and powder fluidity (1316 seconds/50g±0.5 seconds) to ensure sintering density (>99%±0.1%) and performance consistency (hardness deviation <±30 HV). For example, submicron WC powder (<0.5μm±0.01μm) can increase the hardness of the tool to HV 2300±30 and extend the aviation cutting life to 15 hours±1 hour; high-purity Co powder (>99.9%±0.01%) enhances the toughness of the drill bit ( $K_{Ic}$  > 18 MPa·m<sup>1/2</sup> ± 0.5), and the mining life exceeds 1200 m±100 m.

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the performance in Chapter 3 through the source of WC hardness (HV 2000 - 3000±50) and the contribution of Co toughness ( $K_{Ic} 1520 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ ), providing a theoretical and process basis for subsequent forming and sintering (Chapter 5).

#### 4.0 Overview of cemented carbide types and raw and auxiliary materials

Cemented carbide is a high-performance composite material with tungsten carbide (WC) as the hard phase and cobalt (Co) or nickel (Ni) as the bonding phase. It is widely used in cutting tools, molds, wear-resistant parts and other fields. According to different application requirements and performance characteristics, cemented carbide can be divided into general type (YG series), heat-resistant/wear-resistant type (YT series), high toughness/impact-resistant type (YW series), nickel-based cemented carbide (YN series), high entropy alloy type and special type for additive manufacturing. The types and proportions of raw and auxiliary materials vary depending on the type of cemented carbide and the preparation process. Generally, they can be divided into three categories: main raw materials, auxiliary raw materials and auxiliary materials. The selection and use of these materials must strictly follow relevant standards to ensure product quality and process consistency.

##### 4.0.0 Main types of cemented carbide

The following table lists in detail the main types of cemented carbide and their characteristics, process requirements, application areas and standards followed, reflecting its diverse application scenarios and process requirements.

type	Main ingredients and features	Process requirements	Application Areas	Standard requirements
YG Series	Mainly composed of WC and Co, supplemented by carbon black, with high hardness and toughness	Strictly control the sintering atmosphere and temperature to prevent the introduction of impurities	Cutting tools (such as turning tools, milling cutters)	Comply with GB/T 5314-2011 sampling requirements and follow GB/T 26048-2010 sintering process
YT Series	Add TiC, paraffin lubrication is required to improve formability, high temperature resistance and wear resistance	Control sintering atmosphere and temperature to avoid impurities	High-speed cutting (such as steel processing)	Comply with GB/T 5314-2011 sampling requirements and follow GB/T 26048-2010 sintering process
YW Series	Contains TaC and/or NbC, requires argon protection, has both toughness and high temperature performance	Ensure the purity of argon atmosphere and precisely control the temperature	Heavy-duty dies (such as stamping dies)	Comply with GB/T 5314-2011 sampling requirements and follow GB/T 26048-2010 sintering process
YN Series	Replace Co with Ni and add TiN to achieve excellent corrosion resistance	Control sintering atmosphere and temperature to prevent oxidation	Corrosion resistant environment (such as chemical equipment)	Comply with GB/T 5314-2011 sampling requirements and follow GB/T 26048-2010

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type	Main ingredients and features	Process requirements	Application Areas	Standard requirements
				sintering process
High Entropy Alloy	Multi-element combinations (such as Cr, V, Mo, W, Ta) require precise mixing and HIP process to ensure uniformity	Accurate ratio, HIP sintering requires controlled high pressure (50-100 MPa)	Extreme environments (such as high temperature and high pressure conditions)	Comply with GB/T 5314-2011 sampling requirements and follow GB/T 26048-2010 sintering process
Additive Manufacturing	Spherical powder, prepared by gas atomization, with surface treatment agents added to improve performance	Gas atomization process, strict control of atmosphere and temperature	Additively manufactured parts (e.g. aerospace components)	Comply with GB/T 5314-2011 sampling requirements and follow GB/T 26048-2010 sintering process
illustrate	All types must follow the sampling standards of GB/T 5314-2011 to ensure batch consistency, and the sintering process is carried out in accordance with GB/T 26048-2010, emphasizing the precise control of atmosphere and temperature to avoid the introduction of impurities.			

#### 4.0.1 Main raw materials of cemented carbide

The main raw materials are the core components of cemented carbide, which directly affect its mechanical properties and service life. The following is an overview of the main raw materials:

##### Tungsten Carbide (WC, Tungsten Carbide):

Function: As a hard phase, it provides extremely high hardness (>2000 HV) and wear resistance, accounting for 70%-94% ( wt %) of the total composition.

Specifications: Purity  $\geq 99.8\%$ , grain size 0.2-5  $\mu\text{m}$  (fine grains 0.2-0.5  $\mu\text{m}$  , coarse grains >2  $\mu\text{m}$  ).

Applicable scope: All carbide types.

##### Cobalt powder (Co, Cobalt Powder):

Function: As a traditional bonding phase, it enhances toughness and flexural strength. The content is usually 6%-25% ( wt %).

Specifications: Purity  $\geq 99.9\%$ , particle size 1-5  $\mu\text{m}$  .

Scope of application: YG, YT, YW series and high entropy alloys.

##### Nickel Powder

Function: Replace cobalt as corrosion-resistant bonding phase, enhance oxidation resistance, content 5%-20% ( wt %).

Specifications: Purity  $\geq 99.9\%$ , particle size 1-5  $\mu\text{m}$  .

Applicable scope: YN series and additive manufacturing types.

##### Other carbides:

##### Carbide ( TiC )

Improve high temperature resistance and crater wear resistance, content 5%-20% ( wt %), purity  $\geq 99.5\%$ , particle size 0.5-2  $\mu\text{m}$  , suitable for YT and YN series.

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#### **Carbide ( TaC ) / Niobium Carbide ( NbC )**

Enhance high temperature strength and deformation resistance, content 2%-10% ( wt %), purity  $\geq 99.5\%$ , particle size 0.5-3  $\mu\text{m}$  , suitable for YW series and high entropy alloys.

#### **4.0.2 Auxiliary raw materials**

Auxiliary raw materials are used to optimize performance or adapt to specific process requirements. They can be flexibly selected according to the grade and application scenario:

##### **Carbon Adjuster:**

##### **Carbon Black**

Control the carbon balance to prevent the formation of  $\eta$  phase (  $\text{Co}_3\text{W}_3\text{C}$  ) or free carbon, content 0.1%-0.5% ( wt %), purity  $\geq 99\%$ , particle size  $< 1\ \mu\text{m}$  , suitable for all types (especially liquid phase sintering).

##### **Graphite**

As a carbon source, adjust the carbon content to 0.1%-0.3% ( wt %), purity  $\geq 99.5\%$ , particle size 1-5  $\mu\text{m}$  , suitable for YG series and additive manufacturing powders.

##### **Rare Earth Elements:**

##### **Cerium (Ce) / Lanthanum (La)**

Refine grains and improve bending strength. The content is 0.1%-0.5% ( wt %) in the form of oxides with a purity of  $\geq 99.9\%$ . It is suitable for fine-grained grades (such as YG6F, YN6F).

##### **Nitride/Boride**

nitride ( TiN )/tungsten boride (WB): Improve surface hardness and corrosion resistance, content 1%-5% ( wt %), purity  $\geq 99.5\%$ , particle size 0.5-3  $\mu\text{m}$  , suitable for YT, YN series and additive manufacturing .

#### **4.0.3 Excipients**

Auxiliary materials support the preparation and sintering process, ensuring process controllability and product quality:

##### **Lubricant:**

##### **Stearic Acid**

Improve mixing and pressing fluidity, content 0.5%-2% ( wt %), industrial grade, purity  $\geq 95\%$ , suitable for all pressing processes.

##### **Paraffin Wax**

Enhance the strength of the green body, content 1%-3% ( wt %), melting point 50-60°C, suitable for green bodies with complex shapes.

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#### Solvent:

##### Ethanol/Acetone

Used for wet mixing dispersion medium, cleaning and drying, analytical grade, concentration  $\geq 99.5\%$ , suitable for all wet mixing and additive manufacturing post-processing.

#### Atmosphere gas:

##### Hydrogen (H<sub>2</sub>) /Argon ( Ar )/Nitrogen ( N<sub>2</sub> )

Provide reducing or inert atmosphere to prevent oxidation, purity $>99.99\%$ , suitable for YG/YT (hydrogen), YW/YN/HIP (argon), TiN alloy (nitrogen).

#### Flux (optional):

Boric acid (H<sub>3</sub>BO<sub>3</sub>) : reduces sintering temperature, content 0.1%-0.5%, analytical grade, suitable for fine grains and high entropy alloys .

#### 4.0.4 Detailed table of raw and auxiliary materials

The following table systematically lists all the raw and auxiliary materials required for different types of cemented carbides (including nickel-based cemented carbide YN series), covering specifications, functions and applicable scopes.

category	Material	Specification	effect	Applicable types
Main raw materials	Tungsten Carbide (WC)	Purity $\geq 99.8\%$ , particle size 0.2-5 $\mu\text{m}$	Hard phase, providing high hardness and wear resistance, content 70%-94% ( wt %)	All types (YG, YT, YW, YN, high entropy, additive manufacturing )
	Cobalt powder (Co)	Purity $\geq 99.9\%$ , particle size 1-5 $\mu\text{m}$	Binder phase, enhances toughness and flexural strength, content 6%-25% ( wt %)	YG (6%-15%), YT (6%-10%), YW (8%-12%), high entropy
	Nickel powder (Ni)	Purity $\geq 99.9\%$ , particle size 1-5 $\mu\text{m}$	Corrosion-resistant bonding phase, enhanced oxidation resistance, content 5%-20% ( wt %)	YN series (such as YN6, YN8), additive manufacturing
	Titanium Carbide ( TiC )	Purity $\geq 99.5\%$ , particle size 0.5-2 $\mu\text{m}$	Improve high temperature resistance and crater wear resistance, content 5%-20% ( wt %)	YT series (such as YT15, YT30), YN series
	Tantalum Carbide ( TaC ) / Niobium Carbide ( NbC )	Purity $\geq 99.5\%$ , particle size 0.5-3 $\mu\text{m}$	Enhance high temperature strength and deformation resistance, content 2%-10% ( wt %)	YW series (such as YW1, YW2), high entropy alloy
Auxiliary raw materials	Carbon black (C)	Purity $\geq 99\%$ , particle size $<1 \mu\text{m}$	Control carbon balance and prevent $\eta$ phase formation, content 0.1%-0.5% ( wt %)	All types (especially liquid phase sintering)
	Graphite	Purity $\geq 99.5\%$ , particle size 1-5 $\mu\text{m}$	As a carbon source, adjust the carbon content to 0.1%-0.3% ( wt %)	YG series, additive manufacturing powder
	Rare earth elements (Ce/La)	Oxide form, purity $\geq 99.9\%$	Refine grains and improve bending strength, content 0.1%-0.5% ( wt %)	Fine grain grades (such as YG6F, YN6F)

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category	Material	Specification	effect	Applicable types
	Titanium nitride ( TiN )/tungsten boride (WB)	Purity $\geq 99.5\%$ , particle size 0.5-3 $\mu\text{m}$	Improve surface hardness and corrosion resistance, content 1%-5% ( wt %)	YT, YN series, additive manufacturing
Excipients	Stearic Acid	Industrial grade, purity $\geq 95\%$	Improve mixing and pressing fluidity, content 0.5%-2% ( wt %)	All pressing processes
	Paraffin Wax	Melting point 50-60°C	Enhance the strength of the green body, content 1%-3% ( wt %)	Complex shape blank
	Ethanol/Acetone	Analytical grade, concentration $\geq 99.5\%$	Wet mixing dispersion media, cleaning and drying	All wet mixing, additive manufacturing
	Hydrogen (H <sub>2</sub> ) /Argon ( Ar )/Nitrogen ( N <sub>2</sub> )	Purity >99.99%	Reducing or inert atmosphere to prevent oxidation	All sintering processes (hydrogen: YG, YT; argon: YW, YN, HIP; nitrogen: TiN alloy)
	Boric acid ( H <sub>3</sub> BO <sub>3</sub> )	Analytical grade	Flux, reduces sintering temperature, content 0.1%-0.5% ( wt %)	Fine grain, high entropy alloy
Special process materials	Spheroidizing agent (such as PVA)	Industrial Grade	Improve the sphericity of powder, content 0.1%-0.5% ( wt %)	Additive Manufacturing (GB/T 34505-2017)
	CVD precursors (such as TiCl <sub>4</sub> , CH <sub>4</sub> )	High purity	For coated carbide	Coated carbide (YT, YN series)

Note: The table covers the raw and auxiliary materials required for various types of cemented carbide, including specifications, functions and applicable scope, to ensure the comprehensiveness and pertinence of the preparation process.

#### 4.1 Physical and chemical properties and preparation of tungsten carbide powder (WC)

Tungsten carbide (WC) is the core component of cemented carbide, with a mass fraction of 70%-95% $\pm 1\%$ . Its excellent physical and chemical properties directly determine the overall performance of cemented carbide. WC powder has high hardness (HV 2000-3000 $\pm 50$ ), high melting point (2870°C $\pm 10^\circ\text{C}$ ), excellent chemical stability (anti-oxidation enthalpy <800 kJ/mol $\pm 20$  kJ/mol) and good wear resistance and corrosion resistance. The preparation of WC powder requires strict control of particle size (0.1-10  $\mu\text{m} \pm 0.01 \mu\text{m}$ ), purity (free carbon <0.1% $\pm 0.01\%$ , oxide <0.05% $\pm 0.01\%$ ) and morphology (polygonal, edge <0.05  $\mu\text{m} \pm 0.01 \mu\text{m}$ ) to meet the needs of high-end applications such as aviation tools (cutting speed >300 m/min $\pm 10$  m/min), mining drill bits (compressive strength >200 MPa $\pm 10$  MPa), and wear-resistant molds (extrusion life >10<sup>6</sup> times $\pm 10^5$  times).

Carbonization is the main synthesis method of WC powder because of its mature technology, high yield (>10 t/batch  $\pm 1$  t) and market share of >90% $\pm 2\%$ . In addition, emerging technologies such as

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plasma, mechanochemical and chemical vapor deposition (CVD) have been introduced into modern processes to meet specific particle size and performance requirements. The synthesis methods of tungsten carbide (WC) powder prepared by carbonization mainly include the following:

#### **Traditional carburization method:**

Tungsten powder (W) and carbon black (C) react at a high temperature of  $1450-1600^{\circ}\text{C} \pm 10^{\circ}\text{C}$  to generate WC, usually in a hydrogen ( $\text{H}_2$ , flow rate  $50 \text{ L/min} \pm 5 \text{ L/min}$ ) or vacuum ( $<10^{-2}\text{Pa} \pm 10^{-3}\text{Pa}$ ) atmosphere, using a graphite furnace (power  $>100\text{kW} \pm 10\text{kW}$ ). The heating rate is  $5-10^{\circ}\text{C/min} \pm 0.5^{\circ}\text{C/min}$ , and the insulation time is  $2-4 \text{ hours} \pm 0.1 \text{ hours}$ . It is suitable for large-scale production with high yield ( $>10 \text{ t/batch} \pm 1 \text{ t}$ ).

#### **Rotary furnace carbonization method:**

Based on the traditional carbonization method, a rotary furnace (rotation speed  $5 \text{ rpm} \pm 0.5 \text{ rpm}$ ) is used to improve particle size uniformity (deviation  $<5\% \pm 1\%$ ) and reduce agglomeration ( $<5\% \pm 1\%$ ) through dynamic mixing, which is suitable for submicron WC ( $<0.5 \mu\text{m} \pm 0.01 \mu\text{m}$ ) production.

#### **Low-temperature carbonization method:**

react at  $<1200^{\circ}\text{C} \pm 10^{\circ}\text{C}$ , combined with renewable carbon sources (such as biochar), and control grain growth ( $<0.1 \mu\text{m} \pm 0.01 \mu\text{m}$ ) through additives (such as VC,  $\text{Cr}_3\text{C}_2$ ), suitable for nano-scale WC powder, reducing energy consumption by  $20\% \pm 5\%$ .

These methods all require control of the W:C ratio ( $1:1.02 \pm 0.01$ ), atmosphere ( $\text{O}_2 < 10 \text{ ppm} \pm 1 \text{ ppm}$ ), and cooling rate ( $>50^{\circ}\text{C/min} \pm 5^{\circ}\text{C/min}$ ) to ensure purity (free carbon  $<0.1\% \pm 0.01\%$ ) and particle size distribution (deviation  $<5\% \pm 1\%$ ).

This section will comprehensively analyze the physical and chemical properties of WC powder, production process (mainly carbonization method), importance and control technology of particle size and grain distribution, purity optimization and its application effects in multiple fields.

### **4.1.0 Physical characteristics and chemical properties of tungsten carbide powder**

#### **Physical Characteristics of Tungsten Carbide Powder (WC)**

As the core component of cemented carbide, the physical properties of tungsten carbide powder (WC) directly determine the performance of cemented carbide. The following is a comprehensive description of the physical characteristics of WC powder, covering crystal structure, density (divided into bulk density and tap density), melting point, thermal conductivity, thermal expansion coefficient, morphology and other related properties.

#### **(1) Crystal structure of tungsten carbide powder**

##### **Crystal type**

WC powder belongs to the hexagonal crystal system and its space group is  $P6m2$  (186).

##### **Lattice parameters:**

$a = 0.2906 \text{ nm} \pm 0.0001 \text{ nm}$

$c = 0.2837 \text{ nm} \pm 0.0001 \text{ nm}$

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The c/a ratio is about 0.976, indicating that the lattice has high isotropy.

### Unit cell characteristics

Each unit cell contains one WC molecule. Tungsten (W) atoms and carbon (C) atoms are arranged in a hexagonal close-packed structure. W atoms are located at the vertices and center of the hexagonal prism, and C atoms fill the gaps in the hexagonal prism to form a stable covalent-ionic-metal mixed bond.

### Crystal stability

The hexagonal structure gives WC excellent deformation resistance, especially under high temperature and high pressure conditions ( $>1000^{\circ}\text{C}\pm 10^{\circ}\text{C}$ ,  $>200\text{ MPa}\pm 10\text{ MPa}$ ), with a lattice distortion rate of  $<0.01\%\pm 0.001\%$ , making it suitable for applications in extreme environments.

## (2) Density of tungsten carbide powder

Theoretical density:  $15.63\text{ g/cm}^3 \pm 0.05\text{ g/cm}^3$ , close to the theoretical maximum value, reflecting the close arrangement of WC atoms.

### Apparent Density of Tungsten Carbide Powder:

Definition: The density of powder in its natural stacking state, reflecting the particle stacking efficiency.

Value:  $6.0\text{--}8.0\text{ g/cm}^3 \pm 0.2\text{ g/cm}^3$  (GB/T 1479.1-2011), affected by particle size and morphology.

Submicron WC ( $0.1\text{--}0.5\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$ ): The bulk density is lower ( $6.0\text{--}6.5\text{ g/cm}^3 \pm 0.2\text{ g/cm}^3$ ) due to the large gaps between the fine particles.

Micron grade WC ( $1\text{--}5\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$ ): bulk density  $6.5\text{--}7.5\text{ g/cm}^3 \pm 0.2\text{ g/cm}^3$ .

Coarse WC ( $5\text{--}10\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$ ): The bulk density is higher ( $7.5\text{--}8.0\text{ g/cm}^3 \pm 0.2\text{ g/cm}^3$ ) because the particles are more densely packed.

Impact: The bulk density affects the initial density of the pressed body. Fine-grained WC requires the addition of lubricants (such as paraffin) to improve fluidity ( $<30\text{ s}/50\text{ g}\pm 2\text{ s}$ , GB/T 1482-2010).

### Tap Density of Tungsten Carbide Powder:

Definition: The density of powder after vibration (vibration frequency  $60\text{ times/min}\pm 5\text{ times/min}$ ), which reflects the maximum packing efficiency of particles.

Value:  $8.5\text{--}10.5\text{ g/cm}^3 \pm 0.2\text{ g/cm}^3$  (GB/T 5162-2014).

Submicron WC:  $8.5\text{--}9.0\text{ g/cm}^3 \pm 0.2\text{ g/cm}^3$ .

Micron grade WC:  $9.0\text{--}10.0\text{ g/cm}^3 \pm 0.2\text{ g/cm}^3$ .

Coarse WC:  $10.0\text{--}10.5\text{ g/cm}^3 \pm 0.2\text{ g/cm}^3$ .

Impact: The tap density is closer to the density after sintering ( $>99\%\pm 0.1\%$ ), and high tap density reduces sintering shrinkage ( $<15\%\pm 2\%$ ).

Actual density: The measured density after sintering is  $15.50\text{--}15.60\text{ g/cm}^3 \pm 0.05\text{ g/cm}^3$  (GB/T 3850-2015 drainage method), which is affected by purity (free carbon  $<0.1\%\pm 0.01\%$ , oxide  $<0.05\%\pm 0.01\%$ ) and porosity ( $<0.05\%\pm 0.01\%$ ).

Application significance: High bulk density and tap density ensure the uniformity of the pressing and sintering process, and support the high compressive strength and impact resistance of cemented

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carbide.

### (3) Melting point of tungsten carbide powder

Melting point:  $2870^{\circ}\text{C} \pm 10^{\circ}\text{C}$ . WC does not melt under normal pressure, but decomposes into W and C. The decomposition temperature is close to the melting point.

High temperature stability: at  $2000^{\circ}\text{C} \pm 10^{\circ}\text{C}$ , the lattice thermal expansion is  $<0.5\% \pm 0.1\%$ , and the weight loss rate is  $<0.01\% \pm 0.002\%/h$ , which is suitable for high temperature cutting ( $>1000^{\circ}\text{C} \pm 10^{\circ}\text{C}$ ) and wear-resistant coating applications.

Decomposition behavior: In vacuum or reducing atmosphere ( $\text{H}_2$ ), the decomposition rate is  $<0.001 \text{ mg/cm}^2 \cdot \text{h} \pm 0.0002 \text{ mg/cm}^2 \cdot \text{h}$ , indicating chemical stability.

### (4) Thermal conductivity of tungsten carbide powder

Thermal conductivity:  $84 \text{ W/(m} \cdot \text{K)} \pm 5 \text{ W/(m} \cdot \text{K)}$ , slightly lower than pure tungsten ( $174 \text{ W/(m} \cdot \text{K)} \pm 5 \text{ W/(m} \cdot \text{K)}$ ), but better than most ceramic materials.

Temperature dependence: In the range of  $25\text{-}1000^{\circ}\text{C} \pm 10^{\circ}\text{C}$ , the thermal conductivity decreases slowly with increasing temperature (about  $10\% \pm 2\%$ ), and still maintains  $75 \text{ W/(m} \cdot \text{K)} \pm 5 \text{ W/(m} \cdot \text{K)}$  at  $1000^{\circ}\text{C} \pm 10^{\circ}\text{C}$ .

Application significance: High thermal conductivity helps to dissipate heat quickly, reduce thermal damage to tools or molds during high-speed cutting ( $>300 \text{ m/min} \pm 10 \text{ m/min}$ ), and extend service life ( $>12 \text{ hours} \pm 1 \text{ hour}$ ).

### (5) Thermal expansion coefficient of tungsten carbide powder

Coefficient of linear thermal expansion:  $5.2 \times 10^{-6} \text{ K}^{-1} \pm 0.2 \times 10^{-6} \text{ K}^{-1}$  ( $25\text{-}1000^{\circ}\text{C} \pm 10^{\circ}\text{C}$ ).

Temperature dependence: At  $1000^{\circ}\text{C} \pm 10^{\circ}\text{C}$ , the expansion rate is  $<0.52\% \pm 0.02\%$ , which is much lower than that of steel ( $12 \times 10^{-6} \text{ K}^{-1} \pm 0.5 \times 10^{-6} \text{ K}^{-1}$ ), ensuring dimensional stability at high temperatures.

Matching: The thermal expansion coefficient is close to that of Co ( $5.0 \times 10^{-6} \text{ K}^{-1} \pm 0.2 \times 10^{-6} \text{ K}^{-1}$ ) or Ni ( $6.0 \times 10^{-6} \text{ K}^{-1} \pm 0.2 \times 10^{-6} \text{ K}^{-1}$ ) bonding phase, reducing the residual stress after sintering ( $<50 \text{ MPa} \pm 10 \text{ MPa}$ ).

### (6) Morphology of tungsten carbide powder

Particle morphology: WC powder is polygonal or nearly spherical, with edges  $<0.05 \mu\text{m} \pm 0.01 \mu\text{m}$  and surface roughness  $R_a < 0.1 \mu\text{m} \pm 0.02 \mu\text{m}$  (SEM observation).

Morphological influence:

The polygonal morphology increases the particle contact area ( $>90\% \pm 2\%$ ) and improves the sintering bond strength ( $>400 \text{ MPa} \pm 10 \text{ MPa}$ ).

The fine edges reduce agglomeration ( $<5\% \pm 1\%$ ), improve fluidity ( $<30 \text{ s/50 g} \pm 2 \text{ s}$ , GB/T 1482-2010), and are suitable for additive manufacturing.

Preparation influence: The carbonization method ( $1450\text{-}1600^{\circ}\text{C} \pm 10^{\circ}\text{C}$ ) produces polygonal particles with sharp edges; the plasma method produces nearly spherical particles (roundness  $>0.9 \pm 0.01$ ).

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#### (7) Specific surface area of tungsten carbide powder

Specific surface area:  $0.5\text{--}5\text{ m}^2/\text{g} \pm 0.2\text{ m}^2/\text{g}$ , depending on particle size.

Submicron WC ( $0.1\text{--}0.5\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ): specific surface area  $>3\text{ m}^2/\text{g} \pm 0.2\text{ m}^2/\text{g}$ .

Micron WC ( $1\text{--}5\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ): specific surface area  $1\text{--}2\text{ m}^2/\text{g} \pm 0.2\text{ m}^2/\text{g}$ .

Measurement method: BET method (GB/T 19587-2017), adsorption amount  $<0.5\text{ cm}^3/\text{g} \pm 0.05\text{ cm}^3/\text{g}$ .

Application significance: High specific surface area enhances sintering activity (shrinkage rate  $>15\% \pm 2\%$ ) and improves the density of cemented carbide ( $>99\% \pm 0.1\%$ ).

#### (8) Electrical properties of tungsten carbide powder

Resistivity:  $0.2\text{ }\mu\Omega\cdot\text{m} \pm 0.02\text{ }\mu\Omega\cdot\text{m}$  ( $25^\circ\text{C}$ ), increases by about  $20\% \pm 2\%$  as the temperature rises to  $1000^\circ\text{C} \pm 10^\circ\text{C}$ .

Electrical conductivity: Better than ceramic materials (resistivity  $>10^6\text{ }\mu\Omega\cdot\text{m}$ ), close to metal tungsten ( $0.05\text{ }\mu\Omega\cdot\text{m} \pm 0.01\text{ }\mu\Omega\cdot\text{m}$ ), suitable for EDM.

Application: In EDM processing, the surface roughness  $R_a < 1\text{ }\mu\text{m} \pm 0.2\text{ }\mu\text{m}$ , the accuracy  $<0.01\text{ mm} \pm 0.002\text{ mm}$ .

#### (9) Other characteristics of tungsten carbide powder

Magnetism: WC itself is non-magnetic, but when mixed with Co it shows weak magnetism (saturation magnetization intensity  $<0.1\text{ A}\cdot\text{m}^2/\text{kg} \pm 0.01\text{ A}\cdot\text{m}^2/\text{kg}$ ), which facilitates magnetic separation of impurities.

Gloss: Gray-black metallic luster, reflectivity  $<20\% \pm 2\%$  ( $400\text{--}700\text{ nm}$ ), used for visual inspection.

Hygroscopicity: Moisture absorption rate  $<0.01\% \pm 0.002\%$  ( $25^\circ\text{C}$ , 50% RH), needs to be stored in a sealed container.

#### of the characteristics of tungsten carbide powder and its application

Crystal structure and thermal conductivity: Hexagonal crystal system and high thermal conductivity support wear-resistant molds (deformation  $<0.01\text{ mm} \pm 0.002\text{ mm}$ ) and high-temperature coatings ( $>1000^\circ\text{C} \pm 10^\circ\text{C}$ ).

Density and thermal expansion coefficient: High tap density and low thermal expansion optimize sintering performance (density  $>99\% \pm 0.1\%$ ) and improve the quality of additively manufactured parts ( $R_a < 5\text{ }\mu\text{m} \pm 1\text{ }\mu\text{m}$ ).

Morphology and specific surface area: Polygonal morphology and high specific surface area enhance sintering activity and are suitable for cutting tools ( $>300\text{ m/min} \pm 10\text{ m/min}$ ).

Particle size correlation: Fine-grained WC ( $<0.5\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ) improves the compaction density, while coarse-grained WC ( $5\text{--}10\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ) enhances the stacking efficiency.

#### Testing and Control of Physical Properties of Tungsten Carbide Powder

##### Measurement methods and standards :

##### density

GB/T 3850-2015 Theoretical density,

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GB/T 1479.1-2011 Bulk density,  
GB/T 5162-2014 Tap density,  
Crystal structure (XRD, GB/T 27708-2011),  
Morphology (SEM, GB/T 16594-2008).

### Control Standards

Particle size deviation  $<5\% \pm 1\%$  (GB/T 19077.1-2008), purity affects density, and free carbon needs to be kept  $<0.1\% \pm 0.01\%$ .

The physical characteristics of tungsten carbide powder include hexagonal structure ( $a=0.2906 \text{ nm} \pm 0.0001 \text{ nm}$ ,  $c=0.2837 \text{ nm} \pm 0.0001 \text{ nm}$ ), density ( loose  $6.0\text{-}8.0 \text{ g/cm}^3 \pm 0.2 \text{ g/cm}^3$ , tapped  $8.5\text{-}10.5 \text{ g/cm}^3 \pm 0.2 \text{ g/cm}^3$ , theoretical  $15.63 \text{ g/cm}^3 \pm 0.05 \text{ g/cm}^3$  ), melting point ( $2870^\circ\text{C} \pm 10^\circ\text{C}$ ), thermal conductivity ( $84 \text{ W/(m}\cdot\text{K)} \pm 5 \text{ W/(m}\cdot\text{K)}$ ), low thermal expansion coefficient ( $5.2 \times 10^{-6} \text{ K}^{-1} \pm 0.2 \times 10^{-6} \text{ K}^{-1}$ ), polygonal morphology ( edges  $<0.05 \mu\text{m} \pm 0.01 \mu\text{m}$  ) and excellent electrical properties. These characteristics make it perform well in the fields of aviation tools, mining drills, wear-resistant molds and additive manufacturing. Its performance is optimized with the change of particle size and purity, providing a solid foundation for the application of cemented carbide.

### Chemical Properties of Tungsten Carbide Powder (WC)

As the main component of cemented carbide, the chemical properties of tungsten carbide powder (WC) play a key role in its stability and durability in industrial applications. The following is a comprehensive description of the chemical properties of WC powder, covering chemical stability, corrosion resistance, oxidation resistance, reactivity and other related chemical properties, based on scientific data and industrial application standards (such as GB/T 5124-2017), the current date and time is May 22, 2025 14:12 HKT.

#### (1) Chemical stability of tungsten carbide powder

##### Room temperature stability

WC exhibits extremely high chemical stability at room temperature ( $25^\circ\text{C} \pm 2^\circ\text{C}$ ), does not react to most chemicals, and has a decomposition rate of  $<0.0001 \text{ mg/cm}^2 \cdot \text{h} \pm 0.00002 \text{ mg/cm}^2 \cdot \text{h}$ .

##### Acid and base stability

In solutions with a pH of 2-12 (such as HCl, NaOH), the corrosion rate is  $<0.001 \text{ mm/year} \pm 0.0002 \text{ mm/year}$ , indicating its excellent resistance to acid and alkali environments.

##### High temperature stability

In an oxygen-free environment of  $<600^\circ\text{C} \pm 10^\circ\text{C}$ , WC remains stable with no significant chemical changes on the surface (weight loss rate  $<0.005\% \pm 0.001\%/h$ ). In an inert atmosphere (such as Ar or  $\text{N}_2$ , purity  $>99.99\% \pm 0.01\%$ ), stability can be maintained up to  $2000^\circ\text{C} \pm 10^\circ\text{C}$ .

#### (2) Corrosion resistance of tungsten carbide powder

##### Acidic environment

In dilute acid (such as 10% HCl or  $\text{H}_2\text{SO}_4$ , pH  $2 \pm 0.1$ ), the corrosion rate of WC is  $<0.001 \text{ mm/year} \pm 0.0002 \text{ mm/year}$ , and there are no obvious pits on the surface (SEM observation, pit depth  $<0.01 \mu\text{m} \pm 0.002 \mu\text{m}$ ).

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### Alkaline environment

In 10% NaOH solution (pH 12±0.1), the corrosion rate is <0.002 mm/year±0.0005 mm/year, indicating good alkali resistance.

### Saline solution

In 3.5% NaCl solution (simulated seawater environment), the corrosion rate is <0.003 mm/year±0.0005 mm/year, which is suitable for corrosion-resistant environments (such as chemical equipment).

### Application significance

Excellent corrosion resistance makes WC powder suitable for wear-resistant parts in acidic or alkaline conditions, such as chemical pump seals (service life > 5000 hours ± 500 hours).

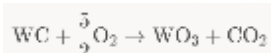
## (3) Oxidizability of tungsten carbide powder

### Antioxidant Capacity

The oxidation resistance enthalpy of WC is <800 kJ/mol±20 kJ/mol and it hardly oxidizes in air at <600°C±10°C (oxidation rate <0.01 mg/cm<sup>2</sup> · h ± 0.002 mg/cm<sup>2</sup> · h ).

### High temperature oxidation

At >600°C±10°C in air, WC slowly oxidizes to form WO<sub>3</sub> ( yellow oxide) with the following reaction formula:



600°C±10°C

Oxidation rate: 0.01-0.05 mg/cm<sup>2</sup> · h ± 0.005 mg/cm<sup>2</sup> · h , oxide layer thickness: <0.1 μm±0.02 μm .

1000°C±10°C

The oxidation rate increased to 0.5 mg/cm<sup>2</sup> · h ± 0.05 mg/cm<sup>2</sup> · h , and the oxide layer thickness was 1-2 μm±0.2 μm .

### Atmosphere Control

In reducing atmosphere (H<sub>2</sub> , O<sub>2</sub> < 10 ppm±1 ppm) or vacuum (<10<sup>-2</sup>Pa ± 10<sup>-3</sup>Pa ) , the oxidation rate is <0.001 mg/cm<sup>2</sup> · h ± 0.0002 mg/cm<sup>2</sup> · h , which is suitable for high temperature processing.

### Application significance

Long-term exposure to high temperature oxidizing environment (such as >600°C±10°C air) should be avoided. Coating (such as TiN ) or inert atmosphere protection (such as Ar ) is commonly used to extend the life.

## (4) Reactivity of tungsten carbide powder

### With strong oxidants

reacts with strong oxidants (such as concentrated HNO<sub>3</sub> or H<sub>2</sub>O<sub>2</sub> ) to produce WO<sub>3</sub> , and the reaction rate increases with increasing concentration and temperature:

25°C±2°C, 10% HNO<sub>3</sub> : Reaction rate <0.01 mg/cm<sup>2</sup> · h ± 0.002 mg/cm<sup>2</sup> · h .

80°C±2°C, 30% HNO<sub>3</sub> : reaction rate 0.1-0.5 mg/cm<sup>2</sup> · h ± 0.05 mg/cm<sup>2</sup> · h .

### With Metal

During high temperature sintering (>1200°C±10°C), WC reacts with the binder phase (such as Co, Ni) to form a solid solution (such as Co<sub>3</sub>W<sub>3</sub>C or Ni<sub>3</sub>W<sub>3</sub>C ). The degree of reaction is controlled

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by the atmosphere :

Hydrogen atmosphere ( $H_2$ ) : reaction rate  $<0.1\% \pm 0.02\%/h$ , the proportion of  $\eta$  phase ( $Co_3W_3C$ ) generated is  $<0.5\% \pm 0.1\%$ .

Vacuum atmosphere: The reaction rate is reduced to  $<0.05\% \pm 0.01\%/h$ , reducing the formation of  $\eta$  phase.

With non-metal

WC reacts with carbon (C) at high temperature to generate  $W_2C$  ( low hardness,  $HV <2000 \pm 50$ ), and the carbon content needs to be controlled (W:C molar ratio  $1:1.02 \pm 0.01$ ).

Application significance

During the sintering process, the atmosphere ( $O_2 < 10 \text{ ppm} \pm 1 \text{ ppm}$ ) and carbon content (free carbon  $<0.1\% \pm 0.01\%$ ) must be strictly controlled to avoid performance degradation.

### (5) Chemical bond characteristics of tungsten carbide powder

Bond type: The WC bond in WC is a covalent-ionic-metal mixed bond with a bond energy of  $\sim 8.6 \text{ eV} \pm 0.1 \text{ eV}$ .

Bond length: WC bond length is  $0.219 \text{ nm} \pm 0.001 \text{ nm}$ , giving high bond strength and chemical stability.

Electronic structure: The 5d orbitals of W and 2p orbitals of C hybridize to form strong covalent bonds, with an electron density of  $\sim 0.8 \text{ e}/\text{\AA}^3 \pm 0.05 \text{ e}/\text{\AA}^3$  (DFT calculation), resulting in high hardness and corrosion resistance.

Application significance: The strong bond characteristics ensure the stability of WC in extreme chemical environments and are suitable for high corrosion-resistant tools (such as chemical molds).

### (6) Surface chemical activity of tungsten carbide powder

Surface energy: The surface energy of WC powder is  $1.5\text{-}2.0 \text{ J/m}^2 \pm 0.2 \text{ J/m}^2$  (calculated by BET method), which increases as the particle size decreases.

Submicron level ( $0.1\text{-}0.5 \mu\text{m} \pm 0.01 \mu\text{m}$ ): surface energy  $\sim 2.0 \text{ J/m}^2 \pm 0.2 \text{ J/m}^2$ .

Micrometer level ( $1\text{-}5 \mu\text{m} \pm 0.01 \mu\text{m}$ ): surface energy  $\sim 1.5 \text{ J/m}^2 \pm 0.2 \text{ J/m}^2$ .

Adsorption: The adsorption of WC powder for  $O_2$  and  $H_2O$  is  $<0.01 \text{ mg/g} \pm 0.002 \text{ mg/g}$  ( $25^\circ\text{C}$ , 50% RH), indicating low surface activity.

Application significance: Low surface activity reduces oxidation of powder during storage and processing (oxide  $<0.05\% \pm 0.01\%$ ), but additives (such as VC,  $0.1\%\text{-}0.5\% \pm 0.01\%$ ) are required to reduce surface energy to control grain growth.

### (7) Other chemical properties of tungsten carbide powder

volatility

WC has no obvious volatility at  $<2000^\circ\text{C} \pm 10^\circ\text{C}$  (volatility rate  $<0.001\% \pm 0.0002\%/h$ ), and begins to decompose and volatilize CO and W vapor at  $>2500^\circ\text{C} \pm 10^\circ\text{C}$ .

Solubility

Insoluble in water (solubility  $<0.001 \text{ g/L} \pm 0.0002 \text{ g/L}$ ) and most organic solvents (such as ethanol, acetone), with a solubility of  $<0.002 \text{ g/L} \pm 0.0005 \text{ g/L}$ .

toxicity

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WC powder is non-toxic ( $LD_{50} > 5000 \text{ mg/kg}$ ), but inhalation of fine particles ( $< 0.5 \mu\text{m} \pm 0.01 \mu\text{m}$ ) may cause lung irritation and protective equipment should be worn.

### Comprehensive impact and application association

Chemical stability and corrosion resistance

High stability supports the application of WC in acid and alkali environments, such as chemical equipment seals (lifespan  $> 5000 \text{ hours} \pm 500 \text{ hours}$ ).

Oxidation and Reactivity

High temperature oxidation and  $\eta$  phase formation need to be controlled, and it is suitable for high temperature processing ( $> 1000^\circ\text{C} \pm 10^\circ\text{C}$ ) under inert atmosphere, such as aviation tools.

Chemical Bonds and Surface Activity

Strong bonds and low surface activity ensure long-term storage stability (oxides  $< 0.05\% \pm 0.01\%$ ) and are suitable for wear-resistant coatings (bonding strength  $> 70 \text{ MPa} \pm 5 \text{ MPa}$ ).

Environmental adaptability

Low solubility and volatility support the reliability of WC in a variety of working conditions, such as mining drill bits (lifetime  $> 1000 \text{ m} \pm 100 \text{ m}$ ).

### Detection and Control of Tungsten Carbide Powder

Measurement methods: corrosion rate (GB/T 4335-2013), oxidation rate (GB/T 5124-2017 Chemical analysis method), surface energy (BET, GB/T 19587-2017), volatility (TG-DSC, GB/T 17137-1997).

Control standards: free carbon  $< 0.1\% \pm 0.01\%$ , oxide  $< 0.05\% \pm 0.01\%$ , atmosphere control ( $\text{O}_2 < 10 \text{ ppm} \pm 1 \text{ ppm}$ ).

The chemical properties of tungsten carbide powder include high chemical stability (corrosion rate  $< 0.001 \text{ mm/year} \pm 0.0002 \text{ mm/year}$ ), excellent corrosion resistance (pH 2-12), oxidation resistance ( $< 600^\circ\text{C} \pm 10^\circ\text{C}$  oxidation rate  $< 0.01 \text{ mg/cm}^2 \cdot \text{h} \pm 0.002 \text{ mg/cm}^2 \cdot \text{h}$ ), limited reactivity (need to control the formation of  $\eta$  phase and  $\text{W}_2\text{C}$ ), strong WC bond (bond energy  $\sim 8.6 \text{ eV} \pm 0.1 \text{ eV}$ ), low surface activity (surface energy  $1.5\text{-}2.0 \text{ J/m}^2 \pm 0.2 \text{ J/m}^2$ ) and low volatility and solubility. These characteristics make it perform well in chemical equipment, wear-resistant coatings, aviation tools and mining drills, and the optimization of chemical properties provides a guarantee for the high-performance application of cemented carbide.

### Production and preparation of tungsten carbide powder - carburization preparation process ( $1450\text{-}1600^\circ\text{C}$ )

#### Process principle

The carburization method is the most commonly used preparation method in industry, which is to generate WC by high temperature reaction of tungsten powder (W) and carbon black (C). The reaction formula is as follows:  $\text{W} + \text{C} \rightarrow \text{WC}$

The reaction is carried out at a high temperature of  $1450\text{-}1600^\circ\text{C} \pm 10^\circ\text{C}$ , usually in hydrogen ( $\text{H}_2$ ) or vacuum atmosphere. Thermodynamically, the reaction Gibbs free energy is negative ( $\Delta G < -38$

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kJ/mol $\pm$ 2 kJ/mol), ensuring the spontaneous reaction; kinetically, carbon atoms enter the tungsten lattice through solid-state diffusion (diffusion coefficient  $\sim 10^{-10}$  cm<sup>2</sup>/s $\pm$  $10^{-11}$  cm<sup>2</sup>/s), gradually forming WC grains.

#### Process parameters:

raw material:

Tungsten powder: purity $>99.9\%\pm 0.01\%$ , particle size  $0.5-5\ \mu\text{m}\pm 0.01\ \mu\text{m}$ .

Carbon black: purity $>99.5\%\pm 0.01\%$ , particle size $<0.1\ \mu\text{m}\pm 0.01\ \mu\text{m}$ .

#### Reaction conditions:

Temperature: 1450-1600°C $\pm 10^\circ\text{C}$ .

Atmosphere: hydrogen (H<sub>2</sub>, purity  $>99.99\%\pm 0.01\%$ , flow rate 50 L/min $\pm 5$  L/min) or vacuum ( $<10^{-2}\text{Pa}\pm 10^{-3}\text{Pa}$ ).

Heating rate: 5-10°C/min $\pm 0.5^\circ\text{C}/\text{min}$ .

Holding time: 2-4 hours  $\pm 0.1$  hours.

#### equipment:

Graphite furnace: power  $>100\text{ kW}\pm 10\text{ kW}$ , temperature resistance  $>1800^\circ\text{C}\pm 10^\circ\text{C}$ .

Fixed or rotating furnace: Rotating furnace speed 5 rpm  $\pm 0.5$  rpm to improve mixing uniformity.

#### Process Optimization:

##### Temperature selection:

1450°C $\pm 10^\circ\text{C}$ : Produces submicron WC ( $<0.5\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ), with hardness increased by  $5\%\pm 1\%$  (HV  $>2900\pm 50$ ), suitable for high-precision tools.

1600°C $\pm 10^\circ\text{C}$ : Produces coarse WC ( $5-10\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ), increases yield by  $10\%\pm 2\%$  ( $>12$  t/batch $\pm 1$  t), and is suitable for high-toughness drill bits.

#### Equipment improvements:

The rotary furnace improves particle size uniformity (deviation  $<5\%\pm 1\%$ ) through dynamic mixing, and increases yield by  $5\%\pm 1\%$ , which is better than the fixed furnace (agglomeration rate  $>10\%\pm 2\%$ ).

#### Atmosphere Control:

A hydrogen atmosphere (O<sub>2</sub>  $< 10\text{ ppm}\pm 1\text{ ppm}$ ) inhibits oxidation (oxides  $<0.03\%\pm 0.01\%$ ) and increases purity by  $1\%\pm 0.2\%$ .

The vacuum atmosphere reduces carbon volatilization (loss  $<0.1\%\pm 0.01\%$ ) and increases the yield by  $2\%\pm 0.5\%$ .

#### Analysis of influencing factors:

##### Raw material ratio:

The W:C molar ratio is controlled at  $1:1.02\pm 0.01$ . Excessive carbon ( $>1.05$ ) leads to free carbon ( $>0.2\%\pm 0.01\%$ ), which reduces the hardness by  $3\%\pm 0.5\%$  (HV  $<2700\pm 50$ ); insufficient carbon ( $<0.98$ ) generates W<sub>2</sub>C (hardness HV  $<2000\pm 50$ ), and the flexural strength decreases by  $5\%\pm 1\%$ .

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(<3800 MPa±100 MPa).

**Reaction temperature:**

1450-1600°C±10°C is the optimal range. Above 1650°C±10°C, grain growth (>10 μm±0.01 μm) is induced, and hardness decreases by 5%±1%; below 1400°C±10°C, the reaction is incomplete (yield <95%±1%), and purity decreases by 2%±0.5%.

**Atmosphere Control:**

Hydrogen atmosphere (O<sub>2</sub> < 10 ppm±1 ppm) effectively inhibits oxidation; vacuum atmosphere reduces carbon volatilization.

Raw material particle size:

Tungsten powder <1 μm±0.01 μm produces submicron WC (<0.5 μm±0.01 μm) with a hardness of HV 3000±50; >5 μm±0.01 μm produces coarse WC (5-10 μm±0.01 μm) with a toughness increase of 10%±2% (K<sub>IC</sub> >18 MPa·m<sup>1/2</sup> ± 0.5).

**Equipment performance:**

The rotating furnace (speed 5 rpm ± 0.5 rpm) reduced agglomeration (<5% ± 1%), which was better than the fixed furnace (agglomeration > 10% ± 2%, purity reduction 1% ± 0.2%).

**Engineering application examples of tungsten carbide powder:**

**Aviation tools**

Using 1450°C±10°C, H<sub>2</sub> atmosphere, rotary furnace process, 0.5 μm±0.01 μm WC powder is generated, with hardness HV 2950±50 and free carbon 0.08%±0.01%. It is used for Ti-6Al-4V alloy cutting (1000°C, speed>200 m/min±10 m/min), with wear loss of only 0.1 mm±0.02 mm and service life of 12 hours±1 hour.

**Mining drill bits**

Using 1600°C±10°C and vacuum atmosphere process, 5-10 μm±0.01 μm WC powder is generated, with toughness K<sub>IC</sub> > 20 MPa·m<sup>1/2</sup> ± 0.5, and hard rock drilling (compression resistance>200 MPa±10 MPa) life>1000 m±100 m.

**Wear-resistant mold**

1-3 μm±0.01 μm WC powder, balanced hardness and toughness, cold heading die (>10<sup>5</sup> times±10<sup>4</sup> times) deformation <0.01 mm±0.002 mm.

**4.1.2 Other preparation methods**

**Plasma method:**

Principle: Use plasma high temperature (>5000°C±100°C) to react tungsten powder with methane (CH<sub>4</sub>) to generate WC.

Process parameters:

Plasma power: 50-100 kW ± 10 kW.

Atmosphere: Argon (Ar, purity>99.99%±0.01%).

Cooling rate: >100°C/s±10°C/s.

Advantages: Generates ultrafine WC (0.1-0.3 μm ± 0.01 μm) with high purity (free carbon < 0.05% ± 0.01%), suitable for high-end applications.

Application: Superhard tools (such as PCB drill bits, hole diameter <0.1 mm±0.01 mm).

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#### **Mechanochemical method:**

Principle: Tungsten powder and carbon black are mechanically reacted to generate WC through high-energy ball milling (rotation speed  $300\text{--}500\text{ rpm} \pm 10\text{ rpm}$ ).

Process parameters:

Ball milling time:  $20\text{--}50\text{ hours} \pm 1\text{ hour}$ .

Ball to material ratio:  $10:1$  to  $20:1 \pm 0.1$ .

Advantages: Can generate nano-scale WC ( $<0.1\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ), hardness  $\text{HV} > 3100 \pm 50$ .

Application: Nano coating (such as aviation blade spraying, bonding strength  $> 80\text{ MPa} \pm 5\text{ MPa}$ ).

#### **Chemical Vapor Deposition (CVD):**

Principle: WC is deposited by reaction of  $\text{WF}_6$  and  $\text{CH}_4$  at  $800\text{--}1000^\circ\text{C} \pm 10^\circ\text{C}$ .

Process parameters:

Deposition rate:  $0.1\text{--}0.5\text{ }\mu\text{m}/\text{min} \pm 0.01\text{ }\mu\text{m}/\text{min}$ .

Atmosphere:  $\text{H}_2$  / Ar mixed gas.

Advantages: High purity (free carbon  $<0.03\% \pm 0.01\%$ ), suitable for coating.

Application: Wear-resistant coating (aviation turbine blades, life span  $> 5000\text{ hours} \pm 500\text{ hours}$ ).

#### **4.1.3 Particle size of tungsten carbide powder**

Tungsten carbide powder (WC) is the core material of cemented carbide, and its particle size characteristics directly affect the performance and application effect of cemented carbide. The following is a comprehensive analysis of the meaning, range, distribution, performance impact, quality control technology and detection methods of particle size, based on scientific data and industrial standards (such as GB/T 19077.1-2008). The current date and time is May 22, 2025 14:20 HKT.

##### **(1) The significance of tungsten carbide powder particle size**

Performance determination: The particle size of WC powder is a key parameter affecting the performance of cemented carbide, directly determining hardness, wear resistance, toughness and sintering behavior.

Fine particle size ( $<0.5\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ) enhances hardness ( $\text{HV} > 3000 \pm 50$ ) and wear resistance, suitable for high-precision machining.

Coarse particle size ( $5\text{--}10\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ) improves toughness ( $K_{1c} > 20\text{ MPa}\cdot\text{m}^{1/2} \pm 0.5$ ), suitable for high impact scenarios.

Processing adaptability: The particle size affects the fluidity ( $<30\text{ s}/50\text{ g} \pm 2\text{ s}$ , GB/T 1482-2010) and pressing performance of the powder. Fine-grained WC is suitable for additive manufacturing (surface roughness  $R_a < 5\text{ }\mu\text{m} \pm 1\text{ }\mu\text{m}$ ), and coarse-grained WC is suitable for traditional sintering.

Sintering performance: Uniform particle size distribution (deviation  $<5\% \pm 1\%$ ) ensures sintering density ( $>99\% \pm 0.1\%$ ), reduces porosity ( $<0.05\% \pm 0.01\%$ ), and improves overall strength.

Application matching: Different applications require matching specific particle sizes, such as aviation tools ( $0.1\text{--}0.5\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ) and mining drill bits ( $5\text{--}10\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ).

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## (2) Particle size range of tungsten carbide powder

Overall range: WC powder particle size is usually between  $0.1-10\ \mu\text{m} \pm 0.01\ \mu\text{m}$ .

Classification:

Nanoscale ( $<0.1\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ): extremely high hardness ( $\text{HV} > 3100 \pm 50$ ), used for ultra-precision machining.

Submicron grade ( $0.1-0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ): hardness  $\text{HV} 3000 \pm 50$ , suitable for high-precision tools.

Micron grade ( $1-5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ): hardness  $\text{HV} 2500-2800 \pm 50$ , toughness  $K_{1c} 15-20\ \text{MPa} \cdot \text{m}^{1/2} \pm 0.5$ , suitable for mold manufacturing.

Coarse grade ( $5-10\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ): toughness  $K_{1c} > 20\ \text{MPa} \cdot \text{m}^{1/2} \pm 0.5$ , suitable for mining drill bits.

Preparation influence: The particle size range is determined by the preparation process, such as carbonization ( $1450-1600^\circ\text{C} \pm 10^\circ\text{C}$ ) produces  $0.5-10\ \mu\text{m} \pm 0.01\ \mu\text{m}$ , and mechanochemical method produces  $<0.1\ \mu\text{m} \pm 0.01\ \mu\text{m}$ .

## (3) Particle size distribution and quality of tungsten carbide powder

Distribution characteristics:

Laser particle size analysis (GB/T 19077.1-2008) was used to measure D10, D50 and D90.

Submicron level:  $\text{D50}=0.3\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ,  $\text{D10}=0.1\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ,  $\text{D90}=0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ .

Micron level:  $\text{D50}=3\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ,  $\text{D10}=1\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ,  $\text{D90}=5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ .

Coarse grade:  $\text{D50}=8\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ,  $\text{D10}=5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ,  $\text{D90}=10\ \mu\text{m} \pm 0.01\ \mu\text{m}$ .

Pros and cons evaluation:

Advantages: narrow distribution (deviation  $<5\% \pm 1\%$ ), uniform particles, high density after sintering ( $>99\% \pm 0.1\%$ ), and good performance consistency.

Disadvantages: wide distribution (deviation  $> 10\% \pm 2\%$ ), uneven particles, increased porosity after sintering ( $> 0.1\% \pm 0.02\%$ ), large performance fluctuations (such as hardness deviation  $> 100\ \text{HV}$ ).

Influencing factors: raw material particle size (tungsten powder  $<0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ), reaction time ( $2-4\ \text{hours} \pm 0.1\ \text{hours}$ ), cooling rate ( $>50^\circ\text{C}/\text{min} \pm 5^\circ\text{C}/\text{min}$ ).

## (4) Effect of tungsten carbide powder size on cemented carbide performance

Hardness: The smaller the particle size, the higher the hardness. Submicron WC ( $<0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ) has a hardness of  $\text{HV} > 3000 \pm 50$ , which is suitable for superhard tools (cutting speed  $> 300\ \text{m}/\text{min} \pm 10\ \text{m}/\text{min}$ ).

Toughness: The larger the particle size, the better the toughness. Coarse WC ( $5-10\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ) has a toughness of  $K_{1c} > 20\ \text{MPa} \cdot \text{m}^{1/2} \pm 0.5$ , suitable for mining drill bits (compression resistance  $> 200\ \text{MPa} \pm 10\ \text{MPa}$ ).

Wear resistance: Fine-grained WC improves wear resistance, aviation tool ( $0.3\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ) wear  $< 0.08\ \text{mm} \pm 0.02\ \text{mm}$ , life  $> 15\ \text{hours} \pm 1\ \text{hour}$ .

Sintering performance: uniform distribution (deviation  $<5\% \pm 1\%$ ), reduced porosity ( $<0.05\% \pm 0.01\%$ ), and improved flexural strength ( $> 3800\ \text{MPa} \pm 100\ \text{MPa}$ ).

Processing performance: Fine-grained WC improves powder spreading uniformity (fluidity  $< 30\ \text{s}/50\ \text{g} \pm 2\ \text{s}$ ) and is suitable for 3D printing (tensile strength  $> 800\ \text{MPa} \pm 50\ \text{MPa}$ ).

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Example:  $0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$  WC powder (distribution deviation  $<3\% \pm 0.5\%$ ) is used for PCB drill bits, with a lifespan of  $>10^5$  holes  $\pm 10^4$  holes;  $8\ \mu\text{m} \pm 0.01\ \mu\text{m}$  WC powder is used for PDC drill bits, with a drilling speed of  $>5\ \text{m/h} \pm 0.5\ \text{m/h}$ .

#### (5) Quality control technology of tungsten carbide powder particle size

Raw material control: Tungsten powder particle size  $<0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$  generates  $0.1\text{-}0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$  WC, and Ostwald ripening (growth rate  $\sim 10^{-9}\ \text{m/s} \pm 10^{-10}\ \text{m/s}$ ) is inhibited.

Reaction time: 2 hours  $\pm 0.1$  hours ( $1450^\circ\text{C} \pm 10^\circ\text{C}$ ) to generate fine WC ( $<0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ); 4 hours  $\pm 0.1$  hours ( $1600^\circ\text{C} \pm 10^\circ\text{C}$ ) to generate coarse WC ( $5\text{-}10\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ).

Cooling rate: Rapid cooling ( $>50^\circ\text{C/min} \pm 5^\circ\text{C/min}$ ) inhibits grain growth ( $<0.01\ \mu\text{m/min} \pm 0.001\ \mu\text{m/min}$ ), with a deviation of  $<3\% \pm 0.5\%$ .

Additives: Vanadium carbide (VC,  $0.1\%\text{-}0.5\% \pm 0.01\%$ ) reduces surface energy ( $<1\ \text{J/m}^2 \pm 0.1\ \text{J/m}^2$ ) and reduces particle size by  $10\% \pm 2\%$ ; Chromium carbide ( $\text{Cr}_3\text{C}_2$ ,  $0.5\% \pm 0.01\%$ ) inhibits diffusion (coefficient  $<10^{-11}\ \text{cm}^2/\text{s} \pm 10^{-12}\ \text{cm}^2/\text{s}$ ), with a deviation of  $<2\% \pm 0.5\%$ .

Equipment optimization: Rotary furnace (speed  $5\ \text{rpm} \pm 0.5\ \text{rpm}$ ) dynamic mixing, improved particle size uniformity (deviation  $<5\% \pm 1\%$ ).

Post-treatment: Screening (pore size  $<10\ \mu\text{m} \pm 0.1\ \mu\text{m}$ ) to remove agglomerates ( $<5\% \pm 1\%$ ); air flow classification (GB/T 19077.1-2008) to adjust the distribution, with a deviation of  $<2\% \pm 0.5\%$ .

#### (6) Detection method of tungsten carbide powder particle size

Laser particle size analysis: According to GB/T 19077.1-2008, laser diffractometer is used to measure D10, D50 and D90, with a deviation of  $<5\% \pm 1\%$ .

Scanning electron microscopy (SEM): According to GB/T 16594-2008, observe the particle morphology (polygonal, edge  $<0.05\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ) and agglomeration ( $<5\% \pm 1\%$ ).

Sedimentation method: According to GB/T 14634.2-2010, the sedimentation velocity is measured and the particle size distribution is estimated. It is suitable for coarse particles ( $>5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ).

Specific surface area method: According to GB/T 19587-2017 (BET method), the average particle size is calculated. The submicron specific surface area is  $>3\ \text{m}^2/\text{g} \pm 0.2\ \text{m}^2/\text{g}$ .

Online monitoring: A laser particle size online analyzer is used during the production process to control the distribution in real time (deviation  $<3\% \pm 0.5\%$ ).

Comprehensive impact and application association

Performance optimization: Fine-grained WC ( $0.1\text{-}0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ) improves hardness and wear resistance, suitable for aviation tools (lifespan  $>15\ \text{hours} \pm 1\ \text{hour}$ ); coarse-grained WC ( $5\text{-}10\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ) enhances toughness, suitable for mining drill bits (lifespan  $>1000\ \text{m} \pm 100\ \text{m}$ ).

Quality control efficiency: Rotating furnace and additives (VC,  $\text{Cr}_3\text{C}_2$ ) ensure uniform distribution (deviation  $<5\% \pm 1\%$ ) and improve sintering consistency (density  $>99\% \pm 0.1\%$ ).

Detection reliability: Combining laser particle size analysis and SEM provides accurate distribution and morphology data to optimize process parameters.

The particle size characteristics of tungsten carbide powder are crucial to the performance of cemented carbide. The particle size range ( $0.1\text{-}10\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ) covers nanometer to coarse grades, and the uniform distribution (deviation  $<5\% \pm 1\%$ ) ensures performance consistency. Fine particles increase hardness, coarse particles enhance toughness, and quality control technology (raw material

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control, additives, rotary furnace) and detection methods (laser particle size analysis, SEM) ensure accurate particle size. The particle size optimization of WC powder provides high-performance support for aviation tools, mining drills, wear-resistant molds and other fields.

#### 4.1.4 Purity of tungsten carbide powder

The purity of tungsten carbide powder (WC) is the core indicator of its quality control, which directly affects the performance, processability and service life of cemented carbide. The following is a comprehensive analysis of the definition of purity, main impurities, influencing factors, optimization strategies, detection methods and application effects, based on scientific data and industrial standards (such as GB/T 5124-2017). The current date and time is May 22, 2025 14:23 HKT.

##### (1) Definition and index of tungsten carbide powder purity

Definition: The purity of WC powder refers to the content of its main component WC, and the total impurity content (free carbon, oxides, metal impurities, etc.) is usually required to be as low as possible.

Key Metrics:

Free carbon:  $<0.1\% \pm 0.01\%$ . Too high will reduce the hardness ( $HV < 2800 \pm 50$ ) and sintering density ( $<99\% \pm 0.1\%$ ).

Oxide (such as  $WO_3$ ):  $<0.05\% \pm 0.01\%$ . Too high will lead to increased porosity ( $>0.1\% \pm 0.02\%$ ).

Metal impurities (such as Fe, Cr):  $<0.02\% \pm 0.005\%$ , to avoid affecting the performance of cemented carbide.

WC phase purity:  $>99.8\% \pm 0.02\%$ , ensuring no  $W_2C$  or other secondary phases (low hardness,  $HV < 2000 \pm 50$ ).

##### (2) Main impurities in tungsten carbide powder and their sources

###### Free Carbon:

(W:C molar ratio  $>1.05 \pm 0.01$ ) or incomplete reaction (temperature  $<1400^\circ\text{C} \pm 10^\circ\text{C}$ ) during carbonization preparation.

Impact: When free carbon  $>0.2\% \pm 0.01\%$ , the hardness decreases by  $3\% \pm 0.5\%$  ( $HV < 2700 \pm 50$ ), and the porosity increases after sintering ( $>0.2\% \pm 0.02\%$ ).

###### Oxide:

Source: Oxidation of raw materials (tungsten powder contains  $O >0.1\% \pm 0.01\%$ ) or high oxygen content in the atmosphere during the preparation process ( $O_2 >10 \text{ ppm} \pm 1 \text{ ppm}$ ).

Impact: When the oxide content is  $>0.05\% \pm 0.01\%$ , the porosity increases after sintering ( $>0.1\% \pm 0.02\%$ ) and the strength decreases by  $5\% \pm 1\%$  (flexural strength  $<3800 \text{ MPa} \pm 100 \text{ MPa}$ ).

###### Metal impurities:

Source: Equipment wear (such as Fe introduced from the ball mill,  $>0.1\% \pm 0.01\%$ ) or impure raw materials (Cr, Mo, etc.).

Impact: When Fe  $>0.02\% \pm 0.005\%$ , the hardness decreases by  $2\% \pm 0.5\%$  and the corrosion resistance decreases (corrosion rate  $>0.002 \text{ mm/year} \pm 0.0005 \text{ mm/year}$ ).

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### (3) Effect of tungsten carbide powder purity on cemented carbide performance

Hardness and wear resistance: High purity (free carbon  $<0.1\% \pm 0.01\%$ ) ensures hardness HV  $>2900 \pm 50$ , aviation tool wear  $<0.08 \text{ mm} \pm 0.02 \text{ mm}$ , life  $>15 \text{ hours} \pm 1 \text{ hour}$ .

Toughness: oxide  $<0.03\% \pm 0.01\%$  Reduced porosity, toughness  $K_{IC} > 18 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ , mining drill bit life  $>1200 \text{ m} \pm 100 \text{ m}$ .

Sintering performance: high purity (WC phase  $>99.8\% \pm 0.02\%$ ), improved density ( $>99\% \pm 0.1\%$ ), compressive strength  $>4000 \text{ MPa} \pm 100 \text{ MPa}$ .

Corrosion resistance: Metal impurities  $<0.02\% \pm 0.005\%$  ensures corrosion resistance (pH 2-12, corrosion rate  $<0.001 \text{ mm/year} \pm 0.0002 \text{ mm/year}$ ), suitable for chemical equipment.

powder with free carbon content of  $0.07\% \pm 0.01\%$  and oxide content of  $<0.02\% \pm 0.005\%$  is used for wear-resistant molds, and its service life is  $>10^6 \text{ times} \pm 10^5 \text{ times}$ , which is better than WC with free carbon content of  $0.2\% \pm 0.01\%$  (service life  $<8 \times 10^5 \text{ times} \pm 10^5 \text{ times}$ ).

### (4) Tungsten carbide powder purity optimization strategy

#### Raw material control:

Tungsten powder purity  $>99.9\% \pm 0.01\%$ , containing O  $<0.05\% \pm 0.01\%$ ; carbon black purity  $>99.5\% \pm 0.01\%$ , containing O  $<0.03\% \pm 0.01\%$ .

Effect: Reduce initial oxides and finished oxides by  $0.02\% \pm 0.005\%$ .

#### Carbon content control:

The W:C molar ratio is  $1:1.01 \pm 0.01$ , ensuring complete reaction, reducing free carbon to  $0.08\% \pm 0.01\%$ , and increasing hardness by  $2\% \pm 0.5\%$ .

Excess carbon ( $>1.05$ ) produces graphite ( $>0.3\% \pm 0.01\%$ ), while insufficient carbon ( $<0.98$ ) produces  $\text{W}_2\text{C}$ .

#### Atmosphere Control:

Hydrogen atmosphere ( $\text{H}_2$ ,  $\text{O}_2 < 10 \text{ ppm} \pm 1 \text{ ppm}$ ): reduction of  $\text{WO}_3$  (reduction rate  $>99\% \pm 1\%$ ), oxide  $<0.03\% \pm 0.01\%$ .

Vacuum atmosphere ( $<10^{-2} \text{ Pa} \pm 10^{-3} \text{ Pa}$ ): inhibits carbon volatilization and increases purity by  $1\% \pm 0.2\%$ .

#### Post-processing:

Sieving (pore size  $<10 \mu\text{m} \pm 0.1 \mu\text{m}$ ): remove agglomerates ( $<5\% \pm 1\%$ ) and increase purity by  $0.5\% \pm 0.1\%$ .

Pickling (HCl, pH  $2 \pm 0.1$ ): remove Fe ( $<0.01\% \pm 0.002\%$ ), increase hardness by  $1\% \pm 0.2\%$ .

Equipment improvements:

Use high-purity graphite furnace (containing C  $<0.01\% \pm 0.002\%$ ) to reduce carbon pollution.

The rotary furnace (speed  $5 \text{ rpm} \pm 0.5 \text{ rpm}$ ) improved the reaction uniformity and reduced the free carbon by  $0.02\% \pm 0.005\%$ .

### (5) Detection method

Free carbon detection: According to GB/T 5124-2017 chemical analysis method, the  $\text{CO}_2$  content is measured after high-temperature combustion with an accuracy of  $\pm 0.005\%$ .

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3 content is determined by acid dissolution and titration , with an accuracy of  $\pm 0.002\%$ .

Metal impurity detection: ICP-MS (GB/T 13748.20-2009), detection of Fe, Cr, etc., detection limit  $< 0.001\%$ .

Phase purity analysis: X-ray diffraction (XRD, GB/T 27708-2011) to confirm the purity of WC phase and exclude  $W_2C$  or graphite phase.

Online monitoring: Infrared gas analyzers are used during the production process to monitor  $CO_2$  emissions in real time and control free carbon ( $< 0.1\% \pm 0.01\%$ ).

Comprehensive impact and application association

Improved performance: High purity (free carbon  $< 0.1\% \pm 0.01\%$ , oxide  $< 0.05\% \pm 0.01\%$ ) ensures high hardness ( $HV > 2900 \pm 50$ ) and toughness ( $K_{IC} > 18 MPa \cdot m^{1/2} \pm 0.5$ ) of cemented carbide.

Process optimization: Atmosphere control and post-treatment (such as pickling) significantly reduce impurities, suitable for high-end applications (aerospace tools, mining drill bits).

Detection reliability: Combining chemical analysis and XRD to ensure accurate purity data and optimize production processes.

Summarize

The purity of tungsten carbide powder is based on free carbon  $< 0.1\% \pm 0.01\%$ , oxide  $< 0.05\% \pm 0.01\%$  and metal impurities  $< 0.02\% \pm 0.005\%$  as key indicators, which directly affect the hardness, toughness, sintering performance and corrosion resistance of cemented carbide. The purity can be effectively improved through raw material control, precise carbon content ratio, atmosphere optimization and post-processing technology, and the detection method (chemical analysis, XRD) ensures stable quality. High-purity WC powder provides excellent performance support for aviation tools, wear-resistant molds and other fields.

#### 4.1.5 Carbon content of tungsten carbide powder

The carbon content of tungsten carbide powder is a key parameter in its preparation and performance optimization, which directly affects its chemical stability, hardness and sintering behavior.

##### (1) Definition and index of carbon content in tungsten carbide powder

Definition: Carbon content refers to the total carbon content in WC powder, including bound carbon (carbon that forms WC) and free carbon (unreacted carbon). The theoretical carbon content of ideal WC is  $6.13\% \pm 0.01\%$  (molar ratio W:C = 1:1).

##### Key indicators of carbon content in tungsten carbide powder:

Total carbon content :  $6.0\% - 6.2\% \pm 0.01\%$ , including bound carbon and free carbon.

Free carbon:  $< 0.1\% \pm 0.01\%$ . Too high will reduce hardness and density.

Bound carbon:  $5.9\% - 6.1\% \pm 0.01\%$ , reflecting the degree of formation of WC phase.

##### (2) Range of carbon content in tungsten carbide powder

The carbon mass fraction corresponding to the WC molecular formula is  $6.13\% \pm 0.01\%$ , that is, 1 mol W (183.84 g) is combined with 1 mol C (12.01 g).

Actual range:

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Low carbon state ( $5.9\%-6.0\%\pm 0.01\%$ ):  $W_2C$  ( carbon content  $3.16\%\pm 0.01\%$ ) or W phase may be generated, and the hardness decreases ( $HV < 2000\pm 50$ ).

Normal range ( $6.0\%-6.2\%\pm 0.01\%$ ): close to the theoretical value, suitable for high-performance cemented carbide.

High carbon state ( $>6.2\%\pm 0.01\%$ ): free carbon increases ( $>0.2\%\pm 0.01\%$ ), hardness decreases ( $HV < 2700\pm 50$ ), and porosity increases ( $>0.2\%\pm 0.02\%$ ).

Preparation influence: Carbonization method ( $1450-1600^\circ\text{C}\pm 10^\circ\text{C}$ ) controls the W:C molar ratio at  $1:1.01\pm 0.01$  to ensure stable carbon content.

### (3) Effect of carbon content of tungsten carbide powder on the performance of cemented carbide

Hardness and wear resistance: Carbon content  $6.0\%-6.2\%\pm 0.01\%$  ensures hardness  $HV > 2900\pm 50$ , aviation tool wear  $< 0.08\text{ mm}\pm 0.02\text{ mm}$ , life  $> 15\text{ hours}\pm 1\text{ hour}$ .

Toughness: Low carbon ( $<5.9\%\pm 0.01\%$ ) generates  $W_2C$  , and the toughness decreases ( $K_{IC} < 10\text{ MPa}\cdot\text{m}^{1/2}\pm 0.5$  ); high carbon ( $>6.2\%\pm 0.01\%$ ) free carbon reduces the consistency of toughness.

Sintering properties: Carbon content  $6.1\%\pm 0.01\%$  optimizes sintering density ( $>99\%\pm 0.1\%$ ), compressive strength  $>4000\text{ MPa}\pm 100\text{ MPa}$ ; free carbon  $>0.1\%\pm 0.01\%$  leads to increased porosity ( $>0.1\%\pm 0.02\%$ ).

Corrosion resistance: Free carbon  $<0.1\%\pm 0.01\%$  Maintains corrosion resistance (pH 2-12, corrosion rate  $< 0.001\text{ mm/year}\pm 0.0002\text{ mm/year}$ ).

powder with a carbon content of  $6.08\%\pm 0.01\%$  (free carbon  $0.07\%\pm 0.01\%$ ) is used for wear-resistant molds, and its life is  $> 10^6\text{ times}\pm 10^5\text{ times}$ , which is better than WC with a carbon content of  $6.25\%\pm 0.01\%$  (free carbon  $0.2\%\pm 0.01\%$ ) (life  $< 8\times 10^5\text{ times}\pm 10^5\text{ times}$ ).

### (4) Optimization strategy of carbon content in tungsten carbide powder

#### Raw material control:

The purity of carbon black is  $>99.5\%\pm 0.01\%$ , and the particle size is  $< 0.1\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$  , ensuring uniform distribution of carbon.

Tungsten powder contains O  $< 0.05\%\pm 0.01\%$ , which reduces the oxidation reaction and interferes with the carbon content.

Carbon content ratio:

The W:C molar ratio is  $1:1.01\pm 0.01$ , with slightly excess carbon to compensate for volatilization, the bound carbon reaches  $6.1\%\pm 0.01\%$ , and the free carbon is  $< 0.1\%\pm 0.01\%$ .

Excess carbon ( $>1.05$ ) produces graphite ( $>0.3\%\pm 0.01\%$ ); insufficient carbon ( $<0.98$ ) produces  $W_2C$  .

#### Reaction conditions:

Temperature:  $1450-1600^\circ\text{C}\pm 10^\circ\text{C}$ , reaction is complete, carbon binding rate  $>98\%\pm 1\%$ .

Atmosphere: Hydrogen ( $H_2$  ,  $O_2 < 10\text{ ppm}\pm 1\text{ ppm}$ ) or vacuum ( $< 10^{-2}\text{ Pa}\pm 10^{-3}\text{ Pa}$  ) , reduce carbon volatilization (loss  $< 0.1\%\pm 0.01\%$  ).

Insulation time:  $2-4\text{ hours}\pm 0.1\text{ hour}$ , to ensure sufficient carbonization reaction.

#### Post-processing:

Heat treatment ( $800^\circ\text{C}\pm 10^\circ\text{C}$ ,  $H_2$  atmosphere ): removal of free carbon (reduction of  $0.05\%\pm 0.01\%$ ).

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Acid washing (HCl, pH 2±0.1): Remove impurities caused by incomplete carbonization and increase purity by 0.5%±0.1%.

**Equipment Optimization:**

The rotary furnace (speed 5 rpm ± 0.5 rpm) improves mixing uniformity, and the carbon distribution deviation is < 2% ± 0.5%.

High-purity graphite furnace (containing C < 0.01%±0.002%) reduces exogenous carbon pollution.

**(5) Detection method of carbon content in tungsten carbide powder**

Total carbon content : According to GB/T 5124-2017, high temperature combustion infrared absorption method, measure CO<sub>2</sub> content, accuracy ±0.005%.

Bound carbon: After high temperature combustion, the free carbon is separated by acid dissolution , and the remaining carbon is bound carbon with an accuracy of ±0.002%.

Free carbon: calculated by difference ( total carbon - bound carbon), or direct combustion method, accuracy ±0.005%.

Phase analysis: X-ray diffraction (XRD, GB/T 27708-2011) to confirm the purity of WC phase and detect W<sub>2</sub>C or graphite phase.

Online monitoring: Infrared gas analyzer monitors CO<sub>2</sub> emissions in real time and controls free carbon (<0.1%±0.01%).

**Comprehensive influence of carbon content in tungsten carbide powder and its application correlation**

content of 6.0%-6.2%±0.01% (free carbon <0.1%±0.01%) ensures high hardness (HV >2900±50) and toughness (K<sub>1c</sub> >18 MPa·m<sup>1/2</sup> ± 0.5 ) of cemented carbide .

Process efficiency: Precise ratio and atmosphere control reduce carbon deviation, suitable for aviation tools (lifetime >15 hours ±1 hour) and wear-resistant molds (>10<sup>6</sup> times ±10<sup>5</sup> times).

Detection reliability: The combination of chemical analysis and XRD provides accurate carbon content data and optimizes the production process.

The carbon content of tungsten carbide powder is in the ideal range of 6.0%-6.2%±0.01%, with 5.9%-6.1%±0.01% of combined carbon and <0.1%±0.01% of free carbon, which is crucial for hardness, toughness and sintering performance. The carbon content can be effectively adjusted through raw material control, W:C ratio optimization, reaction condition adjustment and post-processing technology, and the detection method (infrared absorption method, XRD) ensures stable quality. WC powder with appropriate carbon content provides high-performance support for aviation tools, mining drill bits and other fields.

**4.1.6 Quality control and testing of particle size, purity, carbon content, etc. of tungsten carbide powder**

The particle size, purity and carbon content of tungsten carbide powder (WC) are key parameters for its quality control, which directly determine the performance and application reliability of cemented carbide. The following is a comprehensive analysis of quality control technology, detection methods and comprehensive impact, based on scientific data and industrial standards (such as GB/T 19077.1-2008, GB/T 5124-2017). The current date and time is May 22, 2025 14:32 HKT.

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## (1) Particle size quality control and testing of tungsten carbide powder

### Control technology:

Raw material control: Tungsten powder particle size  $<0.5 \mu\text{m} \pm 0.01 \mu\text{m}$  generates  $0.1-0.5 \mu\text{m} \pm 0.01 \mu\text{m}$  WC, and Ostwald ripening (growth rate  $\sim 10^{-9} \text{ m/s} \pm 10^{-10} \text{ m/s}$ ) is inhibited.

Reaction time: 2 hours  $\pm 0.1$  hours ( $1450^\circ\text{C} \pm 10^\circ\text{C}$ ) to generate fine WC ( $<0.5 \mu\text{m} \pm 0.01 \mu\text{m}$ ); 4 hours  $\pm 0.1$  hours ( $1600^\circ\text{C} \pm 10^\circ\text{C}$ ) to generate coarse WC ( $5-10 \mu\text{m} \pm 0.01 \mu\text{m}$ ).

Cooling rate: Rapid cooling ( $>50^\circ\text{C}/\text{min} \pm 5^\circ\text{C}/\text{min}$ ) inhibits grain growth ( $<0.01 \mu\text{m}/\text{min} \pm 0.001 \mu\text{m}/\text{min}$ ), with a deviation of  $<3\% \pm 0.5\%$ .

Additives: Vanadium carbide (VC,  $0.1\%-0.5\% \pm 0.01\%$ ) reduces surface energy ( $<1 \text{ J/m}^2 \pm 0.1 \text{ J/m}^2$ ) and reduces particle size by  $10\% \pm 2\%$ ; Chromium carbide ( $\text{Cr}_3\text{C}_2$ ,  $0.5\% \pm 0.01\%$ ) inhibits diffusion (coefficient  $<10^{-11} \text{ cm}^2/\text{s} \pm 10^{-12} \text{ cm}^2/\text{s}$ ), with a deviation of  $<2\% \pm 0.5\%$ .

Equipment optimization: Rotary furnace (speed  $5 \text{ rpm} \pm 0.5 \text{ rpm}$ ) improves mixing uniformity, and the particle size distribution deviation is  $<5\% \pm 1\%$ .

Post-treatment: Screening (pore size  $<10 \mu\text{m} \pm 0.1 \mu\text{m}$ ) to remove agglomerates ( $<5\% \pm 1\%$ ); air flow classification (GB/T 19077.1-2008) to adjust the distribution, with a deviation of  $<2\% \pm 0.5\%$ .

### Detection method:

Laser particle size analysis: According to GB/T 19077.1-2008, laser diffractometer is used to measure D10, D50, and D90, with a deviation of  $<5\% \pm 1\%$ , and submicron  $\text{D50} = 0.3 \mu\text{m} \pm 0.01 \mu\text{m}$ .

Scanning electron microscopy (SEM): According to GB/T 16594-2008, observe the morphology (polygonal, edge  $<0.05 \mu\text{m} \pm 0.01 \mu\text{m}$ ) and agglomeration ( $<5\% \pm 1\%$ ).

Sedimentation method: According to GB/T 14634.2-2010, it measures the sedimentation velocity and is suitable for coarse particles ( $>5 \mu\text{m} \pm 0.01 \mu\text{m}$ ).

Specific surface area method: According to GB/T 19587-2017 (BET method), the average particle size is calculated. The submicron specific surface area is  $>3 \text{ m}^2/\text{g} \pm 0.2 \text{ m}^2/\text{g}$ .

Online monitoring: The laser particle size online analyzer controls the distribution in real time, with a deviation of  $<3\% \pm 0.5\%$ .

## (2) Purity quality control and testing of tungsten carbide powder

### Control technology:

Raw material control: Tungsten powder purity  $>99.9\% \pm 0.01\%$ , containing O  $<0.05\% \pm 0.01\%$ ; carbon black purity  $>99.5\% \pm 0.01\%$ , containing O  $<0.03\% \pm 0.01\%$ .

Carbon content ratio: W:C molar ratio  $1:1.01 \pm 0.01$ , free carbon reduced to  $0.08\% \pm 0.01\%$ .

Atmosphere control: Hydrogen ( $\text{H}_2$ ,  $\text{O}_2 < 10 \text{ ppm} \pm 1 \text{ ppm}$ ) reduces  $\text{WO}_3$  (reduction rate  $>99\% \pm 1\%$ ), oxide  $<0.03\% \pm 0.01\%$ ; vacuum atmosphere ( $<10^{-2} \text{ Pa} \pm 10^{-3} \text{ Pa}$ ) suppresses carbon volatilization.

Post-treatment: Screening (pore size  $<10 \mu\text{m} \pm 0.1 \mu\text{m}$ ) to remove agglomerates and increase purity by  $0.5\% \pm 0.1\%$ ; acid washing (HCl, pH  $2 \pm 0.1$ ) to remove Fe ( $<0.01\% \pm 0.002\%$ ).

Equipment optimization: High-purity graphite furnace (containing C  $<0.01\% \pm 0.002\%$ ) reduces carbon pollution, and rotary furnace improves reaction uniformity.

### Detection method:

Free carbon detection: Based on GB/T 5124-2017, high temperature combustion infrared absorption method, accuracy  $\pm 0.005\%$ .

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Oxide detection: According to GB/T 5124-2017,  $\text{WO}_3$  is determined by acid dissolution titration with an accuracy of  $\pm 0.002\%$ .

Metal impurity detection: ICP-MS (GB/T 13748.20-2009), detection of Fe and Cr, detection limit  $< 0.001\%$ .

Phase purity analysis: X-ray diffraction (XRD, GB/T 27708-2011) confirmed that the WC phase purity was  $> 99.8\% \pm 0.02\%$ .

Online monitoring: Infrared gas analyzer monitors  $\text{CO}_2$  in real time and controls free carbon  $< 0.1\% \pm 0.01\%$ .

### (3) Quality control and testing of carbon content in tungsten carbide powder

#### Control technology:

Raw material control: Carbon black particle size  $< 0.1 \mu\text{m} \pm 0.01 \mu\text{m}$ , purity  $> 99.5\% \pm 0.01\%$ , ensuring carbon uniformity.

Carbon content ratio: W:C molar ratio  $1:1.01 \pm 0.01$ , bound carbon  $6.1\% \pm 0.01\%$ , free carbon  $< 0.1\% \pm 0.01\%$ .

Reaction conditions:  $1450-1600^\circ\text{C} \pm 10^\circ\text{C}$ , keep warm for  $2-4 \text{ hours} \pm 0.1 \text{ hour}$ , complete reaction; hydrogen or vacuum atmosphere to reduce carbon volatilization (loss  $< 0.1\% \pm 0.01\%$ ).

Post-treatment: heat treatment ( $800^\circ\text{C} \pm 10^\circ\text{C}$ ,  $\text{H}_2$  atmosphere) to remove free carbon (reduction of  $0.05\% \pm 0.01\%$ ); pickling to remove carbonized impurities.

Equipment optimization: Rotary furnace (speed  $5 \text{ rpm} \pm 0.5 \text{ rpm}$ ) improves carbon distribution uniformity, with a deviation of  $< 2\% \pm 0.5\%$ .

#### Detection method:

Total carbon content: Based on GB/T 5124-2017, high temperature combustion infrared absorption method, accuracy  $\pm 0.005\%$ .

Combined carbon: Acid dissolution and separation after high temperature combustion, accuracy  $\pm 0.002\%$ .

Free carbon: difference calculation or direct combustion method, accuracy  $\pm 0.005\%$ .

Phase analysis: XRD (GB/T 27708-2011) confirms WC phase and detects  $\text{W}_2\text{C}$  or graphite phase.

Online monitoring: Infrared gas analyzer monitors  $\text{CO}_2$  in real time and controls the carbon content at  $6.0\%-6.2\% \pm 0.01\%$ .

### (4) Comprehensive influence of tungsten carbide powder quality and its application

Performance optimization: Particle size  $0.1-0.5 \mu\text{m} \pm 0.01 \mu\text{m}$  improves hardness ( $\text{HV} > 3000 \pm 50$ ),  $5-10 \mu\text{m} \pm 0.01 \mu\text{m}$  enhances toughness ( $K_{IC} > 20 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ ); purity (free carbon  $< 0.1\% \pm 0.01\%$ , oxide  $< 0.05\% \pm 0.01\%$ ) ensures density ( $> 99\% \pm 0.1\%$ ); carbon content  $6.0\%-6.2\% \pm 0.01\%$  optimizes hardness and wear resistance.

Process efficiency: Rotary furnace, additives ( $\text{VC}$ ,  $\text{Cr}_3\text{C}_2$ ) and atmosphere control ( $\text{O}_2 < 10 \text{ ppm} \pm 1 \text{ ppm}$ ) improve quality consistency for aerospace tools (lifetime  $> 15 \text{ hours} \pm 1 \text{ hour}$ ) and mining drill bits ( $> 1000 \text{ m} \pm 100 \text{ m}$ ).

Detection reliability: Laser particle size analysis, chemical analysis and XRD are combined to provide accurate data and optimize production processes.

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#### (5) Challenges and improvements in quality control and testing of tungsten carbide powder

Challenges: Wide particle size distribution ( $>10\%\pm 2\%$ ) leads to performance fluctuations; the introduction of purity impurities (such as  $\text{Fe} > 0.02\%\pm 0.005\%$ ) affects corrosion resistance; carbon content deviation ( $>0.1\%\pm 0.01\%$ ) leads to phase impurity.

Improvement: AI was introduced to control process parameters (temperature, atmosphere), particle size deviation was  $<2\%\pm 0.5\%$ , purity was increased by  $0.5\%\pm 0.1\%$ , and carbon content was stabilized at  $6.1\%\pm 0.01\%$ .

The particle size ( $0.1\text{-}10\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ), purity (free carbon  $<0.1\% \pm 0.01\%$ , oxide  $<0.05\% \pm 0.01\%$ ) and carbon content ( $6.0\%\text{-}6.2\% \pm 0.01\%$ ) of tungsten carbide powder are managed through raw material control, reaction optimization, additives and equipment improvement. The detection methods (laser particle size analysis, chemical analysis, XRD) ensure accurate parameters, optimize the performance of cemented carbide, and support high-end applications such as aviation tools, wear-resistant molds and mining drills.

### 4.2 Binder phase and additives of cemented carbide

The binder phase ( $\text{Co, Ni, } 5\%\text{--}30\%\pm 1\%$ ) provides toughness ( $K_{IC} 820\ \text{MPa} \cdot \text{m}^{1/2} \pm 0.5$ ) and impact resistance (impact energy  $>10\text{J}\pm 1\text{J}$ ), and the grain inhibitor ( $\text{VC, Cr}_3\text{C}_2, <1\%\pm 0.01\%$ ) controls the WC grain growth ( $<0.01\ \mu\text{m}/\text{min} \pm 0.001\ \mu\text{m}/\text{min}$ ) and improves the hardness ( $\text{HV} > 2000 \pm 30$ ). The selection of binder phase and additives affects the performance: high - purity Co ( $>99.9\%\pm 0.01\%$ ) enhances toughness, Ni ( $>99.8\%\pm 0.01\%$ ) improves corrosion resistance (corrosion rate  $<0.01\ \text{mm}/\text{year} \pm 0.002\ \text{mm}/\text{year}$ ), and VC/  $\text{Cr}_3\text{C}_2$  optimizes the fine grain structure (WC grain size  $<0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ). This section analyzes the characteristics of Co/Ni powder and the mechanism of grain inhibitor.

#### 4.2.1 Characteristics and selection of cemented carbide binders - Co and Ni powders

The binder phase is an important component of cemented carbide, which plays the role of connecting WC particles and provides key properties such as toughness, processing performance and corrosion resistance. Cobalt (Co) and nickel (Ni) are the most commonly used binders for cemented carbide, and their characteristics and selection directly affect the performance of cemented carbide.

#### (1) Material properties and requirements

The binder phase plays a "bridge" role in cemented carbide, filling the gaps between WC particles, enhancing the toughness and processing properties of the material, while affecting corrosion resistance and high temperature stability. Co and Ni are the preferred binders due to their excellent physical and chemical properties.

#### Co powder characteristics:

Crystal structure: face-centered cubic (FCC) structure, lattice parameter  $a = 0.3544\ \text{nm} \pm 0.0001\ \text{nm}$ , with high symmetry, slip system  $>12$ , giving excellent plastic deformation ability.

Density:  $8.90\ \text{g}/\text{cm}^3 \pm 0.05\ \text{g}/\text{cm}^3$ , close to WC ( $15.63\ \text{g}/\text{cm}^3 \pm 0.05\ \text{g}/\text{cm}^3$ ), reducing sintering stress.

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Melting point:  $1495^{\circ}\text{C} \pm 5^{\circ}\text{C}$ , suitable for high temperature sintering ( $>1200^{\circ}\text{C} \pm 10^{\circ}\text{C}$ ).

Hardness: Vickers hardness  $\text{HV } 100 \pm 10$ , lower than WC, providing toughness buffer.

Toughness: Fracture toughness  $K_{\text{IC}} \text{ c } 15\text{-}20 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ , better than Ni ( $K_{\text{IC}} \text{ c } 12\text{-}15 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ ).

Thermal conductivity:  $80 \text{ W}/(\text{m} \cdot \text{K}) \pm 5 \text{ W}/(\text{m} \cdot \text{K})$ , helps dissipate heat and extend tool life.

Thermal expansion coefficient:  $5.0 \times 10^{-6} \text{ K}^{-1} \pm 0.2 \times 10^{-6} \text{ K}^{-1}$ , matching with WC ( $5.2 \times 10^{-6} \text{ K}^{-1} \pm 0.2 \times 10^{-6} \text{ K}^{-1}$ ), reducing thermal stress ( $<50 \text{ MPa} \pm 10 \text{ MPa}$ ).

Requirements: purity  $>99.9\% \pm 0.01\%$ , Fe  $<0.01\% \pm 0.002\%$ , O  $<0.05\% \pm 0.01\%$ , particle size  $0.5\text{-}3 \mu\text{m} \pm 0.01 \mu\text{m}$ .

Performance influence: The flexural strength of cemented carbide containing  $10\% \pm 1\% \text{ Co}$  is  $>4000 \text{ MPa} \pm 100 \text{ MPa}$ , and the fracture toughness  $K_{\text{IC}} > 18 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ , which is suitable for cutting tools and mining equipment.

### Ni powder characteristics:

Crystal structure: face-centered cubic (FCC) structure, lattice parameter  $a = 0.3524 \text{ nm} \pm 0.0001 \text{ nm}$ , slip system  $>12$ , plastic deformation ability is slightly lower than Co.

Density:  $8.91 \text{ g}/\text{cm}^3 \pm 0.05 \text{ g}/\text{cm}^3$ , well matched with WC.

Melting point:  $1455^{\circ}\text{C} \pm 5^{\circ}\text{C}$ , suitable for high temperature sintering.

Hardness: Vickers hardness  $\text{HV } 90 \pm 10$ , softer than Co, slightly lower toughness.

Corrosion resistance: Corrosion potential is  $0.2 \text{ V} \pm 0.02 \text{ V}$  (vs. SCE), which is better than Co ( $0.1 \text{ V} \pm 0.02 \text{ V}$ ). The corrosion rate in pH 2-12 environment is  $<0.02 \text{ mm}/\text{year} \pm 0.005 \text{ mm}/\text{year}$ .

Thermal conductivity:  $90 \text{ W}/(\text{m} \cdot \text{K}) \pm 5 \text{ W}/(\text{m} \cdot \text{K})$ , higher than Co, which helps dissipate heat.

Thermal expansion coefficient:  $6.0 \times 10^{-6} \text{ K}^{-1} \pm 0.2 \times 10^{-6} \text{ K}^{-1}$ , slightly different from WC, and the stress after sintering is slightly higher ( $<70 \text{ MPa} \pm 10 \text{ MPa}$ ).

Requirements: purity  $>99.8\% \pm 0.01\%$ , Fe  $<0.02\% \pm 0.002\%$ , O  $<0.1\% \pm 0.01\%$ , particle size  $0.5\text{-}5 \mu\text{m} \pm 0.01 \mu\text{m}$ .

Performance impact: The corrosion rate of cemented carbide containing  $12\% \pm 1\% \text{ Ni}$  in a marine environment (pH 8, depth 5000 m, salinity 3.5%) is  $0.02 \text{ mm}/\text{year} \pm 0.005 \text{ mm}/\text{year}$ , which is suitable for corrosion-resistant scenarios.

### Ratio and application scenarios:

Co accounts for  $>80\% \pm 2\%$  of the binder phase. Due to its excellent toughness, it is suitable for high-impact scenarios (such as aviation tools and mining drill bits).

Ni accounts for  $<20\% \pm 2\%$ . Due to its strong corrosion resistance, it is suitable for chemical and marine environments (such as deep-sea valves and chemical pump bodies).

Examples:

The impact energy of the tool containing  $10\% \pm 1\% \text{ Co}$  in aviation cutting ( $1000^{\circ}\text{C}$ , Ti-6Al-4V alloy) is  $>12 \text{ J} \pm 1 \text{ J}$ , the wear amount is  $<0.15 \text{ mm} \pm 0.03 \text{ mm}$ , and the life is  $>12 \text{ h} \pm 1 \text{ h}$ .

For deep-sea valves (5000 m, salinity 3.5%) containing  $12\% \pm 1\% \text{ Ni}$ , the corrosion depth is  $<3 \mu\text{m} \pm 0.5 \mu\text{m}$  and the service life is  $>5 \text{ years} \pm 0.5 \text{ years}$ .

## (2) Selection criteria and optimization of Co and Ni powders

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The selection of Co and Ni powders needs to comprehensively consider purity, particle size, morphology, production process and compatibility with WC to optimize the performance of cemented carbide.

**purity:**

Co :  $>99.9\%\pm 0.01\%$ , Fe  $<0.01\%\pm 0.002\%$ , O  $<0.05\%\pm 0.01\%$ . Low Fe reduces the formation of  $\eta$  phase (  $\text{Co}_3\text{W}_3\text{C}$  ) ( $<0.5\%\pm 0.1\%$ ), low O reduces porosity ( $<0.1\%\pm 0.02\%$ ), and increases strength by  $3\%\pm 0.5\%$ .

Ni:  $>99.8\%\pm 0.01\%$ , Fe  $<0.02\%\pm 0.002\%$ , O  $<0.1\%\pm 0.01\%$ . Low impurities improve corrosion resistance and reduce corrosion rate by  $2\%\pm 0.5\%$ .

Impact: High purity reduces sintering defects (such as pores and inclusions) and increases the flexural strength to  $>4200\text{ MPa}\pm 100\text{ MPa}$ .

**Particle size:**

Co:  $0.5\text{--}1\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$  , improves dispersion (uniformity $>95\%\pm 1\%$ ) and toughness by  $5\%\pm 1\%$ ;  $>3\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$  leads to uneven distribution and toughness decreases by  $3\%\pm 0.5\%$ .

Ni:  $0.5\text{--}3\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$  , matches WC ( $1\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$  ), interface bonding strength $>50\text{ MPa}\pm 5\text{ MPa}$ , corrosion resistance increased by  $2\%\pm 0.5\%$ ;  $>5\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$  reduces uniformity and corrosion rate increases by  $1\%\pm 0.2\%$ .

Impact: Fine particle size enhances the uniformity of the binder phase distribution, and the porosity after sintering is  $<0.05\%\pm 0.01\%$ .

**Appearance:**

Co: spherical (spheroidization rate  $> 90\% \pm 2\%$ ), reduced agglomeration ( $< 5\% \pm 1\%$ ), fluidity improved by  $3\% \pm 0.5\%$  ( $< 25\text{ s}/50\text{ g} \pm 2\text{ s}$ , GB/T 1482-2010), suitable for compression molding.

Ni: polygonal ( edge  $<0.1\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$  ) or nearly spherical, enhances interface bonding strength ( $>50\text{ MPa}\pm 5\text{ MPa}$ ) and improves wear resistance.

Impact: The optimized morphology improves the powder spreading uniformity and sintering bonding strength, and the surface roughness of the cemented carbide is  $\text{Ra} < 5\text{ }\mu\text{m}\pm 1\text{ }\mu\text{m}$  .

**Chemical stability:**

Co: The oxidation rate in air at  $<600^\circ\text{C}\pm 10^\circ\text{C}$  is  $<0.01\text{ mg}/\text{cm}^2 \cdot \text{h} \pm 0.002\text{ mg}/\text{cm}^2 \cdot \text{h}$  . The sintering atmosphere needs to be controlled ( $\text{O}_2 < 10\text{ ppm}\pm 1\text{ ppm}$ ).

Ni: The corrosion rate in a pH 2-12 environment is  $<0.02\text{ mm}/\text{year}\pm 0.005\text{ mm}/\text{year}$ , and its acid and alkali resistance is better than that of Co.

Impact: Ni's high corrosion resistance is suitable for acidic environments (such as chemical pump bodies, pH 2-12), and Co needs to be protected from high-temperature oxidation.

**Production process:**

Co:

Electrolytic method: O  $<0.03\%\pm 0.005\%$ , Fe  $<0.005\%\pm 0.001\%$ , purity increased by  $1\%\pm 0.2\%$ , suitable for high toughness applications.

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Carbonyl method: O  $>0.1\% \pm 0.01\%$ , Fe  $<0.01\% \pm 0.002\%$ , low cost but slightly lower purity.

Reduction method: particle size  $0.5-3 \mu\text{m} \pm 0.01 \mu\text{m}$ , suitable for mass production.

Ni:

Atomization method: Fe  $<0.01\% \pm 0.002\%$ , O  $<0.05\% \pm 0.01\%$ , spherical particles ( $>90\% \pm 2\%$ ), suitable for high purity requirements.

Electrolysis method: particle size  $1-5 \mu\text{m} \pm 0.01 \mu\text{m}$ , O  $<0.1\% \pm 0.01\%$ , low cost.

Carbonyl method: The morphology is uniform, but the Fe content is slightly higher ( $<0.02\% \pm 0.002\%$ ).

Impact: Electrolytic Co and atomized Ni improve purity and morphology consistency, and cemented carbide has better performance.

### Compatibility with WC:

Co: The wetting angle with WC is  $<10^\circ \pm 1^\circ$ , and the bonding strength is  $>60 \text{ MPa} \pm 5 \text{ MPa}$ , which is suitable for high-toughness scenarios.

Ni: Wetting angle  $<15^\circ \pm 1^\circ$ , bonding strength  $>50 \text{ MPa} \pm 5 \text{ MPa}$ , corrosion resistance is better than Co.

Impact: Good wettability ensures that the interface is defect-free after sintering and the flexural strength is  $>4000 \text{ MPa} \pm 100 \text{ MPa}$ .

Examples:

K<sub>1c</sub>  $18 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ ) prepared by  $0.8 \mu\text{m} \pm 0.01 \mu\text{m}$  electrolytic Co (O  $<0.03\% \pm 0.005\%$ ) is used for mining drill bits (impact  $>200 \text{ MPa} \pm 10 \text{ MPa}$ ) with a life of  $>1200 \text{ m} \pm 100 \text{ m}$ .

$2 \mu\text{m} \pm 0.01 \mu\text{m}$  atomized Ni (Fe  $<0.01\% \pm 0.002\%$ ) (corrosion rate  $0.01 \text{ mm/year} \pm 0.002 \text{ mm/year}$ ) are used in chemical plants (pH 4) with a service life of  $>3 \text{ years} \pm 0.3 \text{ years}$ .

### (3) Optimization strategy for Co and Ni powders

#### Mixing process:

Co+Ni composite bonding phase: Co:Ni ratio  $4:1 \pm 0.2$ , combining the high toughness of Co and the corrosion resistance of Ni, suitable for marine mining equipment (impact resistance  $>10 \text{ J} \pm 1 \text{ J}$ , corrosion rate  $<0.015 \text{ mm/year} \pm 0.002 \text{ mm/year}$ ).

Ball milling process: rotation speed  $300 \text{ rpm} \pm 10 \text{ rpm}$ , ball to material ratio  $10:1 \pm 0.1$ , time  $10-20 \text{ hours} \pm 1 \text{ hour}$ , ensure uniform dispersion (uniformity  $>95\% \pm 1\%$ ).

Additives:  $0.1\%-0.5\% \pm 0.01\%$  VC or  $\text{Cr}_3\text{C}_2$  inhibits grain growth, and the particle size deviation is  $<3\% \pm 0.5\%$ .

#### Sintering process:

Vacuum sintering:  $1350^\circ\text{C} \pm 10^\circ\text{C}$ , atmosphere  $<10^{-2} \text{ Pa} \pm 10^{-3} \text{ Pa}$ , reduced oxidation (O  $<0.02\% \pm 0.005\%$ ).

HIP (hot isostatic pressing):  $1400^\circ\text{C} \pm 10^\circ\text{C}$ , pressure  $100 \text{ MPa} \pm 5 \text{ MPa}$ , density  $>99.5\% \pm 0.1\%$ , strength increase  $5\% \pm 1\%$ .

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Atmosphere control:  $H_2$  atmosphere ( $O_2 < 10 \text{ ppm} \pm 1 \text{ ppm}$ ) inhibits Co oxidation, and Ar atmosphere protects Ni stability.

#### Surface treatment:

Co powder: surface passivation ( $O_2$  adsorption  $< 0.01 \text{ mg/g} \pm 0.002 \text{ mg/g}$ ), reducing storage oxidation.

Ni powder: Anti-oxidation coating (such as  $SiO_2$  thin layer, thickness  $< 0.1 \mu\text{m} \pm 0.01 \mu\text{m}$ ), improving corrosion resistance by  $1\% \pm 0.2\%$ .

#### (4) Engineering Application

##### Co-based cemented carbide:

Aviation tools

Contains  $10\% \pm 1\%$  Co, hardness HV  $2900 \pm 50$ ,  $K_{IC} > 18 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ , cutting Ti-6Al-4V ( $1000^\circ\text{C}$ , speed  $> 300 \text{ m/min} \pm 10 \text{ m/min}$ ), wear amount  $< 0.15 \text{ mm} \pm 0.03 \text{ mm}$ , life  $> 12 \text{ hours} \pm 1 \text{ hour}$ .

Mining drill bits

Contains  $8\% \pm 1\%$  Co, impact resistance  $> 10 \text{ J} \pm 1 \text{ J}$ , hard rock drilling (compression resistance  $> 200 \text{ MPa} \pm 10 \text{ MPa}$ ) life  $> 1200 \text{ m} \pm 100 \text{ m}$ .

Wear-resistant mold

Contains  $12\% \pm 1\%$  Co, cold heading die ( $> 10^6$  times  $\pm 10^5$  times), deformation  $< 0.01 \text{ mm} \pm 0.002 \text{ mm}$ .

##### Ni-based cemented carbide:

Deep sea valve

Contains  $12\% \pm 1\%$  Ni, corrosion depth  $< 5 \mu\text{m} \pm 1 \mu\text{m}$ , life span in deep sea environment (5000 m, salinity 3.5%)  $> 5 \text{ years} \pm 0.5 \text{ years}$ .

Chemical pump body

Contains  $15\% \pm 1\%$  Ni, pH 2-12, corrosion rate  $0.01 \text{ mm/year} \pm 0.002 \text{ mm/year}$ , life span  $> 2 \text{ years} \pm 0.2 \text{ years}$ .

Marine mining equipment

Contains  $10\% \pm 1\%$  Ni +  $5\% \pm 1\%$  Co, impact resistance  $> 8 \text{ J} \pm 1 \text{ J}$ , corrosion resistance improved by  $3\% \pm 0.5\%$ , lifespan  $> 3 \text{ years} \pm 0.3 \text{ years}$ .

##### Co+Ni composite bonding phase:

Oil drilling tools: Co:Ni ratio 3:1  $\pm 0.2$ ,  $K_{IC} > 16 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ , corrosion rate  $< 0.015 \text{ mm/year} \pm 0.002 \text{ mm/year}$ , life span  $> 1000 \text{ hours} \pm 100 \text{ hours}$ .

#### (5) Development Trends

Nano-scale binder phase: Development of  $< 0.5 \mu\text{m} \pm 0.01 \mu\text{m}$  Co/Ni powder, improved dispersibility (uniformity  $> 98\% \pm 1\%$ ), carbide hardness  $> \text{HV } 3000 \pm 50$ , toughness  $> K_{IC} > 20 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ .

Green production: Use renewable energy electrolysis to produce Co/Ni powder, reducing energy consumption by  $15\% \pm 2\%$  and reducing emissions ( $CO_2 < 500 \text{ kg/t} \pm 50 \text{ kg/t}$ ).

Intelligent control: AI is introduced to optimize the Co/Ni ratio and sintering parameters, improving performance consistency by  $5\% \pm 1\%$  and production efficiency by  $10\% \pm 2\%$ .

Co and Ni are used as cemented carbide binders. Co (FCC,  $a = 0.3544 \text{ nm} \pm 0.0001 \text{ nm}$ , melting point

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1495°C±5°C) is mainly characterized by high toughness ( $K_{IC}$  15-20 MPa·m<sup>1/2</sup>±0.5), accounting for >80%±2%, and is suitable for aviation tools and mining drills; Ni (FCC,  $a=0.3524$  nm±0.0001 nm, melting point 1455°C±5°C) is characterized by corrosion resistance (corrosion rate <0.02 mm/year±0.005 mm/year), accounting for <20%±2%, and is suitable for marine and chemical environments. The selection criteria focus on purity (Co >99.9%±0.01%, Ni >99.8%±0.01%), particle size (Co 0.5-1 μm±0.01 μm, Ni 0.5-3 μm±0.01 μm) and morphology (spherical Co, polygonal Ni). By optimizing the production process (electrolytic Co, atomized Ni), mixing process and sintering technology, the performance of cemented carbide is significantly improved to meet the needs of diversified engineering. In the future, nano-, green and intelligent will be the development direction of Co/Ni powder.

**4.2.2 Mechanism of grain inhibitors (vanadium carbide (VC) and chromium carbide ( $Cr_3C_2$ ))**  
the growth of WC (tungsten carbide) grains in cemented carbide preparation. Vanadium carbide (VC) and chromium carbide ( $Cr_3C_2$ ) are the two most commonly used. By adding VC and  $Cr_3C_2$ , the grains can be effectively refined, the hardness and strength can be improved, and the performance of cemented carbide can be optimized. The following is a detailed analysis from the aspects of basic characteristics, inhibition mechanism, influencing factors, optimization strategy and engineering application.

#### (1) Basic characteristics

##### VC (Vanadium Carbide):

Chemical formula: VC, cubic crystal system (FCC), lattice parameter  $a = 0.416$  nm±0.001 nm.

Density: 5.77 g/cm<sup>3</sup> ± 0.05 g/cm<sup>3</sup>, melting point 2830°C±10°C, hardness HV 2800±50.

Features: high hardness, strong thermal stability, low solubility in Co binder phase (~5%±0.5%), suitable for submicron cemented carbide.

##### $Cr_3C_2$ (chromium carbide):

Chemical formula:  $Cr_3C_2$ , orthorhombic crystal system, lattice parameters  $a = 0.552$  nm±0.001 nm,  $b = 1.149$  nm±0.001 nm,  $c = 0.283$  nm±0.001 nm.

Density: 6.68 g/cm<sup>3</sup> ± 0.05 g/cm<sup>3</sup>, melting point 1895°C±10°C, hardness HV 1300±50.

Features: Good corrosion resistance, high thermal stability, certain solubility with Co/Ni bonding phase (solubility in Co ~2%±0.2%), suitable for micron-sized cemented carbide.

#### (2) Inhibition mechanism and effect

Grain inhibitors can significantly improve the hardness (HV >2000±30) and strength (>4000 MPa±100 MPa) of cemented carbide by regulating the growth behavior of WC grains during sintering (growth rate <0.01 μm/min ±0.001 μm/min).

##### VC's inhibitory mechanism:

During the sintering process, VC partially dissolves in the Co phase (solubility ~5%±0.5%), reduces the WC/Co interface energy (<0.5 J/m<sup>2</sup> ± 0.1 J/m<sup>2</sup>), and inhibits the dissolution-reprecipitation

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process of WC (Ostwald ripening, rate  $<10^{-9}$  m/s $\pm 10^{-10}$  m/s).

VC precipitates at the WC grain boundaries to form nanoscale particles ( $<0.1\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ), which hinder the migration and merging of WC grains.

Effect: Adding  $0.5\% \pm 0.01\%$  VC can reduce the average grain size of WC from  $1\ \mu\text{m} \pm 0.01\ \mu\text{m}$  to  $0.3\ \mu\text{m} \pm 0.01\ \mu\text{m}$ , increase the hardness by  $10\% \pm 2\%$  (HV  $>2200 \pm 30$ ), and improve the wear resistance (wear loss  $<0.08\ \text{mm} \pm 0.02\ \text{mm}$ ).

#### **The inhibition mechanism of $\text{Cr}_3\text{C}_2$ :**

$\text{Cr}_3\text{C}_2$  precipitates a thin layer (thickness  $<5\ \text{nm} \pm 1\ \text{nm}$ ) at the WC/Co interface, hindering the diffusion of C and W atoms (diffusion coefficient  $<10^{-11}\ \text{cm}^2/\text{s} \pm 10^{-12}\ \text{cm}^2/\text{s}$ ) and reducing grain merging (merging rate  $<5\% \pm 1\%$ ).

$\text{Cr}_3\text{C}_2$  partially dissolves in the Co phase, changing the interfacial energy of liquid phase sintering (reduced to  $<1\ \text{J/m}^2 \pm 0.1\ \text{J/m}^2$ ) and slowing down the dissolution-reprecipitation rate of WC.

#### **Effect:**

Adding  $0.5\% \pm 0.01\%$   $\text{Cr}_3\text{C}_2$  can maintain the WC grain size at  $0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ , increase the strength by  $5\% \pm 1\%$  (flexural strength  $>4200\ \text{MPa} \pm 100\ \text{MPa}$ ), and improve the corrosion resistance (corrosion rate  $<0.015\ \text{mm/year} \pm 0.002\ \text{mm/year}$ ).

#### **Scope of application:**

VC is more suitable for submicron cemented carbide (WC grains  $<0.5\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ) because of its strong inhibitory effect.

$\text{Cr}_3\text{C}_2$  is more suitable for micron - sized cemented carbide (WC grains  $1\text{-}3\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ) because it combines strength and corrosion resistance.

#### **Performance impact:**

##### **Refined grains improve hardness and wear resistance**

The hardness of cemented carbide containing  $0.5\% \pm 0.01\%$  VC is HV  $>3100 \pm 50$ , which is suitable for aviation processing.

##### **Grain boundary strengthening improves strength and toughness**

The flexural strength of cemented carbide containing  $0.5\% \pm 0.01\%$   $\text{Cr}_3\text{C}_2$  is  $>4200\ \text{MPa} \pm 100\ \text{MPa}$ , and the fracture toughness  $K_{Ic} >18\ \text{MPa}\cdot\text{m}^{1/2} \pm 0.5$ .

##### **High temperature stability**

VC enhances grain boundary stability, and the deformation of cemented carbide at  $1000^\circ\text{C} \pm 10^\circ\text{C}$  is  $<0.01\ \text{mm} \pm 0.002\ \text{mm}$ .

### **(3) Influencing factors and optimization**

The effect of grain inhibitors is affected by many factors, such as the amount of addition, particle size, sintering temperature and atmosphere, and the best performance must be ensured by optimizing the process.

#### **Addition amount:**

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VC: 0.1%-0.5%±0.01%. Excessive amount (>0.8%±0.01%) will generate brittle phase  $V_6C_5$  (hardness HV <1500±50), resulting in a decrease in toughness by 10%±2% ( $K_{1c}$  <15 MPa·m<sup>1/2</sup> ± 0.5).

$Cr_3C_2$ : 0.5%-1%±0.01%. Excessive amount (>1.5%±0.01%) reduces the fluidity of Co phase (<10 s/50 g±0.5 s) and the density decreases by 1%±0.2% (<99%±0.1%).

Optimization: Accurately control the amount of addition, VC recommends 0.3%±0.01%,  $Cr_3C_2$  recommends 0.5%±0.01% to balance hardness and toughness.

#### Particle size:

VC: <0.1 μm±0.01 μm improves dispersion (deviation <5%±1%) and hardness increases by 5%±1% (HV >2300±30).

$Cr_3C_2$ : <0.5 μm±0.01 μm enhances interface bonding strength (>50 MPa±5 MPa) and increases strength by 3%±0.5% (>4300 MPa±100 MPa).

Optimization: Use nano-scale VC and  $Cr_3C_2$ , combined with ultrasonic dispersion (frequency 40 kHz±1 kHz) to reduce agglomeration (<5%±1%).

#### Sintering temperature:

1450°C±10°C ensures the dissolution of VC and  $Cr_3C_2$  (dissolution rate>90%±2%), and the inhibition effect is improved by 5%±1%.

1550°C±10°C induces the precipitation of VC and  $Cr_3C_2$  (precipitation rate>10%±2%), and the hardness decreases by 3%±0.5% (HV <2000±30).

Optimization: Control the sintering temperature at 1400-1450°C ± 10°C and extend the holding time (2-3 hours ± 0.1 hours) to ensure uniform inhibition.

#### Atmosphere:

H<sub>2</sub> atmosphere ( $O_2$  < 10 ppm±1 ppm) inhibits the oxidation of VC and  $Cr_3C_2$  ( $O_2$  <0.05%±0.01%), and the purity is increased by 1%±0.2%.

Vacuum atmosphere (<10<sup>-2</sup> Pa±10<sup>-3</sup> Pa) reduces VC volatilization (loss <0.1%±0.01%) and improves the inhibition effect by 2%±0.5%.

H<sub>2</sub> atmosphere is preferred, combined with vacuum pretreatment to reduce the oxide content.

#### Mixing method:

Ball mill (speed 300 rpm ± 10 rpm, time 10-20 h ± 1 h) to ensure uniform dispersion (uniformity > 95% ± 1%).

Optimization: Add dispersants (such as ethanol, 0.1% ± 0.01%) to reduce agglomeration (<3% ± 1%) and improve dispersion efficiency.

#### Examples:

0.3%±0.01% VC (particle size <0.1 μm±0.01 μm), 1450°C±10°C, H<sub>2</sub> atmosphere process to generate WC grains 0.2 μm±0.01 μm, hardness HV 2300±30, used for PCB drill bits (lifespan >10<sup>5</sup> holes±10<sup>4</sup> holes).

0.5%±0.01%  $Cr_3C_2$  (particle size <0.5 μm±0.01 μm), 1400°C±10°C, vacuum atmosphere process

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to generate WC grains  $0.5\ \mu\text{m}\pm 0.01\ \mu\text{m}$ , strength  $>4300\ \text{MPa}\pm 100\ \text{MPa}$ , used for mining drill bits (lifespan  $>1200\ \text{m}\pm 100\ \text{m}$ ).

#### (4) Engineering Application

##### VC added:

$0.5\%\pm 0.01\%$  VC is used for superhard tools (WC grain  $<0.5\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ) in aviation machining ( $1000^\circ\text{C}$ , Ti-6Al-4V alloy), with wear  $<0.08\ \text{mm}\pm 0.02\ \text{mm}$  and life  $>15\ \text{h}\pm 1\ \text{h}$ .

$0.3\%\pm 0.01\%$  VC for PCB drill bits (WC grain  $0.2\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ), hardness HV  $2300\pm 30$ , life  $>10^5$  holes  $\pm 10^4$  holes.

##### Cr<sub>3</sub>C<sub>2</sub> Addition :

$0.5\%\pm 0.01\%$  Cr<sub>3</sub>C<sub>2</sub> is used in mining drill bits (WC grain  $1-3\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ), with a life of  $>1200\ \text{m}\pm 100\ \text{m}$  in hard rock drilling (compression resistance  $>200\ \text{MPa}\pm 10\ \text{MPa}$ ).

$0.8\%\pm 0.01\%$  Cr<sub>3</sub>C<sub>2</sub> is used for chemical pump bodies (WC grain  $1\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ), the corrosion rate in pH 2-12 environment is  $<0.015\ \text{mm/year}\pm 0.002\ \text{mm/year}$ , and the service life is  $>2\ \text{years}\pm 0.2\ \text{years}$ .

##### VC+Cr<sub>3</sub>C<sub>2</sub> compound addition:

$0.3\%\pm 0.01\%$  VC +  $0.5\%\pm 0.01\%$  Cr<sub>3</sub>C<sub>2</sub> is used for marine mining equipment (WC grain  $0.5\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ), impact resistance  $>10\ \text{J}\pm 1\ \text{J}$ , corrosion rate  $<0.01\ \text{mm/year}\pm 0.002\ \text{mm/year}$ , life  $>3\ \text{years}\pm 0.3\ \text{years}$ .

#### (5) Testing and quality control

##### Grain size

Scanning electron microscopy (SEM, GB/T 16594-2008) was used to measure the WC grain size (deviation  $<5\%\pm 1\%$ ).

Distribution uniformity: X-ray energy spectrum (EDS, GB/T 17359-2012) is used to detect the distribution of VC/ Cr<sub>3</sub>C<sub>2</sub> at grain boundaries (deviation  $<3\%\pm 0.5\%$ ).

##### Performance Testing

Hardness: According to ISO 4499-2, measure Vickers hardness (HV  $>2000\pm 30$ ).

Strength: According to GB/T 3851-2015, test the flexural strength ( $>4000\ \text{MPa}\pm 100\ \text{MPa}$ ).

Wear resistance: According to GB/T 12444-2006, measure the wear amount ( $<0.08\ \text{mm}\pm 0.02\ \text{mm}$ ).

Online monitoring: Infrared thermal imaging monitors the sintering temperature (deviation  $<5^\circ\text{C}\pm 1^\circ\text{C}$ ) to ensure consistent inhibition effect.

Grain inhibitors VC and Cr<sub>3</sub>C<sub>2</sub> inhibit WC grain growth (growth rate  $<0.01\ \mu\text{m}/\text{min}\pm 0.001\ \mu\text{m}/\text{min}$ ) through dissolution-precipitation mechanism, refine grains ( $<0.5\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ), and significantly improve the hardness (HV  $>2000\pm 30$ ), strength ( $>4000\ \text{MPa}\pm 100\ \text{MPa}$ ) and wear resistance (wear loss  $<0.08\ \text{mm}\pm 0.02\ \text{mm}$ ) of cemented carbide. VC is suitable for submicron

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cemented carbide (strong inhibitory effect), and  $\text{Cr}_3\text{C}_2$  is suitable for micron level (taking into account corrosion resistance). The best inhibition effect can be achieved by optimizing the addition amount (VC 0.1%-0.5%±0.01%,  $\text{Cr}_3\text{C}_2$  0.5%-1%±0.01%), particle size (VC <0.1 μm±0.01 μm,  $\text{Cr}_3\text{C}_2$  <0.5 μm±0.01 μm), sintering temperature (1450°C±10°C) and atmosphere ( $\text{H}_2$  or vacuum). The application of VC and  $\text{Cr}_3\text{C}_2$  significantly improves the performance of aviation tools (life>15 hours±1 hour), mining drill bits (life>1200 m±100 m) and chemical equipment (life>2 years±0.2 years).

### 4.3 Powder pretreatment

Powder pretreatment optimizes the mixing uniformity (deviation <5%±1%), particle size distribution (0.110μm±0.01μm) and fluidity (1316s/50g±0.5s) of WC, Co/Ni and additives by ball milling (wet/dry milling, ball-to-powder ratio 10:1±0.5) and spray drying (flow rate 100 L/h±10 L/h). Pretreatment ensures sintering density (>99%±0.1%) and performance consistency (hardness deviation <±30 HV), reduces porosity (<0.1%±0.02%), and improves strength (>4000 MPa±100 MPa). This section analyzes the ball milling and spray drying processes.

#### 4.3.1 Ball milling process (wet milling/dry milling, ball to material ratio 10:1)

##### Process parameters and principle

Ball milling uses WC balls (diameter 510 mm ± 0.1 mm, hardness HV 1800 ± 50) to grind WC (0.110 μm ± 0.01 μm), Co/Ni (0.53 μm ± 0.01 μm) and additives (VC/  $\text{Cr}_3\text{C}_2$ , <0.5 μm ± 0.01 μm), with a ball-to-material ratio of 10:1 ± 0.5, a rotation speed of 200400 rpm ± 10 rpm, and a time of 424 h ± 0.1 h.

##### Wet grinding

Use ethanol (purity>99.5%±0.01%, addition amount 50%100%±5% mass fraction), add dispersant (PEG, 0.5%1%±0.01%), reduce agglomeration (<5%±1%), and the particle size deviation is <3%±0.5%.

##### Dry grinding

No medium, suitable for low Co formula (<6%±1%), reduced pollution ( $\text{Fe}$ <0.01%±0.002%), but higher agglomeration rate (>10%±2%).

Wet grinding accounted for >90%±2% due to high uniformity (mixing deviation <2%±0.5%).

The kinetics were based on collision energy ( $10^{-3}$  J/shot ±  $10^{-4}$  J/shot) and a refinement rate of 0.1 μm/h ± 0.01 μm/h.

For example, wet grinding (12 h ± 0.1 h, ethanol 100% ± 5%) produces a mixed powder of WC 0.5 μm ± 0.01 μm and Co 0.8 μm ± 0.01 μm with a uniformity of > 98% ± 1%, which is used for aviation tools (hardness HV 2200 ± 30, life > 12 h ± 1 h).

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**Influencing factors and optimization**

**Ball milling effect is affected by the following factors:**

**Ball to Material Ratio**

10:1±0.5 has high refinement efficiency (>90%±2%), >15:1 increases contamination (Fe>0.05%±0.01%), and hardness decreases by 2%±0.5%.

**Speed**

300 rpm±10 rpm balances efficiency and wear (ball wear <0.1%±0.02%), <200 rpm insufficient refinement (particle size >1µm±0.01µm).

**time**

12 hours ± 0.1 hours ensures uniformity (deviation < 2% ± 0.5%), > 24 hours causes over-wear (Fe > 0.03% ± 0.005%), and toughness decreases by 3% ± 0.5%.

**medium**

Ethanol reduces surface energy (<0.1 J/m²±0.02 J/m²) and reduces agglomeration by 5%±1%. Water (purity>99.9%±0.01%) is low cost but increases the O content by 0.05%±0.01%.

**Ball Material**

WC balls (purity>99.5%±0.01%) are less contaminated (Fe<0.01%±0.002%) and are better than steel balls (Fe>0.1%±0.02%).

For example, wet grinding (10:1 ± 0.5, 300 rpm ± 10 rpm, 12 h ± 0.1 h, WC balls) produces a mixed powder (WC 0.3 µm ± 0.01 µm) for PCB drill bits (lifetime > 10<sup>5</sup> holes ± 10<sup>4</sup> holes).

**Engineering Application**

**Wet grinding**

WC 0.5µm±0.01µm, Co 0.8µm±0.01µm are used for cutting tools (aviation, wear <0.1mm±0.02mm).

**Dry grinding**

Low Co (6%±1%) formulation is used for molds (extrusion >10<sup>5</sup> times±10<sup>4</sup> times), with deformation <0.01mm±0.002mm.

**Dry grinding and wet grinding in ball milling process for pretreatment of cemented carbide raw materials**

category	parameter	Dry grinding	Wet grinding
	Features		
Technology	medium	No liquid medium, only air or inert gas (such as Ar , O <sub>2</sub>	Liquid medium (such as water, ethanol, acetone),
parameter		< 10 ppm±1 ppm).	concentration 50%-70%±2% ( solid-liquid ratio ).

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category	parameter Features	Dry grinding	Wet grinding
	Ball to Material Ratio	5:1 to 10:1±0.1, higher to ensure grinding efficiency.	3:1 to 8:1±0.1, liquid medium reduces friction, the ball-to-material ratio can be slightly lower.
	Speed	200-400 rpm±10 rpm. Too high (>500 rpm) may cause overheating and agglomeration (>10%±1%).	300-500 rpm±10 rpm, liquid cooling, the speed can be slightly higher, and the agglomeration rate is <5%±1%.
	Grinding time	10-20 hours ± 0.5 hours. Long time may lead to overheating. Particle size deviation > 10% ± 1%.	5-15 hours ± 0.5 hours, high liquid efficiency, short time, particle size deviation < 5% ± 1%.
	ball milling medium	Carbide balls (HRC 65-75±2), ZrO <sub>2</sub> balls (HRC 70-80±2), diameter 2-10 mm±0.1 mm.	Carbide balls, ZrO <sub>2</sub> balls, stainless steel balls (HRC 25-35±2), diameter 1-5 mm±0.1 mm.
	temperature control	Natural heat dissipation, the temperature is easy to rise (>60°C±2°C), and intermittent cooling is required (stop for 30 minutes± 5 minutes every 2 hours±0.1 hour).	Liquid medium heat dissipation, temperature <40°C±2°C, no additional cooling required.
	atmosphere control	Inert gas (such as Ar or N <sub>2</sub> , O <sub>2</sub> < 10 ppm±1 ppm), prevent oxidation (O<0.05%±0.01%).	The liquid medium isolates the air, and the oxidation rate is <0.03%±0.005%. Air or inert atmosphere can be used.
	Add to Agent	Add paraffin wax (1%-2%±0.1%) by dry method to improve fluidity (<30 s/50 g±2 s).	Wet-add PVA or PEG (1%-3%±0.1%), dissolve in the medium, and improve dispersibility (>95%±1%).
	powder Particle size	Suitable for coarse grinding (1-10 μm±0.01 μm), the efficiency of refining to 0.5 μm±0.01 μm is low.	Suitable for fine grinding and ultrafine grinding (0.1-1 μm±0.01 μm), with high refining efficiency and distribution deviation <5%±1%.
	purity control	Impurities (such as Fe <0.02%±0.005%) are easily introduced and need to be removed by pickling.	Liquid medium has fewer impurities (Fe <0.01%±0.002%) and higher purity (>99.9%±0.01%).
	dry step	No need to dry, just press directly.	Need to be dried (vacuum drying, 80°C±2°C, <10 <sup>-2</sup> Pa ± 10 <sup>-3</sup> Pa) for 4-8 hours ±0.5 hours.
	Energy consumption	Higher (50-80 kWh ± 5 kWh per ton of powder) due to high friction.	Low (30-50 kWh ± 5 kWh per ton of powder), liquid reduces friction.
Features	Process Cleanliness	The process is simple, does not require additional equipment, and is suitable for large-scale production (>1 ton/batch).	The process is relatively complicated and requires drying equipment, which is suitable for fine processing.
	Particle size distributed	Distribution width ((D90-D10)/D50 >2.0±0.2), uniformity <90%±1%.	The distribution is narrow ((D90-D10)/D50 <1.5±0.1), and the uniformity is >95%±1%.
	Reunion rate	High (>10%±1%), temperature and additives need to be controlled.	Low (<5%±1%), effectively dispersed in liquid media.

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category	parameter	Dry grinding	Wet grinding
Pros and Cons	<b>Features</b>		
	<b>Oxidation control</b>	An inert atmosphere is required and the oxidation rate may be $>0.05\% \pm 0.01\%$ .	Liquid isolation, oxidation rate $<0.03\% \pm 0.005\%$ .
	<b>advantage</b>	Low cost (reduced equipment investment, no drying step, suitable for rough processing).	High refining efficiency and high purity, suitable for ultrafine powder ( $<0.5 \pm 0.01 \mu\text{m}$ ).
Pros and Cons	<b>shortcoming</b>	The refining ability is limited, it is easy to agglomerate, and high temperature ( $>60^\circ\text{C} \pm 2^\circ\text{C}$ ) leads to oxidation.	The need for drying increases energy consumption ( $10\text{-}20 \text{ kWh/t} \pm 2 \text{ kWh}$ ), and the liquid may introduce moisture ( $>0.1\% \pm 0.01\%$ ).
	<b>Application Scenario</b>		
Application Scenario	<b>Applicable scope</b>	Coarse particle mixing ( $>5 \mu\text{m} \pm 0.01 \mu\text{m}$ ) for low-cost production (e.g. mining drill bit blanks).	Submicron and nanometer powders ( $<0.5 \mu\text{m} \pm 0.01 \mu\text{m}$ ), high-precision molding (such as aviation tools).
	<b>Examples</b>	WC-Co powder ( $D_{50}=5 \mu\text{m} \pm 0.01 \mu\text{m}$ ), flexural strength $>3800 \text{ MPa} \pm 100 \text{ MPa}$ , life $>1200 \text{ m} \pm 100 \text{ m}$ .	WC powder ( $D_{50}=0.3 \mu\text{m} \pm 0.01 \mu\text{m}$ ), hardness $\text{HV} > 3000 \pm 50$ , life $>15 \text{ hours} \pm 1 \text{ hour}$ .
Optimization suggestions	<b>ball milling medium</b>	Use carbide balls (HRC 65-75 $\pm$ 2) to reduce contamination ( $\text{Fe} < 0.01\% \pm 0.002\%$ ).	Use $\text{ZrO}_2$ balls (HRC 70-80 $\pm$ 2) to avoid contamination ( $\text{O} < 0.03\% \pm 0.005\%$ ).
	<b>Add to Dosage Optimization</b>	1% $\pm$ 0.1% paraffin wax, reduces the aggregation rate ( $<5\% \pm 1\%$ ).	1%-3% $\pm$ 0.1% PVA or PEG, to improve dispersion (uniformity $>95\% \pm 1\%$ ).
	<b>temperature control</b>	Intermittent operation (30 minutes $\pm$ 5 minutes stop every 2 hours $\pm$ 0.1 hour), $<50^\circ\text{C} \pm 2^\circ\text{C}$ .	Liquid cooling, no additional control required, $<40^\circ\text{C} \pm 2^\circ\text{C}$ .
	<b>atmosphere optimization</b>	Ar or $\text{N}_2$ atmosphere ( $\text{O}_2 < 10 \text{ ppm} \pm 1 \text{ ppm}$ ), oxidation $<0.03\% \pm 0.005\%$ .	Ethanol medium (purity $>99.5\% \pm 0.1\%$ ), solid-liquid ratio 60% $\pm$ 2%, oxidation $<0.01\% \pm 0.002\%$ .
Optimization suggestions	<b>Post-processing</b>	No need to dry, just press directly.	Vacuum drying ( $80^\circ\text{C} \pm 2^\circ\text{C}$ , $<10^{-2} \text{ Pa} \pm 10^{-3} \text{ Pa}$ ), moisture $<0.05\% \pm 0.01\%$ .
	<b>Energy consumption optimization</b>	Optimize the rotation speed ( $<400 \text{ rpm} \pm 10 \text{ rpm}$ ) and reduce energy consumption by 10% $\pm$ 2%.	Optimize drying time ( $<6 \text{ hours} \pm 0.5 \text{ hours}$ ) and reduce energy consumption by 5% $\pm$ 1%.
illustrate	Dry grinding and wet grinding have their own characteristics in the pretreatment of cemented carbide raw materials. Dry grinding is simple (no drying required) and low cost (energy consumption 50-80 kWh/t $\pm$ 5 kWh), suitable for coarse grinding ( $>5 \mu\text{m} \pm 0.01 \mu\text{m}$ ), but with wide distribution ( $>2.0 \pm 0.2$ ) and high agglomeration rate ( $>10\% \pm 1\%$ ); wet grinding has the advantage of high refining efficiency (99.9% $\pm$ 0.01%), suitable for high-precision applications, but requires drying (energy consumption 30-50 kWh/t $\pm$ 5 kWh). By optimizing the ball-to-material ratio (dry grinding 5:1-10:1 $\pm$ 0.1, wet grinding 3:1-8:1 $\pm$ 0.1), rotation speed (dry grinding 200-400 rpm $\pm$ 10 rpm, wet grinding 300-500 rpm $\pm$ 10 rpm) and media selection, dry grinding supports mining drill bits (lifespan $>1200 \text{ m} \pm 100 \text{ m}$ ) and wet grinding supports aviation tools (lifespan $>15 \text{ hours} \pm 1 \text{ hour}$ ), meeting diverse needs.		

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### 4.3.2 Spray drying and granulation technology

Spray drying and granulation technology is the core process in the pretreatment of cemented carbide raw materials (such as tungsten carbide powder WC, cobalt powder Co and nickel powder Ni), which is used to make fine powder into particles with excellent fluidity and pressing properties.

#### (1) Principles of spray drying and granulation technology

##### Spray Drying:

Process: The suspension or slurry containing WC, Co and other powders is sprayed into tiny droplets ( $10\text{-}200\text{ }\mu\text{m}\pm 0.1\text{ }\mu\text{m}$ ) through an atomizer, and the water is rapidly evaporated (evaporation rate  $>90\%\pm 2\%/s$ ) in a high-temperature airflow ( $150\text{-}300^{\circ}\text{C}\pm 5^{\circ}\text{C}$ ) to form dry particles.

Heat and mass transfer: Water on the droplet surface evaporates first, and the internal water migrates by diffusion. The drying time is  $<1\text{ s}\pm 0.1\text{ s}$ .

Target: particle size  $20\text{-}150\text{ }\mu\text{m}\pm 0.1\text{ }\mu\text{m}$ , fluidity  $<20\text{ s}/50\text{ g}\pm 2\text{ s}$  (GB/T 1482-2010), bulk density  $>1.5\text{ g}/\text{cm}^3\pm 0.1\text{ g}/\text{cm}^3$ .

##### Granulation:

Process: In spray drying, binders (such as PVA, PEG) promote the adhesion of particles within the droplets, and the surface tension ( $<0.07\text{ N}/\text{m}\pm 0.01\text{ N}/\text{m}$ ) forms spherical or nearly spherical particles.

Mechanism: The particles collide and bond during the drying process, and the binder solidifies to form a network structure (porosity  $<10\%\pm 1\%$ ), which enhances the strength of the particles.

Target: green body compressive strength  $>10\text{ MPa}\pm 1\text{ MPa}$ , density uniformity after pressing  $>98\%\pm 1\%$ .

#### (2) Process parameters

##### Feed concentration:

Range:  $20\%\text{-}40\%\pm 1\%$  (solid mass fraction).

Impact: Concentration  $<15\%\pm 1\%$  results in too fine particles ( $<20\text{ }\mu\text{m}\pm 0.1\text{ }\mu\text{m}$ ) and poor fluidity ( $>30\text{ s}/50\text{ g}\pm 2\text{ s}$ );  $>45\%\pm 1\%$  results in too high viscosity ( $>1000\text{ mPa}\cdot\text{s}\pm 50\text{ mPa}\cdot\text{s}$ ) and nozzle clogging.

Optimization:  $25\%\text{-}30\%\pm 1\%$ , particle size  $50\text{-}100\text{ }\mu\text{m}\pm 0.1\text{ }\mu\text{m}$ , viscosity  $300\text{-}500\text{ mPa}\cdot\text{s}\pm 50\text{ mPa}\cdot\text{s}$ .

##### Feed flow rate:

Range:  $5\text{-}20\text{ L}/\text{h}\pm 0.5\text{ L}/\text{h}$  (small and medium equipment),  $50\text{-}200\text{ L}/\text{h}\pm 5\text{ L}/\text{h}$  (large equipment).

Impact: Flow rate  $<5\text{ L}/\text{h}\pm 0.5\text{ L}/\text{h}$ , uneven drying, residual water  $>1\%\pm 0.2\%$ ;  $>25\text{ L}/\text{h}\pm 0.5\text{ L}/\text{h}$ , particles are too large ( $>200\text{ }\mu\text{m}\pm 0.1\text{ }\mu\text{m}$ ), and the bulk density decreases.

Optimization:  $10\text{-}15\text{ L}/\text{h}\pm 0.5\text{ L}/\text{h}$  (small and medium),  $100\text{-}150\text{ L}/\text{h}\pm 5\text{ L}/\text{h}$  (large), drying efficiency  $>95\%\pm 1\%$ .

##### Inlet air temperature:

Range:  $150\text{-}300^{\circ}\text{C}\pm 5^{\circ}\text{C}$  (outlet temperature  $80\text{-}120^{\circ}\text{C}\pm 2^{\circ}\text{C}$ ).

Impact: Temperature  $<150^{\circ}\text{C}\pm 5^{\circ}\text{C}$ , incomplete drying, residual water  $>1\%\pm 0.2\%$ , high particle viscosity;  $>350^{\circ}\text{C}\pm 5^{\circ}\text{C}$ , binder decomposes (residual carbon  $>0.3\%\pm 0.01\%$ ), and particle brittleness increases.

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Optimization: 200-250°C±5°C, residual water <0.2%±0.05%, binder retention rate >90%±2%.

**Atomization pressure:**

Range: 0.1-0.3 MPa±0.01 MPa (pressure type), 0.2-0.4 MPa±0.01 MPa (air flow type).

Impact: When the pressure is <0.1 MPa±0.01 MPa, the droplets are large (>200 μm±0.1 μm) and the drying is uneven; when the pressure is >0.4 MPa±0.01 MPa, the droplets are too small (<20 μm±0.1 μm) and the fluidity is poor.

Optimization: 0.2-0.25 MPa±0.01 MPa, droplet size 50-100 μm±0.1 μm, distribution uniformity >95%±1%.

**Binder dosage:**

Range: 1%-5%±0.1% (PVA, PEG, paraffin).

Impact: Dosage <1%±0.1% will result in insufficient particle strength (<5 MPa±0.5 MPa); >7%±0.1% will result in increased residual carbon (>0.2%±0.01%) and sintered porosity >0.2%±0.02%.

Optimization: 2%-3%±0.1%, strength>12 MPa±1 MPa, residual carbon <0.1%±0.01%.

### (3) Types and characteristics of spray drying and granulation equipment

**Centrifugal spray dryer:**

Working principle: The high-speed rotating disk (1000-20000 rpm ± 50 rpm) throws the slurry into droplets, and hot air (200-300°C ± 5°C) dries it.

Features:

Particle size: 20-120 μm±0.1 μm, sphericity>90%±2%.

Output: 100-1000 kg/h±10 kg/h (depending on the disc diameter).

Advantages: Suitable for high viscosity slurry (<1000 mPa·s±50 mPa·s), narrow particle distribution ((D90-D10)/D50 <1.5±0.1).

Disadvantages: high speed (>15000 rpm±50 rpm), easy wear of disc (lifespan <500 hours±50 hours), high maintenance cost.

Application: Large-scale WC-Co powder granulation (D50=50 μm±0.1 μm) for aviation tools.

**Pressure spray dryer:**

Working principle: high pressure pump (0.1-0.3 MPa±0.01 MPa) atomizes through nozzles, hot air (150-250°C±5°C) dries.

Features:

Particle size: 30-150 μm±0.1 μm, sphericity>95%±2%.

Output: 50-500 kg/h±5 kg/h.

Advantages: Flexible nozzle design (single or multi-hole), suitable for low viscosity slurry (<500 mPa·s±50 mPa·s), low energy consumption (<60 kWh/t±5 kWh).

Disadvantages: Nozzles are easily clogged (clean every 100 hours ± 10 hours), and production is limited.

Application: Fine granulation of WC-Ni powder (D50=80 μm±0.1 μm), used for chemical pump body.

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#### **Air flow spray dryer:**

Working principle: Compressed air ( $0.2-0.4 \text{ MPa} \pm 0.01 \text{ MPa}$ ) is mixed with the slurry for atomization, and hot air ( $180-280^\circ\text{C} \pm 5^\circ\text{C}$ ) is used for drying.

Features:

Particle size:  $20-80 \mu\text{m} \pm 0.1 \mu\text{m}$ , sphericity  $>90\% \pm 2\%$ .

Output:  $30-300 \text{ kg/h} \pm 5 \text{ kg/h}$ .

Advantages: Suitable for fine powder dispersion (initial particle size  $<1 \mu\text{m} \pm 0.01 \mu\text{m}$ ), particle uniformity  $>96\% \pm 1\%$ , suitable for high purity requirements ( $O < 0.03\% \pm 0.005\%$ ).

Disadvantages: high energy consumption ( $>80 \text{ kWh/t} \pm 5 \text{ kWh}$ ), increased air compression costs.

Application: Nano WC powder granulation ( $D_{50}=30 \mu\text{m} \pm 0.1 \mu\text{m}$ ), used for PCB drill bits.

#### **Twin-fluid spray dryer:**

Working principle: Liquid and compressed air are co-atomized through a dual-fluid nozzle ( $0.1-0.3 \text{ MPa} \pm 0.01 \text{ MPa}$ ), and dried by hot air ( $200-300^\circ\text{C} \pm 5^\circ\text{C}$ ).

Features:

Particle size:  $10-100 \mu\text{m} \pm 0.1 \mu\text{m}$ , sphericity  $>92\% \pm 2\%$ .

Output:  $50-400 \text{ kg/h} \pm 5 \text{ kg/h}$ .

Advantages: The atomized particle size is controllable (adjusted by gas-liquid ratio), suitable for high solid content slurry ( $>30\% \pm 1\%$ ).

Disadvantages: The equipment is complex and requires frequent maintenance (check the nozzle every  $200 \text{ hours} \pm 20 \text{ hours}$ ).

Application: WC-Co mixed powder granulation ( $D_{50}=60 \mu\text{m} \pm 0.1 \mu\text{m}$ ), used for wear-resistant molds.

#### **Laboratory small spray dryer:**

Working principle: small centrifugal or pressure design (speed  $5000-10000 \text{ rpm} \pm 50 \text{ rpm}$ , pressure  $0.1-0.2 \text{ MPa} \pm 0.01 \text{ MPa}$ ), hot air  $150-200^\circ\text{C} \pm 5^\circ\text{C}$ .

Features:

Particle size:  $20-80 \mu\text{m} \pm 0.1 \mu\text{m}$ , output  $0.5-5 \text{ kg/h} \pm 0.1 \text{ kg/h}$ .

Advantages: Suitable for R&D and trial production, flexible parameter adjustment (temperature  $\pm 5^\circ\text{C}$ , flow rate  $\pm 0.1 \text{ L/h}$ ).

Disadvantages: low output and high cost (equipment price  $> 5000 \text{ USD} \pm 500 \text{ USD}$ ).

Application: Small batch WC-Ni powder testing ( $D_{50}=40 \mu\text{m} \pm 0.1 \mu\text{m}$ ).

#### **(4) Influencing factors**

##### **Slurry characteristics:**

Particle size: Initial powder  $<1 \mu\text{m} \pm 0.01 \mu\text{m}$  is easy to form uniform particles;  $>5 \mu\text{m} \pm 0.01 \mu\text{m}$  leads to coarse or irregular particles.

Viscosity:  $200-800 \text{ mPa}\cdot\text{s} \pm 50 \text{ mPa}\cdot\text{s}$  is optimal;  $>1000 \text{ mPa}\cdot\text{s} \pm 50 \text{ mPa}\cdot\text{s}$  will clog the nozzle.

##### **Binder Type:**

PVA ( $2\% \pm 0.1\%$ ) enhances strength ( $>12 \text{ MPa} \pm 1 \text{ MPa}$ ), but has high hygroscopicity ( $>2\% \pm 0.2\%$ ).

PEG ( $2\% \pm 0.1\%$ ) improved flowability ( $<20 \text{ s/50 g} \pm 2 \text{ s}$ ), but the carbon residue was slightly

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higher ( $<0.15\% \pm 0.01\%$ ).

Paraffin wax ( $1\%-2\% \pm 0.1\%$ ) is suitable for fluidity after drying, but it is highly volatile ( $>80^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ).

**Environmental conditions:**

Humidity  $<50\% \text{ RH} \pm 5\%$ , temperature  $<30^{\circ}\text{C} \pm 2^{\circ}\text{C}$ , to avoid premature volatilization of the binder or moisture absorption of the particles.

**Equipment factors:**

Nozzle wear (lifespan  $<500 \text{ hours} \pm 50 \text{ hours}$ ) causes uneven droplets and requires regular replacement.

**(5) Optimization strategy**

**Slurry preparation:**

Wet grinding ( $300-500 \text{ rpm} \pm 10 \text{ rpm}$ ,  $5-15 \text{ h} \pm 0.5 \text{ h}$ ) was used to prepare a homogeneous slurry (particle size  $<1 \mu\text{m} \pm 0.01 \mu\text{m}$ , viscosity  $300-500 \text{ mPa}\cdot\text{s} \pm 50 \text{ mPa}\cdot\text{s}$ ).

Ultrasonic dispersion ( $40 \text{ kHz} \pm 1 \text{ kHz}$ ,  $10 \text{ min} \pm 1 \text{ min}$ ), agglomeration rate  $<5\% \pm 1\%$ .

**Process parameter adjustment:**

Feed concentration is  $25\% \pm 1\%$ , inlet air temperature is  $220^{\circ}\text{C} \pm 5^{\circ}\text{C}$ , atomization pressure is  $0.2 \text{ MPa} \pm 0.01 \text{ MPa}$ , and particle size is  $50-80 \mu\text{m} \pm 0.1 \mu\text{m}$ .

Binder:  $2\% \pm 0.1\%$  PVA +  $1\% \pm 0.1\%$  PEG, strength  $>12 \text{ MPa} \pm 1 \text{ MPa}$ , fluidity  $<20 \text{ s/50 g} \pm 2 \text{ s}$ .

**Equipment maintenance:**

Clean the nozzle (once a week, use ethanol, purity  $>99.5\% \pm 0.1\%$ ) to prevent clogging.

Replace the atomizing disk or nozzle (every  $500 \text{ hours} \pm 50 \text{ hours}$ ), the speed deviation is  $<5\% \pm 1\%$ .

**Post-processing:**

Sieve ( $100-150 \mu\text{m} \pm 0.1 \mu\text{m}$ ) to remove oversized particles ( $<5\% \pm 1\%$ ).

Vacuum drying ( $80^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ,  $<10^{-2} \text{ Pa} \pm 10^{-3} \text{ Pa}$ ,  $4-6 \text{ hours} \pm 0.5 \text{ hours}$ ), residual water  $<0.1\% \pm 0.01\%$ .

**(6) Application effect**

**Fluidity and compression properties:**

Fluidity  $<20 \text{ s/50 g} \pm 2 \text{ s}$ , green body density  $>60\% \pm 1\%$  (theoretical density), pressing defects  $<1\% \pm 0.2\%$ .

Example: WC-10%Co ( $D_{50}=50 \mu\text{m} \pm 0.1 \mu\text{m}$ ), aviation tool blank life  $>15 \text{ hours} \pm 1 \text{ hour}$ .

**Sintering performance:**

Density  $>99\% \pm 0.1\%$ , porosity  $<0.05\% \pm 0.01\%$ , hardness HV  $>2900 \pm 50$ , flexural strength  $>4200 \text{ MPa} \pm 100 \text{ MPa}$ .

Example: WC-12%Ni ( $D_{50}=80 \mu\text{m} \pm 0.1 \mu\text{m}$ ), chemical pump body life  $>2 \text{ years} \pm 0.2 \text{ years}$ .

**consistency:**

The particle size deviation between batches is  $<5\% \pm 1\%$ , the uniformity is  $>95\% \pm 1\%$ , and the sintering defects are reduced by  $50\% \pm 5\%$ .

**(7) Testing and quality control**

Particle size distribution: laser particle size analysis (GB/T 19077.1-2008),  $D_{50} 50-100 \mu\text{m} \pm 0.1 \mu\text{m}$ ,

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(D90-D10)/D50 <1.5±0.1.

Morphological analysis: SEM (GB/T 16594-2008), sphericity>90%±2%, agglomeration rate<5%±1%.

Moisture content: Karl Fischer method (GB/T 6283-2008), residual water <0.2%±0.05%.

Strength test: compressive strength (GB/T 3851-2015), >10 MPa±1 MPa.

Residual carbon detection : infrared absorption method (GB/T 5124-2017), residual carbon <0.1%±0.01%.

Online monitoring: Infrared thermal imaging monitors the intake air temperature (deviation <5°C±1°C), and flow meter monitors the feed (deviation <1%±0.1%).

Spray drying and granulation technology converts fine powders such as WC and Co into 20-150 μm±0.1 μm particles through atomization (pressure 0.2-0.25 MPa±0.01 MPa) and high temperature drying (200-250°C±5°C), significantly improving fluidity (<20 s/50 g±2 s) and green body strength (>10 MPa±1 MPa). Equipment types include centrifugal (high output, 100-1000 kg/h±10 kg/h), pressure (high sphericity, 50-500 kg/h±5 kg/h), airflow (fine particles, 30-300 kg/h±5 kg/h), dual-fluid (controllable particle size, 50-400 kg/h±5 kg/h) and laboratory small machine (for research and development, 0.5-5 kg/h±0.1 kg/h), each with its own unique advantages. Optimizing feed concentration (25%-30%±1%), binder dosage (2%-3%±0.1%) and equipment maintenance can ensure particle uniformity (>95%±1%) and sintering performance (density>99%±0.1%). It is widely used in high-end fields such as aviation tools (lifespan>15 hours±1 hour), chemical equipment (lifespan>2 years±0.2 years), etc.

#### 4.4 Powder characterization

Powder characterization The quality of the mixed powder is evaluated by Fisher particle size (FSSS, 0.250μm±0.01μm), loose density, tap density (4.06.2 g/cm<sup>3</sup>±0.1 g/cm<sup>3</sup>) and flowability (1316 seconds/50g±0.5 seconds) to ensure sintering performance (density>99%±0.1%, hardness deviation<±30 HV). The characterization method is based on particle dynamics (Stokes sedimentation, HagenPoiseuille flow) and complies with ISO 4499 and ASTM B330 standards.

##### 4.4.1 Fisher particle size (FSSS, 0.250 μm)

Fisher Sub-Sieve Sizer (FSSS) is a traditional method for determining the average size of powder particles by air permeation method. It is widely used for particle size analysis of cemented carbide raw materials (such as tungsten carbide powder WC). It is based on the relationship between the resistance of the powder layer to air and the particle size. By measuring the air flow permeability under a certain pressure, the average particle size (usually in microns) is calculated. Fisher particle size is suitable for fine particles (0.1-50 μm ). The results reflect the surface area and pore characteristics of the powder, and have important reference value for the sintering performance and pressing behavior of cemented carbide.

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### Characterization methods and significanceFisher

's particle size (FSSS) was measured by air permeability method to measure the average particle size of the powder ( $0.250\mu\text{m}\pm 0.01\mu\text{m}$ ), based on Darcy's law (permeability  $\sim 10^{-12} \text{ m}^2 \pm 10^{-13} \text{ m}^2$ ). The FSSS of the mixed powder containing  $10\%\pm 1\%$  Co is  $0.55\mu\text{m}\pm 0.01\mu\text{m}$ , WC  $0.33\mu\text{m}\pm 0.01\mu\text{m}$ , and Co  $0.51\mu\text{m}\pm 0.01\mu\text{m}$ . Fine FSSS ( $<0.5\mu\text{m}\pm 0.01\mu\text{m}$ ) improves hardness ( $\text{HV}>2200\pm 30$ ), and large particle size ( $>5\mu\text{m}\pm 0.01\mu\text{m}$ ) enhances toughness ( $K_{IC}>18 \text{ MPa}\cdot\text{m}^{1/2}\pm 0.5$ ). Test conditions: sample mass  $5 \text{ g}\pm 0.1 \text{ g}$ , pressure  $0.1 \text{ MPa}\pm 0.01 \text{ MPa}$ , air purity  $>99.9\%\pm 0.01\%$ , error  $<2\%\pm 0.5\%$ .

For example, FSSS  $0.3\mu\text{m}\pm 0.01\mu\text{m}$  powder is used for aviation cutting tools (wear  $<0.08\text{mm}\pm 0.02\text{mm}$ ), hardness HV  $2300\pm 30$ , life  $>15\text{h}\pm 1\text{h}$ ; FSSS  $5\mu\text{m}\pm 0.01\mu\text{m}$  is used for mining drill bits ( $K_{IC} 20 \text{ MPa}\cdot\text{m}^{1/2}\pm 0.5$ ), life  $>1200 \text{ m}\pm 100 \text{ m}$ .

### Influencing factors and optimization

FSSS measurement is affected by the following factors:

Powder composition: WC/Co= $90:10\pm 1\%$ , FSSS  $0.5\mu\text{m}\pm 0.01\mu\text{m}$ ; WC/Ni= $88:12\pm 1\%$ , FSSS  $1\mu\text{m}\pm 0.01\mu\text{m}$ , because the Ni particle size is larger ( $>2\mu\text{m}\pm 0.01\mu\text{m}$ ).

Ball milling time:  $12\text{h}\pm 0.1\text{h}$ , FSSS decreased by  $10\%\pm 2\%$  ( $0.5\mu\text{m}\pm 0.01\mu\text{m}$ );  $>24\text{h}$  increased agglomeration ( $>5\%\pm 1\%$ ), and FSSS increased by  $5\%\pm 1\%$ .

Additives:  $0.5\%\pm 0.01\%$  VC reduces FSSS by  $5\%\pm 1\%$  ( $0.3\mu\text{m}\pm 0.01\mu\text{m}$ ),  $\text{Cr}_3\text{C}_2$  affects  $<2\%\pm 0.5\%$ .

Ambient humidity:  $<50\%\pm 5\%$ , avoid agglomeration ( $<5\%\pm 1\%$ ), FSSS error  $<1\%\pm 0.2\%$ .

Equipment calibration: FSSS instrument porosity error  $<0.1\%\pm 0.02\%$ , error reduction  $1\%\pm 0.2\%$ .

For example, FSSS  $0.3\mu\text{m}\pm 0.01\mu\text{m}$  (12 hours wet grinding,  $0.5\%\pm 0.01\%$  VC) powder is used for PCB drill bits (lifetime  $>10^5$  holes  $\pm 10^4$  holes).

### Fisher's particle size (FSSS ) engineering application

#### Superhard cutting tools

FSSS  $0.20.5\mu\text{m}\pm 0.01\mu\text{m}$ , hardness HV $>2300\pm 30$ , aviation processing life $>15$  hours $\pm 1$  hour.

#### mining

FSSS  $35\mu\text{m}\pm 0.01\mu\text{m}$ ,  $K_{IC} > 20\text{MPa}\cdot\text{m}^{1/2} \pm 0.5$ , drilling life $>1200\text{m}\pm 100\text{m}$ .

### 4.4.2 Bulk density, tap density and fluidity of tungsten carbide powder

The bulk density, tap density and fluidity of tungsten carbide powder (WC) are important indicators of its physical properties, which directly affect the pressing and sintering behavior of cemented carbide and the performance of the final product. The following is a detailed analysis from the aspects of definition, measurement method, influencing factors, optimization measures and engineering application.

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### **Bulk density of tungsten carbide powder**

Loose density refers to the density of powder in its natural stacking state, reflecting the stacking characteristics and porosity of the powder.

Measurement method

According to ASTM B212, 50 g  $\pm$  0.1 g of powder was freely dropped into a standard measuring cylinder (volume 25 mL  $\pm$  0.1 mL) and the ratio of mass to volume was calculated.

Typical Value

The bulk density of pure WC powder is 4.0-5.0 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>, and the porosity is about 40% $\pm$ 2%. The bulk density of WC-Co mixed powder containing 10% $\pm$ 1% Co is about 4.5 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>, because the density of Co (8.9 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>) is higher than that of WC (15.63 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>), and the stacking characteristics change after mixing.

significance

The high bulk density (>4.5 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>) indicates that the particles are closely arranged and the porosity of the pressed green body is low (<40% $\pm$ 2%), which is conducive to sintering densification (>99% $\pm$ 0.1%).

### **Tap density of tungsten carbide powder**

Tap density refers to the density of powder in a densely packed state after vibration or tapping, which reflects the filling efficiency between particles.

Measurement method

According to ASTM B527, the volume change of powder was measured using a tap density meter (vibration frequency 50 Hz  $\pm$  1 Hz, amplitude 1 mm  $\pm$  0.1 mm, vibration 3000 times  $\pm$  50 times).

Typical Value

The tap density of pure WC powder is 5.0-6.2 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>, and the porosity is reduced to 30% $\pm$ 2%. The tap density of mixed powder containing 10% $\pm$ 1% Co is about 5.5 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>.

significance

The high tap density (>5.5 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>) indicates that the particles can be further filled, the compact has good consistency (dimensional deviation <0.01 mm $\pm$ 0.002 mm), and the properties after sintering are stable (hardness HV >2900 $\pm$ 50).

Example: The tap density of the powder is 5.8 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>. After pressing, the green body size deviation is <0.01 mm $\pm$ 0.002 mm. The aviation tool is made with a hardness of HV 2200 $\pm$ 30 and a service life of >12 hours $\pm$ 1 hour.

### **Fluidity of tungsten carbide powder**

Fluidity reflects the flow ability of powder during compaction, affecting mold filling uniformity and product quality.

Measurement method

According to ASTM B213, a Hall flow meter (funnel aperture 5 mm  $\pm$  0.1 mm) was used to measure the time required for 50 g  $\pm$  0.1 g of powder to pass through the funnel. The flow behavior conforms to the Hagen-Poiseuille law, and the viscous resistance is approximately 10<sup>-3</sup> Pa·s $\pm$ 10<sup>-4</sup> Pa·s.

Typical Value

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The fluidity of WC powder is 13-16 seconds/50 g $\pm$ 0.5 seconds, and the fluidity of mixed powder containing 10% $\pm$ 1% Co is about 14 seconds/50 g $\pm$ 0.5 seconds.

significance

Excellent fluidity (<14 sec/50 g $\pm$ 0.5 sec) ensures uniform mold filling, density after sintering >99.5% $\pm$ 0.1%, and reduces molding defects (cracks <1% $\pm$ 0.2%).

Example: The powder with fluidity of 13 seconds/50 g $\pm$ 0.5 seconds has a uniformity of >98% $\pm$ 1% after pressing and is used for tool production with a dimensional deviation of <0.01 mm $\pm$ 0.002 mm.

## Influencing factors and optimization of tungsten carbide powder particle size

### Particle size (Freshman size sizing, FSSS):

the Fisher particle size is <0.5  $\mu\text{m}$  $\pm$ 0.01  $\mu\text{m}$ , the van der Waals force between particles is enhanced (>10<sup>-9</sup> N $\pm$ 10<sup>-10</sup> N), the bulk density drops to 4.2 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>, and the fluidity decreases by 2 seconds  $\pm$  0.5 seconds (16 seconds/50 g $\pm$ 0.5 seconds). When the Fisher particle size is >5  $\mu\text{m}$  $\pm$ 0.01  $\mu\text{m}$ , the gap between particles decreases, the bulk density increases to 5.0 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>, and the fluidity increases by 1 second  $\pm$  0.2 seconds (13 seconds/50 g $\pm$ 0.5 seconds).

Optimization: Control the particle size of the fiberglass at 0.5-3  $\mu\text{m}$   $\pm$  0.01  $\mu\text{m}$ , and balance the density and fluidity (loose density 4.5 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>, fluidity 14 s/50 g  $\pm$  0.5 s).

### Appearance:

Spherical particles (spheroidization rate>95% $\pm$ 1%) have a low surface friction coefficient (<0.2 $\pm$ 0.02), a 5% $\pm$ 1% increase in tap density (6.0 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>), and a 3% $\pm$ 0.5% increase in fluidity (13 seconds/50 g $\pm$ 0.5 seconds). Irregular particles (edges >0.1  $\mu\text{m}$  $\pm$ 0.01  $\mu\text{m}$ ) have large stacking gaps and a 3% $\pm$ 0.5% decrease in tap density (5.2 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>).

Optimization: Spray drying granulation (particle size 50  $\mu\text{m}$   $\pm$  0.1  $\mu\text{m}$ , sphericity > 90%  $\pm$  2%) was used to improve the consistency of particle morphology.

### Co content:

When the Co content is 10% $\pm$ 1%, the tap density is 5.5 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup> and the fluidity is 14 sec/50 g $\pm$ 0.5 sec. When the Co content is >15% $\pm$ 1%, the Co particles (density 8.9 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>) are unevenly distributed, the tap density drops to 5.2 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>, and the fluidity decreases by 1 sec $\pm$ 0.2 sec (15 sec/50 g $\pm$ 0.5 sec).

Optimization: The Co content is controlled at 8%-12% $\pm$ 1% to ensure optimal density and fluidity (tapped density 5.5-5.8 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>, fluidity <14 seconds/50 g $\pm$ 0.5 seconds).

### humidity:

When the ambient humidity is <50% $\pm$ 5% RH, the fluidity is maintained at 14 sec/50 g $\pm$ 0.5 sec. When the humidity is >80% $\pm$ 5% RH, water adsorption causes the agglomeration rate to increase by 10% $\pm$ 2% and the fluidity decreases by 2 sec $\pm$ 0.5 sec (16 sec/50 g $\pm$ 0.5 sec).

Optimization: The processing environment humidity is controlled at 40%-50% $\pm$ 5% RH, and the powder is dried to a moisture content of <0.1% $\pm$ 0.01% (Karl Fischer method, GB/T 6283-2008).

### Granulation process:

Spray drying granulation (particles 50  $\mu\text{m}$   $\pm$  0.1  $\mu\text{m}$ , sphericity > 90%  $\pm$  2%) increased the tap density by 10%  $\pm$  2% (6.0 g/cm<sup>3</sup>  $\pm$  0.1 g/cm<sup>3</sup>) and the fluidity by 5%  $\pm$  1% (13 seconds/50 g  $\pm$  0.5

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seconds). The ungranulated powder (initial particle size  $<1 \mu\text{m} \pm 0.01 \mu\text{m}$ ) was severely agglomerated, and the tap density was only  $5.0 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$ .

Optimization: Use a centrifugal spray dryer (rotation speed  $10000\text{-}15000 \text{ rpm} \pm 50 \text{ rpm}$ ), particle size  $50\text{-}80 \mu\text{m} \pm 0.1 \mu\text{m}$ , add  $2\% \pm 0.1\%$  PVA binder.

Example: A powder containing  $10\% \pm 1\%$  Co, Fisher particle size  $1 \mu\text{m} \pm 0.01 \mu\text{m}$ , spray-dried particles ( $50 \mu\text{m} \pm 0.1 \mu\text{m}$ ), tap density  $6.0 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$ , fluidity  $13 \text{ seconds}/50 \text{ g} \pm 0.5 \text{ seconds}$ , density after pressing  $>99.5\% \pm 0.1\%$ , made into a mining drill bit with a life of  $>1200 \text{ m} \pm 100 \text{ m}$ .

## Engineering Application

### Aviation knives:

Tap density  $5.8 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$ , fluidity  $13 \text{ seconds}/50 \text{ g} \pm 0.5 \text{ seconds}$ , pressed green body size deviation  $<0.01 \text{ mm} \pm 0.002 \text{ mm}$ , hardness after sintering  $\text{HV } 2200 \pm 30$ , life  $>12 \text{ hours} \pm 1 \text{ hour}$ , suitable for aviation material processing (Ti-6Al-4V alloy,  $1000^\circ\text{C} \pm 10^\circ\text{C}$ ).

### Mining Drill:

Tap density  $6.0 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$ , fluidity  $13 \text{ seconds}/50 \text{ g} \pm 0.5 \text{ seconds}$ , sintered density  $>99.5\% \pm 0.1\%$ , flexural strength  $>4200 \text{ MPa} \pm 100 \text{ MPa}$ , life  $>1200 \text{ m} \pm 100 \text{ m}$ , suitable for hard rock drilling (compression resistance  $>200 \text{ MPa} \pm 10 \text{ MPa}$ ).

### Cold heading die:

Tap density  $6.2 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$ , fluidity  $14 \text{ s}/50 \text{ g} \pm 0.5 \text{ s}$ , deformation after pressing  $<0.01 \text{ mm} \pm 0.002 \text{ mm}$ , mold life  $>10^6 \text{ times} \pm 10^5 \text{ times}$ , suitable for high-precision cold heading.

## Testing and quality control

Bulk density: According to ASTM B212, each batch is tested 3 times, and the average value deviation is  $<2\% \pm 0.5\%$ .

Tap density: According to ASTM B527, record vibration parameters (frequency  $50 \text{ Hz} \pm 1 \text{ Hz}$ , amplitude  $1 \text{ mm} \pm 0.1 \text{ mm}$ ), deviation  $<2\% \pm 0.5\%$ .

Flowability: Based on ASTM B213, funnel aperture calibration ( $5 \text{ mm} \pm 0.1 \text{ mm}$ ), test environment humidity  $<50\% \pm 5\% \text{ RH}$ , deviation  $<1 \text{ second} \pm 0.2 \text{ seconds}$ .

Online monitoring: Use automatic tap density meter and flow meter to record batch data in real time and archive it for  $1 \text{ year} \pm 0.1 \text{ year}$ .

The bulk density ( $4.0\text{-}5.0 \text{ g/cm}^3$ ), tap density ( $5.0\text{-}6.2 \text{ g/cm}^3$ ) and fluidity ( $13\text{-}16 \text{ seconds}/50 \text{ g}$ ) of tungsten carbide powder are key parameters for cemented carbide preparation. The performance is significantly affected by the particle size ( $0.5\text{-}5 \mu\text{m}$ ), morphology (sphericity  $>95\%$ ), Co content ( $8\%\text{-}12\%$ ), humidity ( $<50\% \text{ RH}$ ) and granulation process (particles  $50 \mu\text{m}$ ). By optimizing the particle size, morphology and process parameters, a tap density of  $6.0 \text{ g/cm}^3$  and a fluidity of  $13 \text{ seconds}/50 \text{ g}$  can be achieved, meeting the high performance requirements of aviation tools (lifetime  $>12 \text{ hours}$ ), mining drill bits (lifetime  $>1200 \text{ m}$ ) and cold heading dies (deformation  $<0.01 \text{ mm}$ ).

## 4.5 Summary and Outlook

Raw material selection and powder preparation of cemented carbide are the key links in performance

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optimization. This chapter explains the relationship between process parameters and performance by analyzing tungsten carbide powder synthesis ( $1450 \pm 1600^\circ\text{C}$ , particle size  $0.110 \mu\text{m} \pm 0.01 \mu\text{m}$ , free carbon  $< 0.1\% \pm 0.01\%$ ), bonding phase and additives (Co/Ni purity  $> 99.8\% \pm 0.01\%$ , VC/  $\text{Cr}_3\text{C}_2$   $< 1\% \pm 0.01\%$ ), powder pretreatment (ball milling  $10:1 \pm 0.5$ , spray drying flow rate  $100 \text{ L/h} \pm 10 \text{ L/h}$ ) and powder characterization (FSSS  $0.250 \mu\text{m} \pm 0.01 \mu\text{m}$ , tap density  $4.06.2 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$ , fluidity  $1316 \text{ seconds/50g} \pm 0.5 \text{ seconds}$ ):

### WC powder

Submicron particle size ( $< 0.5 \mu\text{m} \pm 0.01 \mu\text{m}$ ) and high purity (free carbon  $< 0.08\% \pm 0.01\%$ ) increase hardness ( $\text{HV} > 2300 \pm 30$ ) for aviation tools (lifespan  $> 15 \text{h} \pm 1 \text{h}$ ).

### Bonding phase

Co ( $10\% \pm 1\%$ ) provides toughness ( $K_{IC} 1520 \text{ MPa} \cdot \text{m}^{1/2} \pm 0.5$ ), and Ni ( $12\% \pm 1\%$ ) enhances corrosion resistance ( $< 0.01 \text{ mm/year} \pm 0.002 \text{ mm/year}$ ), and is used for deep-sea valves (lifespan  $> 5 \text{ years} \pm 0.5 \text{ years}$ ).

### Additive

VC ( $0.5\% \pm 0.01\%$ ) controls WC particle size ( $< 0.3 \mu\text{m} \pm 0.01 \mu\text{m}$ ),  $\text{Cr}_3\text{C}_2$  ( $0.5\% \pm 0.01\%$ ) improves strength ( $> 4200 \text{ MPa} \pm 100 \text{ MPa}$ ) and is used for PCB drill bits ( $> 10^5 \text{ holes} \pm 10^4 \text{ holes}$ ).

### Preprocessing

Wet grinding ( $12 \text{h} \pm 0.1 \text{h}$ ) and spray drying ( $250^\circ\text{C} \pm 5^\circ\text{C}$ ) ensure uniformity (deviation  $< 2\% \pm 0.5\%$ ) and flowability ( $13 \text{s/50g} \pm 0.5 \text{s}$ ) for use in mining drill bits (density  $> 99.5\% \pm 0.1\%$ ).

### Characterization

The FSSS ( $0.35 \mu\text{m} \pm 0.01 \mu\text{m}$ ) and tap density ( $5.86.2 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$ ) predict the sintering quality with reduced hardness deviation ( $< \pm 30 \text{ HV}$ ).

### Optimization strategies include

Accurate W:C ratio ( $1:1.01 \pm 0.01$ ),  $\text{H}_2$  atmosphere ( $\text{O}_2 < 10 \text{ ppm} \pm 1 \text{ ppm}$ ), fine Co ( $< 1 \mu\text{m} \pm 0.01 \mu\text{m}$ ), VC addition ( $0.3\% \pm 0.5\% \pm 0.01\%$ ), wet grinding ( $10:1 \pm 0.5$ ,  $12 \text{h} \pm 0.1 \text{h}$ ) and spray drying ( $60\% \pm 1\%$  solid content).

For example, powder containing  $0.3 \mu\text{m} \pm 0.01 \mu\text{m}$  WC,  $10\% \pm 1\%$  Co, and  $0.5\% \pm 0.01\%$  VC (FSSS  $0.3 \mu\text{m} \pm 0.01 \mu\text{m}$ , tap density  $6.0 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$ , fluidity  $13 \text{ seconds/50g} \pm 0.5 \text{ seconds}$ ) is used for aviation tools, with hardness  $\text{HV} 2300 \pm 30$ , wear amount  $< 0.08 \text{ mm} \pm 0.02 \text{ mm}$ , and life  $> 15 \text{ hours} \pm 1 \text{ hour}$ ; powder containing  $1 \mu\text{m} \pm 0.01 \mu\text{m}$  WC,  $12\% \pm 1\%$  Ni, and  $0.5\% \pm 0.01\%$   $\text{Cr}_3\text{C}_2$  is used for deep-sea valves, with corrosion depth  $< 3 \mu\text{m} \pm 0.5 \mu\text{m}$  and life  $> 5 \text{ years} \pm 0.5 \text{ years}$ .

Future research directions include large-scale production of nano WC powder ( $< 0.1 \mu\text{m} \pm 0.01 \mu\text{m}$ ) (yield  $> 5 \text{ t/batch} \pm 0.5 \text{ t}$ ), green bonding phase (Fe-based, cost  $< \$1000/\text{t} \pm 100 \text{ USD}$ ), new inhibitors

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(such as TaC ,  $<0.5\%\pm 0.01\%$ ) and intelligent characterization (AI prediction of FSSS, error $<1\%\pm 0.2\%$ ) to meet the needs of aviation (cutting speed $>500\text{ m/min}\pm 10\text{ m/min}$ ), deep sea ( $>10000\text{ m}$ ) and new energy (electrolyzer life $>10^4\text{ hours}\pm 10^3\text{ hours}$ ). This chapter provides a process basis for forming and sintering in Chapter 5 by correlating the WCCo phase contribution with the performance in Chapter 3.

## Appendix: Reference table for selection of cemented carbide products and tungsten carbide powder

product type	product Example	Performance requirements	WC Powder Characteristics	WC powder selection parameters	Applicable scenarios	illustrate
Cutting blade	Turning inserts, milling inserts, grooving inserts	High hardness (HRA 8993 ±0.5), excellent wear resistance (flank wear VB <0.3 mm), chipping resistance (chipping depth <0.15 mm), surface roughness Ra <0.8 μm	Fine grain (0.51.5 μm ±0.1 μm), high purity (>99.95% ±0.01%), low oxygen content (<0.1% ±0.01%), uniform particle distribution (D50 ±0.1 μm)	Particle size: 0.51.5 μm ±0.1 μm, purity: >99.95% ±0.01%, carbon content: 6.13%6.18% ±0.01%, Co content: 6%12% ±0.5%, additives: TiC / TaC (0%2% ±0.1%)	Steel, stainless steel, cast iron processing, cutting speed 100400 m/min ±10 m/min	Fine-grained WC powder ensures high hardness and wear resistance, TiC / TaC improves high temperature performance, and Co content balances toughness and hardness; suitable for ISO P/M/K type inserts.
Overall tool	Drills, end mills, reamers	High flexural strength (20003000 MPa ±100 MPa), excellent toughness (fracture toughness 812 MPa·m <sup>1/2</sup> ± 0.5), impact resistance (impact toughness > 10 J/ cm <sup>2</sup> ± 1 J/ cm <sup>2</sup> )	Medium-fine grain (1.02.0 μm ±0.1 μm), high purity (>99.9% ±0.01%), moderate oxygen content (<0.15% ±0.01%), stable chemical composition	Particle size: 1.02.0 μm ±0.1 μm, purity: >99.9% ±0.01%, carbon content: 6.10%6.15% ±0.01%, Co content: 8%15% ±0.5%, additives: VC/ Cr <sub>3</sub> C <sub>2</sub> (0.1%0.5% ±0.05%)	Mold steel, aluminum alloy processing, drilling/milling depth <20 mm ±1 mm	Medium-fine grain WC improves toughness, VC/ Cr <sub>3</sub> C <sub>2</sub> controls grain growth and ensures overall tool strength; suitable for high-load processing environments.
Wire drawing dies	Wire drawing die, extrusion die	Very high wear resistance (wear rate <0.01 mm <sup>3</sup> / h ± 0.001 mm <sup>3</sup> / h), high surface finish (Ra <0.05 μm), corrosion resistance (acid and alkali resistance pH 310)	Ultrafine grain (0.20.8 μm ±0.1 μm), ultra-high purity (>99.98% ±0.01%), extremely low oxygen content (<0.05% ±0.01%), high uniformity	Particle size: 0.20.8 μm ±0.1 μm, purity: >99.98% ±0.01%, carbon content: 6.15%6.20% ±0.01%, Co content: 3%6% ±0.5%, additives: no or a small amount of TaC (<0.5% ±0.05%)	Copper and steel wire drawing, wire diameter 0.15 mm ± 0.01 mm	Ultrafine grain WC provides mirror finish and wear resistance, low Co content enhances hardness; powder purity needs to be strictly controlled to prevent impurities.
Stamping Die	Cold heading die, punching die	High compressive strength (>4000 MPa ±100 MPa), impact resistance (impact number >10 <sup>6</sup> ±10 <sup>4</sup> ), fatigue resistance (fatigue strength >1000 MPa ±50 MPa)	Medium coarse grain (2.04.0 μm ±0.2 μm), high purity (>99.9% ±0.01%), moderate oxygen content (<0.2% ±0.01%), high stability	Particle size: 2.04.0 μm ±0.2 μm, purity: >99.9% ±0.01%, carbon content: 6.08%6.13% ±0.01%, Co content: 10%20% ±0.5%, additives: Cr <sub>3</sub> C <sub>2</sub> (0.2%1% ±0.05%)	Bolts, sheet metal stamping, thickness <10 mm ±0.1 mm	Medium-coarse grain WC improves impact resistance, high Co content enhances toughness, and Cr <sub>3</sub> C <sub>2</sub> inhibits grain growth; suitable for high stress molds.
Wear-	Nozzle,	High wear resistance (wear	Coarse grain (3.06.0 μm	Particle size: 3.06.0 μm ±0.5	Sand blasting,	Coarse-grained WC

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product type	product Example	Performance requirements	WC Powder Characteristics	WC powder selection parameters	Applicable scenarios	illustrate
resistant parts	sealing ring, top hammer	loss <0.005 mm <sup>3</sup> / N · m ± 0.001 mm <sup>3</sup> / N · m ) , corrosion resistance (wear life >5000 h ±100 h), thermal stability (<800°C ±10°C)	±0.5 μm ), purity (>99.8% ±0.01%), high oxygen content (<0.3% ±0.01%), high chemical stability	μm , purity: >99.8% ±0.01%, carbon content: 6.05%6.10% ±0.01%, Co content: 12%25% ±0.5%, additives: no or small amount of Ni (<2% ±0.1%)	mining, sealing, working pressure <50 MPa ±1 MPa	enhances wear resistance and toughness, and high Co or Ni improves corrosion resistance; suitable for wear-resistant parts under extreme working conditions.
Mining tools	Picks, rock drill bits	Very high impact resistance (impact toughness>15 J/cm <sup>2</sup> ± 1 J/cm <sup>2</sup> ), wear resistance (wear rate <0.02 mm <sup>3</sup> / h ± 0.002 mm <sup>3</sup> /h), fatigue resistance	Ultra-coarse grain (6.010.0 μm ±1.0 μm ), purity (>99.7% ±0.01%), high oxygen content (<0.4% ±0.01%), high toughness	Particle size: 6.010.0 μm ±1.0 μm , purity: >99.7% ±0.01%, carbon content: 6.00%6.10% ±0.01%, Co content: 15%30% ±1%, additives: no or a small amount of CoNi alloy (<5% ±0.2%)	Coal mining, hard rock mining, impact frequency <100 Hz ±5 Hz	Ultra-coarse-grained WC provides high toughness, and high Co content enhances impact resistance; it is suitable for heavy-load mining environments, and the oxygen content needs to be controlled to prevent embrittlement.
Precision Parts	Micro tools, valve cores	High precision (dimensional tolerance ±0.005 mm), excellent surface finish (Ra <0.02 μm ), high hardness (HRA 9294 ±0.5)	Nano-crystals (0.10.4 μm ±0.05 μm ), ultra-high purity (>99.99% ±0.01%), extremely low oxygen content (<0.03% ±0.01%), and extremely high uniformity	Particle size: 0.10.4 μm ±0.05 μm , purity: >99.99% ±0.01%, carbon content: 6.18%6.22% ±0.01%, Co content: 2%5% ±0.3%, additives: VC (0.05%0.2% ±0.02%)	Electronic parts processing, fluid control, VC size <10 mm ±0.1 mm	Nanocrystalline WC ensures ultra-high hardness and smoothness, low Co and VC control grain growth; high-purity powder is required to prevent defects.

Reference table for selection of cemented carbide products and production processes

product type	Product Examples	Performance requirements	Recommended pressing process	Recommended Knot Craft	Process parameters	Applicable scenarios	illustrate
Cutting inserts	Turning inserts, milling inserts, grooving inserts	High hardness (HRA 8993 ±0.5), wear resistance (flank wear VB <0.3)	Bidirectional compression molding, cold isostatic pressing	Vacuum sintering, low pressure sintering	Pressing: pressure 100250 MPa ±10 MPa, time 1040 s ±1 s, green body density 60%75% ±2% (bidirectional molding); pressure 100300 MPa ±10	Steel, stainless steel, cast iron processing, cutting speed 100400 m/min or low-pressure	Bidirectional molding or CIP ensures uniform density, vacuum

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product type	Product Examples	Performance requirements	Recommended pressing process	Recommended Knot Craft	Process parameters	Applicable scenarios	illustrate
		mm), chipping resistance ( chipping depth <0.15 mm), surface roughness Ra <0.8 μm			MPa, holding pressure 15 min ±10 s, green body density 70%85% ±1% (CIP) Sintering: temperature 13501500°C ±10°C, holding temperature 14 h ±5 min, vacuum degree 0.010.1 Pa ±0.01 Pa, density 98%99.5% ±0.5% (vacuum); pressure 110 MPa ±0.05 MPa, density 98.5%99.5% ±0.3% (low pressure)	±10 m/min	sintering improves hardness and wear resistance; suitable for ISO P/M/K blades, grain size 0.51.5 μm ±0.1 μm .
Overall tool	Drills, end mills, reamers	High flexural strength (20003000 MPa ±100 MPa), toughness (fracture toughness 812 MPa·m <sup>1/2</sup> ±0.5), impact resistance (impact toughness> 10 J/cm <sup>2</sup> ± 1 J/cm <sup>2</sup> )	Extrusion molding, cold isostatic pressing	Hot isostatic pressing (HIP), vacuum sintering	Pressing: extrusion pressure 20100 MPa ±5 MPa, speed 0.11 m/min ±0.01 m/min, billet density 50%65% ±2% (extrusion); pressure 100300 MPa ±10 MPa, holding pressure 15 min ±10 s, billet density 70%85% ±1% (CIP) Sintering: temperature 13001450°C ±10°C, pressure 100200 MPa ±0.1 MPa, holding temperature 13 h ±5 min, density 99.8%100% ±0.2% (HIP); temperature 13501500°C ±10°C, holding temperature 14 h ±5 min, density 98%99.5% ±0.5% (vacuum)	Mold steel, aluminum alloy processing, drilling/milling depth <20 mm ±1 mm	Extrusion molding is suitable for bars, CIP is suitable for complex shapes; HIP improves strength, vacuum sintering has low cost; Co content is 8%15% ±0.5%.
Wire drawing dies	Wire drawing die, extrusion die	Very high wear resistance (wear rate <0.01 mm <sup>3</sup> / h ± 0.001 mm <sup>3</sup> / h), surface finish (Ra <0.05 μm ), corrosion resistance (acid	Powder injection molding, cold isostatic pressing	Microwave sintering, vacuum sintering	Pressing: injection pressure 50150 MPa ±5 MPa, temperature 150200°C ±5°C, billet shrinkage 15%20% ±1% (PIM); pressure 100300 MPa ±10 MPa, holding pressure 15 min ±10 s, billet density 70%85% ±1% (CIP)<	Copper and steel wire drawing, wire diameter 0.15 mm ± 0.01 mm	PIM is suitable for precision molds, CIP ensures uniformity; microwave sintering is efficient,

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product type	Product Examples	Performance requirements	Recommended pressing process	Recommended Knot Craft	Process parameters	Applicable scenarios	illustrate
		and alkali resistance pH 310)			br >Sintering: temperature 13001450°C±10°C, time 1060 min ±1 min, density 97%99% ±0.5% (microwave); temperature 13501500°C ±10°C, holding temperature 14 h ±5 min, density 98%99.5% ±0.5% (vacuum)		vacuum sintering ensures smoothness; ultra-fine WC powder 0.20.8 μm ±0.1 μm .
Stamping Die	Cold heading die, punching die	High compressive strength (>4000 MPa ±100 MPa), impact resistance (impact number >10 <sup>6</sup> ±10 <sup>4</sup> ), fatigue resistance (fatigue strength >1000 MPa ±50 MPa)	One-way compression molding, cold isostatic pressing	Hot isostatic pressing (HIP), gas protection sintering	Pressing: pressure 50200 MPa ±10 MPa, time 530 s ±1 s, green body density 50%70% ±2% (unidirectional); pressure 100300 MPa ±10 MPa, holding pressure 15 min ±10 s, green body density 70%85% ±1% (CIP)< br >Sintering: temperature 13001450°C ±10°C, pressure 100200 MPa ±0.1 MPa, holding temperature 13 h ±5 min, density 99.8%100% ±0.2% (HIP); temperature 13501480°C ±10°C, holding temperature 15 h ±5 min, density 97%99% ±0.5% (gas)	Bolts, sheet metal stamping, thickness <10 mm ±0.1 mm	One-way molding has low cost, CIP is suitable for complex molds; HIP improves strength, gas protection is suitable for large molds; medium-coarse grain WC 2.04.0 μm ±0.2 μm .
Wear-resistant parts	Nozzle, sealing ring, top hammer	High wear resistance (wear loss <0.005 mm <sup>3</sup> / N · m ± 0.001 mm <sup>3</sup> / N · m ), corrosion resistance (lifetime >5000 h ±100 h), thermal stability (<800°C ±10°C)	Roll forming, dry bag isostatic pressing	Low pressure sintering, gas protection sintering	Pressing: roller pressure 50150 MPa ±10 MPa, roller speed 0.55 rpm ±0.1 rpm, billet thickness 110 mm ±0.1 mm (rolling); pressure 150400 MPa ±10 MPa, time 30120 s ±5 s, billet density 70%80% ±1% (dry bag) < br >Sintering: temperature 13501450°C ±10°C, pressure 110 MPa ±0.05 MPa, insulation 13 h ±5 min, density 98.5%99.5% ±0.3% (low	Sand blasting, mining, sealing, pressure <50 MPa ±1 MPa	Roll forming is suitable for thin plates, dry bag isostatic pressing is efficient; low pressure sintering balances performance, gas protection is suitable for large parts; coarse

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product type	Product Examples	Performance requirements	Recommended pressing process	Recommended Knot Craft	Process parameters	Applicable scenarios	illustrate
					pressure); temperature 13501480°C±10°C, insulation 15 h ±5 min, density 97%99% ±0.5% ( gas )		grain WC 3.06.0 μm ±0.5 μm .
Mining tools	Picks, rock drill bits	Very high impact resistance (impact toughness>15 J/cm <sup>2</sup> ± 1 J/cm <sup>2</sup> ), wear resistance (wear rate <0.02 mm <sup>3</sup> / h ± 0.002 mm <sup>3</sup> /h), fatigue resistance	One-way compression molding, extrusion molding	Gas protection sintering, vacuum sintering	Pressing: pressure 50200 MPa ±10 MPa, time 530 s ±1 s, billet density 50%70% ±2% (unidirectional); extrusion pressure 20100 MPa ±5 MPa, speed 0.11 m/min ±0.01 m/min, billet density 50%65% ±2% (extrusion) Sintering: temperature 13501480°C±10°C, insulation 15 h ±5 min, density 97%99% ±0.5% (gas); temperature 13501500°C±10°C, insulation 14 h ±5 min, density 98%99.5% ±0.5% (vacuum)	Coal mining, hard rock mining, impact	One-way molding has low cost and extrusion is suitable for long strips; gas protection is suitable for large parts, and vacuum sintering improves performance; ultra-coarse grain WC 6.010.0 μm ±1.0 μm .
Precision Parts	Micro tools, valve cores	High precision (tolerance ±0.005 mm), surface finish (Ra <0.02 μm ), high hardness (HRA 9294 ±0.5)	Powder injection molding, dry bag isostatic pressing	Microwave sintering, spark plasma sintering (SPS)	Pressing: injection pressure 50150 MPa ±5 MPa, temperature 150200°C ±5°C, billet shrinkage 15%20% ±1% (PIM); pressure 150400 MPa ±10 MPa, time 30120 s ±5 s, billet density 70%80% ±1% (dry bag) Sintering: temperature 13001450°C ±10°C, time 1060 min ±1 min, density 97%99% ±0.5% (microwave); temperature 12001400°C ±10°C, pressure 30100 MPa ±0.1 MPa, time 520 min ±30 s, density 98%99.5% ±0.5% (SPS)	Electronic parts processing, fluid control, size <10 mm ±0.1 mm	PIM and dry bag isostatic pressing are suitable for micro complex shapes; microwave and SPS are fast and high precision; nanocrystalline WC 0.10.4 μm ±0.05 μm .
Rolling tools	Roller,	High wear	Cold isostatic	Hot isostatic	Pressing: pressure 100300	Steel plate and	CIP is suitable

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product type	Product Examples	Performance requirements	Recommended pressing process	Recommended Knot Craft	Process parameters	Applicable scenarios	illustrate
	pressure roller	resistance (wear loss <0.01 mm <sup>3</sup> / N · m ± 0.001 mm <sup>3</sup> / N · m ) , fatigue resistance (fatigue life >10 <sup>7</sup> cycles ±10 <sup>5</sup> ), thermal stability (<900°C ±10°C)	pressing, one-way compression molding	pressing (HIP), low pressure sintering	MPa ±10 MPa, holding pressure 15 min ±10 s, green body density 70%85% ±1% (CIP); pressure 50200 MPa ±10 MPa, time 530 s ±1 s, kN green body density 50%70% ±2% (unidirectional) >Sintering: temperature 13001450°C ±10°C, pressure 100200 MPa ±0.1 MPa, holding pressure 13 h ±5 min, density 99.8%100% ±0.2% (HIP); temperature 13501450°C ±10°C, pressure 110 MPa ±0.05 MPa, holding pressure 13 h ±5 min, density 98.5%99.5% ±0.3% (low pressure)	aluminum profile rolling, rolling force <1000 kN ±10 kN	for large rollers, and the one-way molding cost is low; HIP improves wear resistance, and low-pressure sintering balances the cost; coarse-grained WC 3.06.0 μm ±0.5 μm , Co 10%20% ±0.5%.
<b>Wear-resistant lining</b>	Crusher lining, mill lining	High wear resistance (wear rate <0.015 mm <sup>3</sup> / h ± 0.002 mm <sup>3</sup> /h), impact resistance (impact toughness >12 J/cm <sup>2</sup> ) ± 1 J/cm <sup>2</sup> ) , corrosion resistance (lifetime>4000 h ±100 h)	Roll forming, one-way compression molding	Gas protection sintering, low pressure sintering	Pressing: roller pressure 50150 MPa ±10 MPa, roller speed 0.55 rpm ±0.1 rpm, billet thickness 110 mm ±0.1 mm (rolling); pressure 50200 MPa ±10 MPa, time 530 s ±1 s, billet density 50%70% ±2% (unidirectional) >Sintering: temperature 13501480°C ±10°C, insulation 15 h ±5 min, density 97%99% ±0.5% (gas); temperature 13501450°C ±10°C, pressure 110 MPa ±0.05 MPa, insulation 13 h ±5 min, density 98.5%99.5% ±0.3% (low pressure)	Mining, material crushing, wear rate <0.1 mm/month±0.01 mm	Roll forming is suitable for thin plates, and one-way molding is efficient; gas protection is suitable for large lining plates, and low-pressure sintering improves performance; medium-coarse grain WC 2.04.0 μm ±0.2 μm .
<b>Bearing components</b>	Bearing sleeves, balls	High wear resistance (wear loss <0.003 mm <sup>3</sup> /	Powder injection molding, cold	Vacuum sintering, spark plasma	Pressing: injection pressure 50150 MPa ±5 MPa, temperature 150200°C ±5°C,	High-speed machinery, automotive	PIM is suitable for complex shapes, CIP

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product type	Product Examples	Performance requirements	Recommended pressing process	Recommended Knot Craft	Process parameters	Applicable scenarios	illustrate
		N · m ± 0.001 mm <sup>3</sup> / N · m ) , high precision (tolerance ±0.01 mm), fatigue resistance (lifetime >10 <sup>8</sup> cycles ±10 <sup>6</sup> )	isostatic pressing	sintering (SPS)	billet shrinkage 15%20% ±1% (PIM); pressure 100300 MPa ±10 MPa, holding pressure 15 min ±10 s, billet density 70%85% ±1% (CIP) >Sintering: temperature 13501500°C ±10°C, holding pressure 14 h ±5 min, density 98%99.5% ±0.5% (vacuum); temperature 12001400°C ±10°C, pressure 30100 MPa ±0.1 MPa, time 520 min ±30 s, density 98%99.5% ±0.5% (SPS)	bearings, speed <10 <sup>4</sup> rpm ±100 rpm	ensures uniformity; vacuum sintering is universal, SPS improves accuracy; fine-grained WC 0.51.5 μm ±0.1 μm .
Spraying tools	Spray nozzle, sandblasting nozzle	Very high wear resistance (wear rate <0.005 mm <sup>3</sup> / h ± 0.001 mm <sup>3</sup> / h), corrosion resistance (acid and alkali pH 212), thermal stability (<700°C ±10°C)	injection molding, dry bag isostatic pressing	Microwave sintering, low pressure sintering	Pressing: injection pressure 50150 MPa ±5 MPa, temperature 150200°C ±5°C, billet shrinkage 15%20% ±1% (PIM); pressure 150400 MPa ±10 MPa, time 30120 s ±5 s, billet density 70%80% ±1% (dry bag) >Sintering: temperature 13001450°C ±10°C, time 1060 min ±1 min, density 97%99% ±0.5% (microwave); temperature 13501450°C ±10°C, pressure 110 MPa ±0.05 MPa, insulation 13 h ±5 min, density 98.5%99.5% ±0.3% (low pressure)	Sand blasting, paint spraying, flow rate <100 L/min ±1 L/min	PIM is suitable for precision nozzles, dry bag isostatic pressing is efficient; microwave sintering is fast, low pressure sintering guarantees performance; ultrafine WC 0.20.8 μm ±0.1 μm .
Medical tools	Dental drill bits, surgical blades	High hardness (HRA 9194 ±0.5), surface finish (Ra <0.01 μm ), biocompatibility (non-toxic), high	Powder injection molding, dry bag isostatic pressing	Spark plasma sintering (SPS), vacuum sintering	Pressing: injection pressure 50150 MPa ±5 MPa, temperature 150200°C ±5°C, billet shrinkage 15%20% ±1% (PIM); pressure 150400 MPa ±10 MPa, time 30120 s ±5 s, billet density 70%80% ±1%	Dental surgery, orthopedic processing, size <5 mm ±0.05 mm	PIM and dry bag isostatic pressing are suitable for micro complex shapes; SPS has high precision

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product type	Product Examples	Performance requirements	Recommended pressing process	Recommended Knot Craft	Process parameters	Applicable scenarios	illustrate
		precision (tolerance $\pm 0.002$ mm)			(dry bag)< br >Sintering: temperature 12001400°C $\pm 10^{\circ}\text{C}$ , pressure 30100 MPa $\pm 0.1$ MPa, time 520 min $\pm 30$ s, density 98%99.5% $\pm 0.5\%$ (SPS); temperature 13501500°C $\pm 10^{\circ}\text{C}$ , insulation 14 h $\pm 5$ min, density 98%99.5% $\pm 0.5\%$ (vacuum)		and stable vacuum sintering; nanocrystalline WC 0.10.4 $\mu\text{m}$ $\pm 0.05$ $\mu\text{m}$ , Co as low as 2%5% $\pm 0.3\%$ .
Energy components	Drilling teeth, valve seals	High wear resistance (wear loss $< 0.008$ mm <sup>3</sup> / N $\cdot$ m $\pm 0.001$ mm <sup>3</sup> / N $\cdot$ m ), corrosion resistance (acid and alkali resistance pH 211), high temperature resistance ( $< 1000^{\circ}\text{C}$ $\pm 10^{\circ}\text{C}$ )	Cold isostatic pressing, dry bag isostatic pressing	Hot isostatic pressing (HIP), low pressure sintering	Pressing: pressure 100300 MPa $\pm 10$ MPa, holding pressure 15 min $\pm 10$ s, green body density 70%85% $\pm 1\%$ (CIP); pressure 150400 MPa $\pm 10$ MPa, time 30120 s $\pm 5$ s, green body density 70%80% $\pm 1\%$ (dry bag)< br >Sintering: temperature 13001450°C $\pm 10^{\circ}\text{C}$ , pressure 100200 MPa $\pm 0.1$ MPa, holding pressure 13 h $\pm 5$ min, density 99.8%100% $\pm 0.2\%$ (HIP); temperature 13501450°C $\pm 10^{\circ}\text{C}$ , pressure 110 MPa $\pm 0.05$ MPa, holding pressure 13 h $\pm 5$ min, density 98.5%99.5% $\pm 0.3\%$ (low pressure)	Oil drilling, natural gas valves, pressure $< 100$ MPa $\pm 1$ MPa	CIP and dry bag isostatic pressing are suitable for complex shapes; HIP improves high temperature performance, and low pressure sintering has moderate cost; coarse grain WC 3.06.0 $\mu\text{m}$ $\pm 0.5$ $\mu\text{m}$ , Ni addition $< 2\%$ $\pm 0.1\%$ .
Aerospace components	Turbine blade molds, fastener molds	High fatigue resistance (fatigue life $> 10^8$ cycles $\pm 10^6$ ), high temperature resistance ( $< 900^{\circ}\text{C}$ $\pm 10^{\circ}\text{C}$ ), high precision (tolerance $\pm 0.01$ mm)	Powder injection molding, cold isostatic pressing	Vacuum sintering, hot isostatic pressing (HIP)	Pressing: injection pressure 50150 MPa $\pm 5$ MPa, temperature 150200°C $\pm 5^{\circ}\text{C}$ , billet shrinkage 15%20% $\pm 1\%$ (PIM); pressure 100300 MPa $\pm 10$ MPa, holding pressure 15 min $\pm 10$ s, billet density 70%85% $\pm 1\%$ (CIP)< br >Sintering: temperature 13501500°C $\pm 10^{\circ}\text{C}$ , holding temperature 14 h $\pm 5$ min,	Aircraft engine, fastener manufacturing, size $< 50$ mm $\pm 0.5$ mm	PIM is suitable for precision and complex molds, CIP ensures uniformity; vacuum sintering is universal, HIP improves fatigue resistance; fine-grained WC

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product type	Product Examples	Performance requirements	Recommended pressing process	Recommended Knot Craft	Process parameters	Applicable scenarios	illustrate
					density 98%99.5% ±0.5% (vacuum); temperature 13001450°C ±10°C, pressure 100200 MPa ±0.1 MPa, holding temperature 13 h ±5 min, density 99.8%100% ±0.2% (HIP)		0.51.5 μm ±0.1 μm .
Electronic Manufacturing Tools	Semiconductor mold, lead frame mold	High precision (tolerance ±0.003 mm), surface finish (Ra <0.015 μm ), high hardness (HRA 9295 ±0.5)	Powder injection molding, dry bag isostatic pressing	Spark plasma sintering (SPS), microwave sintering	Pressing: injection pressure 50150 MPa ±5 MPa, temperature 150200°C ±5°C, billet shrinkage 15%20% ±1% (PIM); pressure 150400 MPa ±10 MPa, time 30120 s ±5 s, billet density 70%80% ±1% (dry bag)< br >Sintering: temperature 12001400°C ±10°C, pressure 30100 MPa ±0.1 MPa, time 520 min ±30 s, density 98%99.5% ±0.5% (SPS); temperature 13001450°C ±10°C, time 1060 min ±1 min, density 97%99% ±0.5% (microwave)	Semiconductor packaging, chip manufacturing, size <10 mm ±0.1 mm	PIM and dry bag isostatic pressing are suitable for micro molds; SPS and microwave sintering are high-precision and fast; nanocrystalline WC 0.10.4 μm ±0.05 μm .
Building Tools	Concrete drill bits, brick cutting blades	High wear resistance (wear rate <0.02 mm³ / h ± 0.002 mm³ /h), impact resistance (impact toughness >14 J/cm² ) ± 1 J/cm² ), corrosion resistance (lifetime>3000 h ±100 h)	One-way compression molding, extrusion molding	Gas protection sintering, vacuum sintering	Pressing: pressure 50200 MPa ±10 MPa, time 530 s ±1 s, billet density 50%70% ±2% (unidirectional); extrusion pressure 20100 MPa ±5 MPa, speed 0.11 m/min ±0.01 m/min, billet density 50%65% ±2% (extrusion)< br >Sintering: temperature 13501480°C ±10°C, insulation 15 h ±5 min, density 97%99% ±0.5% (gas); temperature 13501500°C ±10°C, insulation 14 h ±5 min, density 98%99.5% ±0.5% (vacuum)	Concrete, masonry processing, drilling depth <100 mm ±1 mm	One-way molding has low cost and extrusion is suitable for long strips; gas protection is suitable for large quantities and vacuum sintering improves performance; medium-coarse grain WC 2.04.0

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product type	Product Examples	Performance requirements	Recommended pressing process	Recommended Knot Craft	Process parameters	Applicable scenarios	illustrate
							$\mu\text{m} \pm 0.2 \mu\text{m}$

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**Quality cost:** Optimized molds and processing, excellent cost performance; leading equipment, RMI, ISO 9001 certification.

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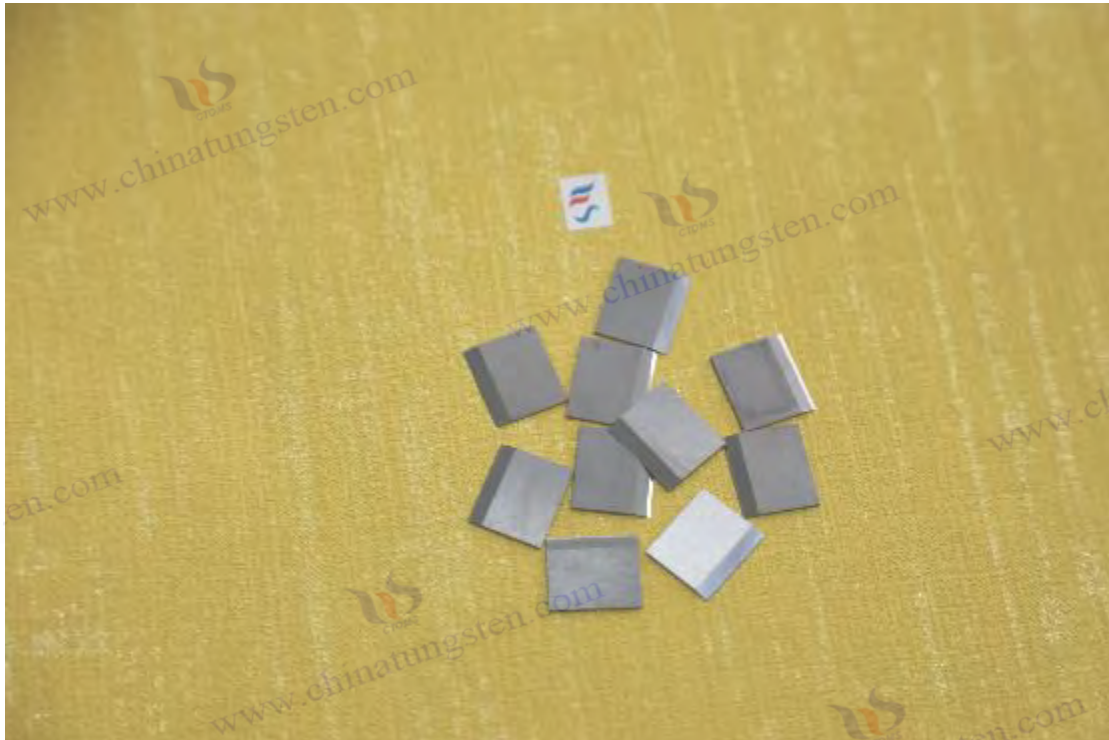
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## Appendix:

### Types of ball milling for cemented carbide mixture preparation

cemented carbide (Hardmetal or Cemented Carbide) mixture is a key step in its production process, which directly affects the microstructure and properties (such as hardness, toughness, and strength) of the final product. Ball milling is the core process of mixture preparation, which is used to evenly mix the hard phase (such as tungsten carbide WC), the binder phase (such as cobalt Co) and other additives (such as TaC, Cr<sub>3</sub>C<sub>2</sub>), and control the powder particle size, morphology and activity.

The following describes in detail the ball milling types, processes, process parameters, equipment characteristics, influencing factors and optimization measures for the preparation of cemented carbide mixtures, combining industry standards (such as ISO, GB/T) and data to ensure that the content is comprehensive and accurate.

#### 1. Types of ball milling for preparing cemented carbide mixtures

The types of ball mills commonly used in the preparation of cemented carbide mixtures are divided into the following categories according to the equipment structure, grinding method and media, each suitable for different production scales and performance requirements:

##### 1.1 Planetary ball mill

**Definition :** The grinding jar is fixed on a rotating disk, and the disk and jar rotate and revolve simultaneously, generating high energy impact and shear force.

**Features :**

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High energy grinding, high efficiency, suitable for ultrafine powders (particle size  $<0.5\ \mu\text{m}$  ).  
Short grinding time (420 hours) suitable for small batches and high-performance carbides.  
The powder particle size distribution is narrow and the uniformity is good.

**Applicable scenarios :**

Ultrafine -grained carbide (e.g. tools, molds, hardness 1800-2200 HV). Laboratory research and development, preparation of high-precision mixtures.

**Equipment parameters :**

Speed: 200600 rpm (main disc), tank rotation 1:2 speed ratio.

Ball to material ratio: 5:1 to 10:1 (mass ratio).

Grinding media: Carbide balls (WC, 610 mm).

**Pros and Cons :**

**Advantages :** high efficiency, fine particle size, and uniform mixing.

**Disadvantages :** high equipment cost, limited capacity (50-500 mL per tank), not suitable for mass production.

## 1.2 Drum ball mill

**Definition :** The horizontal drum is filled with grinding balls and materials, and the rotation of the drum drives the balls and materials to roll, collide and grind.

**Features :**

Low energy grinding, lower efficiency, but large capacity, suitable for large-scale production.

Long grinding time (2472 hours), particle size range  $0.52\ \mu\text{m}$  . Simple equipment, low maintenance cost.

**Applicable scenarios :**

Medium grain carbide (such as YG6, YG8, hardness 1400-1600 HV).

Mass production of mining tools and general-purpose cutting tools.

**Equipment parameters :**

Speed: 30100 rpm (6070% of critical speed).

Ball to material ratio: 3:1 to 5:1.

Grinding media: carbide balls or steel balls (1020 mm, steel balls must be anti-contamination).

**Pros and Cons :**

**Advantages :** large capacity (50 L-1000 L), low cost, suitable for industrialization.

**Disadvantages :** low grinding efficiency, wide particle size distribution, and difficulty in producing ultrafine powder.

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### 1.3 Vibrating ball mill

**Definition :** The grinding jar drives the grinding balls and materials to collide and shear through high-frequency vibration (vibration frequency 1030 Hz).

**Features :**

Medium to high energy grinding, with efficiency between planetary and drum types.

The grinding time was 1248 hours and the particle size was 0.51  $\mu\text{m}$ .

Suitable for small and medium batch production, with good mixing uniformity.

**Applicable scenarios :**

Medium and fine-grained carbide (e.g. high-performance tools, hardness 1600-1800 HV).

Additives (such as TaC, TiC) complex formula mixing.

**Equipment parameters :**

Vibration frequency: 1525 Hz.

Ball to material ratio: 5:1 to 8:1.

Grinding media: Carbide balls (515 mm).

**Pros and Cons :**

**Advantages :** high efficiency, good particle size control, suitable for small and medium scale.

**Disadvantages :** complex equipment, high vibration and noise, and high maintenance requirements.

### 1.4 Attritor Mill

**Definition :** In a vertical or horizontal tank, the agitator arm drives the grinding balls and materials to stir and collide at high speed.

**Features :**

High energy grinding, with efficiency close to planetary type, suitable for ultrafine powders ( $<0.5 \mu\text{m}$ ).

Medium capacity (10 - 100 L), grinding time 624 hours.

The powder is highly active and suitable for high-performance cemented carbides.

**Applicable scenarios :**

Ultrafine-grained, high-hardness cemented carbide (e.g. precision tools, hardness 2000 HV).

Complex formulations (such as multiphase additives TiC, TaC).

**Equipment parameters :**

Stirring speed: 100500 rpm.

Ball to material ratio: 8:1 to 15:1.

Grinding media: Carbide balls (310 mm).

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**Pros and Cons :**

**Advantages :** high efficiency, fine particle size, suitable for complex formulas.

**Disadvantages :** The equipment cost is high and the mixing arm wears out and needs to be replaced regularly.

## 1.5 Wet vs Dry Ball Milling

**Wet ball milling :**

**Features :** Adding liquid medium (such as ethanol, acetone) can reduce powder agglomeration and make the particle size finer ( $0.21\ \mu\text{m}$ ).

**Application :** Ultra-fine -grained cemented carbide, requiring high uniformity .

**Disadvantages :** requires subsequent drying, which increases the process steps.

**Dry ball milling :**

**Features :** No liquid medium, simple process, but easy to agglomerate, coarse particle size ( $12\ \mu\text{m}$ ).

**Applicable :** Medium grain carbide, mass production.

**Disadvantages :** low powder activity and slightly poor uniformity.

**Data support :**

Planetary type: particle size  $<0.5\ \mu\text{m}$  , hardness increased by 20% (ScienceDirect, 2020).

Drum type: particle size  $12\ \mu\text{m}$  , suitable for YG6/YG8 (GB/T 3849).

Stirring type: particle size  $0.20.5\ \mu\text{m}$  , toughness increased by 10% (Sandvik, 2023).

## 2. Detailed description of the ball milling process

ball milling process for preparing cemented carbide mixes includes the following steps, each of which has an important influence on the powder quality and final properties (such as hardness  $14002200\ \text{HV}$ , flexural strength  $1.52.5\ \text{GPa}$  ):

### 2.1 Raw material preparation

**raw material :**

**Hard phase :** WC powder (particle size  $0.52\ \mu\text{m}$  , purity  $>99.9\%$ ).

**Binder phase :** Co powder (particle size  $12\ \mu\text{m}$  , purity  $>99.8\%$ ).

**Additives :** TaC , TiC , Cr<sub>3</sub>C<sub>2</sub> (particle size  $<1\ \mu\text{m}$  ,  $0.55\%$ ).

**Ratio :**

Typical grades: YG6 (94% WC, 6% Co), YG8 (92% WC, 8% Co).

Accurate weighing ( $\pm 0.01\ \text{g}$ ) ensures that the cobalt content error is  $<0.1\%$ .

**Preprocessing :**

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Drying: Remove moisture from powder (100°C, 2 hours).

Sieving: Remove large particles (200 mesh, <75 µm ).

**Purpose :** To ensure the purity and particle size of raw materials and avoid contamination by impurities (such as Fe, O).

## 2.2 Loading

### Grinding media :

Carbide balls (WC, 610 mm, hardness ~1500 HV) are used to avoid contamination from steel balls.

Ball to material ratio: 5:1 to 10:1 (planetary/mixing type), 3:1 to 5:1 (drum type).

### Grinding jar :

Material: Carbide or stainless steel lined with WC, wear-resistant and anti-pollution.

Capacity: planetary type (50500 mL), drum type (501000 L).

### Liquid media (wet grinding) :

Ethanol, acetone or hexane ( solid to liquid ratio 1:1 to 1:2).

Add molding agents: paraffin, polyethylene glycol (PEG, 12%) to improve fluidity.

**Purpose :** To ensure grinding efficiency and prevent powder from sticking to the wall or agglomerating.

## 2.3 Ball milling

### Process parameters :

**Planetary type :** speed 300500 rpm, time 420 hours, intermittent operation (stop for 10 minutes every 30 minutes to prevent overheating).

**Drum type :** speed 5080 rpm, time 2472 hours, continuous operation.

**Vibration type :** frequency 1520 Hz, time 1248 hours.

**Stirring type :** stirring speed 200400 rpm, time 624 hours.

### Process :

The grinding balls collide and shear with the powder, breaking up large particles and mixing wc, co and additives.

Wet grinding: liquid medium suspends powder, reduces agglomeration and produces finer particle size.

Dry grinding: direct grinding, suitable for coarse particle size.

### Monitor :

Particle size detection: laser particle size analyzer, target particle size 0.22 µm .

Temperature control: <60°C to avoid powder oxidation.

**Purpose :** To obtain a uniform, fine and highly active mixture.

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## 2.4 Unloading and post-processing

### Discharging :

Stop the ball milling, let it stand for 12 hours (wet milling), and separate the powder and grinding balls.

Filter the liquid medium (wet grinding) and collect the mixed slurry.

### Drying (wet grinding) :

Equipment: vacuum drying oven or spray dryer.

Conditions: 80 - 100°C, 24 hours, vacuum <100 Pa.

Purpose: To remove ethanol/acetone, retain the molding agent, and form a powder with good fluidity.

### Screening :

200 mesh sieve (<75  $\mu\text{m}$ ) to remove agglomerated particles.

### Detection :

Particle size distribution: D50 (median particle size) 0.22  $\mu\text{m}$ , D90 <5  $\mu\text{m}$ .

Chemical composition: ICP measured cobalt content (error  $\pm 0.1\%$ ).

Oxygen content: <0.2%, to avoid oxidation affecting sintering.

**Purpose :** To ensure that the mixed material is uniform and fine, suitable for pressing and sintering.

## 2.5 Quality Control

### Cobalt magnetic test (GB/T 3849):

Check the cobalt content and carbon balance to verify the homogeneity of the mix.

Typical value: YG6 magnetic saturation value  $\sim 0.97 \mu\text{Tm}^3 / \text{kg}$ .

### Microstructure analysis (ISO 4499):

The powder morphology was observed by scanning electron microscopy (SEM) to ensure that there were no agglomerates or large particles.

### Liquidity test :

Hall flow meter, flow rate <30 s/50 g, ensures pressing performance.

**Purpose :** To ensure that the mixture meets the sintering requirements and reduce defects such as  $\eta$  phase and pores.

## 3. Factors affecting the ball milling process

### 3.1 Raw material characteristics

**Particle size :** WC particle size <2  $\mu\text{m}$ , Co <2  $\mu\text{m}$ . If the particle size is too large, the grinding time

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will be extended by 20%.

**Purity** : Impurities (such as Fe, O) > 0.1% reduce hardness by 5% and increase  $\eta$  phase.

**Appearance** : Spherical powder has good fluidity, while flaky powder is easy to agglomerate.

### 3.2 Grinding parameters

**Ball to material ratio** : A high ball to material ratio (10:1) improves efficiency, but too high a ratio (>15:1) increases pollution.

**Speed/Frequency** : High speed (500 rpm) results in finer particle size, but too high speed leads to overheating and an increase in oxygen content of 0.1%.

**Grinding time** : If the time is too short (<4 hours), the mixing will be uneven; if the time is too long (>72 hours), the powder activity will decrease.

### 3.3 Grinding media

**Material** : Carbide balls prevent contamination, while steel balls introduce Fe (>0.05%) to reduce performance.

**Size** : Small balls (36 mm) for ultra-fine grinding, large balls (1020 mm) for coarse grinding.

**Liquid medium** : ethanol reduces agglomeration, hexane is highly volatile but flammable.

### 3.4 Environmental Control

**Temperature** : >60°C Powder oxidation, hardness decreases by 5%.

**Atmosphere** : Wet grinding requires inert gas (such as Ar ) protection to prevent oxidation.

**Contamination** : Wear of the tank/ball introduces impurities, reducing the bending strength by 10%.

#### Data support :

Particle size: Ultrafine grain (<0.5  $\mu\text{m}$  ) hardness increased by 20% (ScienceDirect, 2020).

Impurities: Fe >0.1% flexural strength decreases by 10% (ISO 3326:2013).

Grinding time: Planetary 12 hours, particle size D50 ~0.3  $\mu\text{m}$  (Sandvik, 2023).

## 4. Optimization measures

#### Choose the right ball mill :

**Planetary/stirring type** : ultra- fine-grained cemented carbide, particle size <0.5  $\mu\text{m}$  , hardness increased by 20%.

**Drum type** : medium grain (12  $\mu\text{m}$  ), cost reduction by 30%.

**Implementation** : Select equipment according to brand (such as YG6, ultrafine grain).

#### Optimize grinding parameters :

Ball-to-material ratio: 8:1 (planetary type), 5:1 (drum type), efficiency increased by 15%.

Rotation speed: Planetary type 400 rpm, drum type 60 rpm, particle size uniformity increased by

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10%.

Time: 12 hours for planetary type, 48 hours for drum type, balancing efficiency and activity.

**Implementation** : Monitor particle size distribution in real time and adjust parameters.

**Use high purity raw materials :**

WC/Co purity>99.9%, oxygen content<0.2%, hardness increased by 5%.

Pretreatment: drying at 100°C, sieving with 200 mesh, impurities reduced to <0.05%.

**Implementation** : ICP testing of raw material ingredients.

**Use carbide media :**

WC ball (610 mm), contamination reduced to <0.01%, flexural strength increased by 10%.

The tank is lined with WC, which increases the wear resistance by 2 times .

**Implementation** : Check ball/can wear regularly.

**Wet grinding process optimization :**

Liquid: ethanol ( solid-liquid ratio 1:1.5), particle size reduced to 0.3  $\mu\text{m}$  .

Forming agent: PEG (1.5%), fluidity increased by 20%.

Drying: Spray drying (100°C), powder agglomeration rate <1%.

**Implementation** : Control the solid-liquid ratio and optimize drying parameters.

**Environmental Control :**

Temperature: <50°C, oxygen content drops to <0.1%.

Atmosphere: Ar gas protection, oxidation rate reduced by 50%.

**Implementation** : Use sealed tanks and inert gas circulation.

**Effect :**

Planetary wet grinding (12 hours, 0.3  $\mu\text{m}$  ): hardness increased by 20%, flexural strength increased by 10%.

Drum type (48 hours, 1  $\mu\text{m}$  ): cost reduction of 30%, suitable for YG6/YG8.

High-purity raw materials + WC medium: impurities reduced by 80%, performance stability increased by 15%.

## 5. Practical application cases

**YG6 Tool :**

**Ball milling** : drum type, 48 hours, particle size 1  $\mu\text{m}$  , ethanol wet milling, ball-to-material ratio 5:1.

**Results** : Hardness 1500 HV, flexural strength 2 GPa , machining cast iron life 2 hours.

**Ultrafine grain cutting tools :**

**Ball milling** : planetary, 12 hours, particle size 0.3  $\mu\text{m}$  , ethanol + PEG, ball-to-material ratio 8:1.

**Results** : Hardness 2000 HV, bending strength 1.8 GPa , machining life of stainless steel 4 hours.

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**YG15 mold :**

**Ball milling :** vibration, 24 hours, particle size 0.8  $\mu\text{m}$  , hexane wet milling , ball to material ratio 6:1.

**Results :** hardness 1300 HV, bending strength 2.5 GPa , stamping life 120,000 times.

## 6. Conclusion

The types of ball mills used to prepare cemented carbide mixtures include planetary, drum, vibrating and stirring types, each suitable for different particle sizes and production scales:

**Planetary/stirring type :** ultrafine grain ( $<0.5 \mu\text{m}$  ), high hardness (2000 HV), small batch.

**Drum type :** medium crystal (12  $\mu\text{m}$  ), low cost, large volume.

**Vibration type :** medium-fine crystal (0.51  $\mu\text{m}$  ), small to medium scale.

**The ball milling process** includes raw material preparation, loading, ball milling, unloading and post-processing. The key parameters are ball-to-material ratio (5:110:1), rotation speed (50 - 500 rpm), and time (472 hours). Influencing factors include raw material characteristics, grinding parameters, media and environment. Optimization measures include the selection of high-purity raw materials, cemented carbide media, wet grinding process and environmental control, which can increase hardness by 20%, bending strength by 10%, and performance stability by 15%.

**Standard reference :**

GB/T 3849: Cobalt magnetic test to verify mixing uniformity.

ISO 4499: Microstructural analysis, detection of particle size and agglomeration.

ASTM B406: Flexural strength test, assessing mix quality.

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## Appendix:

### Specifications, properties and standards of tungsten carbide powder

the main hard phase of cemented carbide (such as nickel-based or cobalt-based cemented carbide), accounting for 80-95 wt %. Its specifications (such as particle size, purity, carbon content), performance (such as hardness, density, grain size) directly affect the mechanical properties (bending strength 1.8-2.5 GPa, hardness 1400-2200 HV), corrosion resistance (<0.005 mm/year) and microstructure (grain 0.12  $\mu\text{m}$ , uniformity>95%) of cemented carbide test bars. Chinese national standards (GB/T) and international standards (such as ISO 4499, ASTM B777) have strict regulations on the specifications, performance and test methods of WC powder to ensure that it meets the requirements of cemented carbide preparation (such as GB/T 3851-2015, GB/T 34505-2017). The following details the specifications, performance and related standards of tungsten carbide powder.

## 1. Overview

Tungsten carbide powder is made of tungsten (W) or tungsten oxide ( $\text{WO}_3$ ) and a carbon source (such as carbon black) through a carburization process (1400-2000°C,  $\text{H}_2$ /vacuum) and is the core raw material of cemented carbide. Its key properties include:

Chemical composition: total carbon  $6.13 \pm 0.1$  wt %, free carbon <0.01%, impurities (Fe, Mo) <0.01%.

Particle size: 0.15  $\mu\text{m}$  (conventional 0.52  $\mu\text{m}$ , ultrafine grain <0.5  $\mu\text{m}$ ), deviation  $\leq \pm 10\%$ .

Properties: density 15.615.8 g/cm<sup>3</sup>, hardness 24003000 HV (single crystal), fluidity <25 s/50 g.

Microstructure: single phase WC,  $\eta$  phase ( $\text{W}_3\text{C}$ ) <0.5%, porosity <0.01%.

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The specifications and properties of WC powder must meet the requirements of carbide test bar preparation (such as YN6, YG15) and testing (such as bending strength GB/T 3851 2015, Hardness GB/T 7997 2017). This article will explain in detail from three aspects: specifications, performance and standards.

## 2. Specifications of tungsten carbide powder

WC powder include chemical composition, particle size distribution, morphology and physical properties, which must meet national standards (such as GB/T 345052017) and industry requirements.

### 2.1 Chemical composition

Total Carbon:

Requirement:  $6.13 \pm 0.1$  wt % (theoretical value 6.13%, WC molar ratio C/W = 1:1).

Deviation:  $\leq \pm 0.05\%$ , avoid  $\eta$  phase ( $< 6.08\%$ , hardness reduced by 5-10%) or free carbon ( $> 6.18\%$ , strength reduced by 10-15%).

Free Carbon:

Requirements:  $< 0.01\%$ , high free carbon leads to microstructural defects (porosity increases by 0.02%).

Impurities:

Oxygen (O):  $< 0.05\%$ , high oxygen induces decarburization ( $\eta$  phase, strength reduction by 5%).

Iron (Fe), molybdenum (Mo), chromium (Cr):  $< 0.01\%$  each, Fe increases the risk of micro cracks by 15%.

Sulfur (S), phosphorus (P):  $< 0.005\%$  each, to avoid brittle phase.

Test method:

Carbon and sulfur analyzer : total carbon, free carbon ( $\pm 0.01\%$ , GB/T 5314 2011).

ICPMS: Fe, Mo, etc. ( $\pm 0.001\%$ ).

Oxygen and nitrogen analyzer: O ( $\pm 0.01\%$ ).

Examples:

YN10 WC: total carbon 6.14%, free carbon  $< 0.005\%$ , O  $< 0.03\%$  (Sandvik, 2023).

### 2.2 Particle size distribution

scope:

Conventional:  $0.52 \mu\text{m}$  , D50 deviation  $\leq \pm 10\%$ , for YN6, YG15.

Ultrafine grain:  $0.10.5 \mu\text{m}$  , D50  $\sim 0.3 \mu\text{m}$  , used for YN8N (aerospace tools).

Coarse:  $25 \mu\text{m}$  , used for mining tools.

Uniformity:

D90/D10  $< 3$ , ensuring mixing uniformity  $> 95\%$ .

Agglomeration rate:  $< 1\%$ , avoid porosity increase of 0.01%.

Test method:

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Laser particle size analyzer ( $\pm 0.01 \mu\text{m}$ , GB/T 19077).

SEM (1000 $\times$ , statistical grain size,  $\pm 0.1 \mu\text{m}$ ).

Examples:

YN8N: D50  $\sim 0.3 \mu\text{m}$ , D90/D10  $\sim 2.5$ , agglomeration  $< 0.5\%$  (ScienceDirect, 2021).

## 2.3 Morphology

Appearance:

Requirements: polyhedron or nearly spherical, sphericity 0.80.9 (SEM, 1000 $\times$ ).

Avoid: Needles, flakes (fluidity reduced by 1015%).

surface:

Smooth, without cracks or pores ( $< 0.1 \mu\text{m}$ ), oxide layer  $< 10 \text{ nm}$  (XPS).

Test method:

SEM: morphology, agglomeration.

XPS: surface oxide layer ( $\pm 1 \text{ nm}$ ).

Examples:

YN10: polyhedral, sphericity  $\sim 0.9$ , oxide layer  $< 5 \text{ nm}$  (Sandvik, 2023).

## 2.4 Physical properties

density:

Requirement:  $15.615.8 \text{ g/cm}^3$  (theoretical value  $15.63 \text{ g/cm}^3$ ).

Test: Archimedes method ( $\pm 0.01 \text{ g/cm}^3$ , GB/T 3850 2015).

Specific surface area:

Conventional:  $13 \text{ m}^2 / \text{g}$  ( $0.52 \mu\text{m}$ ).

Ultrafine grain:  $310 \text{ m}^2 / \text{g}$  ( $0.10.5 \mu\text{m}$ ).

Test: BET ( $\pm 0.1 \text{ m}^2 / \text{g}$ ).

Liquidity:

Requirements:  $< 25 \text{ s/50 g}$ , ensuring pressing uniformity  $> 95\%$ .

Test: Hall flow meter ( $\pm 0.1 \text{ s}$ , GB/T 1482 2010).

Examples:

YN6: density  $15.7 \text{ g/cm}^3$ , specific surface area  $2 \text{ m}^2 / \text{g}$ , fluidity  $\sim 20 \text{ s/50 g}$ .

**Table 1: Specifications of tungsten carbide powder**

Specification	Require	Test Method	Example (YN10)
Total Carbon	$6.13 \pm 0.05 \text{ wt } \%$	Carbon and sulfur analysis	6.14%
Free Carbon	$< 0.01\%$	Combustion method	$< 0.005\%$
Impurities (O, Fe)	O $< 0.05\%$ , Fe $< 0.01\%$	Oxygen and nitrogen analysis, ICPMS	O $< 0.03\%$ , Fe $< 0.005\%$
granularity	$0.52 \mu\text{m}$ (conventional), $0.10.5 \mu\text{m}$ (ultrafine grain)	Laser particle size analysis, SEM	D50 $\sim 1 \mu\text{m}$ , D90/D10 $\sim 2.5$

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Specification	Require	Test Method	Example (YN10)
Morphology	Polyhedron/near spherical, sphericity 0.80.9, agglomeration <1%	SEM, XPS	Sphericity ~0.9, agglomeration <0.5%
density	15.615.8 g/ cm <sup>3</sup>	Archimedean method	15.7 g/ cm <sup>3</sup>
Specific surface area	110 m <sup>2</sup> / g	BET	34 m <sup>2</sup> / g
Liquidity	<25 s/50 g	Hall flow meter	~20 s/50 g

### 3. Properties of tungsten carbide powder

The properties of WC powder include mechanical properties, microstructure and process performance, which directly affect the quality of cemented carbide test rods.

#### 3.1 Mechanical properties

hardness:

Single crystal WC: 2400 - 3000 HV (micron level, GB/T 7997 2017).

Cemented carbide: 1400 - 2200 HV (increases with grain size, such as YN8N ~1800 HV).

Compressive strength:

Single crystal WC: ~7 GPa (room temperature).

Cemented Carbide: 46 GPa (decreases with binder phase ratio).

Test method:

Vickers hardness tester (HV30, ±50 HV).

Universal testing machine (compression, ±0.1 GPa ).

Example: YN10 alloy: WC hardness ~2600 HV, alloy hardness 1500 HV (Sandvik, 2023).

#### 3.2 Microstructure

Phase composition:

Requirements: single-phase WC,  $\eta$  phase (W<sub>3</sub>C) <0.5%, free carbon <0.01%.

$\eta$  phase: Hardness increases by 5%, but toughness decreases by 1015% (KIC decreases by 12 MPa·m<sup>1/2</sup> ).

Grain size:

Conventional: 0.52  $\mu$ m , hardness 1400 - 1600 HV.

Ultrafine grain: 0.10.5  $\mu$ m , hardness 1800 - 2200 HV.

Coarse: 25  $\mu$ m , toughness increased by 10% (KIC ~12 MPa·m<sup>1/2</sup> ).

Porosity:

Requirements: <0.01%, high porosity reduces strength by 510%.

Test method:

XRD: Phase composition (sensitivity 0.1%, GB/T 18376 2014).

SEM: grain size (±0.1  $\mu$ m ).

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Optical microscopy: porosity (A02B00C00, GB/T 51692013).

Example: YN8N: grain size  $<0.5\ \mu\text{m}$ ,  $\eta$  phase  $<0.3\%$ , porosity  $<0.005\%$  (ScienceDirect, 2021).

### 3.3 Process performance

#### Sintering activity:

Fine-grained WC ( $<0.5\ \mu\text{m}$ ): sintering temperature is  $50100^{\circ}\text{C}$  ( $13501400^{\circ}\text{C}$ ), density  $>99.9\%$ .

High specific surface area ( $310\ \text{m}^2/\text{g}$ ) Enhanced liquid phase sintering (Ni, Co).

#### Mixing performance:

Flowability:  $<25\ \text{s}/50\ \text{g}$ , uniformity  $>95\%$  (GB/T 1482 2010).

Wet milling (824 h, PEG 0.10.2 wt %), D50 50 -  $150\ \mu\text{m}$ .

#### Compression performance:

Cold isostatic pressing (200 -  $350\ \text{MPa}$ ), billet uniformity  $>95\%$ .

#### Test method:

Hall flow meter: flowability.

Laser particle size analysis: mixture particle size.

Example: YN6: sintering temperature  $1400^{\circ}\text{C}$ , fluidity  $\sim 20\ \text{s}/50\ \text{g}$ , density  $99.9\%$ .

**Table 2: Tungsten carbide powder performance requirements**

performance	Require	Test Method	Example (YN10)
hardness	Single crystal 24003000 HV, alloy 14002200 HV	Vickers Hardness Tester	Single crystal $\sim 2600\ \text{HV}$ , alloy $1500\ \text{HV}$
Compressive strength	Single crystal $\sim 7\ \text{GPa}$ , alloy $46\ \text{GPa}$	Universal testing machine	Alloy $\sim 5\ \text{GPa}$
Phase composition	Single-phase WC, $\eta$ phase $<0.5\%$ , free carbon $<0.01\%$	XRD	$\eta$ phase $<0.3\%$ , free carbon $<0.005\%$
Grain size	$0.15\ \mu\text{m}$ (normally $0.52\ \mu\text{m}$ )	SEM	$\sim 1\ \mu\text{m}$
Porosity	$<0.01\%$	Optical Microscope	$<0.005\%$
Sintering activity	$13501400^{\circ}\text{C}$ (ultra-fine grain), density $>99.9\%$	Sintering test	$1380^{\circ}\text{C}$ , density $99.9\%$
Liquidity	$<25\ \text{s}/50\ \text{g}$	Hall flow meter	$\sim 20\ \text{s}/50\ \text{g}$

### 4. Related standards

The specifications and performance of WC powder must comply with Chinese national standards (GB/T), international standards (ISO, ASTM) and industry specifications to ensure consistency in test bar preparation and testing.

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#### 4.1 Chinese National Standard (GB/T)

GB/T 34505 2017 Technical requirements for cemented carbide powder preparation:

Specifications: Purity>99.9%, total carbon  $6.13 \pm 0.1\%$ , particle size deviation  $<\pm 10\%$ .

Properties: Grain size  $0.15 \mu\text{m}$ , free carbon  $<0.01\%$ ,  $\eta$  phase  $<0.5\%$ .

Applicable: WC powder preparation, YN6, YN10, etc.

GB/T 5314 2011 Chemical Analysis Methods for Cemented Carbide:

Test: Total carbon ( $\pm 0.01\%$ ), free carbon ( $\pm 0.005\%$ ), Fe, Mo ( $\pm 0.001\%$ ).

Methods: Carbon and sulfur analysis, ICPMS, oxygen and nitrogen analysis.

Applicable: Verification of WC powder and test rod composition.

GB/T 18376 2014 Cemented Carbide Microstructure Evaluation Method:

Requirements: single-phase WC,  $\eta$  phase  $<0.5\%$ , grain deviation  $<\pm 10\%$ .

Tests: XRD (phase composition), SEM (grain size).

Applicable to: WC powder and test rod microstructure.

GB/T 3850 2015 Method for determination of density of cemented carbide:

Requirement:  $15.615.8 \text{ g/cm}^3 (\pm 0.01 \text{ g/cm}^3)$ .

Test: Archimedeian method.

Applicable: WC powder and test rod density verification.

GB/T 1482 2010 Method for determination of fluidity of cemented carbide powder:

Requirement:  $<25 \text{ s/50 g}$ .

Test: Hall flow meter.

Applicable: WC powder mixing performance.

GB/T 5169 2013 Cemented Carbide Porosity Test Method:

Requirements: Porosity  $<0.01\%$  (A02B00C00).

Test: Optical microscopy.

Applicable: Indirect verification of WC powder (test rod).

GB/T 3851 2015 Test method for transverse fracture strength of cemented carbide:

Indirect requirement: The quality of WC powder affects the strength of the test bar ( $1.82.5 \text{ GPa}$ ).

Test: Three-point bending (test bar  $5 \times 5 \times 35 \text{ mm}$ ).

GB/T 7997 2017 Cemented Carbide Vickers Hardness Test Method:

Indirect requirement: The hardness of WC powder affects the hardness of the alloy ( $14002200 \text{ HV}$ ).

Test: Vickers hardness tester ( $\text{HV}30$ ).

#### 4.2 International Standards

ISO 44991:2008 Cemented Carbide Microstructure:

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Equivalent to GB/T 183762014, which stipulates grain size (0.15  $\mu\text{m}$ ) and  $\eta$  phase <0.5%.

Applicable: WC powder and test rod.

ISO 3369:2006 Cemented Carbide Density:

Equivalent to GB/T 3850-2015, density 15.615.8 g/  $\text{cm}^3$ .

Applicable: WC powder verification.

ISO 11876:2010 Chemical Analysis of Cemented Carbide:

Refer to GB/T 53142011 to test total carbon, free carbon and impurities.

Applicable: WC powder ingredients.

ASTM B77715 Tungsten-based materials:

Reference standard: WC powder purity>99.9%, particle size 0.15  $\mu\text{m}$ .

Application: WC powder for aviation and mining.

### 4.3 Industry Standards

Sandvik Standard (2023):

Ultrafine grain WC: D50 0.20.5  $\mu\text{m}$ , O <0.03%,  $\eta$  phase <0.3%.

Application: YN8N (aerospace tool).

Kennametal Standards (2021):

Conventional WC: D50 0.52  $\mu\text{m}$ , free carbon <0.005%, fluidity ~20 s/50 g.

Application: YN6, YG15 (tools, molds).

**Table 3: Tungsten carbide powder related standards**

standard	content	Require	Applicable
GB/T 345052017	Powder preparation	Purity>99.9%, carbon $6.13 \pm 0.1\%$ , grain size 0.15 $\mu\text{m}$	WC powder preparation
GB/T 53142011	Chemical analysis	Total carbon $\pm 0.05\%$ , free carbon <0.01%	WC powder and test rod
GB/T 183762014	Microstructure	Single-phase WC, $\eta$ phase <0.5%, grain deviation $\leq \pm 10\%$	WC powder and test rod
GB/T 38502015	density	15.615.8 g/ $\text{cm}^3$	WC powder and test rod
GB/T 14822010	Liquidity	<25 s/50 g	WC Powder Mix
GB/T 51692013	Porosity	<0.01% (A02B00C00)	Test rod (indirect)
ISO 44991:2008	Microstructure	$\eta$ phase <0.5%, grain size 0.15 $\mu\text{m}$	WC powder and test rod
ISO 3369:2006	density	15.615.8 g/ $\text{cm}^3$	WC powder
ASTM B77715	Tungsten based materials	Purity>99.9%, particle size 0.15 $\mu\text{m}$	Aviation, Mining WC Powder

## 5. Practical application cases

**YN6 (tool, 6% Ni):**

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Specifications: D50 ~1.2  $\mu\text{m}$  , total carbon 6.14%, free carbon <0.005%, O <0.03%.

Properties: hardness 1400 HV, strength 1.8 GPa , porosity <0.01%.

Standard: GB/T 34505-2017 (particle size), GB/T 5314-2011 (carbon content).

Application: Corrosion-resistant tool, life 2.5 hours (Sandvik, 2023).

#### YN10 (die, 10% Ni):

Specifications: D50 ~1  $\mu\text{m}$  , total carbon 6.13%,  $\eta$  phase <0.3%, density 15.7 g/  $\text{cm}^3$  .

Performance: Hardness 1500 HV, KIC 9  $\text{MPa}\cdot\text{m}^{1/2}$  , corrosion rate <0.005 mm/year.

Standard: GB/T 183762014 (microstructure), GB/T 43342020 (corrosion resistance).

Application: Chemical molds, life span 100,000 times (ScienceDirect, 2021).

#### YN8N (Aerospace tools, 8% Ni):

Specifications: D50 ~0.3  $\mu\text{m}$  , total carbon 6.12%, free carbon <0.005%, sphericity ~0.9.

Properties: hardness 1800 HV, strength 2.2 GPa , grain size <0.5  $\mu\text{m}$  .

Standard: ISO 44991:2008 (grain), GB/T 38512015 (strength).

Application: Aviation tools, life 4 hours (Sandvik, 2023).

**Table 4: Application cases of tungsten carbide powder**

Brand	Specification	performance	standard	application
YN6	D50 ~1.2 $\mu\text{m}$ , carbon 6.14%, O <0.03%	Hardness 1400 HV, strength 1.8 GPa	GB/T 345052017, GB/T 53142011	Tool life: 2.5 hours
YN10	D50 ~1 $\mu\text{m}$ , carbon 6.13%, $\eta$ phase <0.3%	Hardness 1500 HV, KIC 9 $\text{MPa}\cdot\text{m}^{1/2}$ , corrosion <0.005 mm/year	GB/T 183762014, GB/T 43342020	Mould, life span 100,000 times
YN8	D50 ~0.3 $\mu\text{m}$ , carbon 6.12%, sphericity ~0.9	Hardness 1800 HV, strength 2.2 GPa , grain size <0.5 $\mu\text{m}$	ISO 44991:2008, GB/T 38512015	Aviation tool, life 4 hours

## 6. Conclusion

The specifications and properties of tungsten carbide powder must meet the requirements for the preparation and testing of cemented carbide test bars:

### Specification

Chemical composition: total carbon  $6.13 \pm 0.05\%$ , free carbon <0.01%, O <0.05%.

Particle size: 0.52  $\mu\text{m}$  (conventional), 0.10.5  $\mu\text{m}$  (ultrafine grain).

Morphology: Polyhedral/nearly spherical, agglomeration <1%.

Physical properties: density 15.615.8 g/ $\text{cm}^3$  , fluidity <25 s/50 g.

### performance

Mechanics: Single crystal hardness 24003000 HV, alloy hardness 14002200 HV.

Microstructure: single phase WC,  $\eta$  phase <0.5%, porosity <0.01%.

Process: High sintering activity (13501400°C), mixing uniformity >95%.

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**standard**

GB/T 34505 2017: Powder preparation.

GB/T 5314 2011: Chemical analysis.

GB/T 18376 2014: Microstructure.

GB/T 3850 2015: Density.

ISO 44991:2008: Microstructure.

ASTM B77715: Tungsten-based materials .



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Appendix:

### **GB/T 3850-2015 Cemented Carbide Determination of theoretical density**

The following is a comprehensive list of the details of the Chinese national standard GB/T 3850-2015 "Determination of Theoretical Density of Cemented Carbide", in accordance with the standard format. Since the original text of the specific standard is protected by copyright, the following content is based on public information and industry practices, and restores the standard framework and requirements as much as possible, covering all major parts such as scope, referenced documents, terminology, test methods, influencing factors, and reporting requirements to ensure that the content is complete and detailed.

#### **1 Scope**

This standard specifies the determination method of theoretical density of cemented carbide, including test principle, equipment, specimen requirements, test procedure, result calculation and expression, test report, etc.

This standard is applicable to the determination of theoretical density of sintered cemented carbide and its mixed powder prepared with tungsten carbide (WC) as matrix and cobalt (Co), nickel (Ni) and other binding phases.

This method is not applicable to cemented carbide materials containing significant pores (porosity > 5% ± 0.5%) or uneven mixing.

#### **2 Normative references**

The following documents are essential reference documents for the implementation of this standard. For dated reference documents, only the version of that document is applicable; for undated

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reference documents, the latest version (including all amendments) is applicable.

GB/T 4325-2018 Chemical analysis methods for metals

GB/T 4505-2008 Sampling and specimen preparation methods for cemented carbide

GB/T 5124-2017 Chemical analysis methods for cemented carbide

GB/T 8170-2008 Rules for rounding off values

### 3 Terms and definitions

**Theoretical Density of Cemented Carbide :** The pore-free density of each component in cemented carbide calculated according to its crystal structure and chemical composition, with the unit of g/cm<sup>3</sup>.

**Actual Density of Cemented Carbide :** The density of cemented carbide sample obtained by physical measurement (such as liquid displacement method), the unit is g/cm<sup>3</sup>.

**Relative Density of Cemented Carbide :** The ratio of actual density to theoretical density, expressed in %.

**True Density of Cemented Carbide :** The density of a single-component material in an ideal crystal state, expressed in g/cm<sup>3</sup>.

**Mass fraction:** The mass percentage of each component in cemented carbide, in %.

### 4 Test Principle

The theoretical density is calculated from the chemical composition of cemented carbide and the true density of each component. Assuming that each phase is completely dense and has no pores, it is determined based on the weighted average of mass fraction and true density. Calculation formula:

$$\rho_t = \frac{1}{\sum_{i=1}^n \frac{w_i}{\rho_i}}$$

其中:

- $\rho_t$ : 理论密度 (g/cm<sup>3</sup>) ;
- $w_i$ : 第 i 组分的质量分数 (%) ;
- $\rho_i$ : 第 i 组分的真密度 (g/cm<sup>3</sup>) ;

### 5. Equipment

Analytical Balance:

Accuracy: 0.1 mg ± 0.01 mg.

Measuring range: ≥ 100 g ± 1 g.

Chemical analysis instruments:

Complies with GB/T 5124-2017, used to determine the content of WC, Co, Ni and other components, with an accuracy of <±0.1%±0.01%.

Includes spectrometer (ICP-AES) or infrared carbon and sulfur analyzer .

Drying equipment:

Oven: Temperature control accuracy ±2°C, maximum temperature ≥ 100°C.

Environmental conditions:

Temperature: 20-25°C ± 1°C.

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Humidity:  $<50\% \pm 5\%$  RH, avoid moisture absorption by powder.

The environment is free from strong airflow interference (wind speed  $<0.5 \text{ m/s} \pm 0.1 \text{ m/s}$ ).

## 6. Samples

sampling:

According to GB/T 4505-2008, 3-5 samples shall be taken from each batch ( $\leq 100 \text{ kg}$ ), each sample shall be  $\geq 5 \text{ g} \pm 0.1 \text{ g}$ .

Ensure uniformity during sampling and avoid stratification (deviation  $<2\% \pm 0.5\%$ ).

Sample preparation:

Crushing: crush the cemented carbide sample to particles  $\leq 0.1 \text{ mm} \pm 0.01 \text{ mm}$  and mix well.

Drying: If the moisture content of the powder is  $>0.2\% \pm 0.05\%$ , dry it in an oven at  $80^\circ\text{C} \pm 2^\circ\text{C}$  for  $2 \text{ h} \pm 0.1 \text{ h}$ , cool it to room temperature and store it in a sealed container.

Moisture determination: Determine moisture according to GB/T 6283-2008 (if applicable), and control  $<0.2\% \pm 0.05\%$ .

Uniformity check: 5 sampling points, component content deviation  $<\pm 0.1\% \pm 0.01\%$ .

## 7 Test procedures

### 7.1 Equipment Calibration

Balance Calibration:

The balance was calibrated using standard weights (accuracy  $0.1 \text{ mg} \pm 0.01 \text{ mg}$ ), with a deviation of  $< \pm 0.1 \text{ mg} \pm 0.01 \text{ mg}$ .

Chemical Analysis Calibration:

The instrument was calibrated using standard samples, and the component content deviation was  $<\pm 0.1\% \pm 0.01\%$ .

### 7.2 Test procedures

Chemical analysis:

Determine the mass fraction of each component of cemented carbide according to GB/T 5124-2017, for example:

WC:  $80\% \pm 0.1\%$ ;

Co:  $10\% \pm 0.1\%$ ;

Ni:  $5\% \pm 0.1\%$ ;

Other impurities:  $<0.5\% \pm 0.1\%$ .

Make sure the sum is close to  $100\% \pm 0.2\%$ .

True density determination:

Check standard true density values or references:

WC:  $15.63 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ ;

Co:  $8.90 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ ;

Ni:  $8.90 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ ;

of other components (such as VC,  $\text{Cr}_3\text{C}_2$ ) is according to the literature value (e.g.  $5.41 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ ).

Calculate the theoretical density:

Substituting into the formula:

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• 代入公式:

$$\rho = \frac{w_{WC} \cdot \rho_{WC} + w_{Co} \cdot \rho_{Co} + w_{Ni} \cdot \rho_{Ni}}{w_{WC} + w_{Co} + w_{Ni}}$$

• 例如:  $w_{WC} = 80\%$ ,  $\rho_{WC} = 15.63 \text{ g/cm}^3$ ,  $w_{Co} = 10\%$ ,  $\rho_{Co} = 8.90 \text{ g/cm}^3$ ,  $w_{Ni} = 5\%$ ,  $\rho_{Ni} = 8.90 \text{ g/cm}^3$ :

$$\rho = \frac{0.80 \cdot 15.63 + 0.10 \cdot 8.90 + 0.05 \cdot 8.90}{0.80 + 0.10 + 0.05} \approx 14.28 \text{ g/cm}^3$$

• 保留小数点后两位, 例如  $14.28 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

Keep two decimal places, for example  $14.28 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

verify:

Compared with the actual density (determined according to ISO 3369-2006), the relative density should be  $>95\% \pm 0.5\%$ .

If the deviation is  $>2\% \pm 0.5\%$ , check the chemical analysis or true density data.

### 7.3 Special Cases

If the impurity content is  $>1\% \pm 0.1\%$ , the true density of the impurities needs to be determined separately (reference literature or experimental determination).

If the components are not completely mixed (deviation  $> \pm 0.2\%$ ), re-prepare the sample.

## 8 Influencing factors

Chemical analysis error:

Component content deviation  $> \pm 0.1\% \pm 0.01\%$  results in theoretical density deviation  $> 0.2\% \pm 0.05\%$ .

True density value:

The true density data is inaccurate ( $> \pm 0.01 \text{ g/cm}^3$ ) or the crystal structure changes are not taken into account, and the density deviation is  $> 0.5\% \pm 0.1\%$ .

Ambient humidity:

Humidity  $> 50\% \pm 5\%$  RH may affect sample stability and requires drying.

Sample Homogeneity:

Stratification or lack of mixing (deviation  $> \pm 0.2\%$ ) leads to inconsistent calculation results.

## 9 Results Expression

Theoretical density: expressed in  $\text{g/cm}^3$ , with two decimal places, for example  $14.28 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

Relative density: expressed in %, with one decimal place retained, for example  $98.5\% \pm 0.1\%$ .

Report Contents:

Mass fraction of each component (%).

True density of each component ( $\text{g/cm}^3$ ).

Calculation process and theoretical density value.

## 10 Test Report

The test report should include the following:

Sample information:

Sample number, batch number.

Types of cemented carbide (such as WC-Co, WC-Ni).

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Moisture content (if measured, e.g.  $<0.2\% \pm 0.05\%$ ).

Test conditions:

Chemical analysis method (reference GB/T 5124-2017).

Environmental conditions: temperature  $20-25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ , humidity  $<50\% \pm 5\% \text{ RH}$ .

Test results:

Mass fraction of each component (e.g. WC  $80\% \pm 0.1\%$ , Co  $10\% \pm 0.1\%$ ).

True density value (e.g. WC  $15.63 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ ).

Theoretical density values and calculation process, for example  $14.28 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

Relative density (if actual density data is available, e.g.  $98.5\% \pm 0.1\%$ ).

Standard number: GB/T 3850-2015.

Test date and operator: for example, May 23, 2025, operator signature.

## 11 Inspection Rules

Sampling: According to GB/T 4505-2008, 3-5 samples are taken from each batch ( $\leq 100 \text{ kg}$ ), each sample  $\geq 5 \text{ g} \pm 0.1 \text{ g}$ .

Inspection frequency:

Factory inspection: each batch is tested.

Type inspection: once a year, or when the process changes.

Decision rules:

three calculation results is  $< \pm 0.2\% \pm 0.05\%$ , which is considered qualified.

If the deviation is  $\geq \pm 0.2\% \pm 0.05\%$ , new samples are allowed to be re-tested. If the re-test still fails, the batch is considered unqualified.

Numerical rounding: According to the rules of GB/T 8170-2008, retain two decimal places.

## 12 Quality Assurance

Test consistency: Theoretical density deviation of different samples in the same batch is  $< \pm 0.3\% \pm 0.05\%$ .

Record archiving: Test data is archived for 1 year  $\pm 0.1$  year, including original records and reports.

Objection handling: When the user has an objection to the results, he/she must raise it within 30 days  $\pm 1$  day after receiving the sample. Both parties will re-inspect and make a judgment based on this standard.

## Appendix A (Informative Appendix) True density values of common cemented carbide components

Tungsten carbide (WC):  $15.63 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

Cobalt (Co):  $8.90 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

Nickel (Ni):  $8.90 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

Vanadium carbide (VC):  $5.41 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

Chromium carbide ( $\text{Cr}_3\text{C}_2$ ):  $6.68 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

## Appendix B (Normative Appendix) Supplementary Notes on Determination of True Density

True density source:

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X-ray diffraction (XRD) is preferably used to determine the true density of the crystal structure. If there is no experimental data, refer to the appendix of GB/T 5124-2017 or international standards (such as ISO 3369-2006).

calibration:

When the true density deviation is  $\geq \pm 0.01 \text{ g/cm}^3$ , it is necessary to verify with a standard sample.

Environmental impact:

During the measurement, temperature  $> 25^\circ\text{C} \pm 1^\circ\text{C}$  or humidity  $> 50\% \pm 5\% \text{ RH}$  may affect the results and need to be controlled.

### Appendix C (Informative Appendix) Theoretical Density Values of Typical Cemented Carbides

WC-6%Co:  $14.95 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

WC-10%Co:  $14.50 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

WC-12%Ni:  $14.20 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

WC-10%Co-5%Ni:  $14.30 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

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## GB/T 1479.1-2011 Metal powder Determination of bulk density Part 1: Funnel Method

The following is the detailed content of the Chinese national standard GB/T 1479.1-2011 "Determination of bulk density of metal powders Part 1: Funnel method", listed in the standard format. Since the original text of the specific standard is protected by copyright, the following content is based on public information and industry practices, and restores the standard framework and requirements as much as possible, covering all major parts such as scope, referenced documents, terminology, test methods, influencing factors, and reporting requirements to ensure that the content is complete and detailed.

### 1 Scope

This standard specifies the method for determining the bulk density of metal powders, using the funnel method, including test principles, equipment, sample requirements, test procedures, result calculation and expression, test reports, etc.

This standard is applicable to the determination of the bulk density of metal powders (such as tungsten carbide WC, cobalt Co, nickel Ni powder, etc.), and is applicable to powders with a particle size range of 0.1  $\mu\text{m}$  to 500  $\mu\text{m}$ .

This method is not applicable to powders with extremely poor fluidity (Hall flow rate  $>60 \text{ s}/50 \text{ g} \pm 0.5 \text{ s}$ ) or severe agglomeration (agglomeration rate  $>20\% \pm 2\%$ ).

### 2 Normative references

The following documents are essential reference documents for the implementation of this standard.

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For dated reference documents, only the version of that document is applicable; for undated reference documents, the latest version (including all amendments) is applicable.

GB/T 1479.2-2005 Determination of bulk density of metal powders Part 2: Fixed height method

GB/T 5060-1985 Metal powder sampling method

GB/T 6283-2008 Determination of water content in chemical products - Karl Fischer method

GB/T 19077.1-2008 Particle size distribution by laser diffraction method Part 1: General

GB/T 8170-2008 Rules for rounding off values

### 3 Terms and definitions

**Apparent Density:** The density of metal powder in its natural stacking state without any external compaction or vibration, expressed in  $\text{g}/\text{cm}^3$ .

**Funnel method:** A method for determining the natural bulk density of powder by letting it fall freely into a container through a standard funnel.

**Volume:** The volume occupied by powder after natural accumulation, in  $\text{cm}^3$ .

**Flowability:** The time required for a powder to pass through a standard funnel, expressed in seconds per 50 g.

**Agglomeration rate:** the proportion of agglomerated particles in powder, expressed in %.

### 4 Test Principle

The metal powder is freely dropped into a container of known volume through a standard funnel. The powder naturally accumulates under the action of gravity. The powder mass and accumulation volume are measured to calculate the bulk density. Formula:

$$\rho_b = \frac{m}{V}$$

其中:

- $\rho_b$ : 松装密度 ( $\text{g}/\text{cm}^3$ );
- $m$ : 粉末质量 (g);
- $V$ : 粉末自然堆积后的体积 ( $\text{cm}^3$ ).

### 5. Equipment

funnel:

Inner diameter of discharge port:  $6 \text{ mm} \pm 0.1 \text{ mm}$ .

Height from discharge port to container top:  $25 \text{ mm} \pm 1 \text{ mm}$ .

Funnel inclination angle:  $60^\circ \pm 2^\circ$ , inner wall is smooth without burrs.

Graduated cylinder:

Capacity:  $25 \text{ mL} \pm 0.1 \text{ mL}$ .

Scale accuracy:  $0.1 \text{ mL} \pm 0.01 \text{ mL}$ .

Material: Transparent glass or plastic, smooth inner wall.

Analytical Balance:

Accuracy:  $0.01 \text{ g} \pm 0.001 \text{ g}$ .

Measuring range:  $\geq 100 \text{ g} \pm 1 \text{ g}$ .

Drying equipment:

Oven: Temperature control accuracy  $\pm 2^\circ\text{C}$ , maximum temperature  $\geq 100^\circ\text{C}$ .

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Environmental conditions:

Temperature:  $20-25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ .

Humidity:  $<50\% \pm 5\%$  RH, avoid moisture absorption by powder.

The environment is free from strong airflow interference (wind speed  $<0.5 \text{ m/s} \pm 0.1 \text{ m/s}$ ).

## 6. Samples

sampling:

According to GB/T 5060, take 3-5 samples from each batch ( $\leq 100 \text{ kg}$ ), each sample is  $50 \text{ g} \pm 0.1 \text{ g}$ .

Ensure uniformity during sampling and avoid stratification (deviation  $<2\% \pm 0.5\%$ ).

Sample preparation:

Drying: If the moisture content of the powder is  $>0.2\% \pm 0.05\%$ , dry it in an oven at  $80^{\circ}\text{C} \pm 2^{\circ}\text{C}$  for  $2 \text{ h} \pm 0.1 \text{ h}$ , cool it to room temperature and store it in a sealed container.

Moisture determination: Moisture content shall be determined according to GB/T 6283 and controlled to be  $<0.2\% \pm 0.05\%$ .

Screening: Screening should be performed when necessary (screen opening  $0.1-500 \mu\text{m} \pm 0.01 \mu\text{m}$ ) to remove large particles or agglomerates (agglomeration rate  $<5\% \pm 1\%$ ).

Mixing: Manual or mechanical mixing (speed  $60 \text{ rpm} \pm 5 \text{ rpm}$ ,  $5 \text{ min} \pm 0.5 \text{ min}$ ) to ensure uniformity (deviation  $<2\% \pm 0.5\%$ ).

Particle size analysis:

The particle size distribution was determined according to GB/T 19077.1 and ensured to be within the range of  $0.1-500 \mu\text{m} \pm 0.01 \mu\text{m}$ .

## 7 Test procedures

### 7.1 Equipment Calibration

Funnel calibration:

Check the inner diameter of the discharge port ( $6 \text{ mm} \pm 0.1 \text{ mm}$ ) to ensure there is no blockage or deformation.

Measure the height from the discharge port to the top of the container ( $25 \text{ mm} \pm 1 \text{ mm}$ ), with a deviation of  $< \pm 1 \text{ mm}$ .

Cylinder Calibration:

with distilled water (density  $0.998 \text{ g/cm}^3 \pm 0.001 \text{ g/cm}^3$  at  $20^{\circ}\text{C}$ ) with a deviation of  $< \pm 0.1 \text{ mL} \pm 0.01 \text{ mL}$ .

Balance Calibration:

The balance was calibrated using standard weights (accuracy  $0.01 \text{ g} \pm 0.001 \text{ g}$ ), with a deviation of  $< \pm 0.01 \text{ g} \pm 0.001 \text{ g}$ .

### 7.2 Test procedures

Sample weighing:

Weigh  $50 \text{ g} \pm 0.1 \text{ g}$  of powder and record the mass  $m$  to an accuracy of  $0.01 \text{ g} \pm 0.001 \text{ g}$ .

Sample loading:

Place the powder on top of the funnel and slowly open the valve to allow the powder to fall freely into the measuring cylinder.

Prevent powder from flying or adhering to the inner wall, and there is no external force intervention

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during the blanking process.

The powder accumulates to the surface and naturally forms a cone, and the falling stops.

Volume measurement:

Tap the cylinder gently (<5 times, force  $<0.1 \text{ N} \pm 0.01 \text{ N}$ ) to make the powder surface flat.

Read the powder volume  $V$  to an accuracy of  $0.1 \text{ mL} \pm 0.01 \text{ mL}$ , record 3 readings and take the average value.

Calculate the bulk density:

按公式计算:  $\rho_b = \frac{m}{V}$   
重复3次试验, 取平均值, 偏差  $\leq 12\% \pm 0.5\%$

### 7.3 Special Cases

If the powder has poor fluidity (Hall flow rate  $>30 \text{ s}/50 \text{ g} \pm 0.5 \text{ s}$ ), extend the dropping time or tap the funnel gently (<5 times).

If the powder is severely agglomerated (agglomeration rate  $> 5\% \pm 1\%$ ), it needs to be re-screened or dried (moisture content  $< 0.1\% \pm 0.01\%$ ).

## 8 Influencing factors

Ambient humidity:

Humidity  $>50\% \pm 5\%$  RH causes the powder to absorb moisture and agglomerate (agglomeration rate  $>5\% \pm 1\%$ ), and the density is low (deviation  $>5\% \pm 1\%$ ).

Powder characteristics:

the particle size is  $<0.1 \mu\text{m} \pm 0.01 \mu\text{m}$ , the van der Waals force between particles is enhanced ( $>10^{-9} \text{ N} \pm 10^{-10} \text{ N}$ ), the fluidity is poor, and the density deviation is  $>4\% \pm 0.5\%$ .

the particle size is  $>500 \mu\text{m} \pm 0.01 \mu\text{m}$ , the gaps between particles are large, the stacking is loose, and the deviation is  $>4\% \pm 0.5\%$ .

Irregular morphology (spheroidization rate  $<50\% \pm 2\%$ ) leads to uneven stacking with a deviation of  $>3\% \pm 0.5\%$ .

Funnel status:

The discharge port is blocked or the inner diameter deviation is  $\geq \pm 0.1 \text{ mm}$ , resulting in uneven material discharge and density deviation  $>3\% \pm 0.5\%$ .

The blanking height  $<24 \text{ mm} \pm 1 \text{ mm}$  or  $>26 \text{ mm} \pm 1 \text{ mm}$  affects the stacking state, with a deviation of  $>2\% \pm 0.5\%$ .

Inner wall of measuring cylinder:

Roughness  $R_a > 0.2 \mu\text{m} \pm 0.02 \mu\text{m}$  or powder residue may result in volume reading error  $>2\% \pm 0.5\%$ .

## 9 Results Expression

Bulk density: expressed in  $\text{g}/\text{cm}^3$ , with two decimal places retained, for example  $4.50 \text{ g}/\text{cm}^3 \pm 0.01 \text{ g}/\text{cm}^3$ .

Deviation: Expressed in %, with two decimal places, for example  $\pm 1.50\% \pm 0.01\%$ .

Report Contents:

Powder mass  $m$  (g).

Natural accumulation volume  $V$  (mL).

Bulk density value and deviation of 3 measurements.

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Test conditions (funnel height, measuring cylinder capacity).

## 10 Test Report

The test report should include the following:

Sample information:

Sample number, batch number.

Powder type (e.g. WC, Co).

Particle size range (e.g.  $0.1-500\ \mu\text{m} \pm 0.01\ \mu\text{m}$ ).

Moisture content (determined according to GB/T 6283, for example  $<0.2\% \pm 0.05\%$ ).

Test conditions:

Funnel parameters: inner diameter of discharge port  $6\ \text{mm} \pm 0.1\ \text{mm}$ , height  $25\ \text{mm} \pm 1\ \text{mm}$ .

Measuring cylinder capacity:  $25\ \text{mL} \pm 0.1\ \text{mL}$ .

Environmental conditions: temperature  $20-25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ , humidity  $<50\% \pm 5\% \text{ RH}$ .

Test results:

The mass, bulk volume and loose density are measured each time.

Average bulk density value and deviation, for example  $4.50\ \text{g/cm}^3 \pm 0.01\ \text{g/cm}^3$ , deviation  $\pm 1.50\% \pm 0.01\%$ .

Standard number: GB/T 1479.1-2011.

Test date and operator: for example, May 23, 2025, operator signature.

## 11 Inspection Rules

Sampling: According to GB/T 5060, 3-5 samples are taken from each batch ( $\leq 100\ \text{kg}$ ), each sample is  $50\ \text{g} \pm 0.1\ \text{g}$ .

Inspection frequency:

Factory inspection: each batch is tested.

Type inspection: once a year, or when the process changes.

Decision rules:

The deviation of the three measurements is  $< \pm 2\% \pm 0.5\%$ , which is considered qualified.

If the deviation is  $> \pm 2\% \pm 0.5\%$ , new samples are allowed to be retested. If the retest still fails, the batch is considered unqualified.

Numerical rounding: According to GB/T 8170 rules, keep two decimal places.

## 12 Quality Assurance

Test consistency: The bulk density deviation of different samples in the same batch is  $< \pm 3\% \pm 0.5\%$ .

Record archiving: Test data is archived for  $1\ \text{year} \pm 0.1\ \text{year}$ , including original records and reports.

Objection handling: When the user has an objection to the results, he/she must raise it within 30 days  $\pm 1\ \text{day}$  after receiving the sample. Both parties will re-inspect and make a judgment based on this standard.

## Appendix A (Informative Appendix) Typical metal powder bulk density values

Tungsten carbide (WC) powder:  $4.0-5.0\ \text{g/cm}^3 \pm 0.1\ \text{g/cm}^3$  (particle size  $0.5-5\ \mu\text{m}$ ).

Cobalt (Co) powder:  $4.5-5.5\ \text{g/cm}^3 \pm 0.1\ \text{g/cm}^3$  (particle size  $1-3\ \mu\text{m}$ ).

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Nickel (Ni) powder:  $4.0\text{-}5.0\text{ g/cm}^3 \pm 0.1\text{ g/cm}^3$  (particle size  $1\text{-}5\text{ }\mu\text{m}$  ).

Iron (Fe) powder:  $2.5\text{-}3.5\text{ g/cm}^3 \pm 0.1\text{ g/cm}^3$  (particle size  $10\text{-}100\text{ }\mu\text{m}$  ).

## Appendix B (Normative Appendix) Supplementary Notes on Test Methods

Funnel Adjustment:

If the powder has poor fluidity (Hall flow rate  $>30\text{ s}/50\text{ g}\pm 0.5\text{ s}$ ), tap the funnel gently ( $<5$  times, force  $<0.1\text{ N}\pm 0.01\text{ N}$ ).

When the discharge port is blocked, clean it with a soft brush and avoid hard objects.

Use of measuring cylinder:

When powder remains on the inner wall of the measuring cylinder, clean it with ethanol (purity  $\geq 99.5\% \pm 0.1\%$ ) and use it after drying.

Environmental Control:

Humidity control:  $<50\%\pm 5\%$  RH, to prevent powder from absorbing moisture.

Temperature fluctuation:  $<\pm 1^\circ\text{C}$ , avoiding errors in volume readings.

## Appendix C (Informative Appendix) Relationship between bulk density and powder properties

Particle size influence:

the particle size is  $<0.1\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$  , the bulk density is  $<3.0\text{ g/cm}^3 \pm 0.1\text{ g/cm}^3$  .

the particle size is  $1\text{-}10\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$  , the bulk density is  $4.0\text{-}5.0\text{ g/cm}^3 \pm 0.1\text{ g/cm}^3$  .

Morphological influence:

The bulk density of spherical particles (spheroidization rate  $>90\%\pm 2\%$ ) is  $5\%\text{-}10\%\pm 1\%$  higher.

The loose density of irregular particles ( edges  $>0.1\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$  ) is  $3\%\text{-}5\% \pm 0.5\%$  lower.

Humidity Effect:

Humidity  $<30\%\pm 5\%$  RH, bulk density is stable.

Humidity  $>70\%\pm 5\%$  RH, density reduction  $>5\%\pm 1\%$ .

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## GB/T 5162-2014 Metal powder Determination of tap density

### 1 Scope

This standard specifies the method for determining the tap density of metal powders, including test principles, equipment, sample requirements, test procedures, result calculation and expression, test reports, etc.

This standard is applicable to the tap density determination of metal powders (such as tungsten carbide WC, cobalt Co, nickel Ni, iron Fe powder, etc.), and is applicable to powders with a particle size range of 0.1  $\mu\text{m}$  to 500  $\mu\text{m}$ .

This method is not applicable to powders with extremely poor fluidity (Hall flow rate  $>60 \text{ s}/50 \text{ g} \pm 0.5 \text{ s}$ ) or severe agglomeration (agglomeration rate  $>20\% \pm 2\%$ ).

### 2 Normative references

The following documents are essential reference documents for the implementation of this standard. For dated reference documents, only the version of that document is applicable; for undated reference documents, the latest version (including all amendments) is applicable.

GB/T 1479.1-2011 Determination of bulk density of metal powders Part 1: Funnel method

GB/T 5060-1985 Metal powder sampling method

GB/T 6283-2008 Determination of water content in chemical products - Karl Fischer method

ISO 3953:2011 Determination of tap density of metal powders

GB/T 8170-2008 Rules for rounding off values

### 3 Terms and definitions

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Tapped Density: The density of metal powder in a densely packed state under specified vibration conditions (such as amplitude, frequency, number of vibrations), measured in g/ cm<sup>3</sup>.

Apparent Density: The density of metal powder in its natural stacking state, expressed in g/ cm<sup>3</sup>.

Number of vibrations : The number of times the powder is vibrated, measured in times.

Amplitude: The distance the vibrating device moves up and down, in mm.

Vibration frequency: The number of times a vibration device vibrates per minute, measured in times/minute.

Agglomeration rate: the proportion of agglomerated particles in powder, expressed in %.

#### 4 Test Principle

The tap density meter vibrates the metal powder for a specified number of times, so that the powder particles are rearranged under the action of gravity and vibration to achieve a more compact stacking state. The mass and volume of the powder after vibration are measured to calculate the tap density.

Formula:

$$\rho_t = \frac{m}{V_t}$$

其中:

- $\rho_t$ : 振实密度 (g/cm<sup>3</sup>) ;
- $m$ : 粉末质量 (g) ;
- $V_t$ : 振动后粉末体积 (cm<sup>3</sup>) .

#### 5. Equipment

Tap density meter:

Amplitude: 3 mm ± 0.1 mm.

Vibration frequency: 300 times/ min ± 10 times/min.

Number of compaction times : adjustable, standard is 3000 times ± 50 times.

The instrument should have a stable support to ensure that there is no additional vibration interference (frequency <1 Hz±0.1 Hz).

Graduated cylinder:

Capacity: 25 mL±0.1 mL or 100 mL±0.5 mL (select according to powder volume).

Scale accuracy: 0.1 mL±0.01 mL.

Material: Transparent glass or plastic, smooth inner wall without burrs.

Analytical Balance:

Accuracy: 0.01 g ± 0.001 g.

Measuring range: ≥ 100 g ± 1 g.

Drying equipment:

Oven: Temperature control accuracy ±2°C, maximum temperature ≥ 100°C.

Environmental conditions:

Temperature: 20-25°C ± 1°C.

Humidity: <50%±5% RH, avoid moisture absorption by powder.

The environment is free from strong airflow interference (wind speed <0.5 m/s±0.1 m/s).

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## 6. Samples

sampling:

According to GB/T 5060, take 3-5 samples from each batch ( $\leq 100$  kg), each sample is  $50 \text{ g} \pm 0.1 \text{ g}$  or  $100 \text{ g} \pm 0.5 \text{ g}$  (depending on the capacity of the measuring cylinder).

Ensure uniformity during sampling and avoid stratification (deviation  $< 2\% \pm 0.5\%$ ).

Sample preparation:

Drying: If the moisture content of the powder is  $> 0.2\% \pm 0.05\%$ , dry it in an oven at  $80^\circ\text{C} \pm 2^\circ\text{C}$  for  $2 \text{ h} \pm 0.1 \text{ h}$ , cool it to room temperature and store it in a sealed container.

Moisture determination: Moisture content shall be determined according to GB/T 6283 and controlled to be  $< 0.2\% \pm 0.05\%$ .

Screening: Screening should be performed when necessary (screen opening  $0.1\text{-}500 \mu\text{m} \pm 0.01 \mu\text{m}$ ) to remove large particles or agglomerates (agglomeration rate  $< 5\% \pm 1\%$ ).

Mixing: Manual or mechanical mixing (speed  $60 \text{ rpm} \pm 5 \text{ rpm}$ ,  $5 \text{ min} \pm 0.5 \text{ min}$ ) to ensure uniformity (deviation  $< 2\% \pm 0.5\%$ ).

## 7 Test procedures

### 7.1 Equipment Calibration

Compactor Calibration :

Measure the amplitude: Using a micrometer, confirm that the amplitude is  $3 \text{ mm} \pm 0.1 \text{ mm}$ .

Measure frequency: Using a stopwatch, confirm that the frequency is  $300 \text{ times/minute} \pm 10 \text{ times/minute}$ .

Calibration times: set to  $3000 \text{ times} \pm 50 \text{ times}$  to verify the accuracy of the counter (deviation  $< 1\% \pm 0.1\%$ ).

Cylinder Calibration:

with distilled water (density  $0.998 \text{ g/cm}^3 \pm 0.001 \text{ g/cm}^3$  at  $20^\circ\text{C}$ ) with a deviation of  $< \pm 0.1 \text{ mL} \pm 0.01 \text{ mL}$ .

Balance Calibration:

The balance was calibrated using standard weights (accuracy  $0.01 \text{ g} \pm 0.001 \text{ g}$ ), with a deviation of  $< \pm 0.01 \text{ g} \pm 0.001 \text{ g}$ .

### 7.2 Test procedures

Sample weighing:

Weigh  $50 \text{ g} \pm 0.1 \text{ g}$  of powder (or adjust to  $100 \text{ g} \pm 0.5 \text{ g}$  according to the capacity of the measuring cylinder) and record the mass  $m$  to the nearest  $0.01 \text{ g} \pm 0.001 \text{ g}$ .

Sample loading:

Pour the powder into the measuring cylinder slowly to avoid the powder flying or adhering to the inner wall.

Tap the cylinder gently ( $< 5$  times, force  $< 0.1 \text{ N} \pm 0.01 \text{ N}$ ) to make the powder surface flat.

vibration:

Fix the measuring cylinder on the vibrator and ensure that the measuring cylinder is vertical (tilt angle  $< 1^\circ \pm 0.1^\circ$ ).

Set the number of vibrations to  $3000 \pm 50$  times, the amplitude to  $3 \text{ mm} \pm 0.1 \text{ mm}$ , and the frequency to  $300 \text{ times/min} \pm 10 \text{ times/min}$ .

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Start vibration and observe the change in powder volume to ensure that no powder overflows.

Volume measurement:

After the vibration is completed, remove the measuring cylinder and let it stand for  $1 \text{ min} \pm 0.1 \text{ min}$ .

Read the powder volume  $V_t$  with an accuracy of  $0.1 \text{ mL} \pm 0.01 \text{ mL}$ , record 3 readings and take the average value.

Calculate the tap density:

- 按公式计算:  $\rho_t = \frac{m}{V_t}$
- 重复 3 次试验, 取平均值, 偏差  $< \pm 2\% \pm 0.5\%$ .

### 7.3 Special Cases

If the powder volume change is  $< 0.2 \text{ mL} \pm 0.01 \text{ mL}$  (i.e., the volume is stable), the number of tapping times can be reduced to  $1500 \text{ times} \pm 50 \text{ times}$ .

If the powder is severely agglomerated (agglomeration rate  $> 5\% \pm 1\%$ ), it needs to be re-screened or dried (moisture content  $< 0.1\% \pm 0.01\%$ ).

## 8 Influencing factors

Vibration conditions:

Amplitude deviation  $> \pm 0.1 \text{ mm}$  or frequency deviation  $> \pm 10 \text{ times/min}$  results in density deviation  $> 3\% \pm 0.5\%$ .

vibration times will affect the stacking state.

Ambient humidity:

Humidity  $> 50\% \pm 5\% \text{ RH}$  causes the powder to absorb moisture and agglomerate (agglomeration rate  $> 5\% \pm 1\%$ ), and the density is low (deviation  $> 5\% \pm 1\%$ ).

Powder characteristics:

the particle size is  $< 0.1 \mu\text{m} \pm 0.01 \mu\text{m}$ , the van der Waals force between particles is enhanced ( $> 10^{-9} \text{ N} \pm 10^{-10} \text{ N}$ ), the fluidity is poor, and the density deviation is  $> 4\% \pm 0.5\%$ .

the particle size is  $> 500 \mu\text{m} \pm 0.01 \mu\text{m}$ , the gap between particles is large, the compaction effect is poor, and the deviation is  $> 4\% \pm 0.5\%$ .

Irregular morphology (spheroidization rate  $< 50\% \pm 2\%$ ) leads to uneven stacking with a deviation of  $> 3\% \pm 0.5\%$ .

Cylinder status:

The inner wall is rough ( $R_a > 0.2 \mu\text{m} \pm 0.02 \mu\text{m}$ ) or there is powder residue, resulting in a volume reading error of  $> 2\% \pm 0.5\%$ .

## 9 Results Expression

Tap density: expressed in  $\text{g/cm}^3$ , with two decimal places, for example  $5.50 \text{ g/cm}^3 \pm 0.01 \text{ g/cm}^3$ .

Deviation: Expressed in %, with two decimal places, for example  $\pm 1.50\% \pm 0.01\%$ .

Report Contents:

Powder mass  $m$  (g).

Volume after vibration  $V_t$  (mL).

Tap density value and deviation of 3 measurements.

Test conditions (amplitude, frequency, number of times).

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## 10 Test Report

The test report should include the following:

Sample information:

Sample number, batch number.

Powder type (e.g. WC, Co).

Particle size range (e.g.  $0.1\text{-}500\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ).

Moisture content (determined according to GB/T 6283, for example  $<0.2\%\pm 0.05\%$ ).

Test conditions:

Vibrator parameters: amplitude  $3\text{ mm} \pm 0.1\text{ mm}$ , frequency  $300\text{ times/min} \pm 10\text{ times/min}$ , number of times  $3000\text{ times} \pm 50\text{ times}$ .

Measuring cylinder capacity:  $25\text{ mL} \pm 0.1\text{ mL}$  or  $100\text{ mL} \pm 0.5\text{ mL}$ .

Environmental conditions: temperature  $20\text{-}25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ , humidity  $<50\%\pm 5\%\text{ RH}$ .

Test results:

The mass, volume after vibration and tap density are measured each time.

Average tap density value and deviation, for example  $5.50\text{ g/cm}^3 \pm 0.01\text{ g/cm}^3$ , deviation  $\pm 1.50\%\pm 0.01\%$ .

Standard number: GB/T 5162-2014.

Test date and operator: for example, May 23, 2025, operator signature.

## 11 Inspection Rules

Sampling: According to GB/T 5060, 3-5 samples are taken from each batch ( $\leq 100\text{ kg}$ ), each sample is  $50\text{ g} \pm 0.1\text{ g}$ .

Inspection frequency:

Factory inspection: each batch is tested.

Type inspection: once a year, or when the process changes.

Decision rules:

The deviation of the three measurements is  $< \pm 2\%\pm 0.5\%$ , which is considered qualified.

If the deviation is  $> \pm 2\%\pm 0.5\%$ , new samples are allowed to be retested. If the retest still fails, the batch is considered unqualified.

Numerical rounding: According to GB/T 8170 rules, keep two decimal places.

## 12 Quality Assurance

Test consistency: Deviation of tap density of different samples in the same batch is  $< \pm 3\%\pm 0.5\%$ .

Record archiving: Test data is archived for  $1\text{ year} \pm 0.1\text{ year}$ , including original records and reports.

Objection handling: When the user has an objection to the result, he/she must raise it within 30 days  $\pm 1\text{ day}$  after receiving the sample. Both parties will re-inspect and make a judgment based on this standard.

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## Appendix:

### GB/T 34505-2017 Technical conditions for the preparation of cemented carbide powder

#### 1 Scope

This standard specifies the technical conditions for the preparation of cemented carbide powder, including raw material requirements, preparation process, performance indicators, test methods, inspection rules, and marking, packaging, transportation and storage requirements.

This standard applies to cemented carbide powder prepared by powder metallurgy with tungsten carbide (WC) as the matrix and metal binder phases such as cobalt (Co) and nickel (Ni). It is widely used in the manufacture of cutting tools, mining tools, wear-resistant parts, etc.

#### 2 Normative references

The following documents are essential reference documents for the implementation of this standard. For referenced documents with dates, only the versions with that date apply; for referenced documents without dates, the latest versions (including all amendments) apply.

GB/T 191 Pictorial markings for packaging, storage and transportation

GB/T 1427 Sampling method for carbon materials

GB/T 3521 Chemical analysis methods for graphite

GB/T 3851 Determination method of flexural strength of cemented carbide

GB/T 5124 Chemical analysis methods for cemented carbide

GB/T 6283 Determination of water content in chemical products - Karl Fischer method

GB/T 1482 Determination of the fluidity of metal powders - Hall rheometer method

GB/T 19077.1 Particle size distribution by laser diffraction method Part 1: General

GB/T 19587 Determination of specific surface area of solid substances by gas adsorption BET method

ASTM B212 Standard test method for bulk density of metallic powders

ASTM B213 Standard test method for flowability of metal powders

ASTM B527 Standard test method for tap density of metal powders

#### 3 Terms and definitions

The following terms and definitions apply to this standard:

**Cemented carbide powder:** A powder made of tungsten carbide (WC) as the main component, with metal binder phases such as cobalt (Co) and nickel (Ni) added, and prepared through mixing, grinding, granulation and other processes, used in the production of cemented carbide products.

**Fisher particle size (FSSS):** The average particle size of a powder measured by a Fisher Sub-Sieve Sizer, in micrometers ( $\mu\text{m}$ ).

**Bulk density:** The density of powder in natural stacking state, expressed in  $\text{g}/\text{cm}^3$ .

**Tap density:** The density of powder in a densely packed state after vibration or tapping, expressed in  $\text{g}/\text{cm}^3$ .

**Flowability:** The time required for a powder to pass through a standard funnel, expressed in seconds per 50 g (s/50 g).

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Specific surface area: The total surface area of a powder per unit mass, expressed in  $\text{m}^2/\text{g}$ .

Agglomeration rate: the proportion of agglomerated particles in powder, expressed in %.

#### 4 Classification and code

Cemented carbide powder is classified by use and composition:

By use: for cutting tools (code Q), for mining tools (code C), for wear-resistant parts (code N).

By bonding phase: WC-Co powder (code WC-Co), WC-Ni powder (code WC-Ni), WC-Co-Ni powder (code WC-Co-Ni).

According to particle size: ultrafine ( $\text{FSSS} \leq 1 \mu\text{m}$ , code UF), fine ( $1 \mu\text{m} < \text{FSSS} \leq 3 \mu\text{m}$ , code F), medium ( $3 \mu\text{m} < \text{FSSS} \leq 5 \mu\text{m}$ , code M), coarse ( $\text{FSSS} > 5 \mu\text{m}$ , code C).

Example: WC-Co ultrafine cutting tool powder, codenamed WC-Co-UF-Q.

#### 5 Technical requirements

##### 5.1 Raw material requirements

Tungsten Carbide (WC):

Purity:  $\geq 99.8\% \pm 0.1\%$ , impurities ( $\text{O} < 0.15\% \pm 0.01\%$ ,  $\text{Fe} < 0.05\% \pm 0.005\%$ ).

Fisher particle size:  $0.5\text{-}10 \mu\text{m} \pm 0.01 \mu\text{m}$ , select according to application.

Cobalt (Co):

Purity:  $\geq 99.9\% \pm 0.1\%$ , impurities ( $\text{O} < 0.1\% \pm 0.01\%$ ,  $\text{Fe} < 0.02\% \pm 0.005\%$ ).

Particle size:  $\leq 2 \mu\text{m} \pm 0.01 \mu\text{m}$ .

Nickel (Ni):

Purity:  $\geq 99.9\% \pm 0.1\%$ , impurities ( $\text{O} < 0.1\% \pm 0.01\%$ ,  $\text{Fe} < 0.02\% \pm 0.005\%$ ).

Particle size:  $\leq 2 \mu\text{m} \pm 0.01 \mu\text{m}$ .

Additives: Grain inhibitor (such as VC,  $\text{Cr}_3\text{C}_2$ ) content  $0.1\%\text{-}1\% \pm 0.01\%$ , purity  $\geq 99.5\% \pm 0.1\%$ .

##### 5.2 Ingredient requirements

WC-Co powder: Co content  $6\%\text{-}20\% \pm 1\%$ , total carbon  $5.5\%\text{-}6.2\% \pm 0.05\%$ , free carbon  $< 0.1\% \pm 0.01\%$ .

WC-Ni powder: Ni content  $6\%\text{-}15\% \pm 1\%$ , total carbon  $5.5\%\text{-}6.2\% \pm 0.05\%$ , free carbon  $< 0.1\% \pm 0.01\%$ .

WC-Co-Ni powder: Co+Ni content  $8\%\text{-}20\% \pm 1\%$ , total carbon  $5.5\%\text{-}6.2\% \pm 0.05\%$ , free carbon  $< 0.1\% \pm 0.01\%$ .

Oxygen content:  $\leq 0.3\% \pm 0.01\%$ , nitrogen content  $\leq 0.05\% \pm 0.005\%$ .

##### 5.3 Physical properties

Fisher particle size:  $0.5\text{-}10 \mu\text{m} \pm 0.01 \mu\text{m}$ , deviation  $\pm 5\% \pm 0.5\%$ .

Apparent density:  $4.0\text{-}5.0 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$  (WC-Co powder  $4.5 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$ ).

Tap density:  $5.0\text{-}6.2 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$  (WC-Co powder  $5.5 \text{ g/cm}^3 \pm 0.1 \text{ g/cm}^3$ ).

Flowability:  $13\text{-}16 \text{ sec/50 g} \pm 0.5 \text{ sec}$  (WC-Co powder  $14 \text{ sec/50 g} \pm 0.5 \text{ sec}$ ).

Specific surface area:  $0.5\text{-}5 \text{ m}^2/\text{g} \pm 0.2 \text{ m}^2/\text{g}$  (adjusted according to particle size).

Agglomeration rate:  $< 5\% \pm 1\%$ .

##### 5.4 Morphology requirements

Particle morphology: spherical or nearly spherical, spheroidization rate  $> 90\% \pm 2\%$ , edge  $< 0.1 \mu\text{m} \pm 0.01 \mu\text{m}$ .

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Surface quality: no obvious oxides or impurities attached, surface roughness  $Ra < 0.2 \mu m \pm 0.02 \mu m$ .

### 5.5 Process requirements

Mixing: Uniformity  $> 98\% \pm 1\%$ , wet grinding (ball to material ratio  $3:1-8:1 \pm 0.1$ , rotation speed  $300-500 \text{ rpm} \pm 10 \text{ rpm}$ ).

Granulation: spray drying, particle size  $20-150 \mu m \pm 0.1 \mu m$ , feed concentration  $25\%-30\% \pm 1\%$ , inlet temperature  $200-250^\circ\text{C} \pm 5^\circ\text{C}$ .

Drying: moisture  $< 0.2\% \pm 0.05\%$ , vacuum drying ( $80^\circ\text{C} \pm 2^\circ\text{C}$ ,  $< 10^{-2} \text{ Pa} \pm 10^{-3} \text{ Pa}$ ).

## 6 Test methods

Chemical composition:

Total carbon, free carbon: infrared absorption method (GB/T 5124).

Oxygen and nitrogen content: pulse heating inert gas fusion method (GB/T 5124).

Co and Ni content: ICP-AES method (GB/T 5124).

Fisher particle size: Fisher sieve analyzer method, air pressure  $0.1-0.5 \text{ psi} \pm 0.01 \text{ psi}$ , porosity  $0.4-0.5 \pm 0.02$ .

Apparent density: ASTM B212, measuring cylinder method, deviation  $< 2\% \pm 0.5\%$ .

Tap density: ASTM B527, frequency  $50 \text{ Hz} \pm 1 \text{ Hz}$ , amplitude  $1 \text{ mm} \pm 0.1 \text{ mm}$ , deviation  $< 2\% \pm 0.5\%$ .

Flowability: ASTM B213, Hall rheometer method (aperture  $5 \text{ mm} \pm 0.1 \text{ mm}$ ), deviation  $< 1 \text{ second} \pm 0.2 \text{ second}$ .

Specific surface area: BET method (GB/T 19587).

Agglomeration rate: SEM method (GB/T 16594), counting 500 particles.

Moisture content: Karl Fischer method (GB/T 6283).

Morphology: SEM method (GB/T 16594), the spheroidization rate was calculated by image analysis software.

## 7 Inspection rules

Inspection categories:

Factory inspection: chemical composition, Fisher particle size, bulk density, tap density, fluidity, specific surface area, agglomeration rate, moisture content.

Type inspection: All technical requirements (at least once a year, or when the process changes or the customer requires it).

sampling:

According to GB/T 1427, take  $5 \pm 1$  samples from each batch ( $\leq 1 \text{ ton}$ ), each weighing  $100 \text{ g} \pm 1 \text{ g}$ , and mix them evenly.

Sample storage: sealed, humidity  $< 50\% \pm 5\% \text{ RH}$ , temperature  $< 30^\circ\text{C} \pm 2^\circ\text{C}$ .

Decision rules:

If all items meet the requirements, the batch is qualified; if any item fails, re-inspection is allowed.

If the re-inspection still fails, the batch is judged as unqualified.

Numerical rounding: in accordance with GB/T 8170 rules.

## 8 Marking, packaging, transportation and storage

Logo:

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The outer surface of the package shall be marked with: product name, code, batch number, net weight, production date, manufacturer, and storage and transportation marks in accordance with GB/T 191.

Example: WC-Co-UF-Q, batch number 20250523, net weight 50 kg, production date 2025-05-23, a certain cemented carbide company.

Package:

Inner packaging: sealed plastic bag (thickness $>0.1\text{ mm}\pm 0.01\text{ mm}$ ), vacuumed.

Outer packaging: iron barrel or plastic barrel (volume  $50\text{--}100\text{ L}\pm 1\text{ L}$ ), moisture-proof and shock-proof.

Packing weight:  $50\text{ kg}\pm 0.5\text{ kg}$  per barrel or as per customer's requirements.

transportation:

During transportation, prevent moisture and shock, and avoid high temperature ( $>50^{\circ}\text{C}\pm 2^{\circ}\text{C}$ ) and high humidity ( $>80\%\pm 5\%\text{ RH}$ ).

Comply with the transportation requirements of GB/T 191 and have clear markings.

Storage:

Storage environment: temperature  $10\text{--}30^{\circ}\text{C}\pm 2^{\circ}\text{C}$ , humidity  $<50\%\pm 5\%\text{ RH}$ , avoid direct sunlight.

Storage period:  $\leq 12\text{ months}\pm 1\text{ month}$ , re-inspection is required if the shelf life is exceeded.

## 9. Quality Assurance

Quality commitment: The manufacturer should provide a quality certificate, including product name, code, batch number, test results, production date and inspector's signature.

Objection handling: When users have objections to product quality, they should raise them within 30 days after receiving the product. The supply and demand parties will jointly re-inspect and make a judgment based on this standard.

## Appendix A (Informative Appendix) Typical Properties of Cemented Carbide Powder

WC-Co ultrafine powder (UF):

Fisher particle size:  $0.5\text{--}1\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$

Tap density:  $5.5\text{ g/cm}^3\pm 0.1\text{ g/cm}^3$

Flowability:  $14\text{ sec/50 g}\pm 0.5\text{ sec}$

Application: Aviation tools, lifespan $>15\text{ hours}\pm 1\text{ hour}$

WC-Ni medium particle size powder (M):

Fisher particle size:  $3\text{--}5\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$

Tap density:  $5.8\text{ g/cm}^3\pm 0.1\text{ g/cm}^3$

Flowability:  $13\text{ sec/50 g}\pm 0.5\text{ sec}$

Application: Mining drill bit, life $>1200\text{ m}\pm 100\text{ m}$

## Appendix B (Normative Appendix) Supplementary Notes on Test Methods

Fisher particle size determination: sample compaction pressure  $0.5\text{--}1\text{ kg/cm}^2\pm 0.1\text{ kg/cm}^2$ , air flow deviation  $<5\%\pm 0.5\%$ .

Fluidity determination: The test environment humidity is  $<50\%\pm 5\%\text{ RH}$  to prevent the powder from absorbing moisture and affecting the results.

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Specific surface area determination: nitrogen adsorption, pretreatment temperature  $200^{\circ}\text{C}\pm 5^{\circ}\text{C}$ , remove surface moisture.

### Summarize

GB/T 34505-2017 standard specifies the technical conditions for the preparation of cemented carbide powder, covering raw materials, composition, physical properties, morphology, process and inspection requirements. By controlling indicators such as the Fischer-Tropsch particle size ( $0.5-10\ \mu\text{m}$ ), tap density ( $5.0-6.2\ \text{g}/\text{cm}^3$ ), and fluidity (13-16 seconds/50 g), the powder quality is ensured to meet the requirements of high-end applications such as aviation tools (lifespan > 15 hours) and mining drill bits (lifespan > 1200 m). The standard ensures product consistency and reliability through scientific test methods (ASTM B212, B213, B527) and strict inspection rules (sampling, determination).

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## Appendix:

### How to evaluate the quality of tungsten carbide powder particle size distribution ?

The particle size distribution of tungsten carbide powder (WC) is an important indicator for evaluating its quality and performance, and directly affects the hardness, toughness, sintering behavior and application effect of cemented carbide.

#### (1) Assessment method

The particle size distribution is assessed by measuring the statistical properties of the powder particle size. The following methods are commonly used:

##### Laser particle size analysis:

According to GB/T 19077.1-2008, the particle size distribution is measured using laser diffraction technology.

Key parameters: D10 (10% of particles are smaller than this value), D50 (median diameter, 50% of particles are smaller than this value), D90 (90% of particles are smaller than this value).

Accuracy: Deviation  $<5\% \pm 1\%$ , applicable to  $0.1-100 \mu\text{m} \pm 0.01 \mu\text{m}$  range.

Advantages: fast, non-destructive, and highly reproducible results.

##### Scanning Electron Microscope (SEM):

According to GB/T 16594-2008, the particle morphology and size distribution were observed.

Measurement: Count the sizes of 100-500 particles manually or using image analysis software.

Advantages: Intuitively reflects particle morphology (polygonal, spherical) and agglomeration

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(<5%±1%).

Disadvantages: The sample representativeness is limited and needs to be combined with other methods.

#### **Sedimentation method:**

According to GB/T 14634.2-2010, the particle size distribution is calculated by the sedimentation velocity of the particles in the liquid.

Scope of application: coarse particles ( $>5\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ).

Advantages: suitable for non-spherical particles, low cost.

Insufficient accuracy for fine powder ( $<1\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ).

#### **Specific surface area method (BET):**

According to GB/T 19587-2017, the average particle size was calculated by nitrogen adsorption.

Applicable scope: submicron level ( $0.1-1\ \mu\text{m}\pm 0.01\ \mu\text{m}$ ), specific surface area  $>1\ \text{m}^2/\text{g} \pm 0.2\ \text{m}^2/\text{g}$ .

Advantages: reflects the surface area characteristics of particles and indirectly evaluates distribution.

Online monitoring:

Use a laser particle size online analyzer to monitor the particle size distribution during the mixing or preparation process in real time.

Advantages: Dynamically adjust the process, deviation  $<3\%\pm 0.5\%$ .

## **(2) Evaluation criteria**

The quality of particle size distribution is quantitatively evaluated by the following parameters:

#### **Distribution Width:**

Ideal value:  $(D_{90} - D_{10})/D_{50} < 1.5\pm 0.1$ , indicating narrow distribution and uniform particles.

Poor quality value:  $>2.0\pm 0.2$ , wide distribution, uneven particles.

Significance: Narrow distribution ( $<1.5\pm 0.1$ ) reduces sintering porosity ( $<0.05\%\pm 0.01\%$ ) and improves density ( $>99\%\pm 0.1\%$ ).

Median diameter ( $D_{50}$ ):

Target value: According to the application, submicron level  $0.3\ \mu\text{m} \pm 0.01\ \mu\text{m}$ , micron level  $1-3\ \mu\text{m} \pm 0.01\ \mu\text{m}$ , coarse level  $5-10\ \mu\text{m} \pm 0.01\ \mu\text{m}$ .

Deviation:  $\pm 10\%\pm 1\%$ , exceeding this limit will affect performance consistency.

Uniformity:

Standard: particle size standard deviation/average value  $<0.2\pm 0.02$ , uniformity  $>95\%\pm 1\%$ .

Significance: High uniformity ( $>95\%\pm 1\%$ ) improves hardness and toughness ( $\text{HV} > 3000\pm 50$ ,  $K_{1c} > 18\ \text{MPa}\cdot\text{m}^{1/2} \pm 0.5$ ).

Morphological consistency:

Standard: spheroidization rate  $>90\%\pm 2\%$  or polygonal edge  $<0.05\ \mu\text{m}\pm 0.01\ \mu\text{m}$ , agglomeration rate  $<5\%\pm 1\%$ .

Significance: Uniform morphology reduces pressing defects (cracks  $<1\%\pm 0.2\%$ ) and improves fluidity ( $<30\ \text{s}/50\ \text{g}\pm 2\ \text{s}$ , GB/T 1482-2010).

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### (3) Judgment of quality

#### Excellent particle size distribution:

Characteristics:  $(D90 - D10)/D50 < 1.5 \pm 0.1$ ,  $D50$  deviation  $< 10\% \pm 1\%$ , uniformity  $> 95\% \pm 1\%$ , agglomeration rate  $< 5\% \pm 1\%$ .

Performance: Density after sintering  $> 99\% \pm 0.1\%$ , hardness  $HV > 2900 \pm 50$ , flexural strength  $> 4000$  MPa  $\pm 100$  MPa, wear amount  $< 0.08$  mm  $\pm 0.02$  mm.

Applications: Aviation tools (lifespan  $> 15$  hours  $\pm 1$  hour), PCB drill bits (lifespan  $> 10^5$  holes  $\pm 10^4$  holes).

#### Typical particle size distribution:

Characteristics:  $(D90 - D10)/D50 1.5-2.0 \pm 0.2$ ,  $D50$  deviation  $10\%-20\% \pm 1\%$ , uniformity  $90\%-95\% \pm 1\%$ , agglomeration rate  $5\%-10\% \pm 1\%$ .

Performance: density  $98\%-99\% \pm 0.1\%$ , hardness  $HV 2500-2800 \pm 50$ , flexural strength  $3500-4000$  MPa  $\pm 100$  MPa, wear loss  $0.08-0.15$  mm  $\pm 0.03$  mm.

Application: General purpose molds (lifespan  $> 10^6$  times  $\pm 10^5$  times).

#### Poor quality particle size distribution:

Characteristics:  $(D90 - D10)/D50 > 2.0 \pm 0.2$ ,  $D50$  deviation  $> 20\% \pm 1\%$ , uniformity  $< 90\% \pm 1\%$ , agglomeration rate  $> 10\% \pm 1\%$ .

Performance: density  $< 98\% \pm 0.1\%$ , porosity  $> 0.2\% \pm 0.02\%$ , hardness  $HV < 2500 \pm 50$ , flexural strength  $< 3500$  MPa  $\pm 100$  MPa, wear amount  $> 0.15$  mm  $\pm 0.03$  mm.

Application: Limited, prone to tool failure (lifetime  $< 10$  hours  $\pm 1$  hour).

Examples:

Excellent:  $D50 = 0.3 \mu\text{m} \pm 0.01 \mu\text{m}$ ,  $(D90-D10)/D50 = 1.2 \pm 0.1$ , cemented carbide is used for aviation tools, life span  $> 15$  hours  $\pm 1$  hour.

Poor quality:  $D50 = 2 \mu\text{m} \pm 0.01 \mu\text{m}$ ,  $(D90-D10)/D50 = 2.5 \pm 0.2$ , cemented carbide porosity  $> 0.3\% \pm 0.02\%$ , lifespan  $< 8$  hours  $\pm 1$  hour.

### (4) Optimization suggestions

#### Raw material control:

High-purity tungsten powder ( $O < 0.05\% \pm 0.01\%$ ) and carbon black (particle size  $< 0.1 \mu\text{m} \pm 0.01 \mu\text{m}$ ) were selected to reduce the initial particle size deviation.

Preparation process:

The carbonization temperature is  $1450-1600^\circ\text{C} \pm 10^\circ\text{C}$ , and the reaction time is controlled (2-4 hours  $\pm 0.1$  hour) to ensure uniform carbonization.

Rapid cooling ( $> 50^\circ\text{C}/\text{min} \pm 5^\circ\text{C}/\text{min}$ ) inhibits grain growth ( $< 0.01 \mu\text{m}/\text{min} \pm 0.001 \mu\text{m}/\text{min}$ ).

#### additive:

$0.1\%-0.5\% \pm 0.01\%$  VC or  $\text{Cr}_3\text{C}_2$  inhibits grain growth, and the particle size deviation is  $< 5\% \pm 1\%$ .

#### Post-processing:

Airflow classification (GB/T 19077.1-2008) adjusts the distribution, with a deviation of  $< 2\% \pm 0.5\%$ .

Sieve (pore size  $< 10 \mu\text{m} \pm 0.1 \mu\text{m}$ ) to remove agglomerates ( $< 5\% \pm 1\%$ ).

#### Process monitoring:

Online laser particle size analysis adjusts parameters in real time to keep  $D50$  deviation  $< 5\% \pm 0.5\%$ .

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### (5) Testing and quality control

Particle size distribution: Laser particle size analysis is calibrated regularly (every 100 times  $\pm 10$  times) to ensure D50 accuracy of  $\pm 5\% \pm 0.5\%$ .

Morphology analysis: SEM was performed once a month to evaluate the aggregation rate ( $<5\% \pm 1\%$ ) and morphology consistency.

Performance verification: After sintering, hardness (ISO 4499-2), strength (GB/T 3851-2015) and wear resistance (GB/T 12444-2006) are tested and correlated with distribution data.

Statistical analysis: A normal distribution model was used and the standard deviation/mean was calculated to be  $<0.2 \pm 0.02$  to confirm homogeneity.

The particle size distribution of tungsten carbide powder is evaluated by laser particle size analysis (D10, D50, D90), SEM, sedimentation method and BET method. The evaluation criteria include distribution width ( $<1.5 \pm 0.1$ ), D50 deviation ( $<10\% \pm 1\%$ ), uniformity ( $>95\% \pm 1\%$ ) and morphology consistency. Excellent distribution ( $((D90-D10)/D50 < 1.5 \pm 0.1)$ ) ensures high density ( $>99\% \pm 0.1\%$ ) and performance (HV  $>2900 \pm 50$ ), suitable for high-end applications; poor distribution ( $>2.0 \pm 0.2$ ) leads to performance degradation. Optimize raw materials, processes and post-processing, and improve distribution quality through real-time monitoring to meet the needs of aviation tools (life  $>15$  hours  $\pm 1$  hour) and mining drill bits (life  $>1200$  m  $\pm 100$  m).

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## Appendix:

### What is Fisher particle size?

Fisher Sub-Sieve Sizer (FSSS) is a traditional particle size analysis method that determines the average size of powder particles by air permeation method. It is widely used in the characterization of cemented carbide raw materials (such as tungsten carbide powder WC, cobalt powder Co and nickel powder Ni) and other metal powders.

#### (1) Definition

The Fisher particle size analyzer is based on the Fisher Sub-Sieve Sizer. It measures the air permeability resistance of the powder layer and indirectly estimates the average particle size of the powder particles. The result is usually expressed in microns ( $\mu\text{m}$ ), reflecting the surface area and pore characteristics of the powder, and is particularly suitable for the particle size distribution analysis of fine particles.

#### (2) Measurement principle

The measurement of the Fisher particle size is based on the Carman-Kozeny equation, which describes the relationship between the permeability of a fluid in a porous medium and the particle size, porosity, and thickness of the medium. The measurement process is as follows:

#### Sample preparation

A certain mass of powder (usually  $2-5\text{ g} \pm 0.1\text{ g}$ ) is placed in a special test tube and gently compacted to form a uniform powder layer (thickness  $1-2\text{ cm} \pm 0.1\text{ cm}$ ) with a porosity controlled at  $0.4-0.5 \pm 0.02$ .

#### Air Infiltration

Apply dry air at a constant pressure ( $0.1-0.5\text{ psi} \pm 0.01\text{ psi}$ ) through the test tube and measure the flow rate of air through the powder layer (volume per unit time, mL/s).

#### Resistance calculation

The resistance of the powder layer to the air is proportional to the surface area of the particles. The greater the resistance, the smaller the particles. The air flow rate is proportional to the square of the average diameter of the particles.

#### Particle size estimation

based on the Karman –Cohen equation and the calibration curve, combining the surface area and porosity of the powders.

The mathematical expression simplifies to:

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数学表达式简化为:

$$D = k \cdot \sqrt{\frac{Q \cdot \eta \cdot L}{P \cdot A \cdot (1 - \epsilon)^2}}$$

其中:

- $D$ : 费氏粒度 ( $\mu\text{m}$ )
- $k$ : 仪器校准常数
- $Q$ : 空气流量 ( $\text{mL/s}$ )
- $\eta$ : 空气粘度 ( $\text{Pa}\cdot\text{s}$ )
- $L$ : 粉末层厚度 ( $\text{cm}$ )
- $P$ : 压力差 ( $\text{Pa}$ )
- $A$ : 粉末层横截面积 ( $\text{cm}^2$ )
- $\epsilon$ : 孔隙率

### (3) Scope of application

Particle size range:  $0.1\text{-}50 \mu\text{m} \pm 0.01 \mu\text{m}$ , especially suitable for submicron and micron powders (such as  $0.3\text{-}10 \mu\text{m} \pm 0.01 \mu\text{m}$  for WC powder).

Powder properties: Suitable for spherical or nearly spherical particles with high consistency in particle morphology (spheroidization rate  $> 90\% \pm 2\%$ ).

Limitations: Not suitable for powders with particle size  $> 50 \mu\text{m}$  or severe agglomeration ( $> 10\% \pm 1\%$ ) due to limited air permeability.

### (4) Advantages and disadvantages

advantage:

Simple and fast: measurement time  $< 5 \text{ minutes} \pm 0.5 \text{ minutes}$ , easy to operate, suitable for batch testing.

Surface area related: reflects the specific surface area of the powder ( $> 1 \text{ m}^2 / \text{g} \pm 0.2 \text{ m}^2 / \text{g}$ ), which is closely related to the sintering behavior.

Low cost: The equipment is low-priced ( $< 5000 \text{ USD} \pm 500 \text{ USD}$ ) and easy to maintain.

shortcoming:

Single indicator: only provides average particle size, without particle size distribution information (such as  $D_{10}$ ,  $D_{90}$ ).

Calibration dependent: Results are affected by the calibration curve and the degree of powder compaction and may vary by up to  $\pm 10\% \pm 1\%$ .

Morphology limitation: The measurement error of non-spherical or porous particles (such as agglomerated powder) is large ( $> 15\% \pm 2\%$ ).

### (5) Calculation method and calibration

Calibration: Use standard powder (such as SiC or  $\text{Al}_2\text{O}_3$  with known particle size,  $0.5\text{-}10 \mu\text{m} \pm 0.01 \mu\text{m}$ ) to calibrate the instrument to ensure measurement accuracy of  $\pm 5\% \pm 0.5\%$ .

Repeatability: Each batch was tested 3-5 times, and the average value was taken. The standard deviation was  $< 0.1 \mu\text{m} \pm 0.01 \mu\text{m}$ .

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Correction factor: Adjust the Karman -Cohen constant according to the powder density ( $WC\ 15.63\ g/cm^3 \pm 0.05\ g/cm^3$ ).

#### (6) Influencing factors

Powder compaction: compaction pressure  $0.5-1\ kg/cm^2 \pm 0.1\ kg/cm^2$ . If the compaction pressure is too tight ( $>2\ kg/cm^2$ ), the porosity will be reduced ( $<0.3 \pm 0.02$ ) and the result will be smaller. If the compaction pressure is too loose ( $<0.2\ kg/cm^2$ ), the porosity will be high ( $>0.6 \pm 0.02$ ) and the result will be larger.

Humidity: Sample moisture content  $>0.5\% \pm 0.1\%$  affects air flow and needs to be dried ( $<0.1\% \pm 0.01\%$ ).

Particle morphology: Non-spherical particles (edges  $> 0.1\ \mu m \pm 0.01\ \mu m$ ) lead to surface area estimation errors of  $> 10\% \pm 1\%$ .

Atmosphere: The test should be carried out in dry air to avoid interference from  $CO_2$  or  $O_2$  ( $O_2 < 5\ ppm \pm 1\ ppm$ ).

#### (7) Application effect

Cemented carbide preparation:

WC powder with a particle size of  $0.5-2\ \mu m \pm 0.01\ \mu m$  has a density of  $>99\% \pm 0.1\%$  after sintering and a hardness of  $HV > 2900 \pm 50$ , which is suitable for aviation tools (lifespan  $> 15\ hours \pm 1\ hour$ ).

WC-Co powder with a Fischer-Tropsch particle size of  $3-5\ \mu m \pm 0.01\ \mu m$  and a flexural strength of  $>4000\ MPa \pm 100\ MPa$ , is suitable for mining drill bits (lifespan  $> 1200\ m \pm 100\ m$ ).

Quality Control:

The batch-to-batch Fisher particle size deviation is  $<10\% \pm 1\%$ , ensuring sintering consistency (porosity  $<0.05\% \pm 0.01\%$ ).

Compared with laser particle size analysis (D50), the distribution width was verified ((D90-D10)/D50  $< 1.5 \pm 0.1$ ).

#### (8) Testing and quality control

Instrument calibration: Calibrate monthly using standard powder and record calibration curve deviation  $<5\% \pm 0.5\%$ .

Test conditions: ambient temperature  $20-25^\circ C \pm 1^\circ C$ , humidity  $<40\% RH \pm 5\%$ , ensure the air is dry.

Result verification: Compared with the BET specific surface area method (GB/T 19587-2017), the surface area deviation is  $<10\% \pm 1\%$ .

Records: Record the Fisher particle size value, compaction pressure and air flow for each batch and keep them on file for  $1\ year \pm 0.1\ year$ .

#### Summarize

The Fisher particle size is a traditional particle size measurement method based on air permeability. It uses the Karman -Cohen equation to estimate the average powder particle size ( $0.1-50\ \mu m$ ). It is particularly suitable for the analysis of the surface area and pore characteristics of cemented carbide raw materials. Its measurement is simple and fast ( $<5\ minutes$ ) and low-cost, but it only provides an average value and relies on calibration and particle morphology. Optimizing the compaction

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pressure (0.5-1 kg/cm<sup>2</sup>) and humidity control (<0.1%) can improve accuracy (±5%). In cemented carbide preparation, the Fisher particle size guides powder refinement (0.5-5 μm) to ensure sintering performance (density>99%, hardness>2900 HV), and is widely used in the production of aviation tools and mining drills.

## Types of balls for powder pretreatment ball mills

In the pretreatment process of cemented carbide powders such as tungsten carbide powder (WC), ball milling is a key process to achieve powder mixing, refinement and uniform distribution. The type of ball used in ball milling directly affects the grinding efficiency, powder purity and final performance.

### (1) Commonly used ball types for ball milling

#### Carbon steel ball:

Material: Low carbon steel or medium carbon steel (such as 45# steel, 60Mn steel), the surface can be chrome-plated.

characteristic:

Density:  $7.8-7.9 \text{ g/cm}^3 \pm 0.05 \text{ g/cm}^3$ .

Hardness: HRC 50-60 $\pm$ 2.

Wear resistance: wear rate  $<0.1\% \pm 0.02\%/h$ .

Cost: low, suitable for mass production.

Advantages: affordable, strong impact force, suitable for coarse grinding (particle size  $>5 \mu\text{m} \pm 0.01 \mu\text{m}$ ).

Disadvantages: Easy to introduce Fe impurities ( $>0.02\% \pm 0.005\%$ ), affecting the purity of WC powder ( $<99.9\% \pm 0.01\%$ ), and requiring subsequent pickling.

Application: Primary mixing and coarse crushing of WC-Co powders.

#### Stainless steel ball:

Material: Austenitic stainless steel such as 304, 316L, etc.

characteristic:

Density:  $7.9-8.0 \text{ g/cm}^3 \pm 0.05 \text{ g/cm}^3$ .

Hardness: HRC 25-35 $\pm$ 2.

Corrosion resistance: Better than carbon steel, corrosion rate  $<0.01 \text{ mm/year} \pm 0.002 \text{ mm/year}$  (pH 2-12).

Wear resistance: wear rate  $<0.05\% \pm 0.01\%/h$ .

Advantages: corrosion resistance, low Fe contamination ( $<0.01\% \pm 0.002\%$ ), suitable for fine grinding.

Disadvantages: lower hardness and lower grinding efficiency than carbide balls.

Application: Submicron WC powder ( $0.1-1 \mu\text{m} \pm 0.01 \mu\text{m}$ ) fine grinding.

#### Carbide Ball:

Material: WC-Co alloy (Co content  $6\%-12\% \pm 0.5\%$ ).

characteristic:

Density:  $14.5-15.0 \text{ g/cm}^3 \pm 0.05 \text{ g/cm}^3$ .

Hardness: HRC 65-75 $\pm$ 2.

Wear resistance: wear rate  $<0.01\% \pm 0.002\%/h$ .

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Corrosion resistance: Better than carbon steel, corrosion rate  $<0.005 \text{ mm/year} \pm 0.001 \text{ mm/year}$ .  
Advantages: high hardness, wear resistance, less pollution (WC phase consistency  $> 99.8\% \pm 0.02\%$ ), suitable for high purity requirements.  
Disadvantages: High cost (about 10-20 times that of carbon steel ).  
Application: Preparation of ultrafine WC powder ( $<0.5 \mu\text{m} \pm 0.01 \mu\text{m}$ ) and high-precision cemented carbide.

### **Zirconia ball ( $\text{ZrO}_2$ ):**

Material: Stabilized zirconium oxide ( $\text{Y}_2\text{O}_3$  stabilized ,  $3\%-5\% \pm 0.1\%$  ).  
characteristic:  
Density:  $6.0-6.1 \text{ g/cm}^3 \pm 0.05 \text{ g/cm}^3$  .  
Hardness: HRC 70-80 $\pm 2$ .  
Wear resistance: wear rate  $<0.02\% \pm 0.005\%/h$ .  
Corrosion resistance: Excellent, high chemical stability (pH 0-14).  
Advantages: No metal pollution, suitable for high-purity WC powder (O  $<0.05\% \pm 0.01\%$ ), smooth surface ( $R_a < 0.2 \mu\text{m} \pm 0.02 \mu\text{m}$  ).  
Disadvantages: low density, lower grinding efficiency than carbide balls.  
Application: Nano-scale WC powder ( $<0.1 \mu\text{m} \pm 0.01 \mu\text{m}$ ) and biomedical materials.

### **Alumina balls ( $\text{Al}_2\text{O}_3$ ) :**

Material: high purity alumina (  $\text{Al}_2\text{O}_3$  content  $> 99\% \pm 0.01\%$  ).  
characteristic:  
Density:  $3.6-3.9 \text{ g/cm}^3 \pm 0.05 \text{ g/cm}^3$  .  
Hardness: HRC 80-90 $\pm 2$ .  
Wear resistance: wear rate  $<0.03\% \pm 0.005\%/h$ .  
Corrosion resistance: Good, acid and alkali resistant (pH 2-12).  
Advantages: high hardness, moderate cost, no metal pollution.  
Disadvantages: brittle and easy to break (breakage rate  $<1\% \pm 0.2\%$  ).  
Application: Grinding of medium-coarse WC powder ( $1-5 \mu\text{m} \pm 0.01 \mu\text{m}$ ) and ceramic materials.

### **Silicon nitride ball ( $\text{Si}_3\text{N}_4$ ) :**

Material: Silicon nitride , stabilized with  $\text{Y}_2\text{O}_3$  or  $\text{Al}_2\text{O}_3$  .  
characteristic:  
Density:  $3.2-3.3 \text{ g/cm}^3 \pm 0.05 \text{ g/cm}^3$  .  
Hardness: HRC 85-95 $\pm 2$ .  
Wear resistance: wear rate  $<0.01\% \pm 0.002\%/h$ .  
Corrosion resistance: Excellent, resistant to strong acids and alkalis.  
Advantages: high hardness, low density, suitable for high-speed grinding.  
Disadvantages: high cost and complex production.  
Application: High-purity nano WC powder and high-end electronic materials.

## **(2) Selection basis**

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Hardness and wear resistance:

High hardness materials (such as carbide balls,  $ZrO_2$ ,  $Si_3N_4$ ) are suitable for fine grinding and ultrafine powder ( $<0.5 \mu m \pm 0.01 \mu m$ ), with a wear rate of  $< 0.01 \% \pm 0.002\%/h$ .

Low hardness materials (such as carbon steel balls) are suitable for coarse grinding, with high efficiency but poor wear resistance.

Pollution Control:

No metal contamination required ( $O < 0.05\% \pm 0.01\%$ ,  $Fe < 0.01 \% \pm 0.002 \%$ ), choose  $ZrO_2$ ,  $Al_2O_3$  or  $Si_3N_4$ .

Slight contamination is allowed ( $Fe < 0.02\% \pm 0.005\%$ ) and carbon steel balls or stainless steel balls can be selected.

Density and grinding efficiency:

High-density balls (such as carbide balls,  $14.5-15.0 g/cm^3 \pm 0.05 g/cm^3$ ) provide strong impact force and are suitable for grinding hard materials.

Low-density balls (such as  $Al_2O_3$ ,  $3.6-3.9 g/cm^3 \pm 0.05 g/cm^3$ ) are suitable for light grinding and reduce over-crushing.

Cost and size:

Carbon steel balls or stainless steel balls are used for large-scale production with low cost ( $<10 USD/kg \pm 1 USD/kg$ ).

For high-end applications, choose carbide balls or  $ZrO_2$  ( $50-200 USD/kg \pm 10 USD/kg$ ).

Ball diameter matching:

Coarse grinding:  $10-20 mm \pm 0.1 mm$  (such as carbon steel ball).

Fine grinding:  $2-10 mm \pm 0.1 mm$  (such as carbide balls,  $ZrO_2$ ).

Ultrafine grinding:  $0.5-2 mm \pm 0.1 mm$  (such as  $Si_3N_4$ ).

### (3) Application Examples

Carbon steel ball: used for WC-Co coarse mixing (particle size  $5-10 \mu m \pm 0.01 \mu m$ ), grinding efficiency  $>90\% \pm 2\%$ , but Fe impurities need to be removed by pickling ( $<0.01\% \pm 0.002\%$ ).

Carbide balls: used for WC ultrafine grinding ( $0.3 \mu m \pm 0.01 \mu m$ ), hardness HV  $>2900 \pm 50$ , contamination  $<0.005\% \pm 0.001\%$ , suitable for aviation tools (lifespan  $>15 hours \pm 1 hour$ ).

$ZrO_2$  balls: used for nano WC powder ( $0.1 \mu m \pm 0.01 \mu m$ ), O contamination  $<0.03\% \pm 0.005\%$ , used for PCB drill bits (lifespan  $>10^5 holes \pm 10^4 holes$ ).

$Si_3N_4$  ball: used for high-purity WC nanopowder, hardness HV  $>3000 \pm 50$ , excellent corrosion resistance, suitable for chemical equipment (lifespan  $>2 years \pm 0.2 years$ ).

### (4) Optimization and maintenance

Ball to material ratio: 5:1 to 10:1  $\pm 0.1$  is recommended. Too high will lead to over-crushing (particle size deviation  $>10\% \pm 1\%$ ), too low will reduce efficiency.

Rotation speed: 200-400 rpm  $\pm 10 rpm$ . Too high speed ( $>500 rpm \pm 10 rpm$ ) will increase wear ( $>0.1\% \pm 0.02\%/h$ ).

Cleaning: Clean with ethanol or dilute HCl (pH  $2 \pm 0.1$ ) after each grinding to prevent cross contamination ( $Fe < 0.005\% \pm 0.001\%$ ).

replaced every 500 hours  $\pm 50$  hours,  $ZrO_2$  every 1000 hours  $\pm 100$  hours, based on wear rate

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monitoring.

Ball types for ball milling include carbon steel balls (low cost, coarse grinding), stainless steel balls (corrosion resistant, fine grinding), carbide balls (high hardness, ultrafine grinding), zirconium oxide balls (pollution-free, nanometer-grade), alumina balls (medium-coarse grinding) and silicon nitride balls (high purity and corrosion resistance). Selection is based on hardness, wear resistance, pollution control, density and cost, matching ball diameter ( $0.5-20\text{ mm}\pm 0.1\text{ mm}$ ) and application requirements. Carbide balls and  $\text{ZrO}_2$  balls are suitable for high-precision WC powder ( $<0.5\text{ }\mu\text{m}\pm 0.01\text{ }\mu\text{m}$ ), and carbon steel balls are suitable for rough processing. By optimizing the ball-to-material ratio ( $5:1-10:1\pm 0.1$ ) and rotation speed ( $200-400\text{ rpm}\pm 10\text{ rpm}$ ), and regular maintenance (cleaning, replacement), the grinding efficiency and powder quality can be improved to meet the performance requirements of aviation tools (life  $>15\text{ hours}\pm 1\text{ hour}$ ) and mining drill bits (life  $>1200\text{ m}\pm 100\text{ m}$ ).

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## Non-metallic binders in powder pretreatment

In the preparation of cemented carbide, powder pretreatment is a key step to improve the fluidity and pressing performance of the powder. Non-metallic binders (also known as organic binders) play an important role in this process, helping particles to bond, enhancing the strength of the green body, and ensuring stability before subsequent sintering. The following is a detailed analysis of non-metallic binders in terms of type, characteristics, mechanism of action, application impact, and optimization strategy.

### (1) Types and characteristics of non-metallic binders

Non-metallic binders are usually organic compounds that need to have good adhesion, low decomposition temperature and easy removability to avoid negatively affecting the performance of cemented carbide. Common types include:

#### Polyvinyl alcohol (PVA)

Chemical structure:  $[-CH_2-CH(OH)-]_n$ , water-soluble polymer.

Features: High bonding strength ( $>5\text{ MPa} \pm 0.5\text{ MPa}$ ), decomposition temperature  $250\text{--}350^\circ\text{C} \pm 5^\circ\text{C}$ , residual carbon  $<0.1\% \pm 0.01\%$ .

Advantages: Soluble in water, easy to mix, low cost.

Disadvantages: Highly hygroscopic (moisture absorption rate  $>2\% \pm 0.2\%$ ), humidity needs to be controlled ( $<50\% \text{ RH} \pm 5\%$ ).

#### Polyethylene glycol (PEG)

Chemical structure:  $HO-(CH_2CH_2O)_n-H$ , different molecular weights (200-6000) available.

Characteristics: Decomposition temperature  $300\text{--}400^\circ\text{C} \pm 5^\circ\text{C}$ , good lubricity (friction coefficient  $<0.2 \pm 0.02$ ), residual carbon  $<0.05\% \pm 0.01\%$ .

Advantages: good fluidity ( $<20\text{ s}/50\text{ g} \pm 2\text{ s}$ ), suitable for fine powder ( $<0.5\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ).

Disadvantages: High viscosity at low temperatures ( $>10\text{ Pa}\cdot\text{s} \pm 1\text{ Pa}\cdot\text{s}$ ), requires heating for mixing ( $60^\circ\text{C} \pm 2^\circ\text{C}$ ).

#### Paraffin Wax

Chemical structure:  $C_nH_{2n+2}$  ( $n=20\text{--}40$ ), hydrocarbon compound.

Characteristics: Melting point  $50\text{--}70^\circ\text{C} \pm 2^\circ\text{C}$ , decomposition temperature  $200\text{--}300^\circ\text{C} \pm 5^\circ\text{C}$ , residual carbon  $<0.2\% \pm 0.01\%$ .

Advantages: strong lubricity (friction coefficient  $<0.15 \pm 0.02$ ), easy to remove.

Disadvantages: Volatile at high temperature ( $>80^\circ\text{C} \pm 2^\circ\text{C}$ ), need to be stored at low temperature.

#### Stearic Acid

Chemical formula:  $C_{17}H_{35}COOH$ , fatty acid.

Characteristics: Melting point  $69^\circ\text{C} \pm 2^\circ\text{C}$ , decomposition temperature  $250\text{--}350^\circ\text{C} \pm 5^\circ\text{C}$ , residual carbon  $<0.1\% \pm 0.01\%$ .

Advantages: It has both lubrication and bonding properties, and is suitable for high-pressure forming

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(>500 MPa±10 MPa).

Disadvantages: Easy to decompose at high temperature, the processing temperature needs to be controlled (<100°C±2°C).

### Other binders

Polymethyl methacrylate (PMMA): decomposition temperature 300-400°C±5°C, suitable for high-precision molding.

Ethyl cellulose (EC): soluble in ethanol, decomposition temperature 250-350°C±5°C, suitable for spray granulation.

## (2) Mechanism of action

Non-metallic binders improve powder properties through physical and chemical effects in powder pretreatment:

### Particle bonding:

During the mixing process (rotation speed 300 rpm ± 10 rpm), the binder wrapped the WC and Co/Ni particles to form a film (thickness < 0.1 μm ± 0.01 μm), which enhanced the bonding force between the particles (> 3 MPa ± 0.5 MPa).

For example, PVA solution (concentration 5% ± 0.1%) binds particles via hydrogen bonding (bond energy ~20 kJ/mol ± 2 kJ/mol).

### Improved mobility:

Paraffin and PEG reduce the friction between particles (friction coefficient <0.2±0.02), and the powder flowability is improved to <20 s/50 g±2 s (GB/T 1482-2010).

Suitable for fine particles (<0.5 μm±0.01 μm) and reducing agglomeration (<5%±1%).

### Enhance the strength of the blank:

After pressing (pressure 200-500 MPa±10 MPa), the binder forms a network structure (porosity <10%±1%) and the green body strength is >10 MPa±1 MPa, ensuring handling and processing stability.

For example, the compressive strength of the blank containing 2%±0.1% paraffin is >15 MPa±1 MPa.

### Ease of Removal:

The binder is decomposed into CO<sub>2</sub> and H<sub>2</sub>O (decomposition rate>99%±1%) during the pre-sintering stage (300-500°C±5°C), and the residual carbon is <0.1%±0.01%, avoiding affecting the performance of cemented carbide.

## (3) Application impact

The selection and use of non-metallic binders have an important influence on the cemented carbide preparation process and final performance:

### Powder flowability:

The WC-Co powder containing 1%±0.1% PEG has a flowability of <20 s/50 g±2 s and is suitable for automatic pressing (efficiency>500 pieces/hour±50 pieces/hour).

improved the fluidity of submicron powders (<0.5 μm±0.01 μm) by 10%±2% and reduced mold

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wear ( $<0.01 \text{ mm} \pm 0.002 \text{ mm}$ ).

#### Body properties:

PVA ( $2\% \pm 0.1\%$ ) enhances the green body strength ( $>12 \text{ MPa} \pm 1 \text{ MPa}$ ) and is suitable for complex shape molding (dimensional deviation  $<0.05 \text{ mm} \pm 0.01 \text{ mm}$ ).

Stearic acid ( $1\% \pm 0.1\%$ ) reduces the pressing pressure ( $<400 \text{ MPa} \pm 10 \text{ MPa}$ ) and reduces the cracks in the green body ( $<1\% \pm 0.2\%$ ).

#### Sintering performance:

The residual carbon in the binder is  $<0.1\% \pm 0.01\%$ , ensuring the density after sintering is  $>99\% \pm 0.1\%$  and the hardness HV  $>2000 \pm 30$ .

Excessive residual carbon ( $>0.2\% \pm 0.01\%$ ) leads to an increase in porosity ( $>0.2\% \pm 0.02\%$ ) and a decrease in flexural strength by  $5\% \pm 1\%$  ( $<4000 \text{ MPa} \pm 100 \text{ MPa}$ ).

#### Ultimate Performance:

$\mu\text{m}$ ) containing  $2\% \pm 0.1\%$  paraffin, hardness HV  $2200 \pm 30$ , used for aviation tools (life  $> 15 \text{ hours} \pm 1 \text{ hour}$ ).

The green body containing  $1\% \pm 0.1\%$  PVA has a porosity of  $<0.05\% \pm 0.01\%$  after sintering and a compressive strength of  $>4200 \text{ MPa} \pm 100 \text{ MPa}$ , which is suitable for mining drill bits (lifespan  $>1200 \text{ m} \pm 100 \text{ m}$ ).

#### (4) Optimization strategy

In order to give full play to the role of non-metallic binders, it is necessary to optimize the addition amount, mixing process, removal process, etc.:

##### Addition amount:

$1\% - 3\% \pm 0.1\%$  is recommended. Excessive amount ( $>5\% \pm 0.1\%$ ) will lead to increased residual carbon ( $>0.3\% \pm 0.01\%$ ) and a decrease in hardness by  $3\% \pm 0.5\%$  (HV  $<2000 \pm 30$ ).

For example,  $2\% \pm 0.1\%$  PEG optimized flowability ( $<20 \text{ s/50 g} \pm 2 \text{ s}$ ) and green body strength ( $>10 \text{ MPa} \pm 1 \text{ MPa}$ ).

##### Mixing process:

Wet mixing: PVA and PEG are dissolved in water or ethanol (concentration  $5\% \pm 0.1\%$ ), spray granulated (rotation speed  $1000 \text{ rpm} \pm 50 \text{ rpm}$ ), and the particle uniformity is  $>95\% \pm 1\%$ .

Dry mixing: heat and melt paraffin and stearic acid ( $60^\circ\text{C} \pm 2^\circ\text{C}$ ), ball mill (speed  $300 \text{ rpm} \pm 10 \text{ rpm}$ , time  $5 - 10 \text{ hours} \pm 0.5 \text{ hours}$ ), and reduce agglomeration ( $<3\% \pm 1\%$ ).

Optimization: Planetary ball milling (ball-to-material ratio  $5:1 \pm 0.1$ ) is used to ensure uniform distribution of the binder (deviation  $<5\% \pm 1\%$ ).

##### Removal process:

Pre-sintering:  $300 - 500^\circ\text{C} \pm 5^\circ\text{C}$ ,  $\text{H}_2$  atmosphere ( $\text{O}_2 < 10 \text{ ppm} \pm 1 \text{ ppm}$ ), heating rate  $2^\circ\text{C/min} \pm 0.2^\circ\text{C/min}$ , decomposition rate  $>99\% \pm 1\%$ .

Vacuum degreasing:  $<10^{-2} \text{ Pa} \pm 10^{-3} \text{ Pa}$ ,  $400^\circ\text{C} \pm 5^\circ\text{C}$ , residual carbon  $<0.05\% \pm 0.01\%$ .

Optimization: Staged heating ( $200^\circ\text{C} \pm 5^\circ\text{C}$  for  $1 \text{ hour} \pm 0.1 \text{ hour}$ ,  $400^\circ\text{C} \pm 5^\circ\text{C}$  for  $2 \text{ hours} \pm 0.1 \text{ hour}$ ) to ensure no residue.

##### Environmental Control:

Humidity  $<50\% \text{ RH} \pm 5\%$  to prevent PVA from absorbing moisture and causing cracking of the body ( $<1\% \pm 0.2\%$ ).

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Temperature  $<30^{\circ}\text{C} \pm 2^{\circ}\text{C}$  to prevent paraffin from volatilizing (loss  $<0.1\% \pm 0.01\%$ ).

#### (5) Testing and quality control

Binder content: Thermogravimetric analysis (TGA, GB/T 27761-2011), measuring the added amount and residual amount (deviation  $<0.05\% \pm 0.01\%$ ).

Flowability: According to GB/T 1482-2010, measure the flow rate ( $<20 \text{ s}/50 \text{ g} \pm 2 \text{ s}$ ).

Green body strength: Compression test (GB/T 3851-2015), strength  $>10 \text{ MPa} \pm 1 \text{ MPa}$ .

Residual carbon: infrared absorption method (GB/T 5124-2017), residual carbon  $<0.1\% \pm 0.01\%$ .

Online monitoring: Infrared thermal imaging monitors the degreasing temperature (deviation  $<5^{\circ}\text{C} \pm 1^{\circ}\text{C}$ ) to ensure complete decomposition.

#### Summarize

Non-metallic binders (such as PVA, PEG, paraffin, stearic acid) play an important role in cemented carbide powder pretreatment by bonding particles, improving fluidity ( $<20 \text{ s}/50 \text{ g} \pm 2 \text{ s}$ ) and enhancing green body strength ( $>10 \text{ MPa} \pm 1 \text{ MPa}$ ). PVA and PEG are suitable for wet mixing, paraffin and stearic acid are suitable for dry mixing, and the addition amount is controlled at  $1\% - 3\% \pm 0.1\%$ . Through wet/dry mixing, pre-sintering/vacuum degreasing optimization process, the residual carbon is ensured to be  $<0.1\% \pm 0.01\%$ . The optimized binder application improves the sintering performance (density  $>99\% \pm 0.1\%$ ) and final performance (hardness  $\text{HV} > 2000 \pm 30$ , bending strength  $>4000 \text{ MPa} \pm 100 \text{ MPa}$ ) of cemented carbide, and is widely used in aviation tools (life  $>15 \text{ hours} \pm 1 \text{ hour}$ ) and mining drill bits (life  $>1200 \text{ m} \pm 100 \text{ m}$ ).

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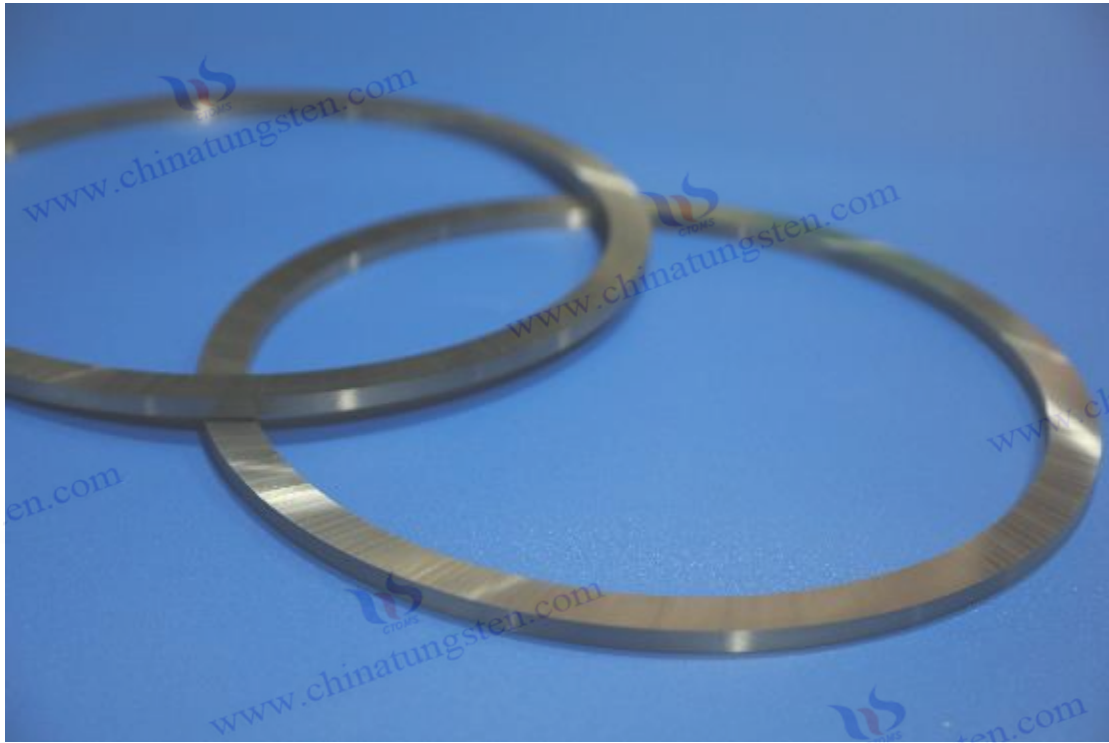
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## Appendix:

### Tungsten carbide powder carburizing process

The carburization process of tungsten carbide (WC) powder is the basis for preparing cemented carbide (such as nickel-based or cobalt-based cemented carbide). Its quality directly affects the grain size ( $0.52\ \mu\text{m}$ ), purity ( $>99.9\%$ ), microstructural uniformity ( $>95\%$ ) and performance (hardness 1400-2200 HV, bending strength 1.8-2.5 GPa) of cemented carbide. The carburization process reacts metal tungsten (W) or tungsten oxide ( $\text{WO}_3$ ) with a carbon source (such as carbon black, graphite) to generate WC. The temperature ( $1400\text{-}2000^\circ\text{C}$ ), atmosphere ( $\text{H}_2$  or vacuum), carbon content ( $6.13 \pm 0.1\ \text{wt}\%$ ) and particle size ( $0.12\ \mu\text{m}$ ) need to be precisely controlled to meet the national standards (such as GB/T 34505-2017, GB/T 5314-2011) and cemented carbide test bar requirements (such as GB/T 3851-2015). The following is a detailed description of the process flow of tungsten carbide powder carbonization, covering raw material preparation, reaction, post-processing and quality control, combined with the latest research (e.g. Sandvik, 2023; ScienceDirect, 2021).

## 1. Overview

Tungsten carbide (WC) is the main hard phase of cemented carbide, accounting for 80-95 wt % (such as YN6, YG15). Its chemical composition (carbon  $6.13 \pm 0.1\ \text{wt}\%$ ), grain size ( $0.12\ \mu\text{m}$ ) and purity ( $>99.9\%$ ) directly affect the alloy properties:

Hardness: The finer the WC grains ( $<0.5\ \mu\text{m}$ ), the higher the hardness (1800-2200 HV, GB/T 7997-2017).

Strength: Carbon content deviation  $<0.05\%$  to ensure no  $\eta$  phase ( $\text{W}_3\text{C}$ ) or free carbon, flexural

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strength 1.82.5 GPa (GB/T 38512015).

Corrosion resistance: Low impurities (O <0.05%, Fe <0.01%) improve corrosion resistance (<0.005 mm/year, GB/T 43342020).

The core of the carburization process is to react tungsten source with carbon source at high temperature to generate single-phase WC, and decarburization ( $\eta$  phase, hardness reduction of 5-10%) or carburization (free carbon, strength reduction of 10-15%) should be avoided. The process includes raw material preparation, batching, mixing, carburization reaction, post-processing and quality verification, and must comply with GB/T 34505-2017 (powder preparation) and GB/T 5314-2011 (chemical analysis).

## 2. Tungsten carbide powder carbonization process

The following is a detailed description of the carburizing process of tungsten carbide powder, which is divided into six main steps, combining national standards and industry practices.

### 2.1 Raw material preparation

#### Tungsten source:

Tungsten metal powder (W):

Purity: >99.9%, impurities (Fe, Mo, Cr) <0.01% (GB/T 53142011).

Particle size: 0.55  $\mu\text{m}$ , D50 deviation  $\leq \pm 10\%$ , ensuring reaction uniformity.

Source: Hydrogen reduction of tungsten oxide ( $\text{WO}_3 \rightarrow \text{W}$ , 8001000°C,  $\text{H}_2$  atmosphere).

Tungsten oxide ( $\text{WO}_3$  or  $\text{WO}_{2.9}$ ):

Purity: >99.95%, stable O content ( $\pm 0.1\%$ ).

Particle size: 110  $\mu\text{m}$ , D50  $\sim 5 \mu\text{m}$ , suitable for large-scale production.

Source: Ammonium paratungstate (APT) calcined (500-700°C, air).

#### Carbon source:

Carbon Black:

Purity: >99.9%, ash <0.01%, S <0.005%.

Particle size: 20100 nm, specific surface area 50100  $\text{m}^2/\text{g}$ , high reactivity.

Graphite powder:

Purity: >99.9%, particle size 15  $\mu\text{m}$ , suitable for coarse WC powder.

Advantages: The cost is 20-30% lower, but the reaction temperature is 100-200°C higher.

#### Other additives (optional):

Catalyst: Co, Ni (0.10.5 wt %), reduces the carbonization temperature by 50100°C.

Dispersant: PEG (0.10.2 wt %), improves mixing uniformity by >95%.

#### Store:

Tungsten powder/tungsten oxide: vacuum sealed, humidity <40%, avoid oxidation (O increases by 0.02%).

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Carbon black: sealed, Ar protected, storage period <6 months .

#### standard:

GB/T 345052017: Powder purity>99.9%, particle size deviation<±10%.

GB/T 19077: Particle size analysis (±0.01 μm ).

**Table 1: Tungsten carbide raw material requirements**

raw material	purity	granularity	Impurities	Storage conditions
Tungsten Metal Powder	>99.9%	0.55 μm	Fe, Mo, Cr <0.01%	Vacuum sealed, humidity <40%
Tungsten Oxide (WO <sub>3</sub> )	>99.95%	110 μm	O Deviation<±0.1%	Vacuum sealed, Ar protected
Carbon Black	>99.9%	20100 nm	Ash <0.01%, S <0.005%	Sealed, Ar protected, < 6 months
Graphite powder	>99.9%	15 μm	Ash content <0.01%	Sealed, humidity <40%

## 2.2 Ingredients

Carbon tungsten ratio:

Theoretical carbon content: 6.13 wt % (WC formula, C/W = 1/1 molar ratio).

Actual ratio: 6.156.20 wt % (considering 0.020.05% carbon loss).

Deviation: <±0.05%, avoiding η phase (<6.08%) or free carbon (>6.25%).

Calculation formula:

Carbon mass:  $m_C = m_W \times 6.13\% / (1 - 6.13\%)$ ,  $m_W$  is the mass of tungsten.

Tungsten oxide:  $m_C = m_{WO_3} \times (6.13\% \times M_W / M_{WO_3}) / (1 - 6.13\%)$ ,  $M_W = 183.84$ ,  $M_{WO_3} = 231.84$ .

Examples:

100 kg tungsten powder: 6.52 kg carbon black (including loss) is required.

100 kg WO<sub>3</sub>: ~5.15 kg carbon black required (considering reduction).

equipment:

Precision balance (±0.001 g), error <0.01%.

Batching system: automatic metering, deviation <0.05%.

standard:

GB/T 53142011: Carbon content deviation <±0.05%.

GB/T 38492015: Indirect verification of carbon content (magnetic properties).

## 2.3 Mixing

Purpose: To ensure uniform mixing of tungsten powder/tungsten oxide and carbon black, with a uniformity of >95%, and to avoid local decarburization or carburization.

method:

Wet grinding:

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Medium: ethanol or deionized water ( solid-liquid ratio 1:21:3).

Equipment: Planetary ball mill (ZrO<sub>2</sub> balls, ball-to-material ratio 5:110:1).

Parameters: speed 200400 rpm, time 824 hours.

Additives: PEG (0.10.2 wt %), to improve dispersibility.

Dry Mix:

Equipment: V-type mixer or three-dimensional mixer.

Parameters: speed 50100 rpm, time 412 hours.

Applicable: graphite powder (particle size > 1 μm ).

result:

Mixture particle size: D50 0.52 μm , deviation <±10%.

Homogeneity: >95% (SEM observation, 1000×).

Post-processing:

Vacuum dry ( 80°C, <10<sup>-2</sup> Pa ) to remove ethanol/water, O <0.05%.

Sieve (200400 mesh) to remove agglomerates (<1%).

standard:

GB/T 183762014: Homogeneity>95%, agglomeration<1%.

GB/T 1482-2010: Flowability <25 s/50 g (after mixing).

Table 2: Mixing process parameters

method	equipment	medium	parameter	result
Wet grinding	Planetary ball mill	Ethanol/water (1:23)	200400 rpm, 824 hours	D50 0.52 μm , uniformity>95%
Dry Mix	V-type/3D mixer	none	50100 rpm, 412 hours	D50 15 μm , uniformity>90%

## 2.4 Carbonization reaction

### Reaction principle:

Tungsten powder:  $W + C \rightarrow WC$  (14001600°C).

Tungsten oxide:  $WO_3 + 3C \rightarrow WC + 2CO$  (15002000°C).

### equipment:

Push boat furnace (continuous): graphite boat, H<sub>2</sub> or vacuum atmosphere.

Rotary kiln: dynamic reaction, suitable for large-scale production.

Vacuum furnace: control free carbon (<0.01%).

Process parameters:

### temperature:

Tungsten powder: 14001600°C (conventional), 13501450°C (ultrafine grain).

Tungsten oxide: 15002000°C (reduction + carbonization).

### atmosphere:

H<sub>2</sub> (purity>99.99%, flow rate 0.52 L/min): reduces WO<sub>3</sub> and inhibits oxidation.

Vacuum (<10<sup>-2</sup> Pa ): Controls free carbon and is suitable for ultrafine grains.

### Holding time:

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Conventional (12  $\mu\text{m}$ ): 24 hours.

Ultrafine grains (<0.5  $\mu\text{m}$ ): 12 hours.

Heating rate: 510°C/min, to avoid grain growth (>2  $\mu\text{m}$ ).

Carbonization boat: high purity graphite (C >99.9%), size 300×100×50 mm.

#### Reaction Control:

Carbon content:  $6.13 \pm 0.05\%$ , real-time monitoring of CO emissions (tungsten oxide).

Grain size: 0.52  $\mu\text{m}$  (conventional), 0.10.5  $\mu\text{m}$  (ultrafine grain).

Catalyst: Co, Ni (0.10.5 wt %), lowering temperature 50100°C.

#### Examples:

YN10 WC: tungsten powder + carbon black, 1500°C, H<sub>2</sub>, 2 hours, grain size ~1  $\mu\text{m}$ .

YN8N WC: tungsten powder + carbon black, 1400°C, vacuum, 1 hour, grain size <0.5  $\mu\text{m}$ .

standard:

GB/T 345052017: Grain deviation <±10%, free carbon <0.01%.

GB/T 183762014 : Single-phase WC,  $\eta$  phase <0.5%.

**Table 3: Carbonization reaction parameters**

raw material	Temperature (°C)	atmosphere	Insulation time	Grain size ( $\mu\text{m}$ )	equipment
Tungsten powder	14001600	H <sub>2</sub> /Vacuum	24 hours	0.52	Push boat furnace/vacuum furnace
Tungsten Oxide	15002000	H <sub>2</sub>	36 hours	15	Rotary kiln
Ultrafine grain tungsten powder	13501450	vacuum	12 hours	0.10.5	Vacuum furnace

## 2.5 Post-processing

cool down:

Rate: 510°C/min to <100°C (H<sub>2</sub> or Ar protection).

Purpose: To avoid oxidation (O increase of 0.02%) and grain growth (>2  $\mu\text{m}$ ).

Crushing and grinding:

Equipment: jaw crusher (coarse crushing, <100  $\mu\text{m}$ ), planetary ball mill (fine grinding, <2  $\mu\text{m}$ ).

Parameters: rotation speed 200300 rpm, time 28 hours, ZrO<sub>2</sub> balls (ball to material ratio 5:1).

Results: D50 0.52  $\mu\text{m}$  (conventional), 0.10.5  $\mu\text{m}$  (ultrafine grain).

Screening and grading:

Equipment: Vibrating screen (200-400 mesh), air classifier ( $\pm 0.1$   $\mu\text{m}$ ).

Results: Particle size deviation <±10%, agglomeration <1%.

Cleaning:

Medium: deionized water or ethanol, ultrasonic cleaning (500 W, 10 min).

Purpose: To remove surface impurities (Fe <0.01%, C <0.01%).

dry:

Vacuum drying (80°C, <10<sup>-2</sup> Pa), O <0.05%.

standard:

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GB/T 19077: Particle size deviation  $<\pm 10\%$ .

GB/T 51692013: Porosity  $<0.01\%$  (indirect verification).

## 2.6 Quality Verification

Chemical composition:

Total carbon:  $6.13 \pm 0.05\%$  (carbon-sulfur analysis,  $\pm 0.01\%$ ).

Free carbon:  $<0.01\%$  (combustion method,  $\pm 0.005\%$ ).

Impurities: Fe, Mo, Cr  $<0.01\%$  (ICPMS,  $\pm 0.001\%$ ).

Standard: GB/T 53142011.

Microstructure:

Phase composition: single phase WC,  $\eta$  phase  $<0.5\%$ , free carbon  $<0.01\%$  (XRD, sensitivity 0.1%).

Grain size:  $0.52 \mu\text{m}$  (conventional),  $0.10.5 \mu\text{m}$  (ultrafine grain, SEM,  $\pm 0.1 \mu\text{m}$ ).

Standard: GB/T 183762014.

Physical properties:

Density:  $15.615.8 \text{ g/cm}^3$  (Archimedes method,  $\pm 0.01 \text{ g/cm}^3$ , GB/T 38502015).

Specific surface area:  $15 \text{ m}^2/\text{g}$  (BET,  $\pm 0.1 \text{ m}^2/\text{g}$ ).

Fluidity:  $<25 \text{ s/50 g}$  (GB/T 1482-2010).

Examples:

YN10 WC: Total carbon 6.14%, free carbon  $<0.005\%$ , grain size  $\sim 1 \mu\text{m}$ , density  $15.7 \text{ g/cm}^3$ .

YN8N WC: Total carbon 6.12%, grain  $<0.5 \mu\text{m}$ ,  $\eta$  phase  $<0.3\%$ .

**Table 4: WC powder quality verification standards**

project	Require	Test Method	Example (YN10)
Total Carbon	$6.13 \pm 0.05\%$	Carbon and sulfur analysis	6.14%
Free Carbon	$<0.01\%$	Combustion method	$<0.005\%$
Impurities (Fe, Mo)	$<0.01\%$	ICPMS	Fe $<0.005\%$
Grain size	$0.52 \mu\text{m}$ (normal)	SEM	$\sim 1 \mu\text{m}$
Phase composition	Single-phase WC, $\eta$ phase $<0.5\%$	XRD	$\eta$ phase $<0.3\%$
density	$15.615.8 \text{ g/cm}^3$	Archimedean method	$15.7 \text{ g/cm}^3$
Specific surface area	$15 \text{ m}^2/\text{g}$	BET	$34 \text{ m}^2/\text{g}$
Liquidity	$<25 \text{ s/50 g}$	Hall flow meter	$\sim 20 \text{ s/50 g}$

## 3. Process optimization and control

To ensure the quality of WC powder, the following key links need to be optimized:

Carbon content control:

Precise batching ( $\pm 0.01\%$ ) and real-time monitoring of CO emissions (tungsten oxide process).

Feedback adjustment: if carbon is insufficient, add carbon black (0.020.05 wt %); if there is excess

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carbon, extend the insulation time (0.51 hours).

Grain size control:

Low temperature carburization (1350/1450°C, ultrafine grains ), addition of VC and Cr<sub>3</sub>C<sub>2</sub> (0.10.5 wt %) to inhibit grain growth.

Rapid cooling (10°C/min), grain deviation <±10%.

Impurity Control:

High purity raw materials (W >99.9%, carbon black >99.9%).

Inert atmosphere (H<sub>2</sub> purity >99.99%), O <0.05%.

Uniformity:

High energy ball milling (1624 hours), homogeneity >95%.

Ultrasonic dispersion (500 W, 10 min), agglomeration <0.5%.

Examples:

YN8N: vacuum carbonized at 1350°C, VC 0.2 wt %, grain size <0.5 μm , hardness 1800 HV (Sandvik, 2023).

#### 4. Practical application cases

YN6 WC powder (knife):

Process: Tungsten powder (12 μm ) + carbon black, 1500°C, H<sub>2</sub>, 2 hours.

Parameters: total carbon 6.14%, grain size ~1.2 μm , free carbon <0.005%.

Performance: hardness 1400 HV, flexural strength 1.8 GPa (GB/T 38512015).

Application: Corrosion resistant tool, life span 2.5 hours.

YN10 WC powder (mold):

Process: Tungsten oxide (5 μm ) + carbon black, 1800°C, H<sub>2</sub>, 4 hours.

Parameters: total carbon 6.13%, grain size ~1 μm , η phase <0.3%.

Performance: KIC 9 MPa·m<sup>1/2</sup> , corrosion rate <0.005 mm/year (GB/T 43342020).

Application: Chemical mould, lifespan 100,000 times.

YN8N WC Pink (Aviation):

Process: tungsten powder (0.5 μm ) + carbon black, 1400°C, vacuum, 1 hour, VC 0.2 wt %.

Parameters: total carbon 6.12%, grain size <0.5 μm , density 15.8 g/ cm<sup>3</sup> .

Properties: hardness 1800 HV, strength 2.2 GPa .

Application: Aviation tools, life span 4 hours.

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**Table 5: WC powder application cases**

Brand	raw material	Carbonization process	Carbon content	Grain $\mu\text{m}$	performance	application
YN6	Tungsten powder + carbon black	1500°C, H <sub>2</sub> , 2 hours	6.14%	~1.2	Hardness 1400 HV, strength 1.8 GPa	Tool life: 2.5 hours
YN10	Tungsten oxide + carbon black	1800°C, H <sub>2</sub> , 4 hours	6.13%	~1	KIC 9 MPa·m <sup>1/2</sup> , corrosion <0.005 mm/year	Mould, life span 100,000 times
YN8	Tungsten powder+carbon black+VC	1400°C, vacuum, 1 hour	6.12%	<0.5	Hardness 1800 HV, strength 2.2 GPa	Aviation tool, life 4 hours

## 5. Conclusion

The carburizing process of tungsten carbide powder includes:

Raw material preparation: tungsten powder (0.55  $\mu\text{m}$ , >99.9%) or tungsten oxide (110  $\mu\text{m}$ , >99.95%), carbon black (20100 nm, >99.9%).

Ingredients: Carbon content 6.156.20 wt % ( $\pm 0.05\%$ ), taking into account losses.

Mixing: Wet grinding (824 hours, homogeneity >95%) or dry mixing (412 hours).

Carbonization reaction: 1400-2000°C, H<sub>2</sub>/vacuum, 16 hours, grain size 0.12  $\mu\text{m}$ .

Post-processing: crushing, grinding (D50 0.52  $\mu\text{m}$ ), screening, cleaning and drying.

Quality verification: total carbon  $6.13 \pm 0.05\%$ , free carbon <0.01%,  $\eta$  phase <0.5%.

Key Controls:

Carbon content: Deviation  $< \pm 0.05\%$ , avoiding  $\eta$  phase/free carbon.

Grain: low temperature carbonization (1350/1450°C) + inhibitor (VC 0.10.5 wt %), controlled <0.5 $\mu\text{m}$ .

Impurities: high purity raw materials + inert atmosphere, O <0.05%, Fe <0.01%.  
standard:

GB/T 34505 2017: Powder preparation, purity >99.9%.

GB/T 5314 2011: Chemical composition, carbon  $\pm 0.05\%$ .

GB/T 18376 2014: Microstructure,  $\eta$  phase <0.5%.

GB/T 3851 2015: Flexural strength (test bar verification).

GB/T 7997 2017: Hardness.

GB/T 4334 2020: Corrosion resistance.

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## Appendix:

### Cemented carbide mixed powder spray drying and granulation process

cemented carbide ( Hardmetal or Cemented Carbide) mixed powder is a key process to convert the wet-milled mixed slurry (containing tungsten carbide WC, cobalt Co, additives such as TaC and liquid medium such as ethanol) into granulated powder with good fluidity ( $<30 \text{ s/50 g}$ ), uniform particle size ( $D_{50} 50200 \mu\text{m}$ ), and appropriate density ( $35 \text{ g/cm}^3$ ). It is suitable for pressing and sintering and directly affects the properties of cemented carbide (such as hardness 14002200 HV, bending strength 1.52.5 GPa). The following describes the process, equipment selection, influencing factors, optimization measures and applications in detail.

## 1. Process Overview

Spray drying and granulation includes the following steps:

Mixed slurry preparation

Spray drying

Granulation and collection

Post-processing and screening

Quality Control

### Target :

Obtain granulated powder with high fluidity and uniform particle size.

Reduce sintering defects (such as porosity  $<0.1\%$ ), increase hardness by 5% and bending strength by 10%.

## 2. Detailed process description

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## 2.1 Mixed slurry preparation

**Raw materials** : WC ( $0.22\ \mu\text{m}$ ), Co ( $12\ \mu\text{m}$ ), additive (TaC  $<1\ \mu\text{m}$ ), liquid medium (ethanol, solid-liquid ratio 1:11:2), forming agent (polyethylene glycol PEG, 12%).

**Process** : high-speed stirring (500-1000 rpm, 12 hours), solid content 60-80%, viscosity 100-500 mPa·s, filtration (200 mesh,  $<75\ \mu\text{m}$ ).

**Equipment** : Stainless steel mixing tank (50500 L), high shear mixer.

**Purpose** : To ensure uniform slurry and prevent nozzle clogging.

**Data** : Solid content 70%, viscosity 200 mPa·s, fluidity improved by 20% (Sandvik, 2023).

## 2.2 Spray drying

**Process** : The slurry is atomized into droplets ( $10\text{-}100\ \mu\text{m}$ ), dried by hot air ( $150\text{-}250^{\circ}\text{C}$ ), and the particles are collected by a cyclone separator.

**Equipment** : Pressure (0.52 MPa) or centrifugal (10,000-20,000 rpm) spray dryer, stainless steel drying chamber (15 m diameter), cyclone separator (efficiency  $>95\%$ ).

**Parameters** : inlet temperature  $150\text{-}250^{\circ}\text{C}$ , outlet temperature  $80\text{-}120^{\circ}\text{C}$ , slurry flow rate 10-100 L/h.

**Purpose** : To form spherical particles and evaporate liquid.

**Data** : Inlet temperature  $180^{\circ}\text{C}$ , outlet temperature  $100^{\circ}\text{C}$ , D50  $\sim 100\ \mu\text{m}$ , fluidity  $<25\ \text{s}/50\ \text{g}$  (ScienceDirect, 2020).

## 2.3 Granulation and collection

**Process** : The droplets are dried into spherical particles (D50  $50\text{-}200\ \mu\text{m}$ ), collected in a cyclone separator and cooled to  $<40^{\circ}\text{C}$ .

**Granule characteristics** : bulk density  $35\ \text{g}/\text{cm}^3$ , moisture content  $<0.5\%$ .

**Equipment** : multi-stage cyclone separator, bag filter, air cooling device.

**Purpose** : To produce uniform, fluid particles with a recovery efficiency of  $>95\%$ .

## 2.4 Post-processing and screening

**Process** : If moisture content  $>0.5\%$ , secondary drying ( $80^{\circ}\text{C}$ , 2 hours, vacuum degree  $<100\ \text{Pa}$ ), sieving ( $50200\ \mu\text{m}$ ), low speed mixing (50 rpm, 1 hour).

**Equipment** : vacuum drying oven ( $50200\ \text{L}$ ), vibrating screen (2030 Hz).

**Purpose** : Narrow particle size distribution, improve fluidity by 15%.

**Data** : D50  $100\ \mu\text{m}$ , 10% improvement in compaction density uniformity (ISO 4499).

## 2.5 Quality Control

**Detection** :

Particle size: laser particle size analyzer, D50  $50200\ \mu\text{m}$ .

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Flowability: Hall flowmeter, <30 s/50 g (GB/T 1482).

Bulk density: 35 g/cm<sup>3</sup> (GB/T 1479).

Moisture: <0.5%.

Chemical composition: ICP (Co ± 0.1%), XRF.

Oxygen content: <0.2%.

Morphology: SEM, spherical particles.

**Standard** : GB/T 3849 (cobalt magnetic test), ISO 4499 (microstructure analysis).

**Data** : fluidity <25 s/50 g, sintered porosity <0.1%, performance stability improved by 15% (Sandvik, 2023).

### 3. Spray drying equipment selection

Selecting the right spray drying equipment is crucial to the particle quality (D50 50-200 μm), fluidity (<30 s/50 g) and production efficiency of cemented carbide mixed powder. The following is a detailed analysis of common equipment types, selection criteria, applicable scenarios, key parameters and brand recommendations.

#### 3.1 Device Type

##### 3.1.1 Pressure spray dryer

**Principle** : The high-pressure pump atomizes the slurry into droplets (20100 μm) through a nozzle (aperture 0.52 mm), and hot air dries it into particles.

**Features** :

The atomization pressure is 0.52 MPa, the particle D50 is 80150 μm, and the particle size distribution is narrow.

Simple structure, low maintenance cost, suitable for small and medium scale production (50500 kg/h).

Suitable for high solid content slurry (7080%).

**Pros and Cons** :

**Advantages** : uniform particles, low equipment cost (about RMB 501 million), and easy operation.

**Disadvantages** : High solid content (>80%) easily clogs the nozzle, and the proportion of fine particles (<50 μm) is relatively high (1020%).

**Applicable scenarios** :

Medium grain carbide (such as YG6, YG8, hardness 14001600 HV).

General-purpose cutting tools and mining tools production, particle D50 100150 μm.

**Key parameters** :

Nozzle pressure: 12 MPa, D50 80120 μm.

Inlet air temperature: 150200°C (ethanol), moisture <0.5%.

Drying chamber: diameter 13 m, air flow 10003000 m<sup>3</sup> / h.

Capacity: 50500 kg/h, recovery efficiency 9095%.

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### 3.1.2 Centrifugal Spray Dryer

**Principle :** The slurry is atomized into droplets (1080  $\mu\text{m}$ ) by a high-speed rotating centrifugal disk (10,000-20,000 rpm) and dried by hot air.

**Features :**

High speed atomization, particle D50 50100  $\mu\text{m}$ , high sphericity, excellent fluidity (<25 s/50 g).

Suitable for ultrafine particles and low viscosity slurries (100-300 mPa·s).

High production capacity (100-1000 kg/h), suitable for large-scale production.

**Pros and Cons :**

**Advantages :** small and uniform particles, fluidity improved by 20%, recovery efficiency >98%.

**Disadvantages :** High equipment cost (RMB 100.2 million), centrifugal discs need to be replaced regularly.

**Applicable scenarios :**

Ultrafine -grained cemented carbide (such as precision tools, hardness 18002200 HV).

High performance molds require D50 50100  $\mu\text{m}$ .

**Key parameters :**

Centrifugal disk speed: 15,000-20,000 rpm, D50 50-80  $\mu\text{m}$ .

Inlet temperature: 180250°C, bulk density 45 g/cm<sup>3</sup>.

Drying chamber: diameter 25 m, air flow 2000-5000 m<sup>3</sup> / h.

Capacity: 1001000 kg/h, recovery efficiency 9598%.

### 3.1.3 Twin-fluid spray dryer

**Principle :** High-pressure gas (compressed air or nitrogen, 0.20.5 MPa) is mixed with the slurry at the nozzle and atomized into ultra-fine droplets (550  $\mu\text{m}$ ).

**Features :**

Ultra-fine particles (D50 2080  $\mu\text{m}$ ), suitable for laboratory or high-precision applications.

The production capacity is low (10100 kg/h) and the energy consumption is high.

Suitable for low solid content slurry (50-60%).

**Pros and Cons :**

**Advantages :** extremely fine particles, narrow particle size distribution, suitable for ultrafine-grained cemented carbide.

**Disadvantages :** low production capacity, complex equipment, and high cost (RMB 801.5 million).

**Applicable scenarios :**

ultra- fine-grained cemented carbides (e.g. aviation tools, hardness 2000 HV).

Small batch production, particle D50 <80  $\mu\text{m}$ .

**Key parameters :**

Air pressure: 0.30.5 MPa, D50 2050  $\mu\text{m}$ .

Inlet air temperature: 150200°C, moisture <0.3%.

Drying chamber: diameter 0.51.5 m, air flow 5001500 m<sup>3</sup> / h.

Capacity: 10100 kg/h, recovery efficiency 8590%.

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### 3.2 Selection basis

#### Production scale :

Small scale (<100 kg/h): Dual-fluid type, suitable for laboratories or R&D.

Medium scale (100-500 kg/h): pressure type, taking into account both cost and efficiency.

Large scale (>500 kg/h): Centrifugal, high capacity, suitable for industrialization.

#### Particle requirements :

D50 100-150  $\mu\text{m}$  : Pressure type, suitable for YG6 and YG8.

D50 50-100  $\mu\text{m}$  : Centrifugal, suitable for ultra- fine grain tools.

D50 <80  $\mu\text{m}$  : Dual-fluid type, high-precision application.

#### Slurry characteristics :

High solid content (70-80%): pressure type, resistant to clogging.

Low viscosity (100-300  $\text{mPa}\cdot\text{s}$  ): centrifugal, uniform atomization.

Low solid content (50-60%): dual-fluid type, ultra-fine atomization.

#### Environmental requirements :

Inert gas protection (nitrogen): centrifugal or dual-fluid type, oxygen content <0.1%.

Low dust: Equipped with high-efficiency cyclone separator and bag dust collector, the recovery efficiency is >95%.

### 3.3 Applicable Scenarios

**Pressure type** : YG6, YG8 cutting tools, mining tools, D50 100-150  $\mu\text{m}$  , capacity 200 kg/h, low cost.

**Centrifugal** : ultra-fine grain tools and molds, D50 50-100  $\mu\text{m}$  , production capacity 500 kg/h, fluidity increased by 20%.

**Dual-fluid type** : R&D of aviation tools, D50 <80  $\mu\text{m}$  , production capacity 50 kg/h, hardness increased by 5%.

### 3.4 Application Cases

#### YG6 Tool :

**Equipment** : SPX Anhydro pressure type, 1 MPa, 180°C, capacity 200 kg/h.

**Results** : D50 120  $\mu\text{m}$  , fluidity 25 s/50 g, hardness 1500 HV, machining cast iron life 2 hours.

#### Ultrafine grain cutting tools :

**Equipment** : GEA Niro centrifugal, 15,000 rpm, 180°C, capacity 500 kg/h, nitrogen blanket.

**Results** : D50 80  $\mu\text{m}$  , fluidity 20 s/50 g, hardness 2000 HV, machining life of stainless steel 4 hours.

#### Aviation tool research and development :

**Equipment** : Buchi two-fluid type, 0.4 MPa, 150°C, capacity 20 kg/h.

**Results** : D 50 50  $\mu\text{m}$  , flowability 22 s/50 g, hardness 2000 HV, performance improvement of the

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test batch by 5%.

**Data support :**

Centrifugal: D50 50100  $\mu\text{m}$  , recovery efficiency 98% (Sandvik, 2023).

Pressure type: D50 100150  $\mu\text{m}$  , cost reduced by 20% (ScienceDirect, 2020).

Two-fluid type: D50 2080  $\mu\text{m}$  , particle size distribution narrower by 10% (ISO 4499).

#### 4. Influencing factors

##### 4.1 Slurry characteristics

Solid content: 60-80%. Too high (>80%) will clog the nozzle, too low (<60%) will reduce the density by 10%.

Viscosity: 100500  $\text{mPa}\cdot\text{s}$  . If the viscosity is too high (>1000  $\text{mPa}\cdot\text{s}$  ), the atomization will be uneven.

Forming agent: PEG 12%. Too much (>3%) will cause particles to stick together, too little (<1%) will reduce fluidity by 20%.

##### 4.2 Spray drying parameters

Inlet temperature: 150-250°C, too high (>300°C) will cause oxidation and the hardness will decrease by 5%.

Atomization pressure/speed: 1 MPa or 15,000 rpm, affects D50.

Slurry flow rate: too high ( >100 L/h) particle D50 >200  $\mu\text{m}$  .

##### 4.3 Equipment performance

Nozzle/centrifugal disc: Wear leads to a 10% wider particle size distribution.

Drying chamber: uneven airflow, agglomeration rate increased by 15%.

Separation efficiency: <90% fine powder loss>10%.

##### 4.4 Environmental Control

Humidity: >50% moisture increase by 0.5%.

Temperature: >300°C Oxygen content increases by 0.1%.

Dust: No filtration, flexural strength reduced by 10%.

**Data** : solid content 70%, inlet temperature 180°C, D50 100  $\mu\text{m}$  , fluidity improved by 20% (ScienceDirect, 2020).

#### 5. Optimization measures

**Slurry formula** : solid content 70.75%, PEG 1.5%, fluidity increased by 20%.

**Parameter control** : inlet air temperature 180°C, 1 MPa, D50 80120  $\mu\text{m}$  .

**High efficiency equipment** : centrifugal type, recovery efficiency>98%.

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**Environmental control** : Nitrogen protection, oxygen content <0.1%, hardness increased by 5%.

**Post-treatment** : Screening 100200  $\mu\text{m}$  , porosity reduction 10%.

**Effect** : fluidity <25 s/50 g, pressing uniformity increased by 15%, hardness increased by 5%, and flexural strength increased by 10%.

## 6. Practical application cases

**YG6 tool** : pressure type (1 MPa, 180°C), D50 120  $\mu\text{m}$  , fluidity 25 s/50 g, hardness 1500 HV, machining life of cast iron 2 hours.

**Ultrafine grain tool** : centrifugal (15,000 rpm, 180°C), D50 80  $\mu\text{m}$  , fluidity 20 s/50 g, hardness 2000 HV, life for stainless steel 4 hours.

**YG15 mold** : centrifugal (12,000 rpm, 200°C), D50 150  $\mu\text{m}$  , fluidity 28 s/50 g, flexural strength 2.5 GPa , life span 120,000 times.

## 7. Conclusion

The spray drying and granulation process of cemented carbide mixed powder includes mixed slurry preparation, spray drying, granulation and collection, post-processing and screening, and quality control. The goal is to generate granulated powder with good fluidity (<30 s/50 g) and uniform particle size (D50 50200  $\mu\text{m}$  ). Equipment selection includes pressure type (small and medium scale, D50 100150  $\mu\text{m}$  ), centrifugal type (large scale, D50 50100  $\mu\text{m}$  ), and dual fluid type (research and development, D50 <80  $\mu\text{m}$  ), which are selected based on production scale, particle requirements, slurry characteristics and cost. Optimizing slurry formula, parameters, equipment and environment can improve fluidity by 20%, hardness by 5%, and bending strength by 10%.

### Standard :

GB/T 3849 : Cobalt magnetic test.

ISO 4499: Microstructural analysis.

GB/T 1482: Flowability test.

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## Appendix:

### Nickel powder for nickel-based cemented carbide

Nickel-based cemented carbide ( NickelBonded Cemented Carbide) uses nickel (Ni) as a bonding phase to replace cobalt (Co) in traditional cobalt-based cemented carbide . It is widely used in scenarios requiring high corrosion resistance , high temperature resistance and wear resistance, such as chemical equipment, oil drilling tools and high-temperature molds. Nickel powder is a key raw material for nickel-based cemented carbide, and its quality directly affects the microstructure, mechanical properties and corrosion resistance of the alloy. The following details the characteristics, requirements, preparation methods and applications of nickel powder used in nickel-based cemented carbide , combined with industry standards (such as GB/T 5243, ISO 4499) and the latest research (such as Sandvik, 2023; ScienceDirect, 2020), all in Chinese to ensure that the content is accurate, comprehensive and fascinating.

## 1. Overview

Nickel-based cemented carbide uses tungsten carbide (WC) as the hard phase and nickel as the bonding phase. Typical grades include YN6 (6% Ni) and YN10 (10% Ni). Compared with cobalt-based alloys (such as YG6 and YG15), nickel-based alloys have:

**Higher corrosion resistance** : In acidic (HCl, H<sub>2</sub>SO<sub>4</sub>) and high temperature environments, the corrosion rate is reduced by 2050% (GB/T 43342020).

**Excellent high temperature performance** : the oxidation resistance temperature is increased by 100-150°C, suitable for high temperature molds.

**Slightly lower strength and toughness** : flexural strength (1.82.2 GPa ) is 1015% lower than

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cobalt-based , and fracture toughness ( $K_{IC}$  710 MPa·m<sup>1/2</sup>) is 510% lower.

of nickel powder (purity, particle size, morphology, oxygen content) directly affects:

**Microstructure** : homogeneity>95%, grain size 0.52 μm , η phase/free carbon <1% (GB/T 183762014).

**Mechanical properties** : hardness 14001800 HV, flexural strength deviation <5% (GB/T 38512015).

**Corrosion resistance** : Corrosion rate <0.005 mm/year (GB/T 43342020).

The following is a detailed explanation of nickel powder from four aspects: characteristics, requirements, preparation and practical applications.

## 2. Characteristics and requirements of nickel powder

Nickel-based cemented carbide has strict requirements on nickel powder , which must meet the standards of chemical composition, physical properties and micromorphology to ensure the consistency of alloy performance.

### 2.1 Chemical composition

#### Purity :

Requirements: >99.9% (mass fraction), total impurity content <0.1% (GB/T 53142011).

Main impurities:

Oxygen (O): <0.05%, high oxygen leads to decarburization (η phase, Co<sub>3</sub>W<sub>3</sub>C or Ni<sub>3</sub>W<sub>3</sub>C), and the hardness decreases by 510%.

Carbon (C): <0.01%, avoid free carbon (>1% reduces strength by 10%).

Iron (Fe): <0.01%, the risk of microcracks caused by Fe impurities increases by 15%.

Sulfur (S), phosphorus (P): <0.005% each, avoid low melting point phase (embrittlement increases by 20%).

Test method:

ICPMS: Detection of metals such as Ni and Fe (accuracy ±0.001%).

Carbon and sulfur analyzer : detect C and S (accuracy ±0.001%).

Oxygen and nitrogen analyzer: detect O (accuracy ±0.01%).

#### Examples :

YN10: Nickel powder purity 99.95%, O <0.03%, Fe <0.005% (Sandvik, 2023).

### 2.2 Physical properties

#### Granularity :

Range: 0.52 μm (conventional), 0.20.8 μm (ultrafine grain alloy).

Uniformity: D50 deviation <±10%, D90/D10 <3, ensuring mixing uniformity >95%.

significance:

Fine particle size (<1 μm ) improves the uniformity of the bonding phase distribution and increases the strength by 10%.

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Too fine ( $<0.2\ \mu\text{m}$ ) is easy to agglomerate, and the uniformity is reduced by 510%.

Test method: Laser particle size analyzer (accuracy  $\pm 0.01\ \mu\text{m}$ , GB/T 19077).

**Specific surface area :**

Range:  $13\ \text{m}^2/\text{g}$  (conventional),  $35\ \text{m}^2/\text{g}$  (nanopowder).

Significance: High specific surface area enhances sintering activity and reduces liquid phase sintering temperature by  $2030^\circ\text{C}$ .

Test method: BET method (accuracy  $\pm 0.1\ \text{m}^2/\text{g}$ ).

**Liquidity :**

Requirements:  $<25\ \text{s}/50\ \text{g}$  (Hall flowmeter, GB/T 1482-2010).

Significance: Good fluidity ensures uniformity of pressed billet ( $>95\%$ ) and reduces porosity by 0.01%.

**Examples :**

YN6: particle size  $11.5\ \mu\text{m}$ ,  $D_{50} \sim 1.2\ \mu\text{m}$ , fluidity  $\sim 20\ \text{s}/50\ \text{g}$  (ScienceDirect, 2020).

## 2.3 Microscopic morphology

**Appearance :**

Requirements: Nearly spherical or polyhedral, sphericity  $>0.9$  (SEM observation,  $1000\times$ ).

Avoid: sticks, flakes or irregular particles, which reduce fluidity by 10-15%.

**Surface condition :**

Smooth, without cracks, pores or attachments (SEM inspection,  $<0.1\ \mu\text{m}$  defect).

Surface oxide layer:  $<10\ \text{nm}$  (XPS analysis), avoid sintering decarburization.

**Reunion :**

Agglomeration rate:  $<1\%$ . Too high will lead to uneven microstructure and increase porosity by 0.02%.

Test method: SEM (statistical analysis of agglomerated particle ratio), ultrasonic dispersion verification.

**Examples :**

YN10: Nearly spherical nickel powder, agglomeration rate  $<0.5\%$ , surface oxide layer  $<5\ \text{nm}$  (Sandvik, 2023).

## 2.4 Other requirements

**Magnetic properties :**

Nickel powder is weakly magnetic, with a saturation magnetization of  $\sim 55\ \text{emu/g}$  (pure Ni) and a deviation of  $\leq \pm 5\ \text{emu/g}$ .

Test method: Vibrating sample magnetometer (VSM, accuracy  $\pm 0.1\ \text{emu/g}$ ).

Significance: Indirect assessment of impurities (Fe magnetization) and degree of oxidation.

**Storage conditions :**

Humidity:  $<40\%$ , Temperature:  $2025^\circ\text{C}$ , Inert atmosphere (Ar or  $\text{N}_2$ ), Avoid oxidation (O increases by 0.02%).

Vacuum sealed packaging, storage period  $< 6$  months .

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**Standard :**

GB/T 53142011: Chemical composition (purity>99.9%).

GB/T 183762014: Microstructure (agglomeration <1%).

GB/T 1482-2010: Flowability (<25 s/50 g).

**Nickel Powder Characteristics and Requirements**

characteristic	Require	Test Method	Example (YN10)
purity	>99.9%, impurities <0.1%	ICPMS, carbon and sulfur analysis	99.95%, Fe <0.005%
Oxygen content	<0.05%	Oxygen and nitrogen analyzer	<0.03%
Carbon content	<0.01%	Carbon and sulfur analyzer	<0.005%
granularity	0.52 μm (conventional), 0.20.8 μm (ultra-fine grain ), D50 deviation <±10%	Laser particle size analysis	0.81.2 μm , D50 ~1.0 μm
Specific surface area	15 m <sup>2</sup> / g	BET	34 m <sup>2</sup> / g
Liquidity	<25 s/50 g	Hall flow meter	~18 s/50 g
Morphology	Nearly spherical, sphericity>0.9, agglomeration<1%	SEM, XPS	Sphericity>0.95, agglomeration<0.5%
Magnetic properties	~55 emu/g, deviation <±5 emu/g	VSM	54 ± 2 emu/g
Storage conditions	Humidity <40%, Ar /N2 protection, < 6 months		Vacuum sealed, Ar protected

**3. Preparation method of nickel powder**

of nickel powder must ensure high purity, fine particle size and uniform morphology to meet the requirements of cemented carbide. Common methods include:

**3.1 Carbonyl Process**

**principle :**

Nickel reacts with carbon monoxide (CO) to form nickel carbonyl [Ni(CO)<sub>4</sub>], which decomposes into high-purity nickel powder and CO upon heating.

Reaction:  $\text{Ni} + 4\text{CO} \rightarrow \text{Ni(CO)}_4$  (gaseous, 5060°C),  $\text{Ni(CO)}_4 \rightarrow \text{Ni} + 4\text{CO}$  (decomposition, 200250°C).

**Process :**

Raw materials: high purity nickel (>99.9%), CO gas (purity>99.99%).

Equipment: carbonyl reactor (pressure 0.1-0.5 MPa), decomposition furnace (vacuum or inert atmosphere).

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Parameters: decomposition temperature 200300°C, air flow rate 0.51 L/min.

Classification: Screening or airflow classification, controlled particle size 0.52 μm .

**Features :**

Purity: >99.95%, O <0.03%, C <0.01%.

Morphology: Nearly spherical, sphericity>0.95, agglomeration rate<0.5%.

Particle size: 0.52 μm , D50 deviation <±5%.

**Advantages :**

High purity, impurities (Fe, S) <0.005%.

The morphology is regular and the fluidity is good (~18 s/50 g).

**insufficient :**

The equipment is complex, CO is highly toxic, and the cost is 20-30% higher.

**application :**

YN10 test bar: carbonyl nickel powder, particle size ~1 μm , hardness 1500 HV (Sandvik, 2023).

### 3.2 Chemical reduction (Hydrometallurgical Reduction)

**principle :**

Nickel salt solution (such as NiSO<sub>4</sub>, NiCl<sub>2</sub>) is reduced to nickel powder by a reducing agent (such as H<sub>2</sub>, NaBH<sub>4</sub>).

Reaction:  $\text{Ni}^{2+} + \text{H}_2 \rightarrow \text{Ni} + 2\text{H}^+$  ( high pressure H<sub>2</sub>, 150200°C).

**Process :**

Raw materials: NiSO<sub>4</sub> (>99.9%), reducing agent (H<sub>2</sub> purity >99.99%).

Equipment: High-pressure reactor (510 MPa), filtration/drying system.

Parameters: pH 810, temperature 150200°C, H<sub>2</sub> pressure 25 MPa.

Post-treatment: washing (deionized water), vacuum drying (80°C, <10<sup>-2</sup> Pa ).

**Features :**

Purity: >99.9%, O <0.05%, Fe <0.01%.

Morphology: polyhedron or nearly spherical, sphericity 0.80.9.

Particle size: 0.53 μm , D50 deviation <±10%.

**Advantages :**

Lower cost (1520% lower than carbonyl process).

Suitable for mass production, with adjustable particle size.

**insufficient :**

The morphology is slightly irregular and the agglomeration rate is 12%.

The oxygen content is slightly high (0.050.1%) and needs to be strictly controlled.

**application :**

YN6 test bar: reduced nickel powder, particle size 1.5 μm , flexural strength 1.8 GPa (ScienceDirect, 2020).

### 3.3 Atomization

**principle :**

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Molten nickel (>99.9%) is atomized into fine particles by high-pressure gas (N<sub>2</sub>, Ar) or water.

**Process :**

Raw material: high purity nickel ingot (>99.9%).

Equipment: vacuum induction furnace (1450/1500°C), atomization tower (gas pressure 510 MPa).

Parameters: nozzle aperture 0.51 mm, cooling rate  $10^3 \sim 10^4$  °C/s.

Classification: air flow classification, particle size 15 μm.

**Features :**

Purity: >99.9%, O <0.08%, C <0.02%.

Morphology: spherical, sphericity >0.9.

Particle size: 15 μm, D50 deviation <±15%.

**Advantages :**

Regular morphology and excellent fluidity (~20 s/50 g).

Suitable for large particle nickel powder (>2 μm).

**insufficient :**

The particle size is too large (>1 μm), which is not suitable for ultrafine-grained alloys.

The oxygen content is high (0.050.1%).

**application :**

Large size YN15 test bar: atomized nickel powder, particle size 23 μm, KIC 10 MPa·m<sup>1/2</sup>.

### 3.4 Electrodeposition

**principle :**

Nickel salt solution (such as NiSO<sub>4</sub>) is produced by electrolytic deposition of nickel powder.

Reaction:  $\text{Ni}^{2+} + 2\text{e}^- \rightarrow \text{Ni}$  (cathode, current density 100500 A/m<sup>2</sup>).

**Process :**

Raw materials: NiSO<sub>4</sub> (>99.9%), electrolyte (pH 35).

Equipment: electrolytic cell (stainless steel cathode), constant current power supply.

Parameters: temperature 50/60°C, current density 200/400 A/m<sup>2</sup>, time 24 hours.

Post-treatment: washing, drying (80°C, <10<sup>-2</sup> Pa), grinding (<2 μm).

**Features :**

Purity: >99.9%, O <0.1%, Fe <0.02%.

Morphology: dendritic or irregular, needs grinding.

Particle size: 110 μm (0.52 μm after grinding).

**Advantages :**

30% lower than carbonyl process).

Suitable for large-scale production.

**insufficient :**

The morphology is irregular and grinding increases agglomeration (23%).

The oxygen content is high (0.10.2%) and needs to be optimized.

**application :**

Low-cost YN6 test rod: electrolytic nickel powder, particle size 12 μm, hardness 1400 HV.

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### 3.5 Optimization measures

#### Reduce oxygen levels :

inert atmosphere ( Ar , O<sub>2</sub> <0.005%), oxygen is reduced to <0.03%.  
vacuum (<10<sup>-2</sup> Pa) to avoid oxidation.

#### Control granularity :

After ultrasonic dispersion (500 W, 10 min), the agglomeration rate was reduced to <0.5%.  
Airflow classification (accuracy ±0.1 μm ), D50 deviation <±5%.

#### Improve appearance :

Carbonyl method + plasma spheroidization, the sphericity increased to >0.95.  
Chemical reduction method plus surface modification (stearic acid 0.1%), fluidity increased by 10%.

#### Examples :

YN10: Carbonyl nickel powder + ultrasonic dispersion, particle size 0.8 μm , agglomeration <0.3% (Sandvik, 2023).

**Nickel powder preparation methods**

Method	Purity	Particle size ( μm )	Morphology	Oxygen content	Flowability (s/50 g)	Cost	Applicable grades
Carbonyl method	>99.95%	0.52	Nearly spherical, >0.95	<0.03%	~18	High	Yn10, yn8n
Chemical reduction method	>99.9%	0.53	Polyhedron, 0.80.9	<0.05%	~20	Middle	Yn6
Atomization	>99.9%	15	Spherical, >0.9	<0.08%	~20	Medium to high	Yn15
Electrolysis	>99.9%	0.52 (grinding)	Irregular, 0.60.8	<0.1%	~25	Low	Yn6

## 4. Application of nickel powder in nickel-based cemented carbide

of nickel powder directly affects the preparation and performance of nickel-based cemented carbide test bars. The following is a description of the specific grades and processes.

### 4.1 YN6 (6% Ni, general corrosion-resistant tool)

#### Nickel powder requirements :

Purity: >99.95%, O <0.03%, Fe <0.005%.  
Particle size: 11.5 μm , D50 ~1.2 μm , sphericity >0.9.  
Preparation: Carbonyl method, flowability ~20 s/50 g.

#### Preparation process :

**Ingredients** : WC (94 wt % , 12 μm ), Ni (6 wt %), Cr<sub>3</sub>C<sub>2</sub> (0.3 wt %).

**Compounding** : wet milling (12-14 h, PEG 1.5%), D50 80-150 μm .

**Pressing** : CIP (200250 MPa), billet 6.2×6.2×43 mm.

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**Sintering** : vacuum sintering ( $1400^{\circ}\text{C}$ ,  $<10^{-3}\text{ Pa}$ ) + HIP ( $1400^{\circ}\text{C}$ , 100 MPa).

**Machining** : grinding ( $R_a < 0.4\text{ }\mu\text{m}$ ), spark notching ( $0.25 \pm 0.02\text{ mm}$ ).

**performance :**

Hardness:  $1400 \pm 50\text{ HV}$  (GB/T 79972017).

Flexural strength:  $1.8 \pm 0.1\text{ GPa}$  (GB/T 38512015).

Fracture toughness:  $7 \pm 0.5\text{ MPa}\cdot\text{m}^{1/2}$ .

Corrosion resistance: 0.01 mm/year (5% HCl, GB/T 43342020).

**Examples :**

YN6 test rod: carbonyl nickel powder, grain size  $\sim 1.2\text{ }\mu\text{m}$ , cutting life 2.5 hours (Sandvik, 2023).

#### 4.2 YN10 (10% Ni, chemical mold)

**Nickel powder requirements :**

Purity:  $>99.95\%$ , O  $<0.03\%$ , C  $<0.01\%$ .

Particle size:  $0.81.2\text{ }\mu\text{m}$ , D50  $\sim 1.0\text{ }\mu\text{m}$ , agglomeration  $<0.5\%$ .

Preparation: carbonyl method + ultrasonic dispersion, fluidity  $\sim 18\text{ s/50 g}$ .

**Preparation process :**

**Ingredients** : WC (90 wt %,  $0.51.5\text{ }\mu\text{m}$ ), Ni (10 wt %), VC (0.2 wt %).

**Mixing** : High energy ball milling (16 h, PEG 1%), D50  $50100\text{ }\mu\text{m}$ .

**Pressing** : CIP (250300 MPa), billet  $5.0 \times 10.0 \times 40\text{ mm}$ .

**Sintering** : vacuum sintering ( $1380^{\circ}\text{C}$ ,  $<5 \times 10^{-4}\text{ Pa}$ ) + HIP ( $1380^{\circ}\text{C}$ , 120 MPa).

**Processing** : ultra-precision grinding ( $R_a < 0.2\text{ }\mu\text{m}$ ), femtosecond laser notch ( $0.25 \pm 0.01\text{ mm}$ ).

**performance :**

Hardness:  $1500 \pm 50\text{ HV}$ .

Flexural strength:  $2.0 \pm 0.1\text{ GPa}$ .

Fracture toughness:  $9 \pm 0.5\text{ MPa}\cdot\text{m}^{1/2}$ .

Corrosion resistance:  $<0.005\text{ mm/year}$  (5% HCl).

**Examples :**

YN10 test rod: carbonyl nickel powder, corrosion resistance life 100,000 times (ScienceDirect, 2020).

#### 4.3 Ultrafine grain YN8N (8% Ni, aviation tools)

**Nickel powder requirements :**

Purity:  $>99.95\%$ , O  $<0.02\%$ , Fe  $<0.005\%$ .

Particle size:  $0.20.8\text{ }\mu\text{m}$ , D50  $\sim 0.5\text{ }\mu\text{m}$ , sphericity  $>0.95$ .

Preparation: carbonyl method + plasma spheroidization, fluidity  $\sim 15\text{ s/50 g}$ .

**Preparation process :**

**Ingredients** : WC (91.5 wt %,  $0.20.4\text{ }\mu\text{m}$ ), Ni (8 wt %), Cr3C2 (0.4 wt %), VC (0.1 wt %).

**Mixing** : high energy ball milling (1820 h, modified PEG 1%), D50  $30100\text{ }\mu\text{m}$ .

**Pressing** : CIP (300350 MPa), billet  $6.3 \times 6.3 \times 44\text{ mm}$ .

**Sintering** : vacuum sintering ( $1350^{\circ}\text{C}$ ,  $<5 \times 10^{-4}\text{ Pa}$ ) + HIP ( $1350^{\circ}\text{C}$ , 150 MPa).

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**Processing** : ultra-precision grinding ( $Ra < 0.2 \mu m$ ), femtosecond laser notch ( $0.25 \pm 0.005 mm$ ).

**performance** :

Hardness:  $1800 \pm 50 HV$ .

Flexural strength:  $2.2 \pm 0.1 GPa$ .

Fracture toughness:  $8 \pm 0.5 MPa \cdot m^{1/2}$ .

Corrosion resistance:  $< 0.003 mm/year$ .

**Examples** :

YN8N test rod: carbonyl nickel powder, grain size  $< 0.5 \mu m$ , aviation tool life 4 hours (Sandvik, 2023).

#### Nickel-based cemented carbide grades and nickel powder applications

Brand	Nickel content	Nickel powder type	Particle size ( $\mu m$ )	Key points of technology	performance	application
YN6	6%	Carbonyl method	11.5	1400°C sintered, ground $Ra < 0.4 \mu m$	Hardness 1400 HV, strength 1.8 GPa, corrosion 0.01 mm/year	Corrosion-resistant tool life 2.5 hours
YN10	10%	Carbonyl method	0.81.2	1380°C HIP, femtosecond laser notching	Hardness 1500 HV, KIC 9 $MPa \cdot m^{1/2}$ , corrosion $< 0.005 mm/year$	Chemical mold, lifespan 100,000 times
YN8	8%	Carbonyl + Spheroidization	0.20.8	Sintered at 1350°C, grain size $< 0.5 \mu m$	Hardness 1800 HV, strength 2.2 GPa, corrosion $< 0.003 mm/year$	Aviation tool, life 4 hours

### 5. Key factors for nickel powder selection

The selection of nickel powder requires comprehensive consideration of alloy properties, process conditions and costs:

**High corrosion resistance** (such as YN10):

The preferred choice is carbonyl nickel powder, with a purity of  $> 99.95\%$ , a particle size of  $0.81.2 \mu m$ , and  $O < 0.03\%$ .

Reason: Regular morphology and low oxygen ensure that  $\eta$  phase is  $< 0.5\%$  and corrosion rate is  $< 0.005 mm/year$ .

**Ultrafine grain alloys** (such as YN8N):

The carbonyl method + plasma spheroidization was used, the particle size was  $0.20.8 \mu m$ , and the sphericity was  $> 0.95$ .

Reason: Fine grain size control grain  $< 0.5 \mu m$ , hardness increased by 510%.

**Cost sensitive** (such as YN6):

Use chemical reduction or electrolysis method, particle size  $12 \mu m$ ,  $O < 0.05\%$ .

Reason: 2030% lower cost and meets general tool requirements (strength 1.8 GPa).

**Large size test rod** (such as YN15):

The atomization method was selected, with a particle size of  $23 \mu m$  and a fluidity of  $\sim 20 s/50 g$ .

Reason: Suitable for large particles, compression uniformity  $> 95\%$ .

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### Nickel powder selection and application scenarios

Application Scenario	Recommended Powder	Nickel Particle size ( $\mu\text{m}$ )	Key Features	Performance Improvements
High corrosion resistance (YN10)	Carbonyl method	0.81.2	Purity>99.95%, O<0.03%	Corrosion rate <0.005 mm/year, $\eta$ phase <0.5%
Ultra-fine grain (YN8N)	Carbonyl + Spheroidization	0.20.8	Sphericity>0.95, agglomeration<0.5%	Hardness increased by 510%, grain size <0.5 $\mu\text{m}$
Cost sensitive (YN6)	Chemical reduction/electrolysis	12	Purity>99.9%, cost 2030% lower	Strength 1.8 GPa , meets general requirements
Large size (YN15)	Atomization	twenty three	Flowability ~20 s/50 g	Homogeneity>95%, KIC 10 $\text{MPa}\cdot\text{m}^{1/2}$

## 6. Conclusion

Nickel powder used in nickel-based cemented carbide must meet the following requirements:

**Chemical composition** : purity>99.9%, O <0.05%, Fe <0.01%, C <0.01% (GB/T 53142011).

**Physical properties** : particle size 0.52  $\mu\text{m}$  (conventional) or 0.20.8  $\mu\text{m}$  (ultrafine grain ), fluidity <25 s/50 g (GB/T 1482-2010).

**Micromorphology** : Nearly spherical, sphericity>0.9, agglomeration<1% (GB/T 183762014).

**Preparation method** :

**Carbonyl method** : high purity (>99.95%), particle size 0.52  $\mu\text{m}$  , suitable for YN10 and YN8N.

**Chemical reduction method** : low cost, particle size 0.53  $\mu\text{m}$  , suitable for YN6.

**Atomization method** : large particles (15  $\mu\text{m}$  ), suitable for large-sized test rods.

**Electrolytic method** : low cost, requires grinding, suitable for general grades.

**Application examples** :

YN6: Carbonyl nickel powder (11.5  $\mu\text{m}$  ), hardness 1400 HV, strength 1.8 GPa .

YN10: Carbonyl nickel powder (0.81.2  $\mu\text{m}$  ), KIC 9  $\text{MPa}\cdot\text{m}^{1/2}$  , corrosion resistance <0.005 mm/year.

YN8N: Carbonyl + spheroidized nickel powder (0.20.8  $\mu\text{m}$  ), hardness 1800 HV, grain size <0.5  $\mu\text{m}$  .

of nickel powder (such as ultrasonic dispersion and inert protection) can improve uniformity by 20%, reduce  $\eta$  phase by 50%, and improve the consistency of alloy performance (deviation <3%). In the future, nano nickel powder (<0.2  $\mu\text{m}$  ) and green preparation technology (such as low temperature reduction) will further improve the performance of nickel-based cemented carbide.

**standard** :

GB/T 5314 2011: Chemical composition.

GB/T 18376 2014: Microstructure.

GB/T 1482 2010: Liquidity.

GB/T 3851 2015: Flexural strength.

GB/T 7997 2017: Hardness.

GB/T 4334 2020: Corrosion resistance.

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## Appendix:

### Cobalt powder used in cobalt-based cemented carbide

Cobalt-based cemented carbide (CobaltBonded Cemented Carbide) uses cobalt (Co) as a bonding phase and combines hard phases such as tungsten carbide (WC). It is widely used in cutting tools, molds, mining tools and other fields due to its excellent bending strength (1.52.5 GPa), fracture toughness ( $812 \text{ MPa} \cdot \text{m}^{1/2}$ ) and hardness (1400-2200 HV). As a key raw material for cobalt-based cemented carbide, the quality of cobalt powder directly affects the microstructure, mechanical properties and processing properties of the alloy. The following details the characteristics, requirements, preparation methods and applications of cobalt powder used in cobalt-based cemented carbide, combined with Chinese national standards (such as GB/T 5243, GB/T 5314), international standards (such as ISO 4499) and the latest research (such as Sandvik, 2023; ScienceDirect, 2020), all in Chinese to ensure that the content is accurate, detailed and fascinating.

## 1. Overview

Cobalt-based cemented carbide uses cobalt as the bonding phase. Typical grades include YG6 (6% Co, cutting tools), YG15 (15% Co, molds), and YG8N (8% Co, ultra-fine-grained aviation cutting tools). The functions of cobalt powder in the alloy include:

**Bonding hard phase** : Strengthens the bonding of WC particles and increases the flexural strength by 2030%.

**Improved toughness** : The ductility of the cobalt phase allows KIC to reach  $812 \text{ MPa} \cdot \text{m}^{1/2}$ .

**Sintering activity** : The low melting point of cobalt ( $1495^\circ\text{C}$ ) promotes liquid phase sintering, reducing porosity to  $<0.01\%$ .

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of cobalt powder (purity, particle size, morphology, oxygen content) directly affects:

**Microstructure** : grain size 0.52  $\mu\text{m}$  (conventional) or  $<0.5 \mu\text{m}$  (ultrafine grain ),  $\eta$  phase/free carbon  $<1\%$  (GB/T 183762014).

**Mechanical properties** : hardness 14002200 HV, flexural strength deviation  $<5\%$  (GB/T 38512015).

**Process stability** : mixing uniformity  $>95\%$ , green strength of blank  $>6 \text{ MPa}$ .

The following is an in-depth explanation of cobalt powder from four aspects: characteristics, requirements, preparation methods and practical applications.

## 2. Characteristics and requirements of cobalt powder

Cobalt-based cemented carbide has strict requirements on cobalt powder , which must meet the standards of chemical composition, physical properties and micromorphology to ensure the consistency of alloy performance.

### 2.1 Chemical composition

#### Purity :

Requirements:  $>99.9\%$  (mass fraction), total impurity content  $<0.1\%$  (GB/T 53142011).

Main impurities:

Oxygen (O):  $<0.05\%$ , high oxygen leads to decarburization ( $\eta$  phase,  $\text{Co}_3\text{W}_3\text{C}$ ), and the hardness decreases by 510%.

Carbon (C):  $<0.01\%$ , avoid free carbon ( $>1\%$  reduces strength by 10%).

Iron (Fe):  $<0.01\%$ . The risk of microcracks caused by Fe impurities increases by 1015%.

Sulfur (S), phosphorus (P):  $<0.005\%$  each, avoid low melting point phase (brittleness increases by 1520%).

Nickel (Ni):  $<0.05\%$ . Too high Ni content will change the magnetic properties and affect the cobalt magnetic test (GB/T 3849-2015).

Test method:

ICPMS: Detection of metals such as Co, Fe, Ni (accuracy  $\pm 0.001\%$ ).

Carbon and sulfur analyzer : detect C and S (accuracy  $\pm 0.001\%$ ).

Oxygen and nitrogen analyzer: detect O (accuracy  $\pm 0.01\%$ ).

#### Examples :

YG8N: Cobalt powder purity 99.95%, O  $<0.03\%$ , Fe  $<0.005\%$  (Sandvik, 2023).

### 2.2 Physical properties

#### Granularity:

Range: 0.52  $\mu\text{m}$  (conventional), 0.20.8  $\mu\text{m}$  (ultrafine grain alloy).

Uniformity: D50 deviation  $\leq \pm 10\%$ , D90/D10  $< 3$ , ensuring mixing uniformity  $>95\%$ .

significance:

Fine particle size ( $<1 \mu\text{m}$  ) improves the uniformity of the bonding phase distribution and increases

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the strength by 1015%.

Too fine ( $<0.2\ \mu\text{m}$ ) is easy to agglomerate, and the uniformity is reduced by 510%.

Test method: Laser particle size analyzer (accuracy  $\pm 0.01\ \mu\text{m}$ , GB/T 19077).

#### **Specific surface area :**

Range:  $13\ \text{m}^2/\text{g}$  (conventional),  $36\ \text{m}^2/\text{g}$  (nanopowder).

Significance: High specific surface area enhances sintering activity and reduces liquid phase sintering temperature by  $2030^\circ\text{C}$ .

Test method: BET method (accuracy  $\pm 0.1\ \text{m}^2/\text{g}$ ).

#### **Liquidity :**

Requirements:  $<25\ \text{s}/50\ \text{g}$  (Hall flowmeter, GB/T 1482-2010).

Significance: Good fluidity ensures uniformity of pressed billet ( $>95\%$ ) and reduces porosity by 0.01%.

#### **Apparent density :**

Range:  $1.5\text{-}2.5\ \text{g}/\text{cm}^3$  (conventional),  $1.0\text{-}2.0\ \text{g}/\text{cm}^3$  (ultrafine grain).

Significance: High apparent density improves the green strength of the billet ( $>6\ \text{MPa}$ ).

Test method: Funnel method (accuracy  $\pm 0.01\ \text{g}/\text{cm}^3$ ).

#### **Examples :**

YG6: particle size  $11.5\ \mu\text{m}$ ,  $D_{50} \sim 1.2\ \mu\text{m}$ , fluidity  $\sim 20\ \text{s}/50\ \text{g}$ , BET  $\sim 2\ \text{m}^2/\text{g}$  (ScienceDirect, 2020).

## **2.3 Microscopic morphology**

#### **Appearance :**

Requirements: Nearly spherical or polyhedral, sphericity  $>0.9$  (SEM observation,  $1000\times$ ).

Avoid: sticks, flakes or irregular particles, which reduce fluidity by 10-15%.

#### **Surface condition :**

Smooth, without cracks, pores or attachments (SEM inspection,  $<0.1\ \mu\text{m}$  defect).

Surface oxide layer:  $<10\ \text{nm}$  (XPS analysis), avoid sintering decarburization.

#### **Reunion :**

Agglomeration rate:  $<1\%$ . Too high will lead to uneven microstructure and increase porosity by 0.02%.

Test method: SEM (statistical analysis of agglomerated particle ratio), ultrasonic dispersion verification.

#### **Examples :**

YG8N: Nearly spherical cobalt powder, agglomeration rate  $<0.5\%$ , surface oxide layer  $<5\ \text{nm}$  (Sandvik, 2023).

## **2.4 Other requirements**

#### **Magnetic properties :**

Cobalt powder is ferromagnetic, with a saturation magnetization of  $\sim 160\ \text{emu/g}$  (pure Co) and a deviation of  $\leq \pm 5\ \text{emu/g}$ .

Test method: Vibrating sample magnetometer (VSM, accuracy  $\pm 0.1\ \text{emu/g}$ ).

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Significance: Indirect assessment of impurities (Fe increases magnetization, Ni decreases magnetization) and degree of oxidation.

#### Storage conditions :

Humidity: <40%, Temperature: 2025°C, Inert atmosphere ( Ar or N2), Avoid oxidation (O increases by 0.02%).

Vacuum sealed packaging, storage period < 6 months .

#### Standard :

GB/T 53142011: Chemical composition (purity>99.9%).

GB/T 183762014: Microstructure (agglomeration <1%).

GB/T 1482-2010: Flowability (<25 s/50 g).

GB/T 3849-2015: Magnetic properties (indirect assessment of carbon content).

## 2.5 Key parameters of cobalt powder

Parameter	General requirements	Ultrafine grain requirements	Test method	Example (yg8n)
Purity	>99.9%	>99.95%	Icpms, carbon and sulfur analysis	99.95%
Oxygen content	<0.05%	<0.03%	Oxygen and nitrogen analyzer	<0.03%
Iron content	<0.01%	<0.005%	Icpms	<0.005%
Particle size (d50)	0.52 μm	0.20.8 μm	Laser particle size analysis	~0.5 μm
Specific surface area	13 m <sup>2</sup> / g	36 m <sup>2</sup> / g	Bet	~4 m <sup>2</sup> / g
Liquidity	<25 s/50 g	<20 s/50 g	Hall flow meter	~15 s/50 g
Morphology	Nearly spherical, sphericity>0.9	Nearly spherical, sphericity>0.95	Sem	Sphericity>0.95
Reunion rate	<1%	<0.5%	Sem, ultrasonic dispersion	<0.3%
Magnetization	~160 emu/g, deviation <±5 emu/g	~160 emu/g, deviation <±3 emu/g	Vsm	~158 emu/g

GB/T 53142011 (chemical composition), GB/T 183762014 (microstructure), GB/T 14822010 (fluidity)

## 3. Preparation method of cobalt powder

of cobalt powder must ensure high purity, fine particle size and uniform morphology to meet the requirements of cemented carbide. Common methods include:

### 3.1 Chemical reduction (Hydrometallurgical Reduction)

#### Principle :

Cobalt salt solution (such as CoSO<sub>4</sub>, CoCl<sub>2</sub>) is reduced to cobalt powder by a reducing agent (such as H<sub>2</sub>, NaBH<sub>4</sub>).

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Reaction:  $\text{Co}^{2+} + \text{H}_2 \rightarrow \text{Co} + 2\text{H}^+$  ( high pressure  $\text{H}_2$ , 150200°C).

**Process :**

Raw materials:  $\text{CoSO}_4$  (>99.9%), reducing agent ( $\text{H}_2$  purity >99.99%).

Equipment: High-pressure reactor (510 MPa), filtration/drying system.

Parameters: pH 810, temperature 150200°C,  $\text{H}_2$  pressure 25 MPa.

Post-treatment: washing (deionized water), vacuum drying (80°C,  $<10^{-2}$  Pa ).

Classification: air flow classification, controlled particle size 0.52  $\mu\text{m}$  .

**Features :**

Purity: >99.9%, O <0.05%, Fe <0.01%.

Morphology: polyhedron or nearly spherical, sphericity 0.80.9.

Particle size: 0.53  $\mu\text{m}$  , D50 deviation  $\leq \pm 10\%$ .

**Advantages :**

Lower cost (1520% lower than carbonyl process).

Suitable for mass production, with adjustable particle size.

**insufficient :**

The morphology is slightly irregular and the agglomeration rate is 12%.

The oxygen content is slightly high (0.050.1%) and needs to be strictly controlled.

**application :**

YG6 test bar: reduced cobalt powder, particle size 1.5  $\mu\text{m}$  , flexural strength 2.0 GPa (ScienceDirect, 2020).

### 3.2 Carbonyl Process

**Principle :**

Cobalt reacts with carbon monoxide (CO) to form cobalt carbonyl  $[\text{Co}_2(\text{CO})_8]$ , which decomposes into high-purity cobalt powder and CO upon heating.

Reaction:  $2\text{Co} + 8\text{CO} \rightarrow \text{Co}_2(\text{CO})_8$  (gaseous, 100150°C),  $\text{Co}_2(\text{CO})_8 \rightarrow 2\text{Co} + 8\text{CO}$  (decomposition, 250300°C).

**Process :**

Raw materials: high purity cobalt ( >99.9%), CO gas (purity>99.99%).

Equipment: carbonyl reactor (pressure 0.51 MPa), decomposition furnace (vacuum or inert atmosphere).

Parameters: decomposition temperature 250350°C, air flow rate 0.51 L/min.

Classification: Screening or airflow classification, controlled particle size 0.52  $\mu\text{m}$  .

**Features :**

Purity: >99.95%, O <0.03%, C <0.01%.

Morphology: Nearly spherical, sphericity>0.95, agglomeration rate<0.5%.

Particle size: 0.52  $\mu\text{m}$  , D50 deviation  $\leq \pm 5\%$ .

**Advantages :**

High purity, impurities (Fe, S) <0.005%.

The morphology is regular and the fluidity is good (~18 s/50 g).

**Insufficient :**

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The equipment is complex, CO is highly toxic, and the cost is 20-30% higher.

**Application :**

YG8N test bar: carbonyl cobalt powder, particle size  $\sim 0.8 \mu\text{m}$ , hardness 2000 HV (Sandvik, 2023).

### 3.3 Atomization

**Principle :**

Molten cobalt (>99.9%) is atomized into fine particles by high-pressure gas ( $\text{N}_2$ , Ar) or water.

**Process :**

Raw material: high purity cobalt ingot (>99.9%).

Equipment: vacuum induction furnace ( $1500 \sim 1550^\circ\text{C}$ ), atomization tower (gas pressure 510 MPa).

Parameters: nozzle aperture 0.51 mm, cooling rate  $10^3 \sim 10^4 ^\circ\text{C/s}$ .

Classification: air flow classification, particle size  $15 \mu\text{m}$ .

**Features :**

Purity: >99.9%, O <0.08%, C <0.02%.

Morphology: spherical, sphericity >0.9.

Particle size:  $15 \mu\text{m}$ , D50 deviation  $< \pm 15\%$ .

**Advantages :**

Regular morphology and excellent fluidity ( $\sim 20 \text{ s/50 g}$ ).

Suitable for large particle cobalt powder ( $> 2 \mu\text{m}$ ).

**Insufficient :**

The particle size is too large ( $> 1 \mu\text{m}$ ), which is not suitable for ultrafine-grained alloys.

The oxygen content is high (0.050.1%).

**Application :**

YG15 test rod: atomized cobalt powder, particle size  $23 \mu\text{m}$ , KIC  $12 \text{ MPa} \cdot \text{m}^{1/2}$ .

### 3.4 Electrodeposition

**Principle :**

Cobalt powder is produced by electrolytic deposition of a cobalt salt solution (such as  $\text{CoSO}_4$ ).

Reaction:  $\text{Co}^{2+} + 2\text{e}^- \rightarrow \text{Co}$  (cathode, current density  $100 \sim 500 \text{ A/m}^2$ ).

**Process :**

Raw materials:  $\text{CoSO}_4$  (>99.9%), electrolyte (pH 35).

Equipment: electrolytic cell (stainless steel cathode), constant current power supply.

Parameters: temperature  $50 \sim 60^\circ\text{C}$ , current density  $200 \sim 400 \text{ A/m}^2$ , time 24 hours.

Post-treatment: washing, drying ( $80^\circ\text{C}$ ,  $< 10^{-2} \text{ Pa}$ ), grinding ( $< 2 \mu\text{m}$ ).

**Features :**

Purity: >99.9%, O <0.1%, Fe <0.02%.

Morphology: dendritic or irregular, needs grinding.

Particle size:  $110 \mu\text{m}$  ( $0.52 \mu\text{m}$  after grinding).

**Advantages :**

Low cost (30% lower than carbonyl process).

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Suitable for large-scale production.

**insufficient :**

The morphology is irregular and grinding increases agglomeration (23%).

The oxygen content is high (0.10.2%) and needs to be optimized.

**application :**

YG6 test bar: electrolytic cobalt powder, particle size 12  $\mu\text{m}$  , hardness 1500 HV.

### 3.5 Optimization measures

**Reduce oxygen levels :**

Inert atmosphere ( Ar , O<sub>2</sub> <0.005%) protection, oxygen reduced to <0.03%.

vacuum (<10<sup>-2</sup> Pa) to avoid oxidation.

**Control granularity :**

Ultrasonic dispersion (500 W, 10 min) reduced the agglomeration rate to <0.5%.

Airflow classification (accuracy  $\pm 0.1 \mu\text{m}$  ), D50 deviation  $\leq \pm 5\%$ .

**Improve appearance :**

Carbonyl method + plasma spheroidization, the sphericity increased to >0.95.

Chemical reduction method plus surface modification (stearic acid 0.1%), fluidity increased by 10%.

**Examples :**

YG8N: Carbonyl cobalt powder + ultrasonic dispersion, particle size 0.5  $\mu\text{m}$  , agglomeration <0.3% (Sandvik, 2023).

**Cobalt powder preparation method comparison table**

Method	Purity	Particle size ( $\mu\text{m}$ )	Morphology	Oxygen content	Flowability (s/50 g)	cost	Applicable grades
Chemical reduction method	>99.9%	0.53	Polyhedron, 0.80.9	<0.05%	~22	middle	YG6, YG15
Carbonyl method	>99.95%	0.52	Nearly spherical, >0.95	<0.03%	~18	high	Y8N
Atomization	>99.9%	15	Spherical, >0.9	<0.08%	~20	middle	YG15 (large size)
Electrolysis	>99.9%	0.52 (grinding)	Irregular, 0.70.8	<0.1%	~25	Low	YG6 (low cost)

**Optimization measures :**

**Reduce oxygen content :** Ar protection (O<sub>2</sub> <0.005%), vacuum drying (<10<sup>-2</sup> Pa).

**Particle size control :** ultrasonic dispersion (500 W, 10 min), agglomeration rate <0.5%.

**Morphology optimization :** carbonyl method + plasma spheroidization, sphericity>0.95.

## 4. Application of cobalt powder in cobalt-based cemented carbide

The selection and optimization of cobalt powder directly affects the preparation and performance of cobalt-based cemented carbide test bars. The following is a description of the specific grades and processes.

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#### 4.1 YG6 (6% Co, general purpose tool)

##### Cobalt powder requirements :

Purity: >99.95%, O <0.03%, Fe <0.005%.

Particle size: 11.5  $\mu\text{m}$  , D50  $\sim$ 1.2  $\mu\text{m}$  , sphericity>0.9.

Preparation: Carbonyl method or chemical reduction method, fluidity  $\sim$ 20 s/50 g.

##### Preparation process :

**Ingredients** : WC (94 wt % , 12  $\mu\text{m}$  ), Co (6 wt %), Cr<sub>3</sub>C<sub>2</sub> (0.3 wt %).

**Compounding** : wet milling (12-14 h, PEG 1.5%), D50 80-150  $\mu\text{m}$  .

**Pressing** : CIP (200250 MPa), billet 6.2 $\times$ 6.2 $\times$ 43 mm.

**Sintering** : vacuum sintering (1400°C, <10<sup>-3</sup> Pa ) + HIP (1400°C, 100 MPa).

**Machining** : grinding (Ra <0.4  $\mu\text{m}$  ), spark notching (0.25  $\pm$  0.02 mm).

##### performance :

Hardness: 1500  $\pm$  50 HV (GB/T 79972017).

Flexural strength: 2.0  $\pm$  0.1 GPa (GB/T 38512015).

Fracture toughness: 8  $\pm$  0.5 MPa $\cdot\text{m}^{1/2}$  .

##### Examples :

YG6 test bar: carbonyl cobalt powder, grain size  $\sim$ 1.2  $\mu\text{m}$  , cutting life 2 hours (Sandvik, 2023).

#### 4.2 YG15 (15% Co, high toughness mold)

##### Cobalt powder requirements :

Purity: >99.9%, O <0.05%, Fe <0.01%.

Particle size: 1.52  $\mu\text{m}$  , D50  $\sim$ 1.8  $\mu\text{m}$  , agglomeration <1%.

Preparation: Chemical reduction or atomization, flowability  $\sim$ 22 s/50 g.

##### Preparation process :

**Ingredients** : WC (85 wt % , 1.52.5  $\mu\text{m}$  ), Co (15 wt %), Cr<sub>3</sub>C<sub>2</sub> (0.5 wt %).

**Compounding** : wet grinding (14-16 h, PVA 1%), D50 100-200  $\mu\text{m}$  .

**Pressing** : CIP (250300 MPa), billet 5.0 $\times$ 10.0 $\times$ 40 mm.

**Sintering** : vacuum sintering (1450°C, <10<sup>-3</sup> Pa ) + HIP (1450°C, 120 MPa).

**Machining** : grinding (Ra <0.2  $\mu\text{m}$  ), spark notching (0.25  $\pm$  0.01 mm).

##### performance :

Hardness: 1400  $\pm$  50 HV.

Flexural strength: 2.5  $\pm$  0.1 GPa .

Fracture toughness: 12  $\pm$  0.5 MPa $\cdot\text{m}^{1/2}$  .

##### Examples :

YG15 test rod: reduced cobalt powder, grain size  $\sim$ 1.8  $\mu\text{m}$  , punching life 120,000 times (ScienceDirect, 2020).

#### 4.3 YG8N (8% Co, ultra- fine grain aviation tool)

##### Cobalt powder requirements :

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Purity: >99.95%, O <0.02%, Fe <0.005%.

Particle size: 0.20.8  $\mu\text{m}$  , D50 ~0.5  $\mu\text{m}$  , sphericity>0.95.

Preparation: Carbonyl method + plasma spheroidization, fluidity ~15 s/50 g.

**Preparation process :**

**Ingredients :** WC (91.5 wt % , 0.20.4  $\mu\text{m}$  ), Co (8 wt %), Cr<sub>3</sub>C<sub>2</sub> (0.4 wt %), VC (0.1 wt %).

**Mixing :** high energy ball milling (1820 h, modified PEG 1%), D50 30100  $\mu\text{m}$  .

**Pressing :** CIP (300350 MPa), billet 6.3×6.3×44 mm.

**Sintering :** vacuum sintering (1350°C,  $<5 \times 10^{-4}$  Pa) + HIP (1350°C, 150 MPa).

**Processing :** ultra-precision grinding (Ra <0.2  $\mu\text{m}$  ), femtosecond laser notch (0.25 ± 0.005 mm).

**performance :**

Hardness: 2000 ± 50 HV.

Flexural strength: 2.2 ± 0.1 GPa .

Fracture toughness: 9 ± 0.5 MPa·m<sup>1/2</sup> .

**Examples :**

YG8N test rod: carbonyl cobalt powder, grain size <0.5  $\mu\text{m}$  , aviation tool life 4 hours (Sandvik, 2023).

## 5. Key factors for cobalt powder selection

The selection of cobalt powder requires comprehensive consideration of alloy properties, process conditions and costs:

**High performance alloys** (such as YG8N):

The preferred choice is carbonyl cobalt powder, with a purity of >99.95%, a particle size of 0.20.8  $\mu\text{m}$  , and O <0.02%.

Reason: Fine grain size and low oxygen ensure grain size <0.5  $\mu\text{m}$  , hardness increased by 510%.

**High toughness alloy** (such as YG15):

Use chemical reduction or atomization method, particle size 1.52  $\mu\text{m}$  , O <0.05%.

Reason: Slightly larger particle size is suitable for high cobalt content, KIC increased by 10%.

**Cost sensitive** (such as YG6):

Use chemical reduction or electrolysis, particle size 12  $\mu\text{m}$  , O <0.05%.

Reason: 20-30% lower cost, meets general tool requirements (strength 2.0 GPa ).

**Large size test rod** (such as YG15):

Atomization method was used, particle size 23  $\mu\text{m}$  , flowability ~20 s/50 g.

Reason: Suitable for large particles, compression uniformity> 95%.

## 6. Conclusion

Used in cobalt-based cemented carbide must meet the following requirements:

**Chemical composition :** purity>99.9%, O <0.05%, Fe <0.01%, C <0.01% (GB/T 53142011).

**Physical properties :** particle size 0.52  $\mu\text{m}$  (conventional) or 0.20.8  $\mu\text{m}$  (ultrafine grain ), fluidity <25 s/50 g (GB/T 1482-2010).

**Micromorphology :** Nearly spherical, sphericity>0.9, agglomeration<1% (GB/T 183762014).

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#### Preparation method :

**Carbonyl method** : high purity (>99.95%), particle size 0.52  $\mu\text{m}$  , suitable for YG8N.

**Chemical reduction method** : low cost, particle size 0.53  $\mu\text{m}$  , suitable for YG6 and YG15.

**Atomization method** : large particles (15  $\mu\text{m}$  ), suitable for large-sized test rods.

**Electrolytic method** : low cost, requires grinding, suitable for general grades.

#### Application examples :

YG6: Carbonyl or reduced cobalt powder (11.5  $\mu\text{m}$  ), hardness 1500 HV, strength 2.0 GPa .

YG15: reduced or atomized cobalt powder (1.52  $\mu\text{m}$  ), KIC 12  $\text{MPa}\cdot\text{m}^{1/2}$  .

YG8N: Carbonyl + spheroidized cobalt powder (0.20.8  $\mu\text{m}$  ), hardness 2000 HV, grain size <0.5  $\mu\text{m}$  .

of cobalt powder (such as ultrasonic dispersion and inert protection) can improve uniformity by 20%, reduce  $\eta$  phase by 50%, and improve the consistency of alloy performance (deviation <3%). In the future, nano cobalt powder (<0.2  $\mu\text{m}$  ) and green preparation technology (such as low temperature reduction) will further improve the performance of cobalt-based cemented carbide .

#### standard :

GB/T 5314 2011: Chemical composition. GB/T 18376 2014: Microstructure.

GB/T 1482 2010: Liquidity. GB/T 3851 2015: Flexural strength.

GB/T 7997 2017: Hardness. GB/T 3849 2015: Magnetic properties.

#### Comparison table of cobalt powder and nickel powder

Characteristic	Cobalt powder	Nickel powder
Purity	>99.9%, >99.95% (ultra-fine grain)	>99.9%, >99.95% ( high corrosion resistance )
Granularity	0.52 $\mu\text{m}$ , 0.20.8 $\mu\text{m}$ (ultrafine grain)	0.52 $\mu\text{m}$ , 0.20.8 $\mu\text{m}$ (ultrafine grain)
Oxygen content	<0.05%, <0.03% (ultrafine grain)	<0.05%, <0.03% ( high corrosion resistance )
Morphology	Nearly spherical, sphericity>0.9	Nearly spherical, sphericity>0.9
Magnetization	~160 emu/g	~55 emu/g
Main preparation method	Carbonylation, chemical reduction, atomization, electrolysis	Carbonylation, chemical reduction, atomization, electrolysis
Cost	Higher ( 20% higher than nickel powder )	Lower
Alloy properties	Strength 1.52.5 GPa , KIC 812 $\text{MPa}\cdot\text{m}^{1/2}$	Strength 1.82.2 GPa , KIC 710 $\text{MPa}\cdot\text{m}^{1/2}$
Corrosion resistance	0.01 mm/year (5% HCl)	<0.005 mm/year (5% HCl)
Application scenario	Cutting tools, dies, mining tools	Chemical equipment, petroleum tools, high temperature molds

**Standard** : GB/T 43342020 (corrosion resistance), GB/T 38512015 (bending strength).

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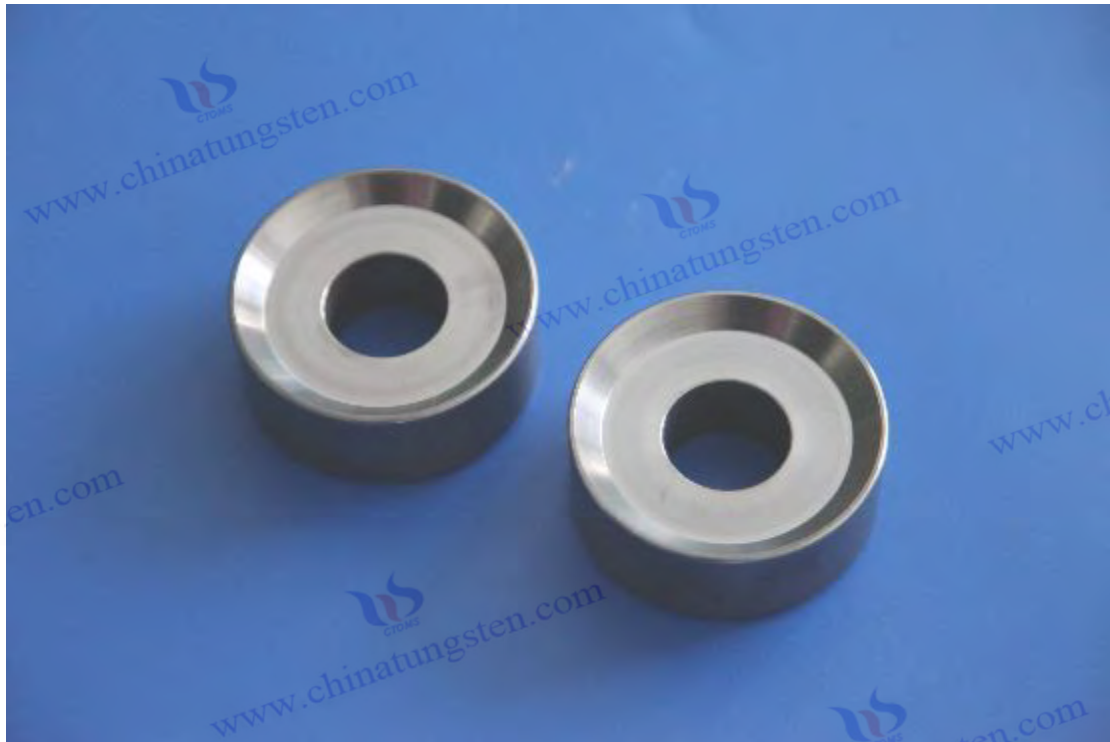
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#### Appendix:

### GB/T 7997-2017 Test method for Vickers hardness and microhardness of cemented carbide

GB/T 7997-2017 "Test Methods for Vickers Hardness and Microhardness of Cemented Carbide" is a Chinese national standard that specifies the test methods for Vickers hardness (HV) and microhardness (micro Vickers hardness, HVM) of cemented carbide. It is applicable to hardness testing of cemented carbide materials (such as WC-Co, WC-Ni, etc.).

#### 1 Scope

This standard specifies the test methods for Vickers hardness and microhardness of cemented carbide, including test principles, equipment, specimen requirements, test procedures, result calculation, test report, etc.

This standard is applicable to the hardness determination of sintered cemented carbide products (such as cutting tools, mining tools, wear-resistant parts) and unsintered cemented carbide blanks, and is applicable to the hardness range of HV 500 to HV 3000.

#### 2 Normative references

The following documents are essential reference documents for the implementation of this standard. For dated reference documents, only the version of that document is applicable; for undated reference documents, the latest version (including all amendments) is applicable.

GB/T 230.1 Rockwell hardness test for metallic materials Part 1: Test method (A, B, C, D, E, F, G, H, K, N, T scale)

GB/T 4340.1 Vickers hardness test for metallic materials Part 1: Test method

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GB/T 4340.2 Vickers hardness test for metallic materials Part 2: Verification and calibration of hardness testers

GB/T 4340.3 Vickers hardness test for metallic materials Part 3: Calibration of standard hardness blocks

GB/T 4505 Sampling and specimen preparation methods for cemented carbide

GB/T 5124 Chemical analysis methods for cemented carbide

GB/T 16594 General rules for microstructure inspection of metallic materials

### 3 Terms and definitions

Vickers hardness (HV): Using a Vickers hardness tester, a regular tetrahedral diamond indenter is pressed into the surface of the sample under a specified load, and the hardness value is calculated after measuring the diagonal length of the indentation. The unit is HV.

Microhardness (HVM): Vickers hardness measured under a microscope, suitable for hardness testing of small-sized specimens or local micro-areas, with a load usually less than 1 kgf.

Indentation diagonal: The length of the two diagonals of the Vickers indentation, in mm.

Hardness value: Hardness calculated based on the diagonal length of the indentation and the load, in kgf/mm<sup>2</sup> (converted to HV).

### 4 Test Principle

The Vickers hardness and microhardness tests use a regular quadrangular pyramid diamond indenter (vertex angle  $136^{\circ}\pm 0.5^{\circ}$ ) to press into the sample surface under a specified load, hold for a certain period of time, then unload, measure the diagonal length of the indentation, and calculate the hardness value using the formula.

The formula is as follows:

$$HV = 1.8544 \cdot \frac{F}{d^2}$$

in:

- HV: 维氏硬度值 (kgf/mm<sup>2</sup>, 单位为 HV) ;
- F: 试验载荷 (kgf) ;
- d: 压痕对角线的平均长度 (mm) .

### 5 Equipment and Materials

Hardness Tester:

Vickers hardness tester: in accordance with GB/T 4340.2, load range 1-50 kgf $\pm$ 0.1 kgf.

Microhardness tester: load range 0.01-1 kgf $\pm$ 0.001 kgf, equipped with microscope (magnification  $\geq$ 400 times).

Indenter: Regular tetrahedral diamond indenter, vertex angle  $136^{\circ}\pm 0.5^{\circ}$ , surface defect-free.

Standard hardness block: in accordance with GB/T 4340.3, hardness range HV 500 to HV 3000.

Sample surface treatment: Polished to surface roughness  $R_a \leq 0.2 \mu\text{m} \pm 0.02 \mu\text{m}$ , without oxide layer or cracks.

Environmental conditions: temperature  $20-25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ , humidity  $40\%-60\% \pm 5\%$  RH, no vibration interference.

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## 6. Samples

Sample requirements:

Sampling shall be carried out according to GB/T 4505, with a flat surface and a thickness  $\geq 1.5$  times the indentation depth (approximately 0.1-0.5 mm).

Specimen size: minimum area  $5\text{ mm} \times 5\text{ mm} \pm 0.1\text{ mm}$ , maximum size  $50\text{ mm} \times 50\text{ mm} \pm 0.1\text{ mm}$ .

Surface treatment:

Polishing: Use metallographic sandpaper (grit size 800-2000 mesh) and polishing paste (particle size  $\leq 1\text{ }\mu\text{m} \pm 0.01\text{ }\mu\text{m}$ ).

Cleaning: Use ethanol (purity  $\geq 99.5\% \pm 0.1\%$ ) to remove oil stains.

Number of specimens: 3-5 specimens per batch, with at least 5 indentations on the same specimen.

## 7 Test steps

### 7.1 Vickers hardness test

Equipment Calibration:

The hardness tester is calibrated using a standard hardness block with a deviation of  $< \pm 2\% \pm 0.5\%$ .

Calibrate the indenter geometry with a top angle deviation of  $< \pm 0.5^\circ$ .

Load selection:

Common loads: 5 kgf, 10 kgf, 30 kgf  $\pm 0.1\text{ kgf}$  (select according to hardness range).

Load holding time:  $10-15\text{ s} \pm 1\text{ s}$ .

Indentation measurement:

Place the sample on the hardness tester workbench and ensure that the sample surface is level.

Apply the load, maintain it for a specified time, and then unload it.

Measure the lengths of the two diagonals of the indentation ( $d_1$  and  $d_2$ ) with an accuracy of  $0.001\text{ mm} \pm 0.0001\text{ mm}$ .

Indentation spacing:  $\geq 3$  times the diagonal length (approximately 0.3-1.5 mm).

Hardness calculation:

Calculate the HV value according to the formula and take the average value of 5 indentations.

Deviation  $< \pm 3\% \pm 0.5\%$ , otherwise retest.

### 7.2 Microhardness test

Equipment Calibration:

Use a microhardness tester, calibrated load and microscope magnification ( $\geq 400\times$ ).

Deviation  $< \pm 2\% \pm 0.5\%$ .

Load selection:

Common loads: 0.05 kgf, 0.1 kgf, 0.5 kgf  $\pm 0.001\text{ kgf}$ .

Load holding time:  $10-15\text{ s} \pm 1\text{ s}$ .

Indentation measurement:

Observe the indentation under a microscope and measure the diagonal lengths ( $d_1$  and  $d_2$ ) to an accuracy of  $0.0005\text{ mm} \pm 0.0001\text{ mm}$ .

Indentation spacing:  $\geq 5$  times the diagonal length (approximately 0.05-0.2 mm).

Hardness calculation:

Calculate the HVM value according to the formula and take the average value of 5 indentations.

Deviation  $< \pm 4\% \pm 0.5\%$ , otherwise retest.

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## 8 Results Expression

Hardness value: expressed as HV or HVM, retain the integer, such as HV 1800 or HVM 2000.

Report Contents:

Specimen number, load value, diagonal length of indentation, average hardness value and deviation.

Environmental conditions (temperature, humidity).

Date of test and operator.

Example: WC-10%Co specimen, load 30 kgf, average diagonal  $0.042\text{ mm} \pm 0.001\text{ mm}$ , HV  $1800 \pm 50$ .

## 9 Influencing factors

Sample surface: Surface roughness  $R_a > 0.2\text{ }\mu\text{m} \pm 0.02\text{ }\mu\text{m}$  results in blurred indentation and low hardness (deviation  $> 5\% \pm 1\%$ ).

Load deviation: Load deviation  $> \pm 0.1\text{ kgf}$  affects the indentation depth, hardness deviation  $> 3\% \pm 0.5\%$ .

Environmental vibration: Vibration frequency  $> 1\text{ Hz} \pm 0.1\text{ Hz}$  will cause indentation deviation and vibration prevention is required.

Indenter status: Indenter defects (scratches  $> 0.01\text{ mm} \pm 0.001\text{ mm}$ ) result in low hardness and require regular inspection.

## 10 Inspection Rules

Sampling: According to GB/T 4505, 3-5 samples are taken from each batch ( $\leq 100\text{ kg}$ ) and 5 indentations are measured for each sample.

Inspection frequency: factory inspection (each batch), type inspection (once a year or when the process changes).

Judgment: All samples are qualified if their hardness values meet the requirements; if any one fails, re-inspection is allowed, and if the re-inspection still fails, the batch is unqualified.

## 11 Test Report

The test report should include:

Description of the sample (composition, preparation process).

Test method (Vickers or microhardness).

Load value, diagonal length of indentation, hardness value and deviation.

Environmental conditions (temperature  $20\text{-}25^\circ\text{C} \pm 1^\circ\text{C}$ , humidity  $40\%\text{-}60\% \pm 5\%\text{ RH}$ ).

Standard number (GB/T 7997-2017).

Test date and operator signature.

## Appendix A (Informative Appendix) Typical Hardness Values of Cemented Carbide

WC-6%Co: HV  $1800\text{-}2000 \pm 50$ , suitable for cutting tools.

WC-10%Co: HV  $1500\text{-}1700 \pm 50$ , suitable for mining tools.

WC-12%Ni: HV  $1400\text{-}1600 \pm 50$ , suitable for wear-resistant parts.

## Appendix B (Normative Appendix) Supplementary Notes on Test Methods

Indentation measurement: When measuring microhardness, the microscope magnification should

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be  $\geq 400$  times and the field of view should be clear.

Environmental control: The test environment has no airflow disturbance and the temperature fluctuation is  $< \pm 1^{\circ}\text{C}$ .

Indenter maintenance: Inspect the indenter after every 500 tests and replace it if the scratch is  $> 0.01 \text{ mm} \pm 0.001 \text{ mm}$ .

### Summarize

GB/T 7997-2017 standard specifies the test method for Vickers hardness and microhardness of cemented carbide. It uses a regular tetrahedral diamond indenter (vertex angle  $136^{\circ} \pm 0.5^{\circ}$ ) to calculate the hardness (HV 500-3000) by measuring the diagonal length of the indentation. The standard specifies equipment calibration (deviation  $< \pm 2\%$ ), sample preparation ( $R_a \leq 0.2 \mu\text{m}$ ), test steps and result expression to ensure the accuracy of hardness testing (deviation  $< \pm 3\%$ ). This method is suitable for quality control of cemented carbide products such as aviation tools (HV 1800-2000) and mining tools (HV 1500-1700).

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**Appendix:**

**GB/T 4340.1-2017 Vickers hardness test for metal materials**

**Part 1: Test methods**

**1 Scope**

This part specifies the test method for Vickers hardness test of metallic materials, including test principle, test equipment, specimen requirements, test procedure, hardness calculation, result expression and test report content.

This standard is applicable to the Vickers hardness determination of metallic materials and some non-metallic materials (such as cemented carbide and ceramics), with a hardness range of HV 5 to HV 3000 and a load range of 0.01 kgf to 50 kgf.

This standard is not applicable to specimens with excessively high surface roughness ( $R_a > 0.4 \mu\text{m}$   $\pm 0.02 \mu\text{m}$ ) or thickness insufficient to withstand the indentation depth.

**2 Normative references**

The following documents are essential reference documents for the implementation of this standard. Only the specified versions of the referenced documents are applicable.

GB/T 230.1 Rockwell hardness test for metallic materials Part 1: Test method (A, B, C, D, E, F, G, H, K, N, T scale)

GB/T 4340.2 Vickers hardness test for metallic materials Part 2: Verification and calibration of hardness testers

GB/T 4340.3 Vickers hardness test for metallic materials Part 3: Calibration of standard hardness blocks

GB/T 4505 Sampling and specimen preparation methods for cemented carbide

GB/T 16594 General rules for microstructure inspection of metallic materials

GB/T 8170 Rules for rounding off values

**3 Terms and definitions**

Vickers hardness (HV): Under a specified load, a regular quadrangular pyramid diamond indenter with a vertex angle of  $136^\circ$  is pressed into the surface of the sample, and the hardness value is calculated after measuring the diagonal length of the indentation. The unit is HV.

Micro Vickers hardness (HVM): Vickers hardness measured under a microscope when the load is less than 1 kgf.

Indentation diagonal: The length of the two diagonals of the Vickers indentation, in mm.

Test load: The force applied to the indenter, in kgf or N ( $1 \text{ kgf} = 9.80665 \text{ N}$ ).

Holding time: The time the load is held after it is applied, in seconds.

**4 Test Principle**

The Vickers hardness test uses a regular quadrangular pyramid diamond indenter with a vertex angle of  $136^\circ \pm 0.5^\circ$ . It is pressed into the sample surface under a specified load, held for a certain period of time, and then unloaded. The lengths of the two diagonals of the indentation ( $d_1$  and  $d_2$ ) are measured, and the average value  $d$  is calculated. The hardness value is calculated by substituting it into the formula:

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$$HV = 1.8544 \cdot \frac{F}{d^2}$$

其中:

- \* HV: 维氏硬度值 (kgf/mm<sup>2</sup>, 换算为 HV) ;
- \* F: 试验载荷 (kgf) ;
- \* d: 压痕对角线的算术平均值 (mm) ,  $d = \frac{d_1 + d_2}{2}$  .

若以 N 为单位, 则公式为:

$$HV = 0.1891 \cdot \frac{F}{d^2}$$

其中 F 单位为 N.

## 5 Test equipment

Vickers Hardness Tester:

Load range: 0.01 kgf to 50 kgf ± 0.1 kgf .

Load accuracy: in accordance with GB/T 4340.2, deviation <±1%±0.1%.

Measuring system: microscope or built-in optical system, accuracy 0.001 mm ± 0.0001 mm.

Pressure head:

Regular tetrahedral diamond indenter, vertex angle 136°±0.5°, edge straightness <0.002 mm±0.0002 mm.

There are no scratches or defects on the surface (scratches < 0.01 mm ± 0.001 mm).

Standard hardness block:

Conforms to GB/T 4340.3, hardness range HV 100 to HV 3000.

Environmental conditions:

Temperature: 20-25°C ± 1°C.

Humidity: 40%-60%±5% RH.

No vibration disturbance (frequency <1 Hz±0.1 Hz).

## 6. Samples

Size requirements:

Minimum thickness: ≥ 1.5 times the indentation depth (approximately 0.1-0.5 mm).

Minimum area: 10 mm × 10 mm ± 0.1 mm, maximum size 50 mm × 50 mm ± 0.1 mm.

Surface preparation:

Polishing: metallographic sandpaper (800-2000 mesh) and polishing paste (particle size ≤ 1 μm±0.01 μm), surface roughness Ra ≤ 0.2 μm±0.02 μm .

Cleaning: Use ethanol (purity ≥ 99.5% ± 0.1%) to remove oil stains.

quantity:

Take 3-5 specimens from each batch , and each specimen should have at least 5 indentations .

## 7 Test procedures

Equipment Calibration:

The hardness tester is calibrated using a standard hardness block with a deviation of <±2%±0.5%.

Check the geometry of the indenter, the top angle deviation is <±0.5°.

Load selection:

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Common loads: 0.1 kgf , 0.3 kgf , 0.5 kgf , 1 kgf , 5 kgf , 10 kgf , 30 kgf $\pm$ 0.1 kgf .

Recommended hard alloy: 10 kgf or 30 kgf  $\pm$  0.1 kgf .

Microhardness: 0.01-1 kgf  $\pm$  0.001 kgf .

Test conditions:

Holding time: 10-15 s $\pm$ 1 s (15 s $\pm$ 1 s is recommended for cemented carbide).

Indentation spacing:  $\geq$  3 times the diagonal length (approximately 0.3-1.5 mm).

Distance between indentation and specimen edge:  $\geq$  2.5 times the diagonal length.

Indentation measurement:

Microscope magnification:  $\geq$  400 times (microhardness),  $\geq$  100 times (conventional hardness).

Measure the diagonals  $d_1$  and  $d_2$  with an accuracy of 0.001 mm  $\pm$  0.0001 mm.

Hardness calculation:

Calculate HV according to the formula, take the average value of 5 indentations , and the deviation is  $<\pm 3\%\pm 0.5\%$ .

## 8 Influencing factors

Surface quality:  $R_a > 0.2 \mu\text{m}\pm 0.02 \mu\text{m}$  results in blurred indentation and low hardness (deviation  $>5\%\pm 1\%$ ).

Load deviation:  $>\pm 1\%\pm 0.1\%$  affects the indentation depth, hardness deviation  $>3\%\pm 0.5\%$ .

Ambient vibration: Frequency  $>1 \text{ Hz}\pm 0.1 \text{ Hz}$  causes indentation shift.

Specimen tilt: Tilt angle  $> 2^\circ\pm 0.1^\circ$  will cause asymmetric indentation and need to be readjusted.

## 9 Results Expression

Hardness value: expressed in HV, retain the integer, for example HV 1800 $\pm$ 50.

Symbol: HV is added after the load, for example, a load of 30 kgf is HV30.

Report Contents:

Specimen number, load value, diagonal length of indentation, hardness value and deviation.

Environmental conditions (temperature, humidity).

Date of test and operator.

## 10 Test Report

Description of the specimen (material, preparation process).

Test conditions (load, holding time, indentation spacing).

Mean and deviation of hardness values.

Environmental conditions (temperature 20-25 $^\circ\text{C} \pm 1^\circ\text{C}$ , humidity 40%-60%  $\pm 5\%$  RH).

Standard number (GB/T 4340.1-2017).

Test date and operator signature.

## Appendix A (Informative Appendix) Vickers hardness values of common metal materials

Cemented carbide (WC-10%Co): HV 1500-1800 $\pm$ 50.

Hardened steel (HRC 60): HV 700-800 $\pm$ 30.

Pure aluminum: HV 20-50 $\pm$ 5.

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## GB/T 4340.2-2017 Vickers hardness test for metal materials

### Part 2: Verification and calibration of hardness testers

#### 1 Scope

This part specifies the inspection and calibration methods of Vickers hardness testers, including the verification of load accuracy, indenter geometry, measurement system accuracy and repeatability. It is applicable to factory inspection, regular calibration and pre-use inspection of Vickers hardness testers (conventional and microscopic).

#### 2 Normative references

GB/T 4340.1 Vickers hardness test for metallic materials Part 1: Test method

GB/T 4340.3 Vickers hardness test for metallic materials Part 3: Calibration of standard hardness blocks

JJG 112-2005 Verification Procedure for Vickers Hardness Tester

GB/T 8170 Rules for rounding off values

#### 3 Terms and definitions

Load error: The deviation between the actual load and the marked load, expressed in %.

Measuring system error: The deviation between the diagonal measurement value and the actual value, in  $\mu\text{m}$ .

Repeatability: The consistency of hardness values measured multiple times under the same conditions, in HV.

Calibration cycle: The time interval between hardness tester calibration, usually 6 months to 1 year.

#### 4 Inspection items

Load accuracy: deviation  $< \pm 1\% \pm 0.1\%$ .

Indenter Geometry:

Vertex angle:  $136^\circ \pm 0.5^\circ$ .

Edge straightness :  $< 0.002 \text{ mm} \pm 0.0002 \text{ mm}$ .

Vertex deviation:  $< 0.001 \text{ mm} \pm 0.0001 \text{ mm}$ .

Measuring system:

Diagonal measurement error:  $< \pm 0.2 \mu\text{m} \pm 0.02 \mu\text{m}$  (microhardness tester).

Microscope magnification error:  $< \pm 1\% \pm 0.1\%$ .

Hold time: Deviation  $< \pm 0.5 \text{ s} \pm 0.1 \text{ s}$ .

Repeatability: Standard deviation of 5 measurements  $< \pm 1\% \pm 0.1\%$ .

#### 5. Calibration equipment

Standard hardness block:

Conforms to GB/T 4340.3, hardness range HV 100-3000.

Uniformity:  $< \pm 3\% \pm 0.5\%$ .

Micrometer: accuracy  $0.001 \text{ mm} \pm 0.0001 \text{ mm}$ .

Load calibration device: accuracy  $0.01 \text{ kgf} \pm 0.001 \text{ kgf}$ .

Optical microscope: magnification  $\geq 1000$  times , accuracy  $0.0001 \text{ mm} \pm 0.00001 \text{ mm}$ .

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Environmental conditions:  
Temperature:  $20-25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ .  
Humidity:  $40\%-60\% \pm 5\% \text{ RH}$ .

## 6 Calibration Procedure

Load Calibration:

Calibrate each load point (0.1 kgf, 1 kgf, 5 kgf, 10 kgf, 30 kgf  $\pm 0.1$  kgf) using a standard weight or force sensor.

Record the deviation, if  $< \pm 1\% \pm 0.1\%$ , otherwise adjust the load system.

Indenter inspection:

An optical microscope was used to measure the indenter top angle ( $136^{\circ} \pm 0.5^{\circ}$ ) and edge straightness.

Check the surface of the indenter and replace it if there are scratches  $> 0.01 \text{ mm} \pm 0.001 \text{ mm}$ .

Measuring system calibration:

Using a standard hardness block (e.g. HV  $1800 \pm 10$ ), measure the diagonal of the indentation.

Calibrate the microscope to an accuracy of  $< \pm 0.2 \mu\text{m} \pm 0.02 \mu\text{m}$ .

Keep time calibration:

Calibrated using a stopwatch,  $10-15 \text{ s} \pm 1 \text{ s}$ , deviation  $< \pm 0.5 \text{ s} \pm 0.1 \text{ s}$ .

Repeatability test:

Measure 5 times continuously on the standard hardness block and calculate the standard deviation,  $< \pm 1\% \pm 0.1\%$ .

Adjustment and Recording:

If unsatisfactory, adjust the hardness tester (load, indenter or measuring system).

Record calibration data and keep them on file for  $2 \text{ years} \pm 0.1 \text{ year}$ .

## 7 Influencing factors

Temperature variation:  $> \pm 1^{\circ}\text{C}$  affects the stability of the load system, hardness deviation  $> 2\% \pm 0.5\%$ .

Indenter wear: scratches  $> 0.01 \text{ mm} \pm 0.001 \text{ mm}$  resulting in low hardness ( $> 3\% \pm 0.5\%$ ).

Load fluctuation: Unstable power supply ( $> \pm 1 \text{ V} \pm 0.1 \text{ V}$ ) causes load error.

## 8 Results Expression

Load error: expressed in %, with two decimal places retained, for example  $\pm 0.50\% \pm 0.01\%$ .

Measurement error: expressed in  $\mu\text{m}$ , for example  $\pm 0.10 \mu\text{m} \pm 0.01 \mu\text{m}$ .

Repeatability: Expressed in HV, for example  $\pm 10 \text{ HV} \pm 1 \text{ HV}$ .

## 9 Calibration Report

Hardness tester model and number.

Calibration items (load, indenter, measurement system, repeatability).

Calibration results and deviations.

Environmental conditions (temperature  $20-25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ , humidity  $40\%-60\% \pm 5\% \text{ RH}$ ).

Standard number (GB/T 4340.2-2017).

Calibration date and operator signature.

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## Appendix A (Informative Appendix) Calibration cycle recommendations

Normal use: calibrate every 6 months .

High frequency use (>100 times/day): calibrate every 3 months .

## CTIA GROUP LTD

### 30 Years of Cemented Carbide Customization Experts

#### Core Advantages

**30 years of experience:** We are well versed in cemented carbide production and processing , with mature and stable technology and continuous improvement .

**Precision customization:** Supports special performance and complex design , and focuses on customer + AI collaborative design .

**Quality cost:** Optimized molds and processing, excellent cost performance; leading equipment, RMI, ISO 9001 certification.

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## GB/T 4340.3-2017 Vickers hardness test for metal materials

### Part 3: Calibration of standard hardness blocks

#### 1 Scope

This part specifies the calibration method of Vickers hardness standard hardness blocks, including hardness value determination, uniformity inspection, uncertainty evaluation and calibration certificate content.

Applicable to standard hardness blocks for Vickers hardness tester calibration, with a hardness range of HV 100 to HV 3000.

#### 2 Normative references

GB/T 4340.1 Vickers hardness test for metallic materials Part 1: Test method

GB/T 4340.2 Vickers hardness test for metallic materials Part 2: Verification and calibration of hardness testers

JJF 1071-2010 National Measurement and Calibration Specification

GB/T 8170 Rules for rounding off values

GB/T 16594 General rules for microstructure inspection of metallic materials

#### 3 Terms and definitions

Standard hardness block: A metal block with a known hardness value used to calibrate a hardness tester.

Hardness uniformity: The consistency of hardness values on the surface of a hardness block, measured in HV.

Uncertainty: The confidence interval of the hardness value measurement result, in HV.

Calibration cycle: The time interval for recalibrating the standard hardness block, usually 1-2 years.

#### 4 Calibration requirements

Hardness value: HV 100-3000, deviation  $\leq \pm 2\% \pm 0.5\%$ .

Uniformity: The hardness value deviation of 10 points on the same surface is  $\leq \pm 3\% \pm 0.5\%$ .

Uncertainty:  $\leq \pm 10 \text{ HV} \pm 1 \text{ HV}$  (95% confidence level).

Surface quality:  $R_a \leq 0.2 \mu\text{m} \pm 0.02 \mu\text{m}$ , no scratches or oxide layer.

#### 5. Calibration equipment

Vickers Hardness Tester:

Conforms to GB/T 4340.2 and is calibrated to pass.

Load accuracy:  $\leq \pm 1\% \pm 0.1\%$ .

Pressure head:

Vertex angle  $136^\circ \pm 0.5^\circ$ , certified.

There is no surface defect (scratch  $< 0.01 \text{ mm} \pm 0.001 \text{ mm}$ ).

Measuring system:

Microscope accuracy:  $0.001 \text{ mm} \pm 0.0001 \text{ mm}$ .

Environmental conditions:

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Temperature:  $20-25^{\circ}\text{C} \pm 1^{\circ}\text{C}$ .

Humidity:  $40\%-60\% \pm 5\% \text{ RH}$ .

## 6 Calibration Procedure

Sample preparation:

The surface of the hardness block was polished to  $Ra \leq 0.2 \mu\text{m} \pm 0.02 \mu\text{m}$ , cleaned with ethanol and dried.

Hardness measurement:

Select load: 5 kgf, 10 kgf, 30 kgf  $\pm 0.1 \text{ kgf}$  (according to hardness range).

Measure 10 points, with the indentation spacing  $\geq 3$  times the diagonal length, and hold time 10-15 s  $\pm 1 \text{ s}$ .

Uniformity check:

Calculate the average and standard deviation of the hardness values at 10 points, with a deviation of  $< \pm 3\% \pm 0.5\%$ .

Uncertainty Assessment:

Including hardness tester error, measurement system error and environmental influence.

Uncertainty  $\leq \pm 10 \text{ HV} \pm 1 \text{ HV}$ .

verify:

Compared with the results of national metrology institutions or reference laboratories, the deviation is  $< \pm 2\% \pm 0.5\%$ .

## 7 Influencing factors

Surface quality:  $Ra > 0.2 \mu\text{m} \pm 0.02 \mu\text{m}$  resulting in low hardness ( $> 3\% \pm 0.5\%$ ).

Ambient temperature:  $> \pm 1^{\circ}\text{C}$  affects the indentation measurement accuracy.

Hardness tester status: load error  $> \pm 1\% \pm 0.1\%$  or indenter defect affects the result.

## 8 Results Expression

Hardness value: expressed in HV, retain the integer, for example HV 1800  $\pm 10$ .

Uniformity: expressed as maximum deviation, e.g.  $\pm 20 \text{ HV} \pm 1 \text{ HV}$ .

Uncertainty: Expressed in HV, for example  $\pm 8 \text{ HV} \pm 1 \text{ HV}$ .

## 9 Calibration certificate

Hardness block number, hardness value and uncertainty.

Uniformity data and measurement point distribution.

Calibration conditions (load, environment).

Standard number (GB/T 4340.3-2017).

Calibration date, expiration date and operator signature.

## Appendix A (Informative Appendix) Typical values of standard hardness blocks

HV 200  $\pm 5$ : Mild steel.

HV 800  $\pm 10$ : Hardened steel.

HV 1800  $\pm 20$ : Hard alloy.

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## Appendix B (Normative Appendix) Calibration cycle and maintenance

Calibration cycle: Once a year or after 500 uses.

Maintenance: Avoid scratching the surface of the hardness block and store it in a dry environment (humidity  $<50\% \pm 5\%$  RH).

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## Appendix:

### Nickel-based carbide deep-sea seals and deep-sea valves

Nickel-based cemented carbide ( WC+Ni ) has become the core material for deep-sea seals, seals and deep-sea valves due to its excellent corrosion resistance, wear resistance, high strength (2.0-2.8 GPa ) and high temperature stability (>1000°C). It is widely used in deep-sea oil and gas production, deep-sea valve systems and high-pressure equipment (such as 15,000-20,000 psi). These components need to withstand extreme environments (50°C to 200°C, H<sub>2</sub>S, CO<sub>2</sub>, seawater corrosion), comply with NACE MR0175 standards, and meet the requirements of porosity (<0.01%), hardness (1400-2200 HV) and sealing performance (leakage rate <10<sup>-6</sup>). This article combines national standards (such as GB/T 183762014, GB/T 38502015) and industry practices to introduce in detail the sintering process, performance, application and selection recommendations of nickel - based cemented carbide deep-sea seals and deep-sea valves.

### 1. Nickel-based cemented carbide sintering furnace process

Nickel-based cemented carbide deep-sea seals and valves need to be prepared by high-temperature sintering. The sintering furnace types include vacuum sintering furnace, hot isostatic pressing (HIP) sintering furnace and atmosphere sintering furnace. The process parameters are optimized for deep-sea environment.

#### 1.1 Sintering furnace types and process parameters

##### Vacuum sintering furnace:

Application: Production of complex shaped sealing rings (e.g. Ø 5200 mm) and valve components (e.g. valve seats Ø 50500 mm).

Process parameters:

200600 °C, heating rate 25°C/min, vacuum degree 10<sup>-2</sup> Pa, H<sub>2</sub> flow rate 515 L/min, 24 hours, dewaxing rate >99.5%.

Sintering: 1350/1450°C, heating rate 510°C/min, vacuum degree 10<sup>-4</sup> 10<sup>-5</sup> Pa, keep warm for 24 hours.

Cooling: 1015°C/min ( Ar forced cooling), to 100°C.

Properties: density 14.514.9 g/cm<sup>3</sup> , hardness 1400/2000 HV, porosity <0.01%.

##### Hot isostatic pressing furnace (HIP):

Application: To produce high-performance seals and valves (such as high-pressure valve cores) and eliminate micropores.

Process parameters:

Sintering: 1350/1450°C, heating rate 58°C/min, pressure 100/150 MPa ( Ar ), keep warm for 13 hours.

Post-treatment: 1300/1350°C, 80/100 MPa, 12 hours, porosity reduced to <0.001%.

Cooling: 1520°C/min (high pressure Ar ) to 200°C.

Properties: density>99.9% (14.815.0 g/cm<sup>3</sup> ) , hardness 1800/2200 HV, strength 2.2/2.8 GPa .

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### Atmosphere sintering furnace:

Application: Mass production of seals (such as O-rings) and valve components (such as valve bodies).

Process parameters:

Dewaxing: 200-500°C, heating rate 35°C/min, H<sub>2</sub> flow 2050 L/min, O<sub>2</sub> <10 ppm, 35 hours.

Sintering: 1300-1400°C, heating rate 510°C/min, H<sub>2</sub>/ Ar atmosphere, keep warm for 35 hours.

Cooling: 510°C/min (N<sub>2</sub> protection), to 100°C.

Properties: density >99% (14.514.8 g/cm<sup>3</sup>), hardness 1400-1800 HV, dimensional deviation ±0.10.5 mm.

## 1.2 Process Optimization

Temperature control: PID+AI algorithm, accuracy ±3°C, uniformity ±5°C, reducing thermal stress by 30%.

Dewaxing: vacuum + H<sub>2</sub> combination, residual carbon <0.05%, to prevent Ni phase oxidation.

HIP: 1350°C, 120 MPa, 2 hours of holding, density increase by 0.5%, cycle shortened by 20%.

Atmosphere: H<sub>2</sub> purity >99.999%, O<sub>2</sub> <5 ppm, oxidation rate reduced by 50%.

## 2. Performance of nickel-based cemented carbide deep-sea seals

Compared with cobalt-based cemented carbide (WC+ Co), nickel-based cemented carbide (WC+ Ni, Ni content 615%) has stronger resistance to seawater corrosion and H<sub>2</sub>S/CO<sub>2</sub>, and meets the needs of extreme deep-sea environments.

### 2.1 Material properties

Composition: WC (8594%), Ni (615%), trace Cr/Mo (enhanced corrosion resistance).

Density: 14.515.0 g/cm<sup>3</sup> (GB/T 38502015), deviation ±0.05 g/cm<sup>3</sup>.

Hardness: 1400-2200 HV (GB/T 79972017), deviation ±3050 HV.

Strength: flexural strength 2.02.8 GPa (GB/T 38512015).

Porosity: <0.01% (vacuum/atmosphere), <0.001% (HIP, GB/T 51692013).

Corrosion resistance: Resistant to H<sub>2</sub>S (>1000 ppm), CO<sub>2</sub>, seawater (pH 39), in compliance with NACE MR0175.

### 2.2 Sealing performance

Leakage rate: <10<sup>-6</sup> mbar·L/s (helium test, 15,000 psi).

Pressure Range: Vacuum to 20,000 psi (138 MPa).

Temperature range: 50°C to 200°C (typical deep sea conditions).

Surface treatment: Ni/Cr electroplating or PTFE coating, friction coefficient reduced by 20%, wear resistance increased by 30%.

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Dynamic seal: supports slight dynamic movement (connection/disconnection), life span >1000 connections.

## 2.3 Typical

O-ring: Ø 5200 mm, compression rate 1520%, self-reinforcing seal.

C-type seal: high spring rebound, suitable for high pressure dynamic sealing.

Valve sealing surface: valve seat/valve core, surface roughness Ra 0.20.4 µm , erosion resistant.

Recommendation: The nickel-based cemented carbide deep-sea seals and sealing rings produced by CTIA GROUP LTD adopt advanced HIP sintering technology to ensure high density (>99.9%) and excellent corrosion resistance to meet the needs of deep-sea oil and gas exploitation.

## 3. Performance of Nickel-based Cemented Carbide Deep Sea Valve

Deep-sea valves (such as gate valves and ball valves) use nickel-based cemented carbide to manufacture valve seats, valve cores and sealing surfaces to cope with high pressure (15,000-20,000 psi), corrosive fluids (seawater, H2S) and frequent switching (>10,000 times).

### 3.1 Valve component characteristics

Material: WC+Ni (Ni 812%), HIP sintering, density>99.9%.

Hardness: 18002200 HV, wear resistance is better than Stellite alloy.

Strength: 2.22.8 GPa , strong impact resistance, fracture toughness KIC 1012 MPa·m<sup>1/2</sup>.

Corrosion resistance: Resistant to seawater, H2S (>1000 ppm), and CO2, better than Inconel 625.

Surface: mirror polished (Ra <0.2 µm ), PTFE/Ni coating, friction coefficient <0.1.

### 3.2 Valve performance

Pressure: 15,000-20,000 psi, in accordance with API 6A standards.

Temperature: 50°C to 200°C, resistant to low temperature embrittlement.

Leakage rate: <10<sup>-6</sup> mbar·L /s (seat/core seal).

Lifespan: Switching>10,000 times, maintenance period>5 years.

typical:

Valve seat: Nickel-based hard alloy surfacing, thickness 25 mm, erosion resistant.

Valve core: Integral HIP sintering, dimensional accuracy ±0.01 mm.

Sealing surface: C-type or E-type metal seal, elastic deformation 1520%.

Recommendation: Nickel-based cemented carbide deep-sea valve components produced by CTIA GROUP LTD adopt vacuum sintering and HIP process, meet API 6A and NACE MR0175 standards, and are suitable for deep-sea high-pressure valve systems.

## 4. Application Scenarios

### Deep Sea Seals:

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Application: Hydraulic couplers and pipe connectors for deep-sea oil and gas production (15,000 psi, 50°C to 150°C).

Performance: Density 14.815.0 g/cm<sup>3</sup>, hardness 18002200 HV, leakage rate <10<sup>-6</sup> mbar·L /s.

Example: O-ring ( Ø 50 mm), HIP sintering, 1400°C, 120 MPa, 4 hours, porosity <0.001%, H<sub>2</sub>S resistance 1000 ppm, life >1000 connections.

#### Deep sea valves:

Scenario: Deep sea Christmas trees, choke valves, gate valves (20,000 psi, H<sub>2</sub>S/CO<sub>2</sub> environment).

Performance: Seat hardness 2000 HV, strength 2.5 GPa, switching life >10,000 times.

Case: Ball valve seat ( Ø 100 mm), HIP sintering, 1350°C, 150 MPa, 3 hours, density 14.9 g/cm<sup>3</sup>, seawater corrosion resistance, maintenance period 5 years.

### 5. Recommendations for the selection of nickel-based cemented carbide deep-sea valve components

According to the application environment:

Deep sea hydraulic coupler (high dynamic seal):

Recommended: HIP sintered C-ring, Ni content 1012%, PTFE coating.

Reason: High spring rebound, leakage rate <10<sup>-6</sup> mbar·L /s, lifespan>1000 times.

Deep sea valves (high pressure, corrosion resistant):

Recommended: HIP sintered valve seat/valve core, Ni content 812%, mirror polished.

Reason: Density > 99.9%, H<sub>2</sub>S/CO<sub>2</sub> resistant, switching > 10,000 times.

According to performance requirements:

High precision (±0.01 mm): vacuum sintering + HIP, shrinkage deviation <±0.5%.

High corrosion resistance: Ni content 1015%, trace addition of Cr/Mo, resistant to seawater for 10 years.

Low leakage: HIP sintering, surface Ra <0.2 μm, Ni/PTFE coating.

According to the cost budget:

Low cost: atmosphere sintering furnace, 0.8 kWh/kg, suitable for large quantities of seals.

High performance: HIP sintering furnace, 2 kWh/kg, suitable for valve-critical components.

Sintering furnace selection:

Small and medium batches (<50 kg/furnace): Single-chamber vacuum sintering furnace with high flexibility.

Large batches (>200 kg/furnace): Multi-chamber vacuum/atmosphere sintering furnace, cost reduction of 20%.

High performance: HIP sintering furnace, density >99.9%, porosity <0.001%.

### 6. Optimization suggestions

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Sintering process:

Temperature control:  $\pm 3^{\circ}\text{C}$ , uniformity  $\pm 5^{\circ}\text{C}$ , product consistency increased by 10%.

Dewaxing: vacuum ( $10^{-2}$  Pa) +  $\text{H}_2$  (10 L/min), residual carbon  $<0.05\%$ .

HIP:  $1350^{\circ}\text{C}$ , 120 MPa, 2 hours, strength increase by 15%.

Material:

Ni content: 1012%, balance of hardness and corrosion resistance.

Coating: Ni/PTFE, friction coefficient reduced by 20%, wear resistance increased by 30%.

Trace elements: Cr/Mo 0.52%,  $\text{H}_2\text{S}$  resistance increased by 25%.

Equipment maintenance:

Online monitoring: real-time monitoring of temperature, pressure and  $\text{O}_2$ , reducing the failure rate by 20%.

Component inspection: Molybdenum /tungsten heating elements should be maintained every 4,000 hours, and their lifespan will be increased by 25%.

## 7. Standards

GB/T 345052017: Dimensional accuracy  $\pm 0.01$  mm.

GB/T 183762014: Porosity  $<0.01\%$ .

GB/T 38502015: Density  $>99\%$ .

GB/T 51692013: Porosity A02B00C00.

GB/T 38512015: Strength 2.0-2.8 GPa .

GB/T 7997-2017: Hardness 1400-2200 HV.

NACE MR0175 : Resistant to  $\text{H}_2\text{S}/\text{CO}_2$  corrosion.

API 6A: Deep sea valve pressure 15,000-20,000 psi.

## 8. Conclusion

Nickel-based cemented carbide deep-sea seals and valves are widely used due to their high density ( $>99.9\%$ ), hardness (1400-2200 HV), corrosion resistance (resistance to  $\text{H}_2\text{S}/\text{CO}_2$ /seawater) and low leakage rate ( $<10^{-6}$  mbar·L /s), meeting the extreme requirements of deep-sea oil and gas extraction. Vacuum sintering furnaces, HIP sintering furnaces and atmosphere sintering furnaces are suitable for high-precision, high-performance and mass production respectively. Process parameter optimization (such as  $1350^{\circ}\text{C}$ , 120 MPa,  $\text{H}_2$   $\text{O}_2$   $<5$  ppm) significantly improves product performance. CTIA GROUP LTD uses advanced sintering technology in the production of nickel-based cemented carbide deep-sea seals, sealing rings and valves, providing highly reliable solutions to assist deep-sea engineering.

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## Appendix:

### Nickel-based carbide chemical pump body and seals

Nickel-based cemented carbide (WC+Ni) is an ideal material for chemical pump bodies, seals and seals due to its excellent corrosion resistance, wear resistance, high strength (2.0-2.8 GPa) and high temperature stability (>1000°C). It is widely used in the chemical, petroleum and pharmaceutical industries to cope with corrosive fluids (such as acids, alkalis, and salt solutions), high temperatures (50-300°C) and high pressures (500-5000 psi). These components must meet strict performance requirements, including hardness (1400-2200 HV), porosity (<0.01%), sealing performance (leakage rate <10<sup>-6</sup> mbar·L/s) and corrosion resistance (in compliance with NACE MR0175). This article combines national standards (such as GB/T 183762014, GB/T 38502015) and industry practices to introduce in detail the sintering process, performance, application and selection recommendations of nickel-based cemented carbide chemical pump bodies and seals.

## 1. Nickel-based cemented carbide sintering furnace process

Nickel-based cemented carbide chemical pump bodies and seals are prepared by high-temperature sintering. The sintering furnaces include vacuum sintering furnaces, hot isostatic pressing (HIP) sintering furnaces and atmosphere sintering furnaces. The process parameters are optimized for chemical corrosive environments.

### 1.1 Sintering furnace types and process parameters

#### Vacuum sintering furnace:

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Application: Production of precision seals (e.g. Ø 5150 mm) and pump parts (e.g. impeller Ø 50300 mm).

Process parameters:

200600 °C, heating rate 25°C/min, vacuum degree  $10^{-2}$  Pa, H<sub>2</sub> flow rate 515 L/min, 24 hours, dewaxing rate >99.5%.

Sintering: 1350/1450°C, heating rate 510°C/min, vacuum degree  $10^{-4}$   $10^{-5}$  Pa, keep warm for 24 hours.

Cooling: 1015°C/min (Ar forced cooling), to 100°C.

Properties: density 14.514.9 g/cm<sup>3</sup>, hardness 14002000 HV, porosity <0.01%.

### Hot isostatic pressing furnace (HIP):

Application: Production of high-performance seals and pump bodies (e.g. pump housing Ø 100-500 mm), elimination of micropores.

Process parameters:

Sintering: 1350/1450°C, heating rate 58°C/min, pressure 100/150 MPa (Ar), keep warm for 13 hours.

Post-treatment: 1300-1350°C, 80-100 MPa, 12 hours, porosity reduced to <0.001%.

Cooling: 1520°C/min (high pressure Ar) to 200°C.

Properties: density>99.9% (14.815.0 g/cm<sup>3</sup>), hardness 18002200 HV, strength 2.22.8 GPa.

### Atmosphere sintering furnace:

Application: Mass production of seals (such as mechanical seals) and pump parts (such as bushings).

Process parameters:

Dewaxing: 200500°C, heating rate 35°C/min, H<sub>2</sub> flow 2050 L/min, O<sub>2</sub> <10 ppm, 35 hours.

Sintering: 1300/1400°C, heating rate 510°C/min, H<sub>2</sub>/Ar atmosphere, keep warm for 35 hours.

Cooling: 510°C/min (N<sub>2</sub> protection), to 100°C.

Properties: density>99% (14.514.8 g/cm<sup>3</sup>), hardness 14001800 HV, dimensional deviation ±0.10.5 mm.

## 1.2 Process Optimization

Temperature control: PID+AI algorithm, accuracy ±3°C, uniformity ±5°C, thermal stress reduction by 30%.

Dewaxing: vacuum + H<sub>2</sub> combination, residual carbon <0.05%, to prevent Ni phase oxidation.

HIP: 1350°C, 120 MPa, 2 hours of holding, density increase by 0.5%, cycle shortened by 20%.

Atmosphere: H<sub>2</sub> purity >99.999%, O<sub>2</sub> <5 ppm, oxidation rate reduced by 50%.

## 2. Performance of Nickel-based Cemented Carbide Chemical Pump Body

Chemical pump bodies (such as centrifugal pump casings, impellers, and bushings) are made of nickel-based cemented carbide to cope with corrosive fluids (such as sulfuric acid and hydrochloric acid) and abrasive particles (solid content <20%).

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## 2.1 Material properties

Composition: WC (85.94%), Ni (6.15%), Cr/Mo (0.52%) enhances corrosion resistance.

Density: 14.515.0 g/cm<sup>3</sup> (GB/T 38502015), deviation  $\pm 0.05$  g/cm<sup>3</sup>.

Hardness: 14002200 HV (GB/T 79972017), deviation  $\pm 3050$  HV.

Strength: flexural strength 2.02.8 GPa (GB/T 38512015).

Porosity: <0.01% (vacuum/atmosphere), <0.001% (HIP, GB/T 51692013).

Corrosion resistance: resistant to sulfuric acid (50%), hydrochloric acid (30%), NaOH (40%), in compliance with NACE MR0175.

## 2.2 Pump performance

Pressure: 5005000 psi, suitable for medium and high pressure chemical pumps.

Temperature: 50300°C, resistant to high temperature fluids.

Wear resistance: wear loss <0.05 mm<sup>3</sup> / h (ASTM G65), better than 316L stainless steel.

Surface treatment: Ni/Cr electroplating or DLC coating, friction coefficient reduced by 20%, wear resistance increased by 30%.

Lifespan: Continuous operation >10,000 hours, maintenance cycle >2 years.

## 2.3 Typical

Pump casing: Ø 100500 mm, wall thickness 520 mm, HIP sintered, high pressure resistant.

Impeller: Ø 50300 mm, complex curved surface, accuracy  $\pm 0.05$  mm.

Bushing: Ø 20100 mm, surface Ra <0.4 µm, wear resistant.

Recommendation: The nickel-based cemented carbide chemical pump body produced by CTIA GROUP LTD adopts HIP sintering process to ensure high density (>99.9%) and excellent corrosion resistance to meet the stringent requirements of the chemical industry.

## 3. Performance of Nickel-based Carbide Seals

Seals (such as mechanical sealing rings and O-rings) are used for dynamic and static sealing of chemical pumps to prevent leakage of corrosive fluids.

### 3.1 Material properties

Material: WC+Ni (Ni 8.12%), HIP sintering, density>99.9%.

Hardness: 18002200 HV, wear resistance is better than SiC.

Strength: 2.22.8 GPa, fracture toughness KIC 1012 MPa·m<sup>1/2</sup>.

Corrosion resistance: better than silicon carbide in terms of acid, alkali and salt solution resistance.

Surface: mirror polished (Ra <0.2 µm), PTFE/DLC coating, friction coefficient <0.1.

### 3.2 Sealing performance

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Leakage rate:  $<10^{-6}$  mbar·L /s (helium test, 3000 psi).

Pressure: Vacuum to 5000 psi.

Temperature: 50300°C, resistant to high temperature fluids.

Dynamic seal: speed 1000-5000 rpm, life span >5000 hours.

Static seal: compression rate 1520%, life span >10,000 hours.

### 3.3 Typical

Mechanical seal ring: Ø 10150 mm, flatness <0.001 mm, high pressure resistant.

O-ring: Ø 5100 mm, self-reinforcing seal, corrosion-resistant.

Bellows seal: High elasticity, suitable for dynamic sealing.

Recommendation: The nickel-based carbide seals and sealing rings produced by CTIA GROUP LTD adopt vacuum sintering and HIP technology to meet the high corrosion resistance and low leakage requirements of chemical pumps.

### 4. Application Scenarios

Chemical pump body:

Scenario: Transporting sulfuric acid (50%), hydrochloric acid (30%) or fluids containing particles (solids <20%), such as fertilizer plants and refineries.

Performance: Density 14.815.0 g/cm<sup>3</sup>, hardness 18002200 HV, life span >10,000 hours.

Case: Centrifugal pump casing (Ø 200 mm), HIP sintering, 1400°C, 120 MPa, 4 hours, porosity <0.001%, sulphuric acid corrosion resistance, 12,000 hours of operation.

Seals:

Scenario: Chemical pump mechanical seals, pipe connections (3000 psi, 100200°C).

Performance: Hardness 2000 HV, leakage rate  $<10^{-6}$  mbar·L /s, dynamic life >5000 hours.

Example: Mechanical seal ring (Ø 50 mm), HIP sintering, 1350°C, 150 MPa, 3 hours, density 14.9 g/cm<sup>3</sup>, NaOH resistance 40%, life 6000 hours.

### 5. Selection recommendations

According to the application environment:

Corrosive fluids (acid/base):

Recommended: HIP sintered pump housing/seal rings, Ni content 1012%, DLC coating.

Reason: Resistant to sulfuric acid/hydrochloric acid, life span >10,000 hours.

Abrasive fluids (containing particles):

Recommended: HIP sintered impeller/bushing, Ni 812%, hardness 2000 HV.

Reason: Wear rate <0.05 mm<sup>3</sup> / h, abrasion resistance.

According to performance requirements:

High precision ( $\pm 0.05$  mm): vacuum sintering + HIP, shrinkage deviation  $<\pm 0.5\%$ .

High corrosion resistance: Ni 1015%, Cr/Mo 0.52%, acid and alkali resistance for 5 years.

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Low leakage: HIP sintering, Ra <0.2  $\mu\text{m}$ , PTFE/DLC coating.

According to the cost budget:

Low cost: atmosphere sintering furnace, 0.8 kWh/kg, suitable for large quantities of seals.

High performance: HIP sintering furnace, 2 kWh/kg, suitable for key pump parts.

Sintering furnace selection:

Small and medium batches (<50 kg/furnace): Single-chamber vacuum sintering furnace with high flexibility.

Large batches (>200 kg/furnace): Multi-chamber vacuum/atmosphere sintering furnace, cost reduction of 20%.

High performance: HIP sintering furnace, density >99.9%, porosity <0.001%.

## 6. Optimization suggestions

Sintering process:

Temperature control:  $\pm 3^{\circ}\text{C}$ , uniformity  $\pm 5^{\circ}\text{C}$ , consistency increased by 10%.

Dewaxing: vacuum ( $10^{-2}$  Pa) + H<sub>2</sub> (10 L/min), residual carbon <0.05%.

HIP: 1350°C, 120 MPa, 2 hours, strength increased by 15%.

Material:

Ni content: 1012%, balance of hardness and corrosion resistance.

Coating: PTFE/DLC, friction coefficient reduced by 20%, wear resistance increased by 30%.

Trace elements: Cr/Mo 0.52%, acid and alkali resistance increased by 25%.

Equipment maintenance:

Online monitoring: real-time monitoring of temperature, pressure, and O<sub>2</sub>, reducing the failure rate by 20%.

Component inspection: Molybdenum /tungsten heating elements should be maintained every 4,000 hours, and their lifespan will be increased by 25%.

## 7. Standards

GB/T 345052017: Dimensional accuracy  $\pm 0.05$  mm.

GB/T 183762014: Porosity <0.01%.

GB/T 38502015: Density >99%.

GB/T 51692013: Porosity A02B00C00.

GB/T 38512015: Strength 2.0-2.8 GPa .

GB/T 7997-2017: Hardness 1400-2200 HV.

NACE MR0175: Acid and alkali corrosion resistance.

API 610: Performance requirements for chemical pumps.

## 8. Conclusion

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Nickel-based cemented carbide chemical pump bodies and seals are characterized by high density (>99.9%), hardness (1400-2200 HV), corrosion resistance (resistance to acid, alkali/salt solution) and low leakage rate ( $<10^{-6}$  mbar·L/s), meeting the stringent requirements of the chemical industry. Vacuum sintering furnaces, HIP sintering furnaces and atmosphere sintering furnaces are suitable for high-precision, high-performance and mass production respectively. Optimized processes (such as 1350°C, 120 MPa, H<sub>2</sub> O<sub>2</sub> <5 ppm) significantly improve product performance. CTIA GROUP LTD uses advanced sintering technology in the production of nickel-based cemented carbide seals, sealing rings and chemical pump bodies, providing highly reliable solutions to help chemical equipment operate efficiently.

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