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NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 77.160

CCS H 21

GB/T 3249-2022

Replacing GB/T 3249-2009

**Test Method for Fisher Number of Metal Powders and
Related Compounds**

金属及其化合物粉末费氏粒度的测定方法

Issued on: March 09, 2022

Implemented on: October 01, 2022

Issued by: State Administration for Market Regulation;

Standardization Administration of the People's Republic of China.

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Test Method for Fisher Number of Metal Powders and Related Compounds

1 Scope

This Document specifies a test method for Fisher number of metal powders of related compounds.

This Document is applicable to the determination of the Fisher number of metal powders and related compounds (carbides, nitrides and oxides, etc.) with a powder particle size of 0.5 μ m to 50 μ m.

This Document does not apply to powder particles with poor axial symmetry and irregular shapes (such as flakes and fibers, etc.), neither does it apply to mixtures of different powders and powders containing binders or lubricants. If necessary, it shall be adopted by the relevant parties after consultation.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in this Document. For the dated documents, only the versions with the dates indicated are applicable to this Document; for the undated documents, only the latest version (including all the amendments) is applicable to this Document.

GB/T 3500 Powder Metallurgy - Vocabulary

GB/T 37561 Standard Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis

3 Terms and Definitions

For the purposes of this Document, the terms and definitions given in GB/T 3500 and the following apply.

3.1 Fisher sub-sieve sizer

An instrument that uses the air permeability principle to measure the envelope surface area of the powder.

3.2 Envelope-specific surface area

The surface area of the powder measured according to the measurement method specified in ISO 10070.

3.3 Air permeability

The pressure-drop of air through the powder sample layer.

3.4 De-agglomeration

The process of breaking down agglomerated powder particles.

3.5 Fisher number

The average particle size calculated by conversion on the premise that all particles are spherical and single in size.

3.6 Fisher calibration tube

Ruby standard tube with precise aperture, the value of the standard tube is precisely calibrated.

3.7 Porosity of a bed of powder

The ratio of the powder bed pore volume to the total powder bed volume.

3.8 Agglomerate

Aggregation formed by several particles sticking together.

4 Principle

The determination of Fisher number adopts the air permeability method. It is assumed that the powder is spherical particles with uniform particle size, smooth surface and no internal pores. Under the condition of constant flow or pressure, the air permeability or resistance is measured. Since the air permeability of the powder sample layer is related to the envelope-specific surface area of the powder, from which the specific surface area can be calculated; and then converted into the average diameter of the volume surface area, which is used to express the Fisher number.

According to the air permeability method, the powder Fisher number is calculated according to Formula (1):

$$d_{vs} = \frac{60\,000}{14} \sqrt{\frac{\eta C L^2 \rho M^2 F}{(A L \rho - M)^3 (P - F)}} = c \sqrt{\frac{L^2 \rho M^2 F}{(A L \rho - M)^3 (P - F)}} \dots\dots\dots (1)$$

Where:

The Fischer calibration tube, also known as the ruby standard tube, is a standard sample used to measure the average particle size, so that the measurement data of different operators can be compared. The ruby standard tube has been tested three times when leaving the factory, and the measurement results and their associated porosity are engraved on the ruby standard tube.

5.3 Sample filling device

5.3.1 Adjust according to the instruction manual of the instrument. When adjusting, the porous plug and filter paper shall not be inserted into the sample tube, but shall be stacked together and placed between the support column and the bottom of the holder.

5.3.2 A special baseline gauge can be used to replace the porous plug and filter paper for adjustment. The height of the gauge is $19.30\text{ mm} \pm 0.10\text{ mm}$.

5.4 Balance

The weighing range of the balance is at least 50g, and the calibration division value is 0.001g.

6 Sample

6.1 The samples to be tested should be uniform and representative.

6.2 The total amount of samples shall be more than twice the true density value.

6.3 The sample shall be dry and there shall be no agglomeration. If the powder sample has agglomeration, it shall affect the measurement result. It is recommended to de-agglomerate or grind the powder and conduct the test. The de-agglomeration shall be carried out in accordance with the provisions of GB/T 37561.

6.4 The sample shall not be deformed, agglomerated or broken when pressure is applied; and shall be properly stored to prevent oxidation.

7 Inspection and Calibration of Instrument

7.1 Inspection

7.1.1 Inspect that the position of the beam of the particle size indicator needle shall be parallel to the baseline of the reading plate, and it shall coincide with the porosity indicator needle when falling. The rack shall be perpendicular to the baseline of reading plate when moving up and down.

7.1.2 The height of the porous plug shall be inspected regularly to ensure that the height of the porous plug is consistent.

7.1.3 The pressure gauge and fine-tuning needle valve shall be inspected regularly and kept

clean.

7.1.4 Inspect whether the upper and lower rubber pads holding the sample tube are in good condition, and ensure that there is no air leakage; otherwise, the rubber pads shall be replaced in time.

7.1.5 Inspect whether the position requirements of the support column for pressing the sample are appropriate, that is, the distance between the plane of the rack and the upper plane of the support rod of the base shall be kept equal to the total thickness of two porous plugs and two pieces of fast filter paper; at this time, the particle size indicator needle on the rack just points to the baseline of the reading plate.

7.1.6 Inspect whether the desiccant is effective (if using color-changing silica gel, it shall remain blue).

7.1.7 Regularly inspect the air pump and add lubricating oil.

7.1.8 Turn on the power and allow the instrument to warm up for more than 20 min.

7.1.9 Inspect the zero position of the pressure gauge: move the rack down to align the particle size indicator needle with the baseline of the reading plate; then turn the water level adjustment knob of the pressure gauge to align the water level meniscus with the upper edge of the beam of the particle size indicator needle. The adjustment of the pressure gauge shall be carried out before the operation of the instrument. After the adjustment is completed, keep it for at least 5min. During the test, because the water is adsorbed in the glass tube of the pressure gauge, the water level cannot return to the zero point, which is a normal phenomenon. Therefore, each time the zero point of the water level is inspected, the instrument shall be left vacant for more than 15 min. After adjusting the zero point of the water level, the instrument shall be re-calibrated.

7.1.10 Inspect the water level line of the steady pressure tube: Set the shift valve to the low gear; install the empty sample tube between the rubber pads on the right side of the support column; adjust the regulator valve; and control the bubbling speed of the vertical tube in the steady pressure tube to 2 ~ 3 bubbles per second. Keep the inlet pressure at $50.0\text{cmH}_2\text{O} \pm 0.4\text{cmH}_2\text{O}$. The water level of the pressure gauge rises gradually; and the height of the water level reached within 1min~2min shall be 25cm. The water level marking line in the steady pressure tube can only be used as a reference, and cannot be used as the basis for the pressure calibration of the steady pressure tube.

7.1.11 Abrasion of the sample tube may lead to erroneous results. When such a situation is suspected, a spare tube that has not been used shall replace the sample tube, and the measurement shall be repeated. If the sample tube is obviously worn or scratched, it shall be discarded immediately. The inner diameter of the sample tube can be inspected regularly with an inner micrometer, and the inner diameter shall be $12.70\text{mm} \pm 0.05\text{mm}$.

7.1.12 Inspect the handwheel with a pressure calibrator (equivalent device) at least once a

8.2 The determination of the Fisher number and the calibration of the instrument shall be performed by the same person.

8.3 After the instrument is calibrated, adjust the shift valve to the desired position.

8.4 Put a filter paper on one end of the sample tube; use a porous plug to press the end with holes into the sample tube together with the filter paper; and then put the porous plug in the sample tube on top of the support column of the Fischer sub-sieve sizer until it is the same height of the support column; and then place the sample tube upright on the rubber support seat; keeping the filter paper above the porous plug.

8.5 Take a sample equal to the true density of the powder, accurate to 0.01g.

8.6 With the help of the funnel, pour the weighed sample into the sample tube; tap the edge of the sample tube and the funnel to make the powder sample completely pour into the sample tube; then put another piece of filter paper on the mouth of the sample tube and press it into another porous plug; and they shall enter into sample tube together until completely submerged.

8.7 Put the sample tube on the support column under the rack and pinion, and the lower porous plug is in contact with the upper edge of the support column.

8.8 Drop the rack until the bottom plane of the rack is in contact with the porous plug above the sample tube; turn the handle; apply pressure; and compact the sample. Until the pressure value rises to 222N (50l bf), repeat the force three times to the maximum pressure value; remove the pressure. After the last maximum pressure is applied, inspect the rack to ensure that it does not move up when the force is withdrawn. Move the reading plate of particle size so that the particle size indicator needle coincides with the height line of the sample. After that, the reading plate of particle size shall not be touched again until the test is completed; and record the porosity indicated by the porosity indicator needle.

8.9 Remove the sample tube; at this time, the sample tube should not vibrate, lay flat or upside down. Clamp the sample tube between the rubber pads on the right side of the support column; and clamp the sample tube tightly to ensure that both ends of the sample tube are sealed and airtight.

8.10 At this time, the liquid level in the pressure gauge tube gradually rises; maintains for at least 5min; and rises to a maximum value. Read the Fisher number of the particle size indicator needle on the reading plate; record the measurement results and the ambient temperature.

8.11 Remove the sample tube and take out the porous plug from the sample tube.

9 Test Data Processing

9.1 Weigh two samples for measurement; and take the arithmetic mean of the two measurements as the result. Test reports for the supplied powder samples include the results of two measurements, both performed on the same sample; while recording the porosity and Fisher

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