IS 7512 (2006): Method for the determination of average particle size of metal powders by fisher sub-sieve sizer [MTD 25: Powder Metallurgical Materials and Products]

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(पहला पुनरीक्षण)

Indian Standard

METHOD FOR THE DETERMINATION OF AVERAGE PARTICLE SIZE OF METAL POWDERS BY FISHER SUB-SIEVE SIZER

(First Revision)

ICS 77.160

BUREAU OF INDIAN STANDARDS
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Price Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Powder Metallurgical Materials and Products Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1974. While reviewing this standard in the light of experience gained during these years, the Committee decided to revise it to bring it in line with the present practices followed in determination of average particle size of metal powders by Fisher sub-sieve sizer.

In preparation of this standard assistance has been derived from the following:

a) ASTM B 330-1998 'Standard test method for average particle size Fisher number of metal powders of refractory metal and their related compound by Fisher sub-sieve size'

b) ISO 10070 : 1991 'Metallic powder — Determination of envelope specific surface area from measurement of permeability to air of a powder bed under steady state flow condition'.

c) MPIF Standard — Method for determination of average partial size of metal powder using this Fisher sub-sieve size

In reporting the result of a test or analysis made in accordance with this standard, if the final values, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHOD FOR THE DETERMINATION OF AVERAGE PARTICLE SIZE OF METAL POWDERS BY FISHER SUB-SIEVE SIZER

(First Revision)

1 SCOPE

1.1 This standard prescribes the method of determining the average particle size of metal powder by Fisher sub-sieve sizer.

1.2 This test method uses air permeability to determine an envelope specific surface area and its associated average equivalent spherical diameter of metal powders and related compounds. The powders may be analyzed in their as supplied (shipped, received or processed) condition or after they have been de-agglomerated or milled by a laboratory procedure. The values obtained are not intended to be absolute but are generally useful on a relative basis for control purposes.

2 REFERENCES

The standards listed below contain provisions, which through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revisions and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

IS No.	*Title*
5432 : 1982	Glossary of terms relating to powder metallurgy (*first revision*)
6492 : 1972	Methods for sampling of powders for powder metallurgical purposes

3 TERMINOLOGY

For the purpose of this standard, the definition given in IS 5432 and the following shall apply.

3.1 Fisher Sub-sieve Sizer — A commercially available permeability instrument for measuring envelope-specific surface area.

3.2 Envelope-Specific. Surface Area — The specific surface area of a powder as determined by gas permeametry.

3.3 Air Permeability — The measurement of air pressure drop across a packed bed of powder.

3.4 De-agglomeration — Process used to break up agglomerates of practicles.

3.5 Fisher Number — A calculated value equated to an average particle diameter, assuming all the particles are spherical and of uniform size.

3.6 Fisher Calibrator Tube — A jewel with a precision orifice mounted in a tube similar to a sample tube. The calibrator tube value is directly traceable to the master tube maintained by Fisher.

3.7 Porosity of a Bed of Powder — The ratio of the volume of the void space in the powder bed to that of the overall volume of the powder bed.

3.8 Agglomerate — Several particles adhering together.

4 PRINCIPLE OF TEST

4.1 This test method provides a procedure for determining the envelope-specific surface area of powders which is equated to an average particle diameter by calculation assuming the particle are monosize, smooth surface, non-porous spherical particles. For this reason, values obtained by this test method will be defined as a Fisher number. The degree of correlation between the results of this test method and the quality of powders in use will vary with each particular application and has not been fully determined.

4.2 This test method is generally applicable to all metal powders and related compounds, including carbides, nitrides and oxides, for particles having diameters between 0.2 and 50 μm. It should not be used for powders composed of particles whose shape is too far from equiaxed, that is, flakes or fibres. In these cases, it is permissible to use the test method described only by agreement between the parties concerned. This test method shall not be used for mixtures of different powders, nor for powders containing binders or lubricants. When the powder contain agglomerates, the measured surface area may be affected by the degree of agglomeration.

4.3 When an average particle size of powders is determined

using the Fisher sub-sieve sizer, it should be clearly kept in mind that this average size is derived from the determination of the specific surface area of the powder using a relationship that is true only for powders of uniform size and spherical shape.

5 APPARATUS

5.1 The Fisher sub-sieve sizer consists of an air pump, an air-pressure reglating device, a precision-bore sample tube, a standardized double range air flowmeter, and a calculator chart. Included is accessory equipment consisting of a plug manipulator, powder funnel, two porous plugs, a supply of paper disks, and a rubber tube support stand.

NOTE — Necessary replacement parts should be obtained from the manufacturer, especially in the case of the precision manometer which is a part of the air flowmeter.

5.2 The manufacturer also furnishes directions which should be followed except as amended as follows. Particular attention should be given to proper maintenance of the instrument with special reference to the instructions on: (a) periodic checking of the water level in the pressure regulator standpipe, (b) manometer level before the sample tube is inserted, and (c) the sample packing assembly.

5.3 Jewel Calibrator Tube

A standard for average particle size measurement. It allows operators to relate their data to that of other analysts. Each calibrator is factory tested three times with the resulting readings and associated porosity recorded on the tube.

NOTE — Adjust the sample packing assembly (a) as described in the manufacturer's directions with the exception that the plugs and paper disks are not inserted in the sample tube but are merely stacked together and placed between the brass support and the 'flat' of the bottom of the rack, of (b) as previously described except that a specially made base line gauge is used instead of the plugs and paper disks. This base line gauge shall have a height of 19.30 ± 0.10 mm. Check all plug heights when new plugs are purchased and periodically thereafter to make sure all are equal in height.

5.4 Balance

Having a capacity of at least 50 g and a sensitivity of 0.001 g.

6 STANDARDIZATION OF APPARATUS

6.1 Before proceedings with standardization of the Fisher sub-sieve sizer, the following items shall be checked:

a) Chart shall be properly aligned horizontally with the indicator pointer;

b) Rack and pinion shall be properly aligned vertically with the chart;

c) Sample tube or plugs shall not be worn;

d) Manometer and air resistors shall be free of visible contamination;

e) Rubber sample tube seals shall not be worn to the point where leakage occurs;

f) Sample packing post shall be properly adjusted;

g) Drying agent shall be in proper condition; and

h) Manometer and standpipe levels shall be checked.

6.1.1 Adjust the manometer only when the machine is not operating and with the pressure released for minimum of 5 min to allow the manometer tube to drain complete.

6.2 The standardization of the Fisher sub-sieve sizer shall be made using the Fisher jewel calibrator tube (jewel orifice tube) as the primary standard. Specification shall be made at both ranges of the machine.

The Fisher jewel calibrator tube used for standardization shall be checked under a microscope at least once a month to determine the condition and cleanliness of the orifice.

If the orifice is not clean, clean as described in the Fisher sub-sieve sizer instruction manual.

6.3 With the sub-sieve sizer properly adjusted and set to the proper range, proceed follows:

a) Mount the Fisher jewel calibrator tube between the rubber seal supports just to the right of the brass post. Clamp the upper cap down onto the tube so that an airtight seal is obtained at both ends.

b) Adjust the calculator chart so that the porosity reading corresponds to the value indicated on the jewel calibrator use.

c) Switch on the machine and allow it to warm up for a minimum of 20 min. Adjust the pressure control knob, located near the bubble observation window at the lower left of the panel, until the bubbles rise in the stand pipe at the rate of 2 to 3 bubbles per second. This will cause the water line to rise above the calibration mark on the upper end of the stand pipe. This is normal and does not mean the calibration is in error.

d) The liquid level in the manometer tube will rise slowly until it reaches a maximum. Allow at least 5 min for this to happen. At the end of this period, using care not to disturb the chart, turn the rack up until the upper edge of the cross bar coincides with the bottom of the liquid meniscus in the manometer. The Fisher number is indicated by the location of the pointer tip in relation to the curves on the calculator chart. Record the ambient temperature to the nearest 1°C. Release the clamp on the upper end of the tube slowly so the manometer returns to its zero position slowly with very little overshoot. This limits the formation of liquid droplets on the inside of the manometer tube.

e) The value obtained in the manner must correspond

to the Fisher number indicated on the jewel calibrator tube within ± 1percent.

f) If the Fisher number value as indicated on the chart does not correspond to ± 1 percent of the value indicated on the jewel calibrator tube, calibrate the sub-sieve as follows:

Adjust either the high needle valve or the low needle valve as required to bring the Fisher number indicated on the chart to the value indicated on the jewel calibrator tube. After adjustment is made, repeat **6.3(d)**.

NOTE – Because only one flow meter is used for the low (0.2 to 20.0 µm) Fisher number range, while both flow meters are used for the high (20.0 to 50.0 µm) Fisher number range, the low range should be standardized first. After the low range is standardized, the high range is then standardized, making adjustments only to the one flow meter opened up by the range-control knob.

g) Standardization with the jewel calibrator tube is recommended before and after any series of determinations or at least very 4 h of continued operation. Warm up of the machine is required if it has been off for more than 30 min.

7 PROCEDURE

7.1 Temperature of Test

Make Fisher number determinations with ± 2°C of the temperature at which standardization of the Fisher sub-sieve was made. Re-standardize, if the temperature of the test varies more than ± 2°C.

7.1.1 The sampling of powder for conducting the test shall be in accordance with IS 6492.

7.2 Size of Test Sample

The mass of the sample used for tests should be equal in grams (within ± 0.01 g) to the theoretical density of the powder (Tungsten, 19.3 g; molybdenum 10.22 g; tantalum 16.6 g, etc).

7.3 Fisher Number Determination

The Fisher number determination shall be made by the same operator who make the standardizations, and is started after standardization or the determination of another sample. Proceed as follows.

7.3.1 With the sub-sieve sizer properly adjusted, set the range control to the range desired.

7.3.2 Lay a paper disk over one end of the sample tube using one of the porous plugs with the perforated surfaces of the plug against the surface of the paper disk. This crimps the paper around the edges and the paper precedes the plug into the sample tube. Push the plug into the tube until it is even with the end of the sample tube. Place the sample tube in a vertical position in a support with the paper side of the plug up.

7.3.3 *Determine the Mass of the Sample*

With the aid of the powder funnel, completely transfer the sample into the sample tube, tapping the side of the tube and funnel 2 or 3 times each to settle the powder. Lay a second paper disk over the top of the sample tube, and using another porous brass plug, force the plug and paper disk down into the sample tube until it is just inside the sample tube. Place the sample tube on the brass post beneath the rack and pinion with the lower plug in contact with the upper end of the brass post.

7.3.4 Lower the rack, guiding it until the flat-bottom end comes in contact with the upper plug. Pack the sample firmly by turning down the pinion knob with the torque wrench or torque screwdriver until a compressive force of 222 N (50 lbf) is applied to the sample. After this force is applied, the sample tube should not be touching the block in which the brass post is mounted. In cases where the tube tends to move down and kept on the block during compression, the tube can be held temporarily by hand or a spacer can be used until most of the compressive force has been applied. The spacer is then removed when the maximum force is actually applied. Apply and release maximum force a total of the three times. After the load maximum compression force has been applied, check the rack to make sure it has not been removed upward with the final release of pressure. Check torque wrench or torque screwdriver for standardization at least once every month using sample pressure calibrator or an equivalent device.

7.3.5 Shift the calculator chart laterally until the extreme tip of the pointer just coincides with the sample height curve on the chart. The pointer should be midway between the top and bottom of the line. The chart must not be moved after this setting until the determination is finished. Record the porosity value indicated at the bottom of the chart.

7.3.6 Without disturbing the sample in any way, mount the sample tube between the rubber cushioned supports just to the right of the brass post. Clamp the upper cap down onto the sample tube so that an airtight seal is obtained at both ends.

NOTE — The sample tube may eventually wear and cause faulty values. When this condition is suspected, replace the tube. Sample tube with obvious wear or scratches, or both, should be discarded.

7.3.7 Determine the Fisher number, switching on the machine and allowing the liquid level in the manometer tube to rise until it reaches a maximum. Allow a minimum of 5 min for this to happen. The Fisher number is indicated

by the location of the tip of the pointer in relation to the curves on the calculator chart. Record this value along with the porosity for the sample and the ambient temperature at which the measurement was made.

NOTE — Formulas for calculating the Fisher number or the equivalent spherical diameter and porosity values from sample and manometer heights are as follows:

a) Where sample mass is equal to the theoretical density of the material being tested:

$$\text{Porosity} = \frac{(LA - 1)}{LA} \text{ and} \quad \ldots\ldots (1)$$

$$\text{Fisher number, } \mu\text{m} = CL\sqrt{\frac{F}{(P - F)(AL - 1)^3}} \quad \ldots\ldots (2)$$

where

L = sample height after compaction, in cm;
A = cross-sectional area of the sample tube, cm^2 (= 1.267cm^2);
M = mass of sample for 1 cm^3;
D = theoretical density of material being tested, in g/cm^3;
C = cross-sectional constant = 3.80;
F = pressure difference of water, cm (NOTE $F = 2H$, where H = height of water column above base line, in cm); and
P = overall air pressure (determined by standpipe) = 50 cm of water.

b) Where sample mass is not equal to the theoretical density of material being tested, as in the case when the sample size is less than the tube density:

$$\text{Porosity} = \frac{\frac{(LA - M)}{D}}{LA}, \text{ and} \quad \ldots\ldots (3)$$

$$\frac{CLM}{D}\sqrt{\frac{F}{(P - F)(AL - M/D)^3}} \quad \ldots\ldots (4)$$

where

M = mass of sample, in g; and
D = true density of material being tested, in g/cm^3.

For a powder in which all particles are spherical and of uniform size, the particle side d in micrometers may be calculated from the volume-specific surface area S_v by the following formula:

$$d = \frac{6 \times 10^6}{S_v} \quad \ldots\ldots (5)$$

A calculation of an equivalent spherical diameter based on this equation is performed automatically by the calculator chart of the Fisher sub-sieve sizer from the values related to the porosity and to the permeability of the powder bed measured by the instrument. In other words, what is determined with the instrument is the specific surface area of the powder. When an equivalent spherical diameter is determined using the Fisher sub-sieve size, it should be clearly kept in mind that this equivalent spherical diameter is derived from the determination of the specific surface area of the powder using a relationship that is true only for powders of uniform size and spherical shape. Hence, the term 'Fisher number' is preferred to describe the results of this test, rather than particle size or equivalent spherical diameter.

8 REPORTING OF RESULTS

8.1 Report the Fisher number value of as supplied powders as the average of two determinations, each made on separate portions of the sample, if the two values agree within 3 percent. If the values do not agree within 3 percent. If the values do not agree within 3 percent, review the equipment and procedure and repeat the test using separate portions of the sample. Report the average porosity of the checked samples along with the average Fisher number, and identify the values as being determined on as supplied powder.

8.2 If the powder is de-agglomerated or milled in the laboratory prior to analysis in accordance with practice only one determination of the Fisher number, need be made and reported along with the porosity value determined as identified as lab milled. If another laboratory method it was to de-agglomerate or mill the powder, sufficient information to described the procedure completely must also be included with the results.

8.3 Table 1 provides limitations of Fisher number.

Table 1 Reporting Limitations

Sl No.	Range Fisher Number	Porosity	Range Control	Chart Division (Fisher Number)	Read and Report to Fisher Number
(1)	(2)	(3)	(4)	(5)	(6)
i)	0.2 to 0.5	0.60 to 0.70	Read direct	0.1	0.1
ii)	0.2-0.5	0.70-0.80	Read direct	0.1	0.05
iii)	0.5-1.0	0.55-0.80	Read direct	0.1	0.02
iv)	1.0-4.0	0.45-0.80	Read direct	0.1	0.02
v)	4.0-8.0	0.40-0.80	Read direct	0.2	0.05
vi)	8.0-15.0	0.40-0.65	Read direct	0.5	0.2
vii)	15.0-20.0	0.40-0.75	Read direct	1.0	0.5
viii)	20.0-50.0	0.40-0.60	Read direct	1.0	0.5

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Review of Indian Standards

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