Designation: B 330 – 02

Standard Test Method for Fisher Number of Metal Powders and Related Compounds[1]

This standard is issued under the fixed designation B 330; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope *

1.1 This test method uses air permeability to determine an envelope-specific surface area and its associated average equivalent spherical diameter (from 1 to 50 μm) 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 ("lab milled") such as that specified in Practice B 859. The values obtained are not intended to be absolute but are generally useful on a relative basis for control purposes.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

B 243 Terminology of Powder Metallurgy[2]

B 859 Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis[2]

E 456 Terminology Relating to Quality and Statistics[3]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO/DIS Document:*

10070 Metallic Powders Determinations of Envelope-Specific Surface Area from Measurements of the Permeability to Air of a Powder Bed Under Steady-State Flow Conditions[4]

3. Terminology

3.1 *Definitions*—Many terms used in this test method are defined in Terminology B 243.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *Fisher sub-sieve sizer*, *n*—a commercially available permeability instrument for measuring envelope-specific surface area.

3.2.2 *envelope-specific surface area*, *n*—the specific surface area of a powder as determined by gas permeametry in accordance with ISO/DIS 10070.

3.2.3 *air permeability*, *n*—the measurement of air pressure drop across a packed bed of powder.

3.2.4 *de-agglomeration*, *n*—process used to break up agglomerates of particles.

3.2.5 *Fisher Number*, *n*—a calculated value equated to an average particle diameter, assuming all the particles are spherical and of uniform size.

3.2.6 *Fisher calibrator tube*, *n*—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.

3.2.7 *porosity of a bed of powder*, *n*—the ratio of the volume of the void space in the powder bed to the that of the overall volume of the powder bed.

3.2.8 *agglomerate*, *n*—several particles adhering together.

4. Significance and Use

4.1 This test method provides a procedure for determining the envelope-specific surface area of powders, from which is calculated an "average" particle diameter, assuming the particles are monosize, smooth surface, nonporous, 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 1 and 50 μm. It should not be used for powders composed of particles whose shape is too far from equiaxed, that is, flakes or fibers. 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

[1] This test method is under the jurisdiction of ASTM Committee B09 on Metal Powders and Metal Powder Products and is the direst responsibility of Subcommittee B09.03on Refractory Metal Powders.

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[2] *Annual Book of ASTM Standards*, Vol 02.05.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

***A Summary of Changes section appears at the end of this standard.**

contains agglomerates, the measured surface area may be affected by the degree of agglomeration. Methods of de-agglomeration such as that referenced in 1.1 may be used if agreed upon between the parties concerned.

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[5] consists of an air pump, an air-pressure regulating 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 1—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 (*1*) periodic checking of the water level in the pressure regulator standpipe, (*2*) manometer level before the sample tube is inserted, and (*3*) 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 2—Adjust the sample packing assembly (1) 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 (2) as previously described except that a specially made baseline gage is used instead of the plugs and paper disks. This baseline gage 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 proceeding with standardization of the Fisher sub-sieve sizer, the following items shall be checked:

6.1.1 The chart shall be properly aligned horizontally with the indicator pointer.

6.1.2 The rack and pinion shall be properly aligned vertically with the chart.

6.1.3 The sample tube or plugs shall not be worn.

6.1.4 The manometer and air resistors shall be free of visible contamination.

6.1.5 The rubber sample tube seals shall not be worn to the point where leakage occurs.

6.1.6 The sample packing post shall be properly adjusted.

6.1.7 The drying agent shall be in proper condition.

6.1.8 The manometer and standpipe levels shall be checked.

6.1.8.1 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 completely.

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 as follows:

6.3.1 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.

6.3.2 Adjust the calculator chart so that the porosity reading corresponds to the value indicated on the jewel calibrator tube.

6.3.3 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 standpipe at the rate of two to three bubbles per second. This will cause the water line to rise above the calibration mark on the upper end of the standpipe. This is normal and does not mean the calibration is in error.

6.3.4 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 crossbar 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.

6.3.5 The value obtained in this manner must correspond to the Fisher Number indicated on the jewel calibrator tube within ±1 %.

6.3.6 If the Fisher Number value as indicated on the chart does not correspond to ±1 % 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 6.3.4.

NOTE 3—Because only one flowmeter is used for the low (1- to

[5] The manufacturer of the Fisher sub-sieve sizer #14-311A, is Fisher Scientific, Instrument Division, 2000 Park Lane, Pittsburgh, PA 15275. Besides supplying the basic instrument, they also supply accessories of: calibrator tube #14-313-7 and sample calibrator, #14-311-2.

20.0-μm) Fisher Number range while both flowmeters 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 flowmeter opened up by the range-control knob.

6.3.7 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 sizer was made. Restandardize if the temperature of the test varies more than ±2°C.

7.2 *Size of Test Sample*—The mass of 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; and so forth).

7.3 *Fisher Number Determination*—The Fisher Number determination shall be made by the same operator who makes 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 surface 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.

7.3.4 With the aid of the powder funnel, completely transfer the sample into the sample tube, tapping the side of the tube and funnel two or three 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.

7.3.5 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 in which the tube tends to move down and rest 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 three times. After the final 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.

7.3.6 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.

7.3.7 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 4—The sample tube may eventually wear and cause faulty values. When this condition is suspected, replace the tube. Sample tubes with obvious wear or scratches, or both, should be discarded.

7.3.8 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 5—Formulas for calculating the Fisher Number or the equivalent spherical diameter and porosity values from sample and manometer heights are as follows:

(*1*) Where sample mass is equal to the theoretical density of the material being tested:

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

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

where:

L = sample height after compaction, cm;
A = cross-sectional area of the sample tube, cm^2 (=1.267 cm^2);
M = mass of sample for 1 cm^3;
D = theoretical density of material being tested, 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, cm); and
P = overall air pressure (determined by standpipe) = 50 cm of water.

(2) 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 true density:

$$\text{Porosity} = \frac{LA - \left(\frac{M}{D}\right)}{LA} \text{ and} \tag{3}$$

$$\text{Fisher Number } (\mu\text{m}) = \frac{CLM}{D}\sqrt{\frac{F}{(P - F)\left(AL - \frac{M}{D}\right)^3}} \tag{4}$$

where:

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

For a powder in which all particles are spherical and of uniform size, the particle size d in micrometres may be

calculated from the volume-specific surface area S_v:

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

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 sizer, 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 result of this test, rather than "particle size" or "equivalent spherical diameter."

8. Report

8.1 Report the Fisher Numbers and porosities of "as-supplied" powders as the results of two determinations, each made on separate portions of the sample. Report both measured porosities of the packed samples along with both Fisher Numbers and identify the results as being determined on "as supplied" powder.

8.2 If the powder is de-agglomerated or milled in the laboratory before analysis in accordance with Practice B 859, only one determination of the Fisher Number need be made and reported along with the porosity value determined and identified as "lab milled." If another laboratory method is used to de-agglomerate or mill the powder, sufficient information to describe the procedure completely must also be included with the results.

8.3 Table 1 provides limitations of Fisher Number.

TABLE 1 Reporting Limitations

Range (Fisher Number)	Porosity	Range Control	Chart Division (Fisher Number)	Read and Report to (Fisher Number)
0.2 to 0.5	0.60 to 0.70	read direct	0.1	0.1
0.2 to 0.5	0.70 to 0.80	read direct	0.1	0.05
0.5 to 1.0	0.55 to 0.80	read direct	0.1	0.02
1.0 to 4.0	0.45 to 0.80	read direct	0.1	0.02
4.0 to 8.0	0.4 to 0.80	read direct	0.2	0.05
8.0 to 15.0	0.40 to 0.65	read direct	0.5	0.2
15.0 to 20.0	0.40 to 0.75	read double	1.0	0.5
20.0 to 50.0	0.40 to 0.60	read double	1.0	0.5

9. Precision and Bias [6]

9.1 *Precision*—The results of an interlaboratory study to determine the precision of this test method are available in ASTM Research Report No. B09–1010, a report on a study done in five laboratories on tungsten carbide powders in both the as-supplied and laboratory-milled conditions. Although this is not in conformance with the requirements of Practice E 691 (six laboratories are required), the user of this test method may infer its precision from this interlaboratory study. The pertinent conclusions are presented in 9.1.1 and 9.1.2.

9.1.1 The within-laboratory repeatability limit, *r*, as defined by Terminology E 456, was estimated to be 2 to 6 % of the measured Fisher Number. Duplicate results from the same laboratory should not be considered suspect unless they differ by more than *r*.

9.1.2 The between-laboratory reproducibility limit, *R*, as defined by Terminology E 456, was found to be estimated by the following equation:

$$R = 0.173F - 0.042 \quad (6)$$

where:

R = the reproducibility limit and
F = the measured Fisher Number.

Results from two different laboratories should not be considered suspect unless they differ by more than *R*.

9.2 *Bias*—The Fisher Number is a calculated estimate of average particle diameter in a powder. No absolute method of determining powder particle size exists, nor are there any universally recognized standard or reference powders for this measurement; therefore, it is not possible to discuss the bias of results by this test method.

10. Keywords

10.1 envelope-specific surface area; Fisher Number; metal powder; particle size; porosity; powder; specific surface

[6] Supporting data are available from ASTM Headquarters. Request RR: B09–1010.

SUMMARY OF CHANGES

Committee B09 has identified the location of selected changes to this standard since the last issue (B 330 – 00) that may impact the use of this standard.

(1) Paragraph 8.1 was modified to remove the 3 % duplication requirement and to report both results. Rationale: It has been found that sampling and precision differences with fine metal powders preclude meeting the former 3 % duplication criterion for "as-supplied" materials. The decision was made at a meeting of Subcommittee B09.03 on April 30, 2002, to remove the 3 % requirement, retaining the duplicate *test* requirement, and reporting *both* results.

(2) Paragraphs 1.1 and 4.2 and Note 3 were changed to limit applicability to >1 µm. Rationale: Discussions at the same subcommittee meeting mentioned above indicated that the range of applicability of this method does not extend to the submicrometer particle size regime.

(3) Paragraph 4.1 was editorially modified.

(4) Equation 3 in Note 5 was changed to correct a typographical error.