Designation: B 330 – 00

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.

1. Scope

1.1 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 ("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]

2.3 *MPIF Standard:*

32 Method for Determination of Average Particle Size of Metal Powders Using the Fisher Subsieve Sizer[5]

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 which is equated to an "average" particle diameter by calculation 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 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 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

[1] This test method is under the jurisdiction of ASTM Committee B-9 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.

[5] Available from Metal Powder Industries Federation, 105 College Rd. East, Princeton, NJ 08540–6692.

for powders containing binders or lubricants. When the powder 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[6] 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.

[6] 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.