Designation: B859 – 21

Standard Practice for De-Agglomeration of Refractory Metal Powders and Their Compounds Prior to Particle Size Analysis[1]

This standard is issued under the fixed designation B859; 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 practice covers the de-agglomeration of refractory metal powders and their compounds in preparation for particle size analysis.

1.2 Experience has shown that this practice is satisfactory for the de-agglomeration of elemental tungsten, molybdenum, rhenium, and tantalum metal powders, and tungsten carbide. Other metal powders (for example, elemental metals, carbides, and nitrides) may be prepared for particle size analysis using this practice with caution as to effectiveness until actual satisfactory experience is developed.

1.3 *Units*—With the exception of the values for mass, for which the use of the gram (g) unit is the long-standing industry practice, the values stated in SI units are to be regarded as standard. No other units of measure are included in this standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see Note 2.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B243 Terminology of Powder Metallurgy

B330 Test Methods for Estimating Average Particle Size of Metal Powders and Related Compounds Using Air Permeability

B761 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by X-Ray Monitoring of Gravity Sedimentation

B821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis

B822 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering

3. Terminology

3.1 *Definitions*—Definitions of powder metallurgy terms can be found in Terminology B243.

4. Significance and Use

4.1 Refractory metal powders, such as tungsten and molybdenum, are usually produced by hydrogen reduction at high temperatures. Thus, they usually contain numerous large, strongly-sintered agglomerates. Many of the manufacturing processes using these powders involve a milling step or some similar treatment or depend on the individual particulate size, not on the agglomerate size.[3] Thus, a knowledge of the individual particulate size distribution, not the agglomerate size distribution, is usually desired from a particle size analysis of these powders. This practice provides a procedure for breaking down agglomerates into their constituent particles (de-agglomeration), without excessive fracture of the individual particles. The procedure is often referred to as *laboratory milling* or *rod milling*.

4.2 The laboratory milling conditions specified in this guide have been in use since 1965, initially as part of a particle size analysis test method. This guide was first published as a separate, stand-alone standard in 1995 because of its applicability in preparing powder samples for analysis by other methods as well (for example, Test Methods B761 and B822).

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Michaels, A. I., "Turbidimetric Particle Size Distribution Theory: Application to Refractory Metal and Oxide Powders," *1958 Symposium on Particle Size Measurement, ASTM STP 234*, ASTM, 1959, pp. 207–244.

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

Information on the development and establishment of the milling conditions here specified can be found in the footnoted reference.[4]

4.3 The milling procedure described in this practice does not necessarily break down only agglomerates without fracturing individual particles; some particle fracture may occur in certain powders. However, use of this practice *does* provide consistent particle size analysis results that have been found to relate well to powder behavior in numerous manufacturing processes.

4.4 This practice shall be used for the de-agglomeration of the refractory metal powders and compounds listed in 1.2, when an evaluation of the individual particulate size distribution is required from the subsequent particle size analysis. It shall not be used when the *agglomerate* (as-is or as-supplied) size distribution is desired.

4.5 This practice may be used in preparing samples for Test Methods B330, B761, B822, and other particle size analysis methods, prior to the dispersion procedure of Guide B821, if used.

5. Apparatus

5.1 *Milling Bottle*—There are two alternative materials for the milling container:

5.1.1 *Glass Bottle*—Round laboratory solution bottle, 250 mL capacity, or a 250 mL, 140 mm high, 60 mm diameter, wide-mouth, flat-bottom centrifuge bottle, with cap or stopper, or

5.1.2 *Stainless Steel Bottle*—Fabricated according to the detailed drawings in Appendix X1.

5.2 *Tungsten Rods*—Fifty rods 75 ± 3 mm long by 4.0 ± 0.3 mm in diameter, ground surface.

5.3 *Laboratory Jar Roll Mill,* capable of rotating the milling bottle at 145 rpm.

NOTE 1—If a jar roll mill is not available to give a bottle rpm of 145, the bottle can be either (*1*) set up on a lathe, or (*2*) built up in diameter and used on a faster rpm mill.

5.4 *Screen, No. 20 (850 µm), and Bottom Pan.*

6. Procedure

6.1 Place 30 ± 0.1 g of tungsten, molybdenum, rhenium, or tantalum metal, or 50 ± 0.1 g of tungsten carbide powder in the milling bottle containing the 50 tungsten milling rods.

6.2 Seal the milling bottle and rotate on the jar roll mill for 60 min ± 15 s at a bottle speed of 145 ± 5 rpm. After the first 5 min, check to be sure the rods are cascading inside the bottle by listening for the fast-paced, repeated "clinking" sound that the cascading rods make. If the rods are not cascading, stop the mill, set the bottle upright momentarily, then replace the bottle on the mill and continue milling for the remaining 55 min (if the rods are now cascading; if not, repeat setting the bottle upright and restarting until they are).

6.3 After milling, immediately screen the powder through a No. 20 (850 µm) screen to remove the milling rods. Dislodge any milled powder that might remain in the bottle by placing a couple of screened rods in the bottle and "whipping" for a few turns.

NOTE 2—**Warning:** The fresh metal surfaces produced during milling may have a tendency to rapidly oxidize when the milling bottle is opened. Use caution when opening the bottle to avoid fire or explosion.

6.4 Remove all the milled powder from the bottom pan and place in a sample container.

7. Particle Size Analysis

7.1 If necessary, disperse the milled powder according to Guide B821.

7.2 Immediately perform the desired particle size analysis.

NOTE 3—Since milled powder has a greater tendency than as-supplied powder to pick up moisture and oxidize, the analysis procedure should be initiated immediately after milling is completed. For all practical purposes, however, two runs can be made in succession on each milled powder. If more than two runs on the same milled powder are desired, provisions may be taken to lessen (elimination is not possible) the effect of humidity on the milled powder, such as immediate splitting of the sample and storage under dry nitrogen or in a desiccator.

8. Keywords

8.1 de-agglomeration; laboratory-milled; laboratory milling; molybdenum; particle size analysis; powders; refractory metals; rhenium; rod-milled; rod milling; tantalum; tungsten; tungsten carbide

[4] Buerkel, W. A., "Turbidimetric Particle Size Analysis as Applied to Tungsten Powder and the Carbide Industry," *Handbook of Metal Powders*, A. Poster, ed., Reinhold Publishing Corp., New York, NY, 1966, pp. 20–37.

APPENDIX

(Nonmandatory Information)

X1. FABRICATED STAINLESS STEEL MILLING BOTTLE

X1.1 The stainless steel milling bottle mentioned in 5.1.2 may be fabricated according to the following detailed drawings (Figs. X1.1-X1.4):

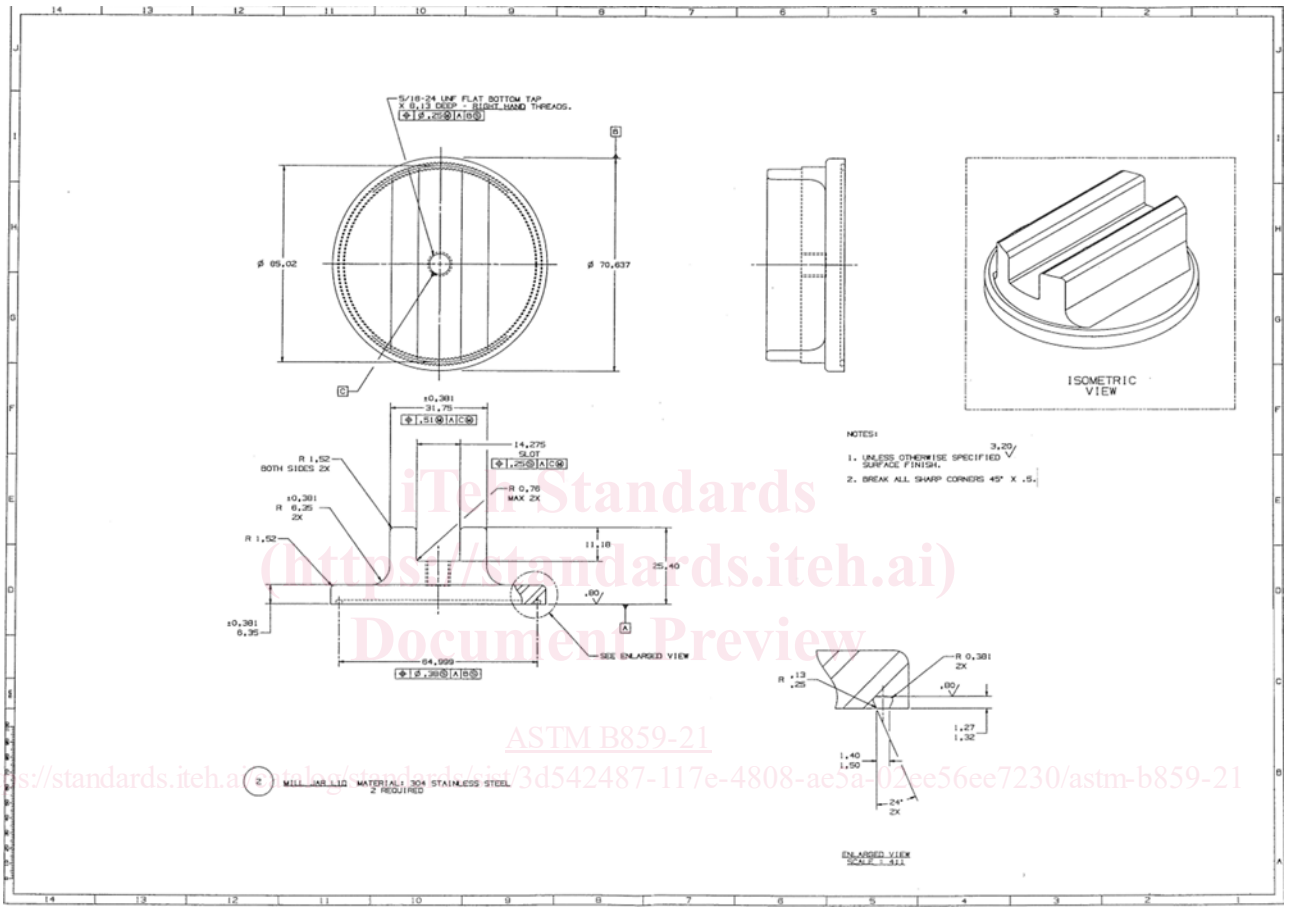

FIG. X1.1 Milling Bottle End Cap

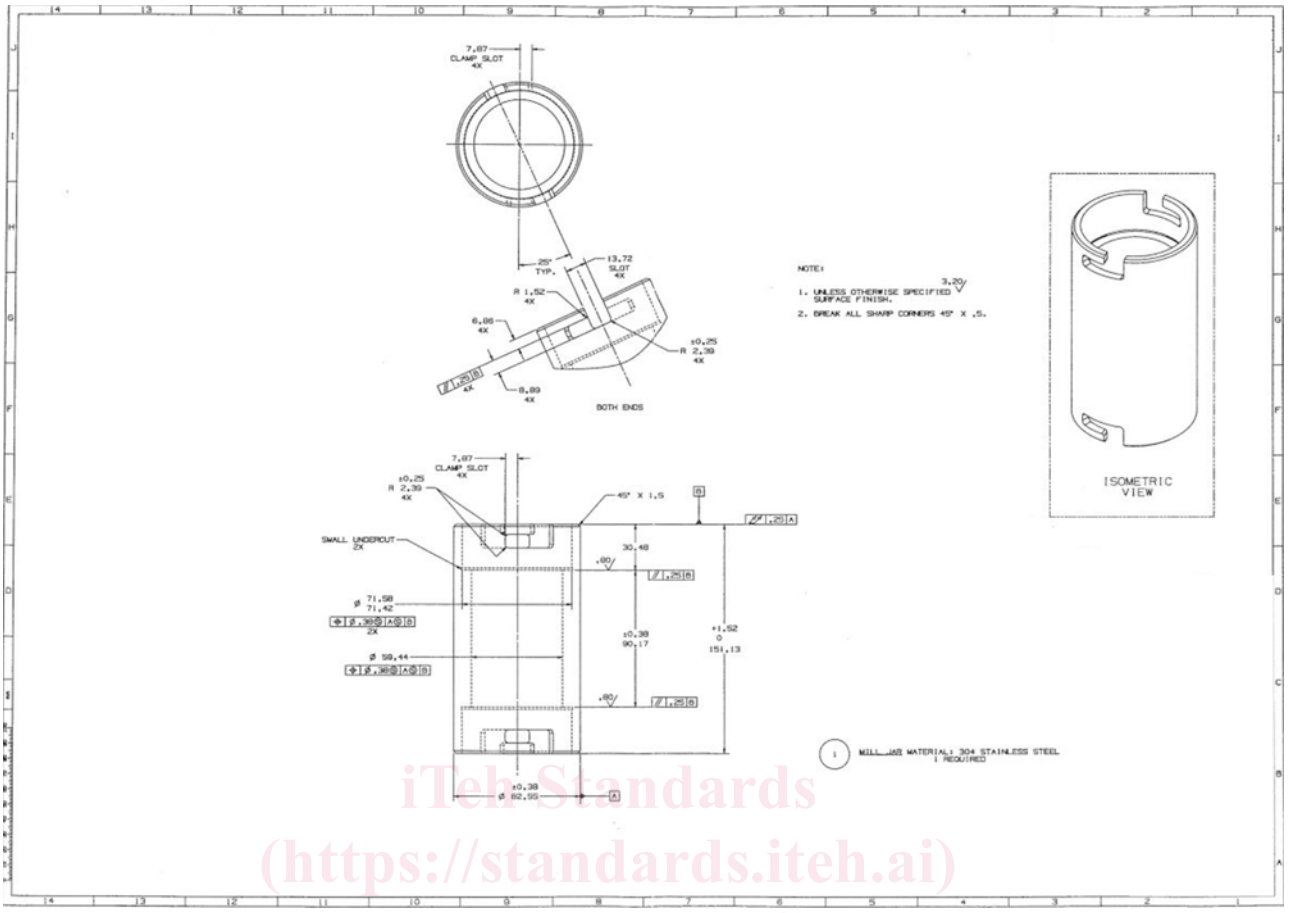

FIG. X1.2 Milling Bottle Body