Designation: B 859 – 03

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 B 859; 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 *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.* For specific precautionary statements, see Note 2.

2. Referenced Documents

2.1 *ASTM Standards:*

B 243 Terminology of Powder Metallurgy[2]

B 330 Test Method for Fisher Number of Metal Powders and Related Compounds[2]

B 430 Test Method for Particle Size Distribution of Refractory Metal Powders and Related Compounds by Turbidimetry[2]

B 761 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by X-Ray Monitoring of Gravity Sedimentation[2]

B 821 Guide for Liquid Dispersion of Metal Powders and Related Compounds for Particle Size Analysis[2]

B 822 Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering[2]

2.2 *ASTM Adjunct:*

ADJB0859 Detailed Drawings of Alternative Steel Milling Bottles[3]

3. Terminology

3.1 *Definitions*—Definitions of powder metallurgy terms can be found in Terminology B 243.

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.[4] 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 as part of Test Method B 430. 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 (for example, Test Methods B 761 and B 822), in addition to Test Method B 430. Information on the development and establishment of the milling conditions here specified can be found in the footnoted reference.[5]

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

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

[3] Available from ASTM International Headquarters. Order Adjunct No. ADJB0859.

[4] 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.

[5] 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.

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