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Standard Test Method for Metal Powder Skeletal Density by Helium or Nitrogen Pycnometry[1]

This standard is issued under the fixed designation B923; 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 covers determination of skeletal density of metal powders. The test method specifies general procedures that are applicable to many commercial pycnometry instruments. The method provides specific sample outgassing procedures for listed materials. It includes additional general outgassing instructions for other metals. The ideal gas law forms the basis for all calculations.

1.2 This test method does not include all existing procedures appropriate for outgassing metal materials. The included procedures provided acceptable results for samples analyzed during an interlaboratory study. The investigator shall determine the appropriateness of listed procedures.

1.3 *Units*—With the exception of the values for density and the mass used to determine density, for which the use of the gram per cubic centimetre (g/cm^3) and gram (g) units is the longstanding industry practice, the values in SI units are to be regarded as 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.*

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]

B215 Practices for Sampling Metal Powders
B243 Terminology of Powder Metallurgy
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions:*

[1] This test method 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.

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

3.1.1 Refer to Terminology B243 for additional definitions relating to metal powders.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *density, n*—the mass per unit volume of a material.

3.2.2 *density, skeletal, n*—the ratio of mass of discrete pieces of solid material to the sum of the volumes of the solid material in the pieces and closed pores within the pieces.

3.2.3 *outgassing, n*—the evolution of gas from a material in a vacuum or inert gas flow, at or above ambient temperature.

3.2.4 *skeletal volume, n*—the sum of the volumes of the solid material in the pieces and closed pores within the pieces.

4. Summary of Test Method

4.1 An appropriately sized sample (to provide at least the minimum skeletal volume required for reliable results for the instrument or apparatus used) is outgassed under appropriate conditions prior to analysis.

4.2 The sample is weighed to nearest 0.0001 g. It is important to use an analytical balance to determine the sample mass. The pycnometer measures the total displaced skeletal volume of the sample under analysis. The sample mass is then used to calculate the skeletal density of the metal. Any error in the sample mass will affect the calculated density. Some cleaning of the sample surface may take place inside the pycnometer. Therefore, it is best to reweigh the sample after analysis and use the final mass when calculating skeletal density.

4.3 Sample skeletal volume is determined a minimum of five times. Skeletal volume average and standard deviation are calculated using standard statistical methods.

4.4 Calculations are based on the ideal gas law, as required by the instrument being used for the determination. The assumption of ideal behavior is accepted as valid at analytical temperatures and pressures. For instruments designed with two pressure chambers, one a sample compartment, and the other a gas expansion chamber, the equation for sample volume calculation takes the form:

$$V_{sample} = V_{cell} - V_{exp} \cdot \left(\frac{P_2}{P_1 - P_2} \right) \quad (1)$$

where:

V_{sample} = calculated sample volume,
V_{cell} = calibrated sample compartment volume,
V_{exp} = calibrated expansion chamber volume,
P_1 = measured gas pressure when only V_{cell} is filled with analysis gas, and
P_2 = measured gas pressure after expansion of the analysis gas into V_{exp}.

5. Significance and Use

5.1 Both suppliers and users of metals can benefit from knowledge of the skeletal density of these materials. Results of many intermediate and final processing steps are controlled by or related to skeletal density of the metal. In addition, the performance of many sintered or cast metal structures may be predicted from the skeletal density of the starting metal powder, for all or a portion of the finished piece.

6. Interferences

6.1 This test method can be used to determine the skeletal volume of a powder or solid only after the open pores have been emptied of any physically adsorbed molecules. Such adsorbed species (for example, water or volatile organic compounds) prevent entry of the gas probe molecules into the open porosity of the sample. Therefore, it is necessary to remove these adsorbed contaminants prior to pycnometry analysis. Generally, such outgassing is performed by evacuating or flushing the sample. Outgassing can be accelerated by using elevated temperatures, provided no irreversible sample changes occur. Typical minimum vacuum levels attained are 10^{-1} Pa. Typical flushing gases are those used for analysis. Outgassing is complete when duplicate skeletal volume

analyses produce results within expected instrument repeatability limits. Some commercial instruments include capabilities for automated evacuation, or flushing of the sample, or both. Elevated temperatures should not be used when outgassing samples inside the pycnometer.

6.2 This test method can be used to determine the volume of a sample whose pores have been deliberately filled with a second phase. In this case, removal of the second phase should be avoided. Vacuum degassing or flushing of the sample is not necessary in this case.

7. Apparatus

7.1 Commercial instruments are available from several manufacturers for the measurement of skeletal volume by gas pycnometry. Some instruments perform calculations of skeletal volume, or density, or both, upon completion of the analysis. Others require manual calculation of skeletal volume and density.

7.2 *Analytical Balance*—A balance readable to 0.0001 g, with a capacity adequate for the mass of the test portion, and capable of determining the mass of the test portion to the nearest 0.001 g.

8. Reagents and Materials

8.1 *Helium,* 99.999 mole percent, with the sum of N_2, O_2, argon, CO_2, hydrocarbons (as CH_4), and H_2O totaling less than 10 parts per million; dry and oil-free; cylinder, or other source of purified helium.

8.2 *Nitrogen,* 99.999 mole percent, with the sum of O_2, argon, CO_2, hydrocarbons (as CH_4), and H_2O totaling less than 10 parts per million; dry and oil-free; cylinder, or other source of purified nitrogen.

8.3 *Other High Purity Gas,* dry and oil-free; cylinder, or other source of gas, if other gas is to be used as the analysis or flushing gas. The actual composition of the gas shall be known.

9. Hazards

9.1 Precautions applying to the use of compressed gases should be observed.

10. Sampling, Test Specimens, and Test Units

10.1 No specific instructions are given. Nevertheless, it is important that the test portion being analyzed represent the larger bulk sample from which it is taken. The bulk sample should be homogenized before any sampling takes place. Best results are obtained when a flowing bulk material is temporarily diverted into a collector for an appropriate time. It is better to sample the entire flow for a short time than to sample a portion of the flow for a longer time. Collecting several small test portions and combining them improves the reliability of the sampling process. Rotating rifflers are available that satisfy these requirements. Refer to Practices B215 for information on the use of a chute sample splitter.

10.2 While there is no specific requirement for the test specimen size, result reliability increases as the percentage of the pycnometer sample capacity used increases. To this end, it is recommended that the test specimen have a skeletal volume greater than or equal to 10 % of the pycnometer sample capacity.

11. Calibration and Standardization

11.1 Follow manufacturer's instructions for calibration and operational verification of the instrument.

12. Conditioning

12.1 Weigh (to nearest 0.0001 g) a clean, empty sample holder. Record the empty sample holder mass.

12.2 Add sample aliquant to empty sample holder. Sample quantity should be sufficient to satisfy minimum skeletal volume as required by manufacturer. Weigh (to nearest 0.0001 g) and record sample and sample holder mass.