



APPLICATION NOTE 182

PARTICLE SIZE FOR PROCESS AND QUALITY CONTROL OF REFRACTORY METAL POWDERS

Refractory metals, particularly tungsten and molybdenum, are often produced in powder form by chemical reduction from ores and oxides, using high-temperature furnaces in a reducing atmosphere, usually hydrogen. Because of their high melting points, these materials do not lend themselves well to conventional melt-and-cast metallurgical processes. Powder metallurgical techniques – consolidation by mechanical pressing and sintering to reduce or eliminate porosity – have been used to produce solid bodies and parts for high-strength, high-temperature applications. In recent years, metal injection molding (MIM) and additive manufacturing (MAM) processes are being increasingly used to consolidate the powders and produce complex metal shapes, with very little machining necessary.

In all of these consolidation and shaping processes, the particle size of the powders is critical. Because of its simplicity and short analysis time, an air permeability technique for estimating the particle

size has been used to control powder production processes and assess the quality of the powders. The technique measures the pressure of air transmitted through a packed bed of powder under a specified input pressure; the transmitted pressure is then related to the specific surface area of the powder, from which an average particle size is calculated.

The air permeability technique is quick and simple, requiring only an amount of powder numerically equal to its density in g/cm^3 , thus using one cubic centimeter of solid material. The sample can be weighed, packed, and analyzed in a matter of minutes, so the method is often used to control process conditions: Samples are taken periodically from the process furnaces, their particle size measured, and process conditions, such as temperature, atmosphere concentration, and speed through the furnace, are adjusted to produce the desired final particle size, according to the particle size specifications set by the supplier and powder purchaser.

For many years, the refractory metals industry had depended on an instrument called the Fisher SubSieve Sizer (FSSS) for this particle size estimate, using ASTM Standard Test Method B330. However, that instrument is not very precise due to its estimation of pressures and packing factors by “eyeball” estimates of packed sample volumes and standpipe manometer pressures; and the Fisher SubSieve Sizer is no longer available or supported.

The Micromeritics® Sub-Sieve AutoSizer II particle size, surface area, and powder bed porosity analyzer has been developed to replace the Fisher SubSieve Sizer, exhibiting much better precision because of its use of traceable, calibrated pressure transducers and precise sample height measurement (Figures 1 and 2). Procedures for the use of the Sub-Sieve AutoSizer II are now included in ASTM Standards B330 (for refractory metals), C721 (for certain ceramic powders), and E2980 (a general standard applying to nearly all classes of powder materials).



Figure 1. Micromeritics Sub-Sieve AutoSizer II Particle Size, Surface Area, and Powder Bed Porosity Analyzer

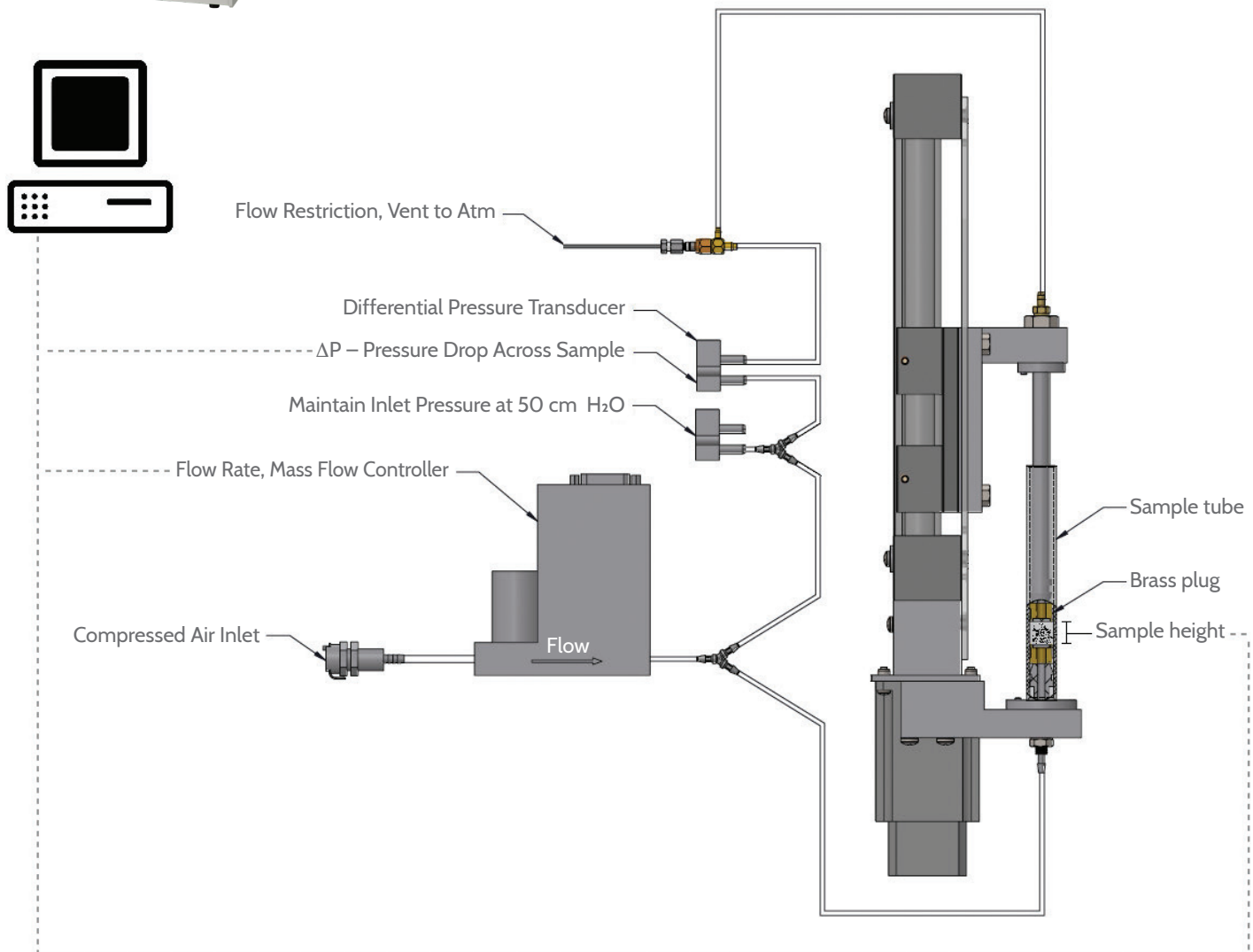


Figure 2. Micromeritics Sub-Sieve AutoSizer II Schematic

An example of the Sub-Sieve AutoSizer's greater precision can be seen in Figure 3, which is the result of measurements on several tungsten powders on both the Fisher SubSieve Sizer and the Micromeritics Sub-Sieve AutoSizer II. The figure shows the variability of the measurements based on a measure of repeatability, defined as the range of variability of repeated measurements of the same material, on the same instrument, by the same operator. The variability of the Sub-Sieve AutoSizer II measurement is considerably lower over the entire range of measurement.

A more dramatic example of the Sub-Sieve AutoSizer II greater precision can be seen

in Figure 4, the result of measurements on the same powders as in Figure 3, but performed on different instruments. Here the reproducibility is defined as the variability of measurements performed on different instruments, often by different operators in different laboratories. Having the supplier and purchaser of powder materials agree on particle size measurements, within narrow limits, has always been the goal for setting specifications for these materials. Figure 4 shows that the Sub-Sieve AutoSizer II gives more reasonable limits for agreement; keeping within the lower limits of the Sub-Sieve AutoSizer II will avoid serious conflicts, while the larger variability of the FSSS often can exceed the specification limits, leading to much confusion and disagreement.

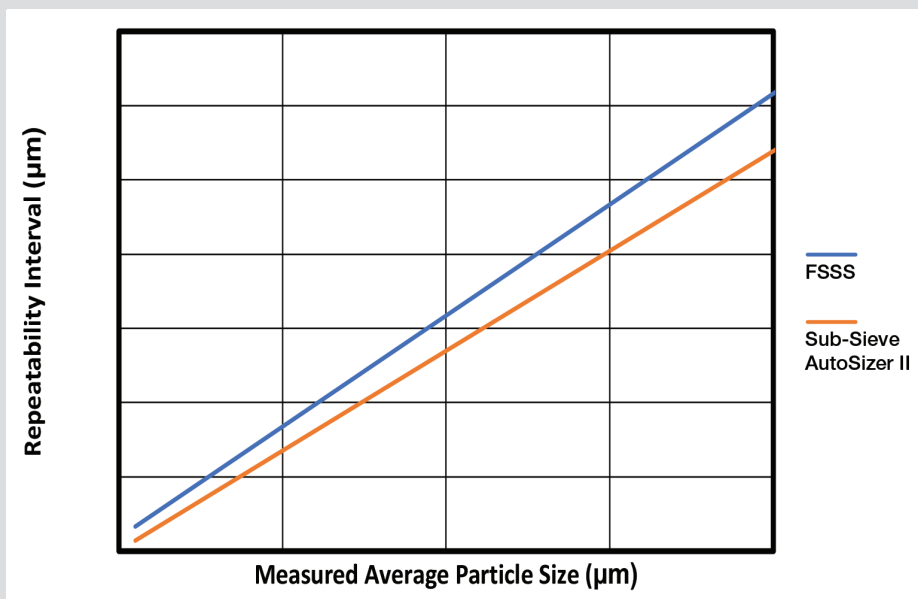


Figure 3. Repeatability Comparison

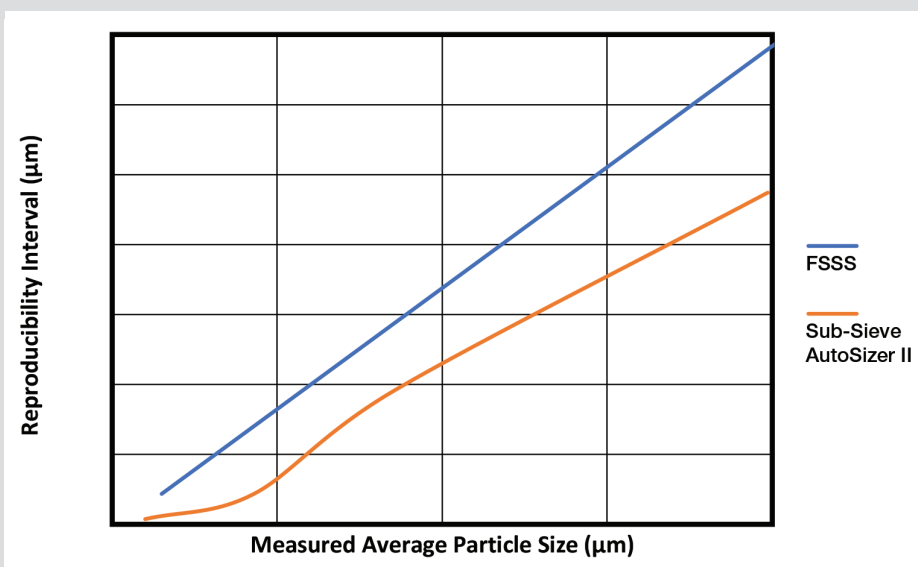


Figure 4. Reproducibility Comparison

Key Features and Benefits

- Guided step-by-step procedure allows quick set-up and reduces user errors
- Fisher Mapping
 - Calibration with FSSS data on the same material ensures the Micromeritics Sub-Sieve AutoSizer II particle size, surface area, and powder bed porosity analyzer can produce the same Fisher Number Size
 - Seamless transition from the manual FSSS system to the Sub-Sieve AutoSizer II system without changing powder specifications
- High level of user flexibility as the unit can be used via touchscreen or a computer through a web browser, regardless of PC operating system
- Uses high quality mass flow controller (MFC) and pressure sensors to give the highest level of air flow control and sensitivity for monitoring small changes in powder bed porosity, specific surface area, and particle size
- Provides a quick check of the relative fineness of a powder ideal for in-process quality control

Using the Sub-Sieve AutoSizer II air permeability method of particle size estimation results in significant analysis time savings compared to other particle size measurement techniques, which require more elaborate, time-consuming sample preparation and dispersion methods. Samples can be analyzed quickly, without holding up production processes. Metal powder production processes are thus more tightly controlled, resulting in improved quality control and fewer false rejections of in-spec material. And the Sub-Sieve AutoSizer II is very simple to use, requiring very little operator training and education.

Because of the Sub-Sieve AutoSizer II much better precision, refractory metals producers can now better control the particle size of their powders, resulting in significantly less rework, and powder users can have confidence in their specifications, decreasing rejection of good material, and lowering the incidence of using out-of-spec powders. The ease and speed of analysis of the Micromeritics Sub-Sieve AutoSizer II particle size, surface area, and powder bed porosity analyzer results in higher material throughput, with very little delay caused by analysis time.