AIR PERMEABILITY: A NEW (OLD) TECHNIQUE FOR PARTICLE SIZE MEASUREMENT

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Abstract:
The permeability of air through a packed powder bed has been used for many years to measure particle size, via pressure measurements converted to surface area and subsequent conversion to particle size. Instruments using this method have been used extensively in the refractory metals industry due to their fast and easy operation.

Older instruments using this technique suffer from poor precision due to uncertain control of compaction forces and the lack of accurate pressure measurement. Thus, older instruments have fallen out of use in recent years, and are no longer available or supported. Newer instruments have now surfaced that provide precise control of compaction forces and accurate pressure measurement, exhibiting much better precision.

This presentation describes the air permeability method and compares the precision of the newer instruments with older ones, outlining how the newer instruments can be useful. Comparison with other particle size analysis methods is also presented.

Introduction:
For many years, the permeability of air through a bed of packed powder has been used to measure powder specific surface area, converting the measured surface area into a particle size based on assumed geometry and material density. Indeed, the refractory metals industry has relied on air permeability measurements of particle size* for over 50 years, because of the quick and easy operation of instruments using this technique. In fact, many specifications of metal and carbide particle size have been written based on these measurements. Problems have arisen, however, due to the poor precision of the instruments used, where material that is measured to be within the particle size specification limits is actually out of spec, and vice versa. Recent developments in instrument technology, as described below, may be able to minimize these problems.

It should be noted that no measurement technique actually measures the particle size of a powder, which is almost always a distribution of a range of particle sizes, in various shapes. All measurement methods rely on some other characteristic that can be related to particle size, and usually express their results in terms of an "equivalent spherical diameter" based on those characteristics. Thus, many standard test methods for particle size contain the following cautionary caveat[1] :

> *Reported particle size measurement is a function of both the actual dimension and/or shape factor as well as the particular physical or chemical properties of the particle being measured. Caution is required when comparing data from instruments operating on different physical or chemical parameters or with different particle size measurement ranges. Sample acquisition, handling, and preparation can also affect reported particle size results.*

Nevertheless, these "particle size" measurements can be very useful in a relative sense for estimating the particle size of many powder materials, including metal powders and their compounds like carbides and oxides. The measurement methods can be the basis for particle size specifications when the precision and variability of the measurements are taken into account, if the variability is not too large.

Particle Size Measurement by Air Permeability:
The air permeability method of particle size measurement has the advantage of being fast and easy to perform, taking just a few minutes without a great deal of sample preparation. In this method, a quantity of powder equal to 1 cm^3 of actual solid material (sample mass numerically equal to the density of the material) is packed to a specified force in a tube of known inside diameter. The porosity of the powder bed is then measured by means of the height of the powder column, thereby measuring the total (apparent) volume of the powder bed and comparing that to the 1 cm^3 of material in the tube. Air is then passed through the powder bed at a specified pressure, and the transmitted pressure is measured. The specific surface area of the powder is then determined using

the Kozeny-Carman equation[2] , which relates the surface area to the porosity and the pressure drop through the compacted powder. The "average particle size" can then be determined from a simple relation of surface area to particle size:

$$d = 6 / \rho S \qquad (1)$$

where:

d = the average particle diameter in µm
ρ = the density of the powder material in g/cm^3
S = the specific surface area of the powder material in m^2/g, and
6 = a factor which converts the units of d to µm.

Old Method:
The older methods of air permeability particle size measurement, typified by the Fisher Sub-Sieve Sizer† instrument (Figure 1), use analog estimation techniques to arrive at an average particle size.

Figure 1: Fisher Sub-Sieve Sizer[3]

The packing force in this instrument is applied using a "needle-scale" torque wrench, or one that is "clickable", stopping and releasing the force when the specified torque is reached. The input pressure is controlled by means of an observed bubbling rate in a water standpipe at the back of the instrument (viewed through the hole at the upper left of the instrument).

Figure 2 shows a closer look at the chart on the Fisher Sub-Sieve Sizer:

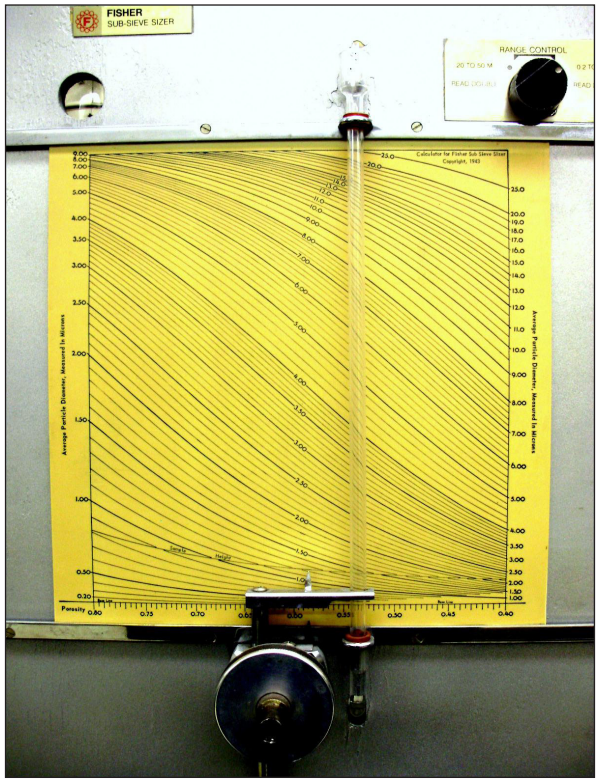

Figure 2: Fisher Sub-Sieve Sizer Particle Size Chart[3]

The chart shows curves representing the calculation of the average particle diameter via the KozenyCarman equation and Equation (1). The sample height is also read from the chart by aligning the point of the arrow on the metal crossbar with the nearly-horizontal "Sample Height" line near the bottom, by sliding the chart to the left or right. The average particle diameter (often called the "Fisher Number") is then read from the chart by aligning the metal crossbar with

† The Fisher Sub-Sieve Sizer is no longer commercially available, nor is it supported with parts and service. Some instruments are still being used, especially in the refractory metals industry.

the bottom of the water meniscus in the glass standpipe (a measure of the transmitted pressure) and reading the line closest to the pointer tip (or interpolating between lines).

It is quite obvious that poor precision can arise from all these "eyeball" estimations:

1. The packing force is unreliable because of the variability of operating and reading the torque wrench.
2. The bubble rate is not a very precise measure of pressure.
3. The sample height, and thus the porosity, is very difficult to estimate - just a slight difference in aligning the pointer with the sample height line of the chart can result in a large difference in porosity. That shift then affects the position of the chart lines in relation to the pressure standpipe and the pointer when measuring the transmitted pressure.
4. It is very difficult to locate the bottom of the meniscus in the standpipe.
5. It is very difficult to read the position of the pointer with respect to the chart lines, depending greatly on the angle of view, the eyesight of the viewer, and the skill of the operator in interpolation.

New Method

The new method of air permeability particle size measurement is performed by an instrument called the Subsieve AutoSizer (Figure 3).

Figure 3: Subsieve AutoSizer (SAS)

This new instrument makes use of all-digital control and measurement:

1. The packing force is controlled by calibrated pressure transducers.
2. The sample height is measured by the precise position of a piston that does the compaction, thus accurately determining the porosity.
3. The input pressure is accurately and precisely controlled, also by calibrated pressure transducers.
4. The transmitted air pressure is measured accurately and precisely by calibrated, traceable pressure transducers.
5. The Kozeny-Carmen equation is used directly to calculate the surface area from the porosity and transmitted pressure.
6. Equation (1) is used to directly calculate the average particle diameter.

Precision:

Because of the more precise control and measurement capabilities of the newer method, its test variability would be expected to be lower. Figure 4 shows a comparison of the new method's repeatability precision with that of the old method. There it can be seen that the new method does indeed show a lower repeatability interval[5] – a measure of the single-instrument, single-operator variability of the measurement[6,7]- across the entire range of measured particle sizes.

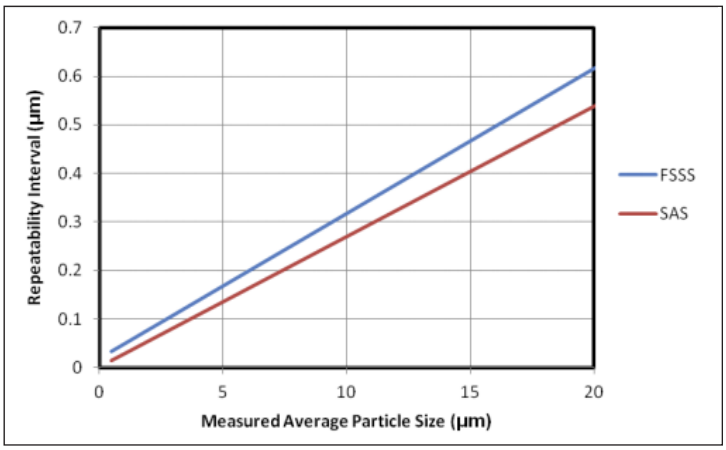

Figure 4: Repeatability Comparison[5,8]

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Figure 5 shows a similar comparison with regard to reproducibility – a measure of the between instruments, between-laboratories variability[6,7]. Here the reproducibility interval is clearly lower for the new method.

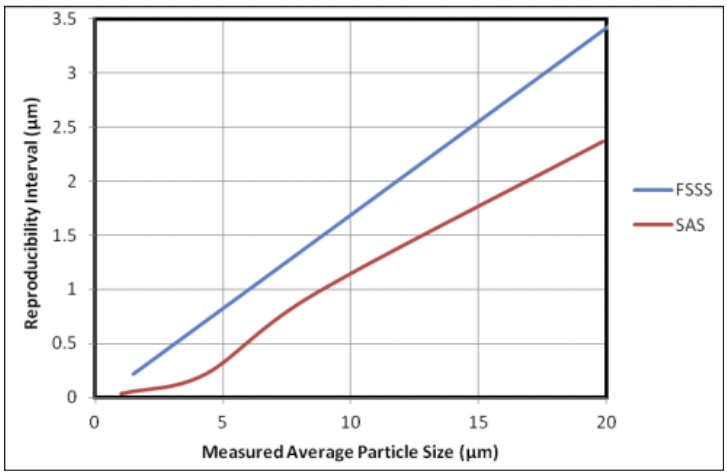

Figure 5: Reproducibility Comparison[5,10]

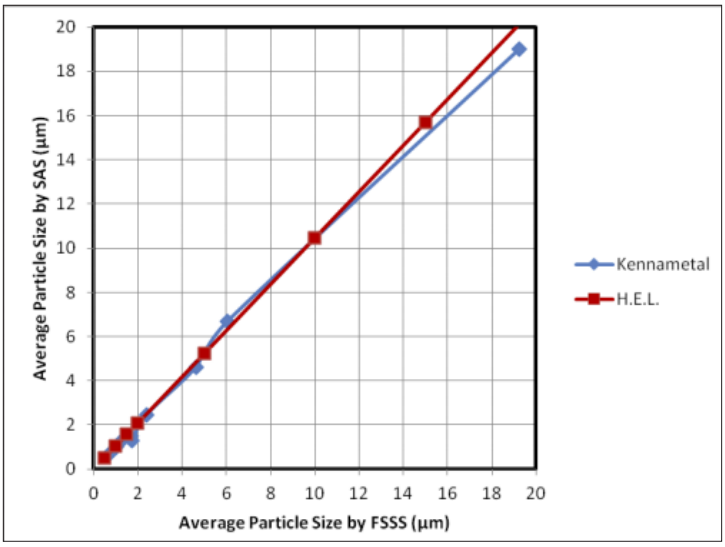

Figure 6: Comparison of Results from Old (FSSS) and New (SAS) Methods[8,9]

In Figure 6, a comparison of particle size results shows a nearly 1:1 relationship between the old and new methods in two different sets of data, indicating that the new method is a suitable replacement for the old with regard to measured average particle size.

Comparison With Other Methods:
Figure 7 shows a preliminary comparison of the new method with laser diffraction particle size analysis. Although the absolute values of particle size differ, the correlation is quite good. Note that the correlation is logarithmic, not surprising as particle size distributions measured by laser diffraction tend to be log normal distributions; thus, a technique that measures only an average particle size might be expected to produce results that are logarithmically related to the laser diffraction distribution.

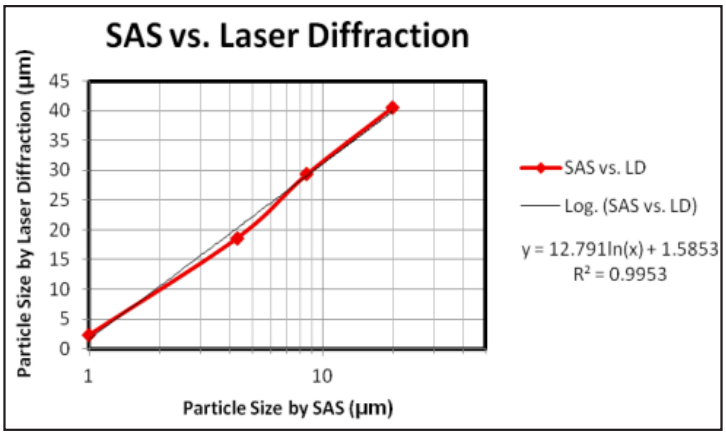

Figure 7: Comparison of the New Method with Laser Diffraction Particle Size Analysis[10]

The differences here serve to point out what was mentioned earlier: that different particle size measurement methods, based on different physical and chemical principles, can produce different results. A likely reason for the large differences here is related to dispersion of the powder: This preliminary comparison was done on tungsten powder, which has a tendency to bind finer particles together into relatively large, strongly-bound, multi-particle agglomerates. Standard procedure when analyzing these materials for primary particle size is to gently mill the powders to break up the agglomerates11, followed by ultrasonic treatment in a surfactant solution to keep the primary particles dispersed12. This procedure was not followed in this preliminary comparison.

Summary and Conclusion:
Table 1 summarizes how the intermediate measurements are determined and their method of application. There it is quite obvious that the new method should be superior to the old in precise and accurate application of forces and pressures, and in obtaining more reliable results. The new method's superior precision is borne out by Figures 4 and 5.

Measurementw Parameters	Old Method	New Method
Packing Force	Needle-Scale Torque Wrench	Calibrated Digital Pressure Transducers
Input Pressure	Visually-Timed Bubble Rate	Calibrated Digital Pressure Transducers
Sample Height	Visual Alignment with Sample Height Curve on Chart	Position of Piston
Transmitted Pressure	Visually Estimated Level of Meniscus in Standpipe	Calibrated Digital Pressure Transducers
Particle Size	Visual Alignment and Interpolation of Curves on Chart	Direct Calculation

Table I: Determination and Application of Intermediate Measurement Parameters

Since the introduction of the new method, several standard test methods have been revised and written to include the new technique:

- MPIF Standard 32, Methods for Estimating Average Particle Size of Metal Powders Using Air Permeability[13]
- ASTM B330, Standard Test Methods for Estimating Average Particle Size of Metal Powders and Related Compounds Using Air Permeability[5]
- ASTM C721, Standard Test Methods for Estimating Average Particle Size of Alumina and Silica Powders by Air Permeability[14]
- ASTM E2980, Standard Test Methods for Estimating Average Particle Size of Powders Using Air Permeability[15]
- ISO 10070, Metallic powders – Determination of envelope-specific surface area from measurements of the permeability to air of a powder bed under steady-state flow conditions[16]

MPIF 32, ASTM B330, and ASTM C721 now include the new method, but continue to include the old method, as several of the older instruments are still in use throughout the world. ASTM E2980 is a general standard that specifies only the new method, and as such can be used for many different classes of materials, including organic materials. ISO 10070 is currently in the process of revision, and will continue to include the old method, as well as some even older methods that are probably no longer used.

The new air permeability technique has been shown to be a viable and more precise method for estimating the particle size of powder materials. Although the method cannot give any indication of the full particle size distribution of a powder, it can be useful for assessing the relative particle sizes of a particular powder material produced in a broad range of sizes; for example, tungsten and tungsten carbide. The new air permeability technique can be used for many different classes of materials, such as ceramics, pigments, and even pharmaceutical drugs. The new method can be especially useful in production control and quality control of metal powders, where particle size specifications can now be written with confidence in their applicability, avoiding the confusion stemming from the poor precision of the old method.

Future Work:
Considerably more work needs to be done to further evaluate the precision of the new air permeability method on a wider range of materials (here only tungsten was used), on a broader range of particle sizes, and in multiple unrelated laboratories. Work also needs to be done to compare the new method with other particle size analysis techniques, including but not limited to laser diffraction, using better dispersion methods to yield particle size results that are not only correlatable, but closer in absolute size.

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