



PARTICLE SIZE AND SHAPE ANALYSIS OF POWDERED METALS

Metal powders are highly engineered materials used to manufacture precision metal parts with a wide range of performance characteristics. Virtually any metal can be made into a powder through processes including atomization, electrolysis, chemical reduction, and size comminution (pulverization). The choice of production method is determined by cost analysis, raw material supply, and the particle size and shape distributions most beneficial to performance. The metal powder can then be used to fabricate parts from simple single level shapes to complex multi level shapes. The technique and study of these processes is known as powder metallurgy, a field of study well acquainted with particle characterization.

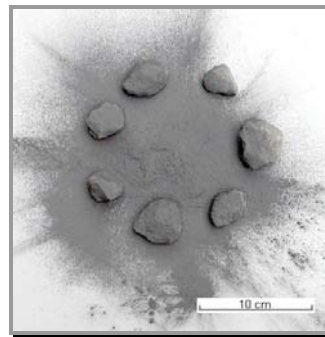
Effect of Particle Size and Shape

The size and shape distribution of the metal particles impacts powder behavior during die filling, compaction, and sintering, and therefore influences the physical properties of the parts created. Laser diffraction is now the most common particle sizing technique for powdered metals as it is for most all micronized particle measurement. Image analysis instruments are growing in popularity, however, as they can quantify both the particle size and shape of the powdered metals.

Spherical, or otherwise rounded particles, are preferred for reasons of powder flow and improved compaction leading to structurally sound parts. Larger, more spherical particles reduce inter-particle friction – a major concern with powder metallurgists.

The current marketplace uses powders with median diameters between 0.8 – 3.0 μ m as that range represents the best price-performance ratio¹. Smaller, nanoscale powders prove too costly to buy and use although they exhibit improved hardness and strength. Larger particles, on the other hand, may offer significantly reduced product performance with little to no savings in raw material cost. Particle size and shape affect bulk flow rate (tied to the density of finished parts) and the amount of material delivered to press-and-sinter machinery (tied to throughput and product consistency).

Powder Production



The most widely used method for creating metal powders is atomization where the raw material is melted and then broken into smaller particles. After the raw material is melted in a furnace it flows into a tundish which supplies a constant flow of metal into an atomization chamber. The metal stream exits the tundish and collides with a high velocity stream of water, air, or inert gas. The molten metal stream disintegrates into fine droplets that solidify as they fall through the atomization chamber.

An electrolysis process uses electricity to remove impurities and create a fine, pure metals powder. Electricity moves the soon-to-be powdered metal from the anode (made of the metal) to the cathode, covering the cathode in a film which is then washed and dried to make the metal powder. A variety of chemical reactions reduce metal into elemental powders. A common process liberates metals from their oxide forms through the use of reducing agents that attach to the oxygen in the oxide and render the metal powder. Simple size reduction



Applications Note

Powdered Metals

via mechanical comminution is used to reduce the size of large particles to the desired range. As implied previously, every powdering process maintains certain advantages and disadvantages - including a tendency towards creating particle size distributions and shapes of a specific kind – which can impact final product performance and predictability.

Parts Production

Powder metal is used to create a wide variety of parts for a range of industries. The two most popular techniques currently used to consolidate and form metal powder into parts are metal injection molding and sintering. The powder is typically injected into a mold or passed through a die producing a weakly cohesive structure close to the dimensions of the manufactured part. A combination of pressure, temperature, and setting time then finishes the part production.



Particle Characterization

Physical properties important to metal powder processing and performance include particle size distribution, shape and inter-particle friction. Powder flow and compaction are highly dependent on the friction between the particles. The most common way to quantify inter-particle friction is by measuring the angle of repose: the angle formed by pouring the powder through a funnel onto a flat surface – see Figure 1. Higher angles indicate greater friction while smaller angles suggest less friction.

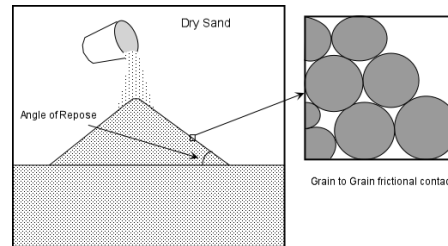


Figure 1: Angle of Repose example for solid particles

Particle size analysis of metal powders has been performed using various techniques through the years including microscope, sieves, aerodynamic time of flight, and laser diffraction. Laser diffraction has now replaced sieve analysis as the most popular technique for most particle size distribution measurements owing to faster measurement, greater ease of use, and improved return on investment.

Laser diffraction instruments measure the intensity and angle of light after interaction with the particle and transform this information into the particle size distribution. The [LA-950 Particle Size Analyzer](#) features the fastest measurement time on the market (less than 1 minute), widest measurement range (10nm to 3mm), and most flexible accessory set with samplers to handle dry powdered metals or wet dispersions.

ASTM B822 - 02 Standard Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering¹ provides guidance on the use of laser diffraction for measuring metal powders. The ASTM standard notes the requirement for a representative sample – suggesting a micro-sample splitter to minimize sampling errors. When measuring the powder in a liquid suspension it is important to create a stable dispersion².

Automated optical microscopy offers a valuable advantage over laser diffraction: shape characterization of micronized powders. Intrinsic properties of the metal may preferentially drive particle formation towards one of several shapes which effect material behavior during processing and final product performance. The [PSA300 Image Analyzer](#) allows the user to easily classify grades of material according to shape.

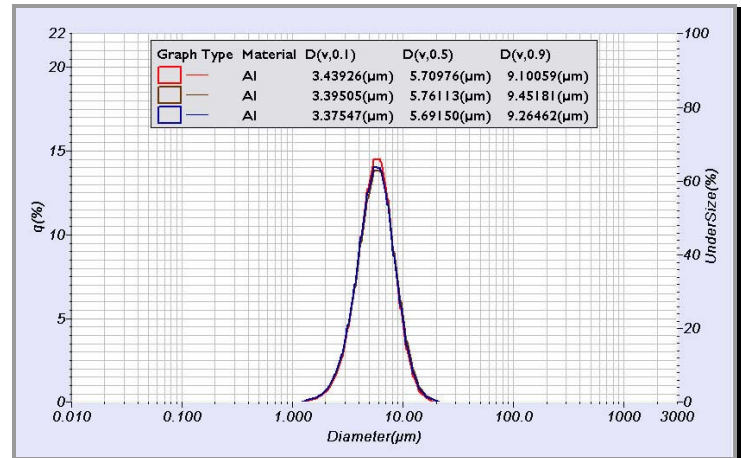
**Experimental Results**

All of the measurement examples presented here are materials used in press-and-sinter processes. Particle size and shape produce a noticeable effect on the production process as well as final product characteristics such as hardness, strength, porosity, etc.

Example 1 : Aluminum Precision

An aluminum alloy possessing a surface modification making wet dispersion unsuitable was tested instead as a dry dispersion using the [LA-950 PowderJet Dry Feeder](#). Three consecutive measurements of the aluminum powder were obtained from individual samplings and the result overlay is displayed to the right. Such a high degree of precision is made possible through the use of state of the art optics and a revolutionary feedback loop to control the mass flow rate through the measurement zone. Coefficient of variation is automatically calculated in the LA-950 software so the User can easily monitor repeatability and reproducibility.

Verifying that the powder has been produced in a narrow size distribution with median diameter at a desired value guarantees predictable material behavior and consistent final product performance.



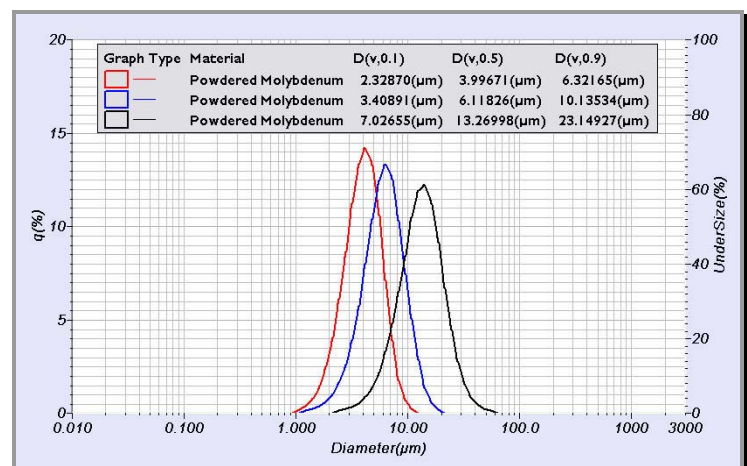
| Material | Test or Assay. | D(v,0.1) | D(v,0.5) | D(v,0.9) |
|--------------------------|----------------|---------------|---------------|---------------|
| Aluminum | 1 | 3.439 | 5.710 | 9.101 |
| Aluminum | 2 | 3.395 | 5.761 | 9.452 |
| Aluminum | 3 | 3.375 | 5.692 | 9.265 |
| Average | | 3.403 | 5.721 | 9.273 |
| Std. Dev. | | 0.033 | 0.036 | 0.176 |
| CV (%) | | 0.962 | 0.626 | 1.894 |
| Custom (10.0, 6.0, 10.0) | | PASSED | PASSED | PASSED |

Example 1: Three consecutive Aluminum powder measurements using the LA-950 PowderJet Dry Feeder. Note the ultra low CV% quantifying high precision.

Example 2 : Grades of Molybdenum

The changing product performance of varying powder particle sizes allows manufacturers to market and sell several grades of raw material appropriate to the consumer's application. The LA-950 measured three different sizes (and therefore different grades) of powdered molybdenum and the result overlay is presented in Example 3.

Verifying that the powder has been produced in a narrow size distribution with median diameter at a desired value guarantees predictable material behavior and consistent final product performance.



Example 2: Three different grades of Molybdenum powder accurately characterized through particle size analysis

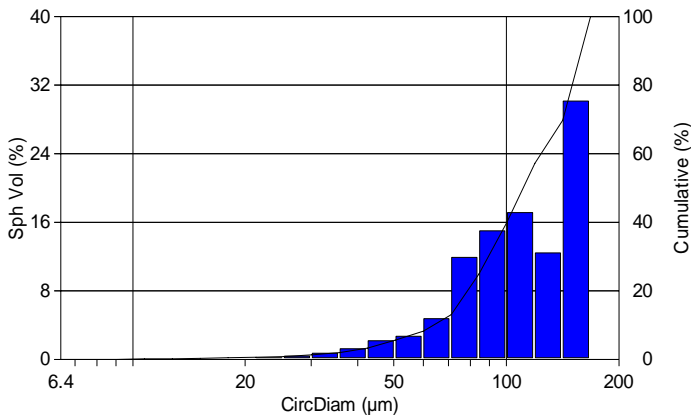


Applications Note

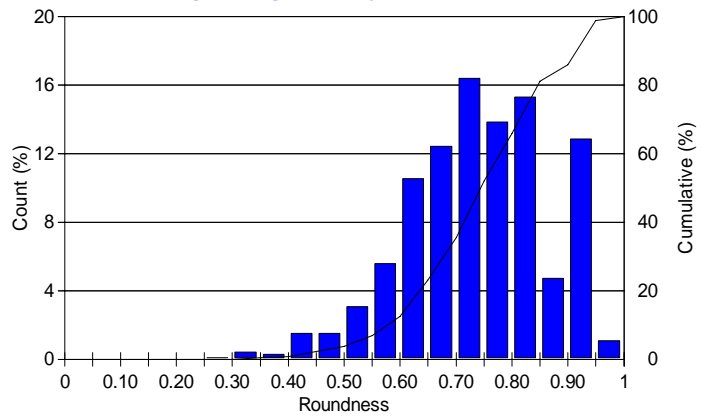
Powdered Metals

Example 3

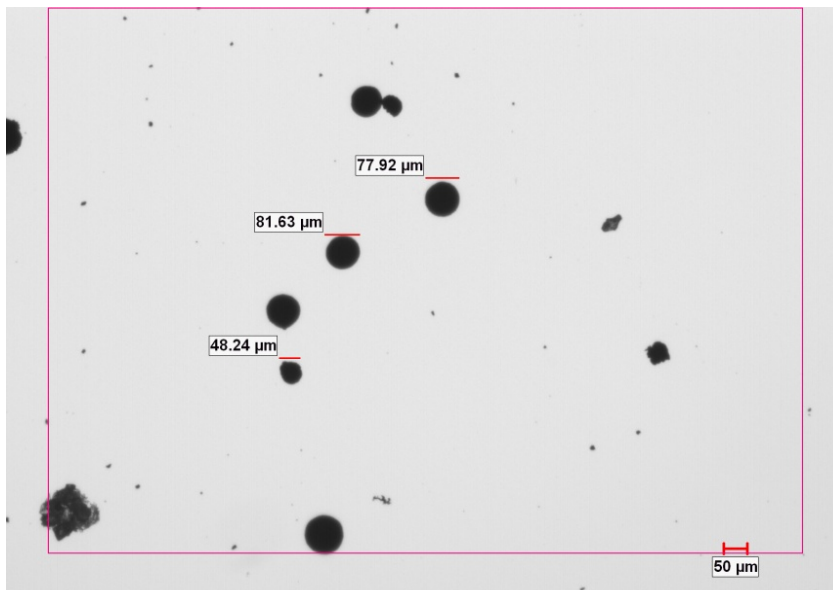
Nickel alloy powder was analyzed on the PSA300 image analysis system in order to characterize particle size and shape. Particle size distributions created using image analysis start as number distributions (i.e. Count), but can easily be converted into volume based distributions (i.e. Spherical Volume). Particle shape parameters such as roundness (graph on the right) are more often displayed on a number rather than volume basis. The sample analysis displayed here has identified this batch of material as low-quality (labeled as such because it would produce low-performance final parts) owing to small amounts of very round particles and a large amount of the largest particles. For more information regarding shape parameters refer to [TN150 Size and Shape Parameters](#) and [AN163 Particle Size and Shape Measurement of Micronized Powders using Image Analysis](#)^{3,4}.



| | |
|-----------|----------|
| Minimum: | 6.4 µm |
| Maximum: | 164.2 µm |
| Std Dev.: | 36.6 µm |
| D10: | 65.1 µm |
| D50: | 111.4 µm |
| D90: | 153.7 µm |



| | |
|-----------|-------|
| Minimum: | 0.29 |
| Maximum: | 0.97 |
| Mean: | 0.74 |
| Std Dev.: | 0.127 |
| D10: | 0.58 |
| D50: | 0.74 |
| D90: | 0.93 |



Example 3: Data and image captured using the PSA300 Image Analyzer. Ruler annotations included for reader's benefit



Conclusions

The accurate classification and confirmation of size and shape for powdered metals directly relates to quality control of the final press-and-sinter product. The long acquaintance of powder metallurgists with particle characterization technologies speaks to this critical correlation. Automated particle analysis for metal materials ranging from micronized powders to millimetres-wide final products provides powerful troubleshooting and process optimization tools. Many powdered metal manufacturers and consumers have successfully switched from older techniques to laser diffraction and image analysis particle characterization instruments and are experiencing the benefits of reduced expense and improved data quality.

References

1. Economics and the nano powder story that just won't sell, available at www.metal-powder.net/newsview/Sept07.asp
2. ASTM B822 - 02 Standard Test Method for Particle Size Distribution of Metal Powders and Related Compounds by Light Scattering, available at www.astm.org/Standards/B822.htm
3. See HORIBA Applications Note AN151 Wet Method Development for Laser Diffraction Particle Size Measurements, available at www.horibalab.com/
4. See HORIBA Technical Note TN150 Size and Shape Parameters Defined in the PSA300 available at www.horibalab.com/



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