

Quality Control for Metal Powders and Powder Metallurgy Processes Based on Particle Size and Morphology

The industries of Metal Powders manufacturing and Powder Metallurgy parts manufacturing came into modern day prominence about 70 years ago. Since then many different types of both metal powder and powdered parts manufacturing processes have been developed and are still practiced. The major processes used are described below.

Ceramic powders and ceramic parts manufacturing are similar industries which also use the same powder quality control technologies.

Metal Powders Processes: Direct Reduction (sponge iron), Gas Atomization, Liquid Atomization, and Centrifugal Atomization are all processes in use today.

Direct Reduction: Purified iron oxide ore, combined with a carbon source like coke, is heated to high temperatures in a rotary kiln. The product is sponge iron which is removed from excess solid carbon, ground, annealed (to remove excess contained carbon and oxygen) and reground for final use for manufactured parts.

Gas Atomization: A molten metal, which can be pure or alloyed metals, passes through an orifice under high pressure into a gas filled chamber where it cools and solidifies as it falls through the chamber. The powder is collected and annealed for subsequent parts manufacture.

Liquid Atomization: Similar to Gas Atomization, but the metal stream is hit by a high-pressure liquid spray which cools and solidifies the droplets more rapidly, resulting in smaller, less porous, cleaner particles with a wider size distribution compared to gas atomized powders. The product is then annealed.

Centrifugal Atomization: A rod of the metal to be powdered enters a chamber into a rotating spindle. An electric arc across the gap melts the end of the rod from which melted droplets are thrown into the surrounding chamber and solidified. This method can produce a much narrower size distribution than either atomization method.

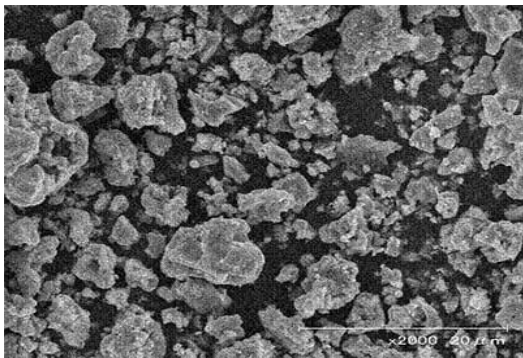


Figure 1. Direct Reduced Powder: Blocky, rough – results in high green strength after compaction.

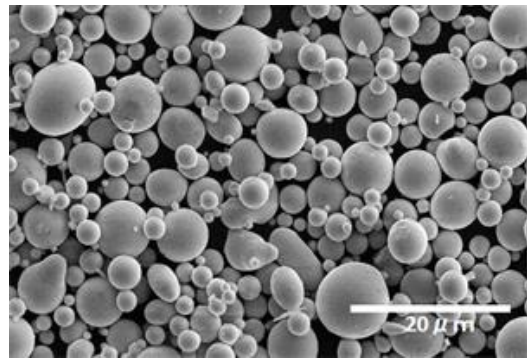


Figure 2. Liquid Atomized Powder: Spherical, smooth (required for Selective Laser Melting).

Commonly available metal powders include aluminum, bronze, metal carbides, chromium, cobalt, copper, hafnium, iron, molybdenum, nickel, niobium, platinum, rhenium, silicon, silver, tantalum, tungsten, vanadium any many different alloys of these.

Powder Metallurgy Parts Manufacturing: Powdered metal components are made from powdered metal using a variety of manufacturing techniques. These techniques include pressing and sintering, powder forging, hot isostatic pressing, electric current assisted sintering, metal injection molding, and selective laser melting.

Pressing & Sintering: The part is first compressed by die compaction at room temperature. In some cases, this is enough to create a finished part. In most cases, die compaction (pressing) is followed by sintering, at high enough temperatures for the particles to diffuse or coalesce together, not melt completely. The final part has some porosity to it, unlike a molten cast part. The lower the porosity of the final part, the higher the final strength and hardness.

Powder Forging: A pressed and sintered part is heated to high temperatures and then hot-forged. The final part has properties near those of wrought parts.

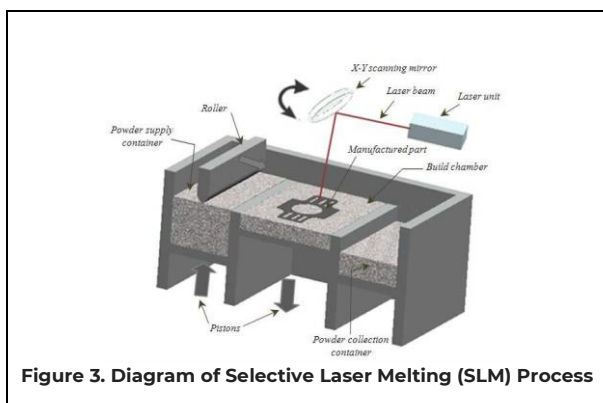
Hot Isostatic Pressing (HIP): Powder fills a mold which is evacuated and heated to high temperatures while it's subjected to external gas pressure up to 15,000psi. The final part has near wrought density and strength.

Electric Current Assisted Sintering (EACS): Similar to HIP except the heat is electrical localized and massive resistive heat, sometimes complemented by electric currents which can activate other mechanisms like surface oxide removal. The massive heat concentrates at particle surfaces, and the localized heat enhances plastic deformation during sintering.

Metal Injection Molding (MIM): MIM can produce more complex parts, because a mixture of the powder with a binder gives it fluid properties which can flow into small spaces and passages. The mixture gets compacted into a "green" part, after which the binder is removed, either thermally or chemically, to produce a "brown" part, which is sintered and shrinks to give a complex part with 97 – 99% density.

Selective Laser Melting (SLM): SLM is the latest, and considered by most to be the most advanced, PM process technology. (See diagram below) It uses a rotating mirror, which, by following a CAD pattern, directs a laser beam onto the top powder layer, melting the powder layer on top of the previous layer of the part. All particles not melted onto the part are scraped off while the next layer is loaded. Attempts are made to successfully re-use the un-melted particles for as many cycles as possible before it shows too much wear to meet size and shape criteria. It can take 10 pounds of metal powder to produce a 1-pound part if the left-behind powder can't be recycled.

Below right is an example of the complexity of a metal part that can be made by laser melting. Parts can also be very durable as they're made in one structural piece requiring no subassembly. They are also much more customizable on demand. One machine supplied with a number of different available CAD programs can be used to make individual custom parts on demand saving the large expense of tooling to produce a single part type.



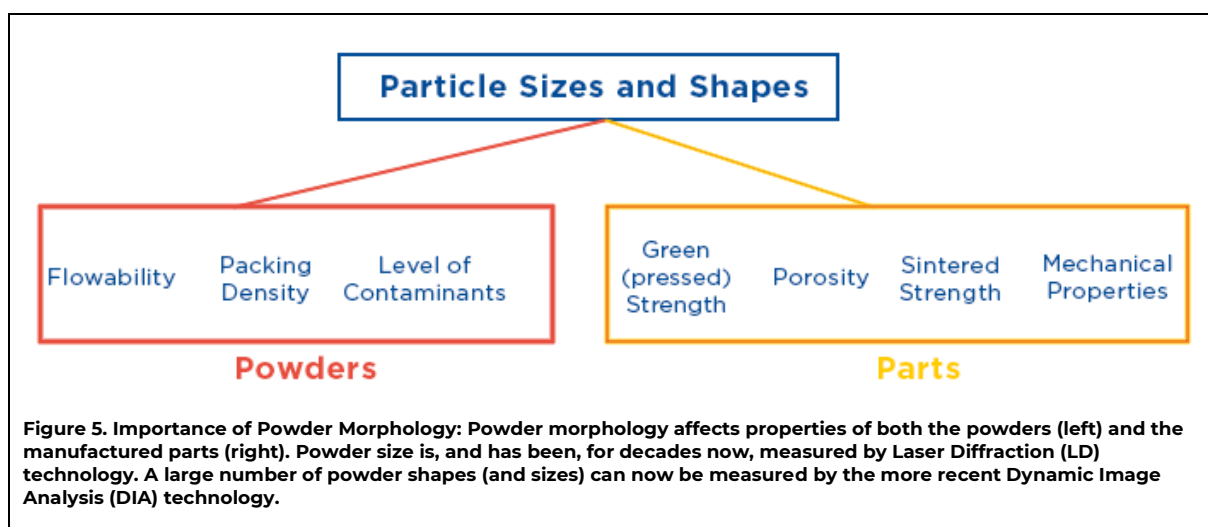
The size specifications for an atomized metal powder are often tighter than for most other parts manufacturing processes. The mean size might be smaller, and the distribution narrower for a complex part with very thin surfaces. Or a bimodal distribution might be called for to maximize the loose-packed density on the bed of the laser melter, which would maximize the density and strength and minimize voids of the finished part.

The individual particle shapes are now also very important to control. The particles must be highly spherical and smooth-surfaced for 1) good flowability and packing as the bed of the laser melter is recreated after

every layer is deposited, and 2) the most consistent structural integrity as the part is fused. And, as contaminants are detrimental in any metal powder, they're especially a problem in feed to laser melting, because even a single contaminant could cause a point defect in a very thin section of a part. Contaminants can be identified by image analysis if they are non-spherical, rough-surfaced, or translucent. They can also be quantified as a proportion of the sample by volume or number.

Recycling the metal powder means the powder will wear and pick up some contaminants on each recycle. So the recycle stream must be re-measured for both size and shape before re-use. When it goes out of spec it must be melted and atomized into quality powder again.

Quality Control for Metal Powders: Metal powders need to meet quality specifications of both the powder manufacturers (outgoing inspection) and the powder metallurgy parts manufacturers (incoming inspection). Basic powder morphology (sizes and shapes) is a specification in itself and influences all other specifications, depicted in the chart below.



Laser Diffraction (LD): Laser light hitting a stream of flowing powder is scattered at higher angles and lower intensities, the smaller the particle. Detectors at many angles around the sample stream measure the distribution of the scattered light, and an iterative algorithm calculates the size distribution which scattered it. Laser Diffraction has become the de facto standard method for QC size measurement in both the metal powders and the powder metallurgy industries.

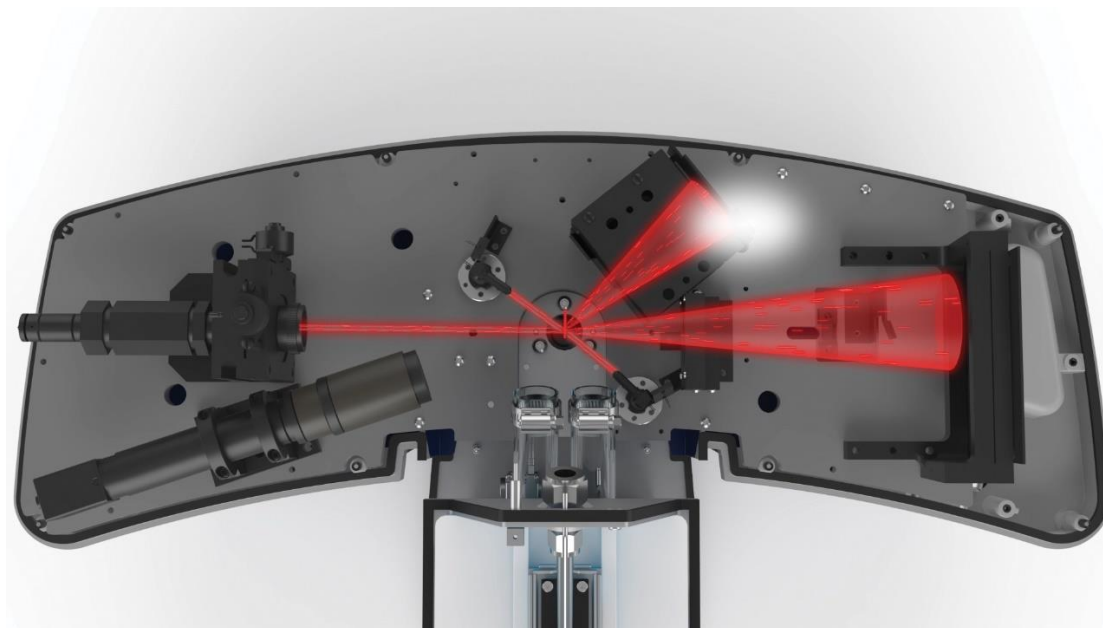


Figure 6. Diagram of Laser Diffraction (LD) Technology: The tri-laser diodes at different angles provide scattered light to the array detectors at angles from 0 to 165 degrees. The blue lasers, at lower wavelengths, detect the smallest particles more accurately.

Dynamic Image Analysis (DIA): Particles flow through a sample cell between a high-speed strobe light and a digital camera. A video file of the particle images is sent to a computer. All the analysis takes place on the recorded images. The size of the pixels is calibrated so all size and shape data are easily calculated and reported. The video image file is saved and can be re-measured under different Standard Operating Conditions (SOP).

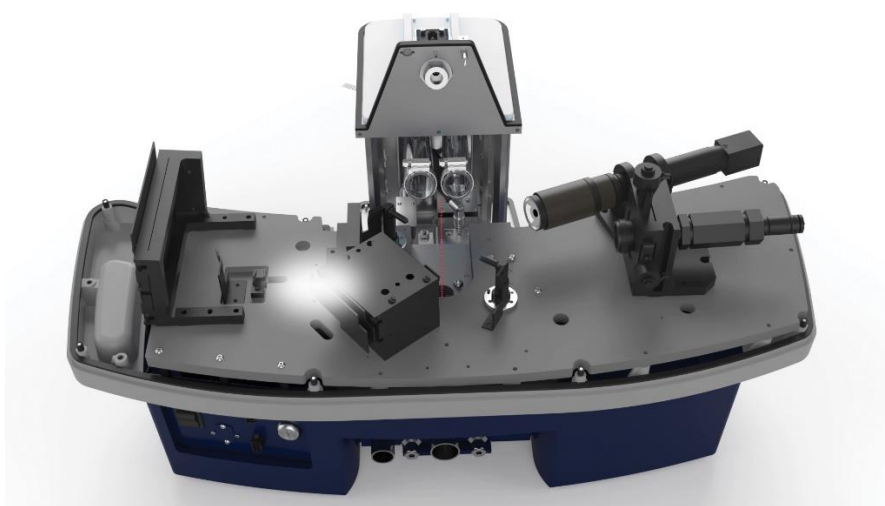


Figure 7. Diagram of Dynamic Image Analysis (DIA) Technology: Rapid strobe on left illuminates the sample cell. Particles flowing through cell are photographed by digital camera on the right. Video image output is recorded in image file in computer.

Microtrac Sync
Combination LD and DIA
Analyzer for Metal Powders



Figure 8. Synchronous Size and Shape Analyzer: Each measuring unit measures and reports all results simultaneously on the same sample.

The instrument pictured in Figure 8. measures one sample simultaneously using both the Laser Diffraction and Dynamic Image Analysis technologies.

They report all parameters as they were discussed previously. This is the only combination LD/DIA system commercially available today.

Summary

- Sizes and shapes (morphology) of metal powders need to be measured:
 - To meet suppliers and users' QC requirements
 - To identify/quantify off-spec and contaminant quantities for all processes
 - To monitor recycle streams in laser melting additive manufacturing
- LD is the size technology predominantly used in the metal powders/powder metallurgy industries for qc data
- DIA is the technology used for morphological data
- A combination LD/DIA system can now be used to make both measurements simultaneously on the same sample in a matter of minutes