Total Solutions in Particle Characterization

APPLICATION NOTE

# Applying Particle Size and Morphology Analysis for R&D and QC in the Additive Manufacturing Processes for Powder Metallurgy Parts

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Application Note

SL-AN-59 Revision A

Provided By:

Microtrac, Inc.

Particle Size Measuring Instrumentation

## Introduction

Metal Powders manufacturing and Powder Metallurgy (PM) parts manufacturing came into prominence about 70 years ago. Since then many different processes for both industries have been developed, with more and more technical sophistication and specialization. The advent of 3D printing technology, now labeled Additive Manufacturing, has brought about the latest PM manufacturing technology, Selective Laser Melting.

## Selective Laser Melting (SLM)

Selective Laser Melting 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, oxidation and agglomeration 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.

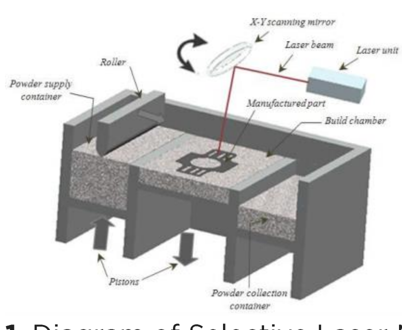


**Figure 1.** Diagram of Selective Laser Melting (SLM) Process

**Figure 2.** Complex part made by SLM

The size specifications for an atomized metal powder used in SLM 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 wider 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 also now 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.

Particle shapes can't be detected by size analysis, because they're independent of size. An elongated or rough-surfaced particle can exist in any of the size fractions in the distribution. It's necessary then to measure the complete morphology, sizes and shapes, of metal powders to control their quality. Specifications can be set on shape parameters to ensure not only the proper size distribution, but also the proper shape distributions for high-quality powder.

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 in SLM means the powder will wear and possibly 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 used in SLM**

The metal powder needs to meet quality specifications regarding size and shape. Size has been measured for decades by Laser Diffraction technology. Morphology, sizes and shapes, is now being measured by Dynamic Image Analysis technology.

**Laser Diffraction (LD):** Coherent 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.

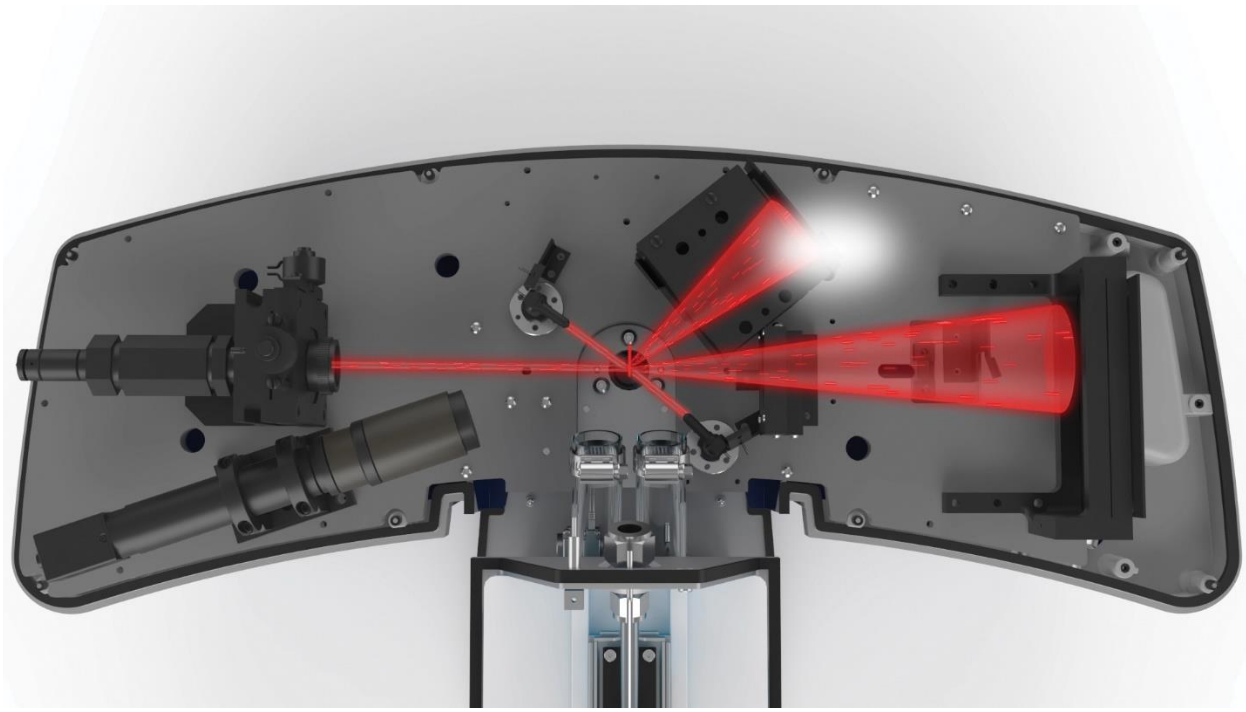

**Figure 3.** Diagram of Laser Diffraction (LD) technology: Three laser diodes at different angles provide scattered light to the array detectors at angles from 0 to over 160 degrees.

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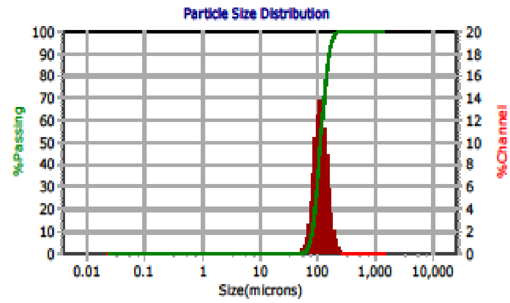
## - Particle Size Analysis -

### -Tabular Data -

| Size(um) | %Chan | %Pass | Size(um) | %Chan | %Pass | Size(um) | %Chan | %Pass | Size(um) | %Chan | %Pass |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1408 | 0.00 | 100.00 | 67.86 | 1.34 | 2.35 | 3.27 | 0.00 | 0.00 | 0.1580 | 0.00 | 0.00 |
| 1291 | 0.00 | 100.00 | 62.23 | 0.70 | 1.01 | 2.999 | 0.00 | 0.00 | 0.1450 | 0.00 | 0.00 |
| 1184 | 0.00 | 100.00 | 57.06 | 0.31 | 0.31 | 2.750 | 0.00 | 0.00 | 0.1330 | 0.00 | 0.00 |
| 1086 | 0.00 | 100.00 | 52.33 | 0.00 | 0.00 | 2.522 | 0.00 | 0.00 | 0.1220 | 0.00 | 0.00 |
| 995.6 | 0.00 | 100.00 | 47.98 | 0.00 | 0.00 | 2.312 | 0.00 | 0.00 | 0.1110 | 0.00 | 0.00 |
| 913.0 | 0.00 | 100.00 | 44.00 | 0.00 | 0.00 | 2.121 | 0.00 | 0.00 | 0.1020 | 0.00 | 0.00 |
| 837.2 | 0.00 | 100.00 | 40.35 | 0.00 | 0.00 | 1.945 | 0.00 | 0.00 | 0.0940 | 0.00 | 0.00 |
| 767.7 | 0.00 | 100.00 | 37.00 | 0.00 | 0.00 | 1.783 | 0.00 | 0.00 | 0.0860 | 0.00 | 0.00 |
| 704.0 | 0.00 | 100.00 | 33.93 | 0.00 | 0.00 | 1.635 | 0.00 | 0.00 | 0.0790 | 0.00 | 0.00 |
| 645.6 | 0.00 | 100.00 | 31.11 | 0.00 | 0.00 | 1.499 | 0.00 | 0.00 | 0.0720 | 0.00 | 0.00 |
| 592.0 | 0.00 | 100.00 | 28.53 | 0.00 | 0.00 | 1.375 | 0.00 | 0.00 | 0.0660 | 0.00 | 0.00 |
| 542.9 | 0.00 | 100.00 | 26.16 | 0.00 | 0.00 | 1.261 | 0.00 | 0.00 | 0.0610 | 0.00 | 0.00 |
| 497.8 | 0.00 | 100.00 | 23.99 | 0.00 | 0.00 | 1.156 | 0.00 | 0.00 | 0.0560 | 0.00 | 0.00 |
| 456.5 | 0.00 | 100.00 | 22.00 | 0.00 | 0.00 | 1.060 | 0.00 | 0.00 | 0.0510 | 0.00 | 0.00 |
| 418.6 | 0.00 | 100.00 | 20.17 | 0.00 | 0.00 | 0.972 | 0.00 | 0.00 | 0.0470 | 0.00 | 0.00 |
| 383.9 | 0.00 | 100.00 | 18.50 | 0.00 | 0.00 | 0.892 | 0.00 | 0.00 | 0.0430 | 0.00 | 0.00 |
| 352.0 | 0.00 | 100.00 | 16.96 | 0.00 | 0.00 | 0.818 | 0.00 | 0.00 | 0.0390 | 0.00 | 0.00 |
| 322.8 | 0.00 | 100.00 | 15.56 | 0.00 | 0.00 | 0.750 | 0.00 | 0.00 | 0.0360 | 0.00 | 0.00 |
| 296.0 | 0.00 | 100.00 | 14.27 | 0.00 | 0.00 | 0.688 | 0.00 | 0.00 | 0.0330 | 0.00 | 0.00 |
| 271.4 | 0.00 | 100.00 | 13.08 | 0.00 | 0.00 | 0.630 | 0.00 | 0.00 | 0.0300 | 0.00 | 0.00 |
| 248.9 | 0.26 | 100.00 | 12.00 | 0.00 | 0.00 | 0.578 | 0.00 | 0.00 | 0.02790 | 0.00 | 0.00 |
| 228.2 | 0.58 | 99.74 | 11.00 | 0.00 | 0.00 | 0.530 | 0.00 | 0.00 | 0.02550 | 0.00 | 0.00 |
| 209.3 | 1.09 | 99.16 | 10.09 | 0.00 | 0.00 | 0.486 | 0.00 | 0.00 | 0.02340 | 0.00 | 0.00 |
| 191.9 | 2.08 | 98.07 | 9.25 | 0.00 | 0.00 | 0.446 | 0.00 | 0.00 | | | |
| 176.0 | 3.69 | 95.99 | 8.48 | 0.00 | 0.00 | 0.409 | 0.00 | 0.00 | | | |
| 161.4 | 5.88 | 92.30 | 7.78 | 0.00 | 0.00 | 0.375 | 0.00 | 0.00 | | | |
| 148.0 | 8.77 | 86.42 | 7.13 | 0.00 | 0.00 | 0.344 | 0.00 | 0.00 | | | |
| 135.7 | 11.24 | 77.65 | 6.54 | 0.00 | 0.00 | 0.315 | 0.00 | 0.00 | | | |
| 124.5 | 13.28 | 66.41 | 6.00 | 0.00 | 0.00 | 0.2890 | 0.00 | 0.00 | | | |
| 114.1 | 13.69 | 53.13 | 5.50 | 0.00 | 0.00 | 0.2650 | 0.00 | 0.00 | | | |
| 104.7 | 12.48 | 39.44 | 5.04 | 0.00 | 0.00 | 0.2430 | 0.00 | 0.00 | | | |
| 95.96 | 10.28 | 26.96 | 4.62 | 0.00 | 0.00 | 0.2230 | 0.00 | 0.00 | | | |
| 88.00 | 7.14 | 16.68 | 4.24 | 0.00 | 0.00 | 0.2040 | 0.00 | 0.00 | | | |
| 80.70 | 4.62 | 9.54 | 3.89 | 0.00 | 0.00 | 0.1870 | 0.00 | 0.00 | | | |
| 74.00 | 2.57 | 4.92 | 3.57 | 0.00 | 0.00 | 0.1720 | 0.00 | 0.00 | | | |




### - Measurement Info -

| Title | |
|---|---|
| Microtrac | |
| Identifiers | |
| Metal Powder | |
| MT12595, File S2811 | |
| Database Record | 8 |
| Run Number | Avg Of 3 |
| Date | 5/5/2016 |
| Time | 9:39 AM |
| Acquired Date | 5/5/2016 |
| Acquired Time | 9:39 AM |
| Serial Number | S3501 |
| Sample Cell ID | 2067 |
| Calculated Data | |
| Above Residual | 0 |
| Below Residual | 0 |
| Loading Factor | 0.0697 |
| Transmission | 0.980 |
| RMS Residual | 1.113% |
| User Defined Calculations | |
| Name | Value |
| Recalculation Status | |
| DB-Meas : : Original : | |

### -SOP Info-

| MICROTRAC METAL POWDER | |
|---|---|
| Timing | |
| Setzero Time | 30 (sec) |
| Run Time | 30 (sec) |
| Number of Runs | 3 |
| Multi-Run Delay | 0 (min) |
| Delay First Meas. | Disabled |
| Analysis | |
| Metal Powder | |
| Refractive Index | N/A |
| Transparency | Absorb |
| Shape | Irregular |
| WATER | |
| Refractive Index | 1.33 |
| Options: | |
| Filter | Enabled |
| Analysis Gain | Default(2) |
| Analysis Mode | S3000/3500 |
| Perspective | |
| Progression | Geom 8 Root |
| Distribution | Volume |
| Upper Edge(um) | 1408 |
| Lower Edge(um) | 0.0215 |
| Residuals | Disabled |

| Summary | |
|---|---|
| Data | Value |
| MV(um): | 115.8 |
| MN(um): | 95.86 |
| MA(um): | 108.6 |
| CS: | 5.50E-02 |
| SD: | 28.44 |
| Mz: | 114.5 |
| σi: | 28.96 |
| Ski: | 0.1796 |
| Kg: | 1.039 |

| Percentiles | |
|---|---|
| %Tile | Size(um) |
| 10.00 | 81.21 |
| 20.00 | 90.70 |
| 30.00 | 98.09 |
| 40.00 | 105.1 |
| 50.00 | 111.9 |
| 60.00 | 119.2 |
| 70.00 | 127.8 |
| 80.00 | 138.7 |
| 90.00 | 155.4 |
| 95.00 | 171.4 |

| Peaks Summary | | |
|---|---|---|
| Dia(um) | Vol% | Width |
| 111.9 | 100 | 56.88 |

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11.0.0.5

- Notes -

-

| Sample System(Wet) | |
|---|---|
| Number of Rinses | 4 |
| Flow Rate (%) | 65% |
| Deaeration Cycles | 3 |

**Figure 4. Metal Powder Size Quality Control Report - Laser Diffraction Data:** Clockwise from upper left: Tabular Size Distribution Data, Graphical Distribution, Standard Operating Procedure, Measurement Information, Percentiles, Summary Data.

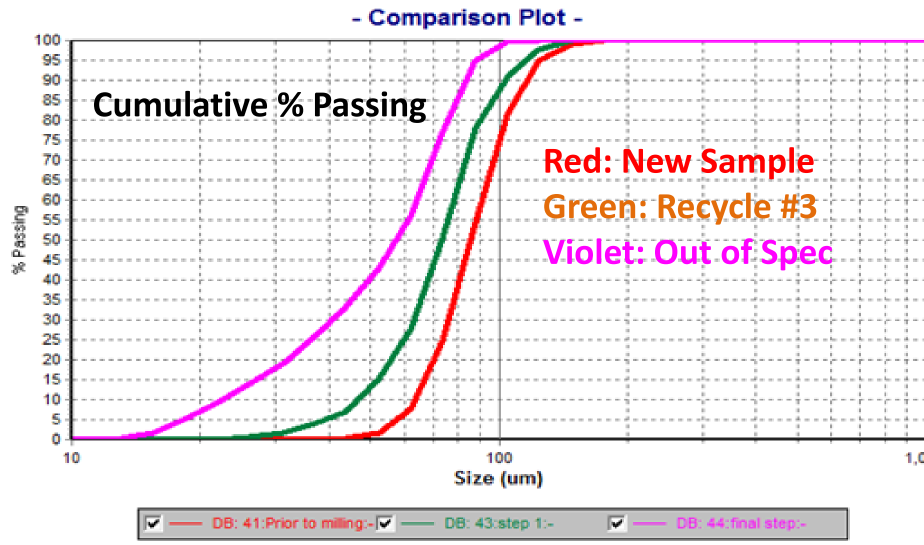


**Figure 5.** Comparison size distribution plots of series of recycled powder from LD software report. Individual tabular data, percentiles and summary data available in individual sample reports.

**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).

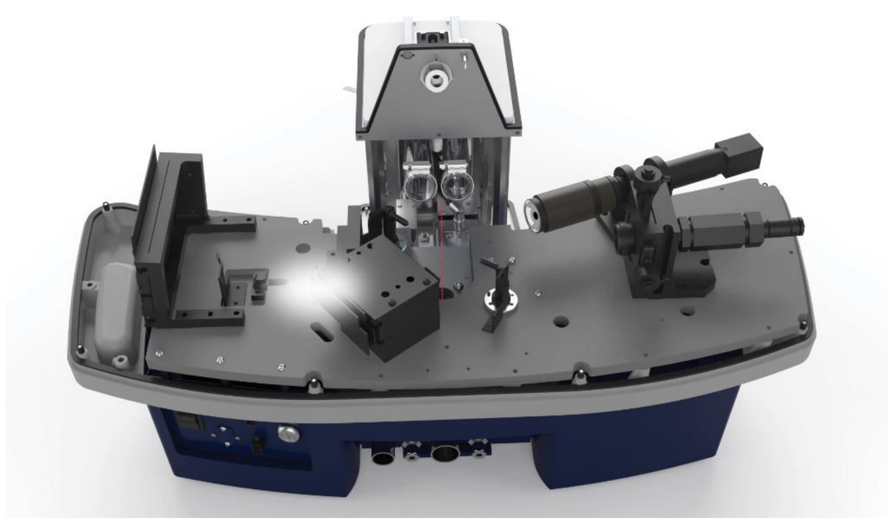

**Figure 6. Diagram of Dynamic Image Analysis (DIA) Technology:** Rapid strobe on left illuminates the sample cell. Particles flowing though cell are photographed by digital camera on the right. Video image output is recorded in image file in computer.

## Microtrac Sync

Combination LD and DIA
Analyzer for Metal Powders


**Figure 7. Synchronous Size and Shape Analyzer:** Each measuring unit measures and reports all results simultaneously on the same sample. The instrument pictured measures one sample using both the Laser Diffraction and Dynamic Image Analysis technologies. They report all size and shape parameters of the metal powder. This is the only combination LD/DIA system commercially available today.

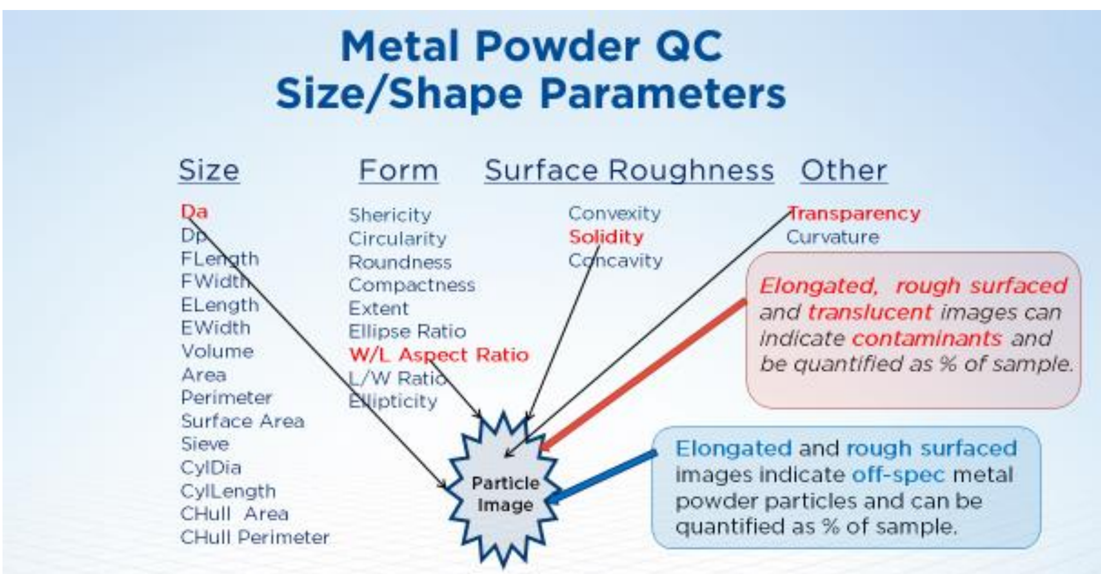


**Figure 8. Morphological Parameters Measured by DIA:** Size, Form, Surface Roughness and Intensity parameters are measured. Form parameters quantify the overall shape of particles, roughness parameters quantify the surface roughness, and the other parameters quantify the intensity and distribution of light coming through the particles from the strobe light.

The blue outline above of a particle image will be used as an example of parameters which can indicate off-spec or contaminant particles in a metal powder sample. The blue image shown above represents a particle which is elongated, as measured by W/L Aspect Ratio, rough-surfaced, as measured by Solidity, and translucent, as measured by Transparency. The shape parameters here are on a scale of 0 to 1. A W/L Aspect Ratio of 1 is a shape with equal width and length. A Solidity of 1 represents a perfectly smooth particle with no concave indentations. A Transparency of 1 is a perfectly transparent particle. No more than 2 or 3 parameters are ever needed to evaluate key shapes for most applications.  Any of these 3 parameters can indicate a contaminant. An opaque metal powder particle which is elongated or rough-surfaced can be out of specification regarding the limits set for W/L Aspect Ratio and Solidity.  Out-of-spec powder, or contaminants, can be identified and quantified as % by volume or number of the sample.

## Identifying Off-Spec Metal Powder

- *W/L Aspect Ratio*, 0 to 1.0, lower values identify elongated images
- *Solidity*, 0 to 1.0, lower values identify rough surfaced images
- *Transparency,* 0 to 1.0, 1 is completely transparent, maybe glass or plastic contaminant
- Images show powder particles fused during cooling following atomization

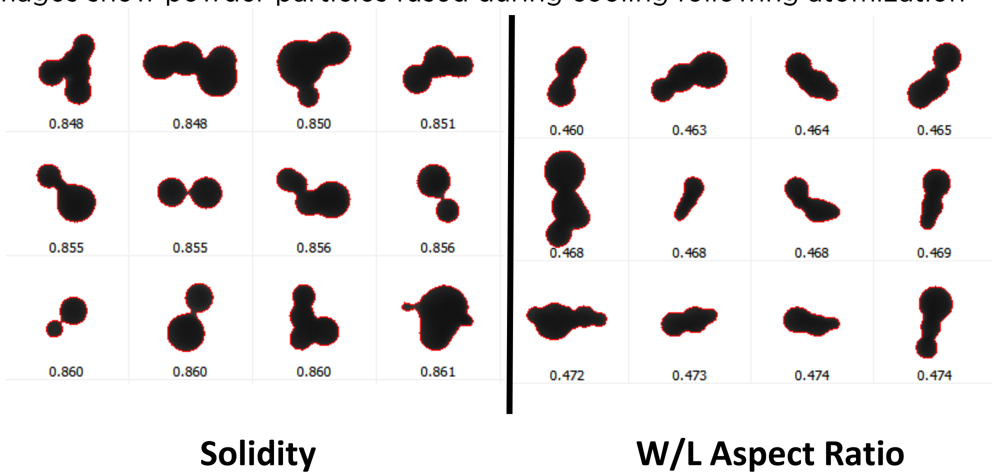


**Figure 9.** An example of how shape analysis can be used to identify and report off-spec powder or contaminants. Segments of a metal powder image file are shown above, sorted by the two parameters listed, indicating images with low Solidity and W/L Aspect Ratio.

Width/Length Aspect Ratio, on a scale of 0 to 1, lower values indicate more elongated images, representing powder with non-spherical shape. Solidity, 0 to 1, lower values identify images with rough surfaces. The Transparency parameter, on a scale of 0 to 1, 1 indicates a completely transparent particle. Transparency can identify and quantify contaminants like glass or plastic from packaging or handling. And contaminants in metal powder would typically have much lower W/L and Solidity values *vs* those that are simply out of spec.

The images above, with their W/L and Solidity values displayed, are obviously fused individual powder particles which formed during cooling following atomization. Fused groups of metal powder particles like these are detrimental to the part manufacturing process. They lower the packing density, increase the porosity and lower the green and sintered strength of more conventional parts, and can cause point structural defects in laser melted parts.

Image analysis provides the QC manager with all the tools needed to determine the specification limits of these shape parameters for a particular powder. Size and shape specifications for metal powders would depend on the part complexity, the process, the desired yield rate and other considerations.

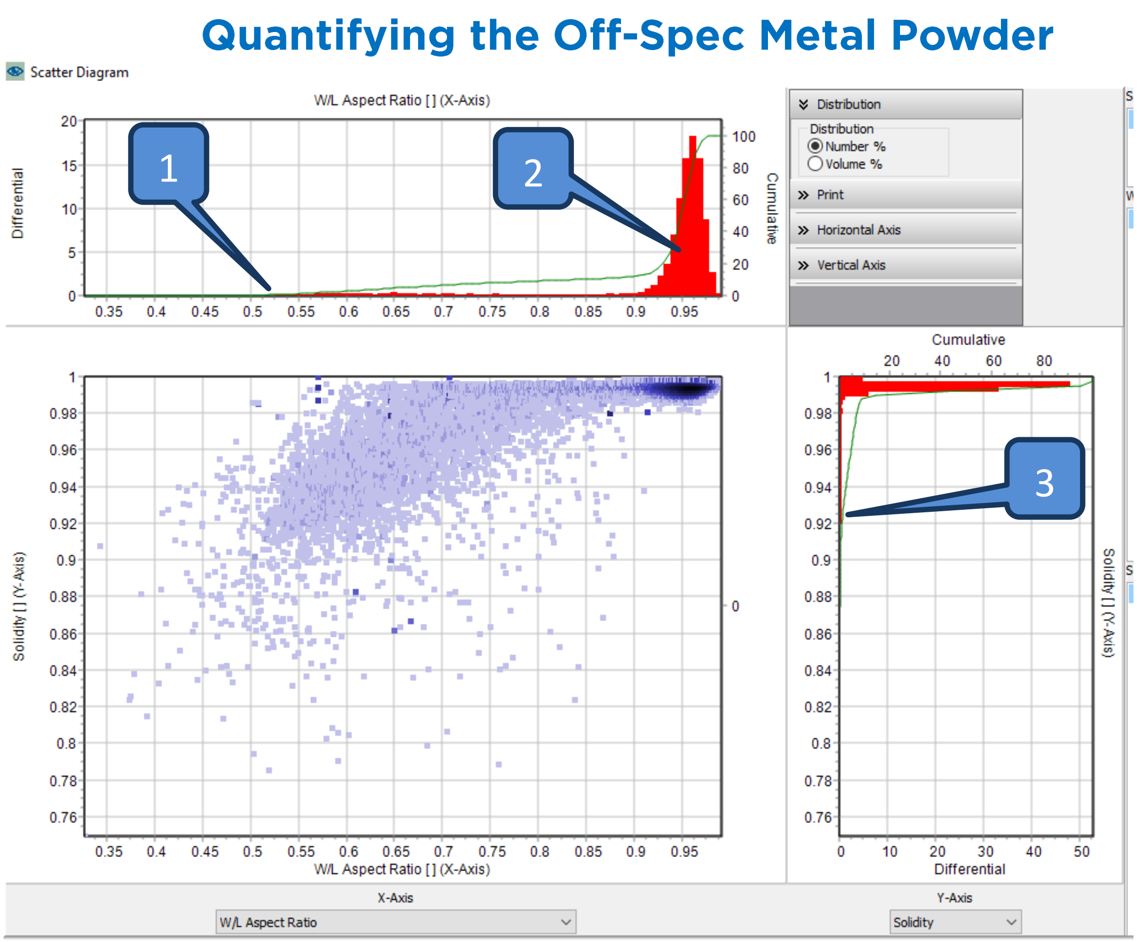


**Figure 10.** W/L vs Solidity shows W/L values down to about 0.5, very elongated (1)

But bulk of particles are above 0.9, very spherical as desired (2). Solidity is all above about 0.9, very smooth particles in entire sample (3).

The oblong particles can be easily identified and quantified using the View Particles Search feature or Filter feature to be discussed.

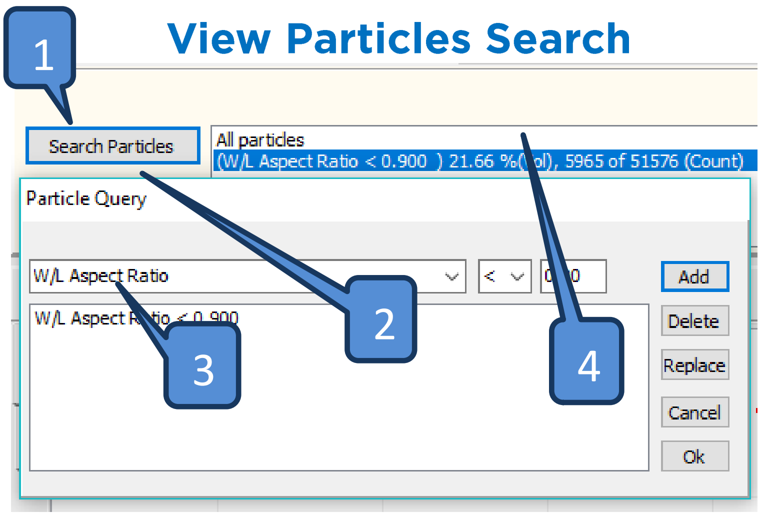


**Figure 11.** View Particles display. Search Particles button selected (1), brings up the Particle Query window below (2). Particle Query window allows setting up any number of limits for parameters. W/L particles less than 0.900 has been set (3), and quantified in result (4), 21.66 % Vol, 5965 of 51,576 or 11.6 % Count.

Here's a function in the View Particles display which can be used for isolating groups of images matching a user search criterion. We'll use it to search for the group of images which fall into the low W/L range we just identified in the Scatter Diagram. Select the Search Particles button, callout 1, upper left. That opens the Particle query window, callout 2. Here we chose W/L from a list of all parameters, and set limits of less a value of 0.900, callout 3, and initiated that search.
Callout 4 points to one result of that search which comes up in the Search Particles window – it reports that fraction of particles meeting the search criterion is 21.66 % by volume of the whole sample, and has a particle count of 5965 out of a total of 51,576 particle for a count or number % of 11.6. This group of particles were found to consist of fused individual metal powder particles, and their quantity might indicate a batch of powder which is out of spec with regard to shape.

## Automating the Query

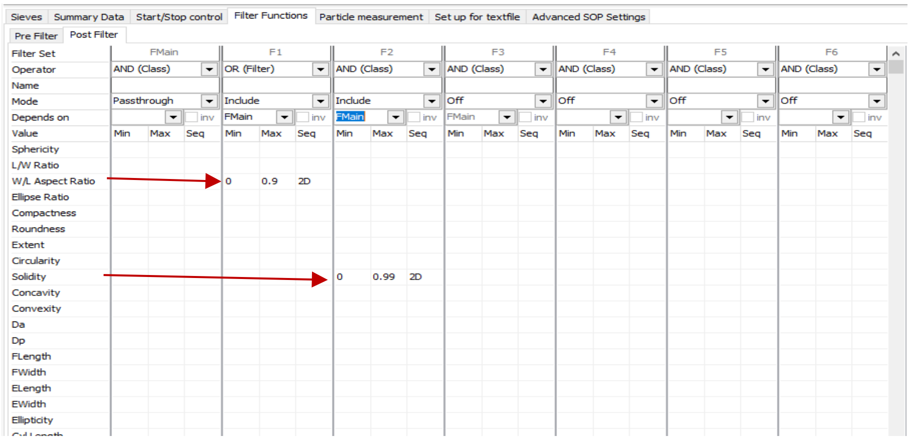


**Figure 12.** Up to six different search queries can be set up in this Filter Functions display and set in the SOP. Example here searches for W/L results 0 to 0.900, and Solidity 0 to 0.990

Up to six different searches which can isolate and quantify six different components in a sample can be programmed here and assigned to an SOP. Components can be contaminants in general, different types of materials that are purposely combined to create a desired mix of properties in a product, or any out-of-spec particles which didn't meet size or shape requirements. As long as these components are measured for those different sizes and shapes, they can be quantified and separately viewed, as individual sub-distributions, in one measurement.  When a search is automated and set in the SOP, the various filters can be selected to report all the quantified sub-distributions.

## X-Y Graph and Table

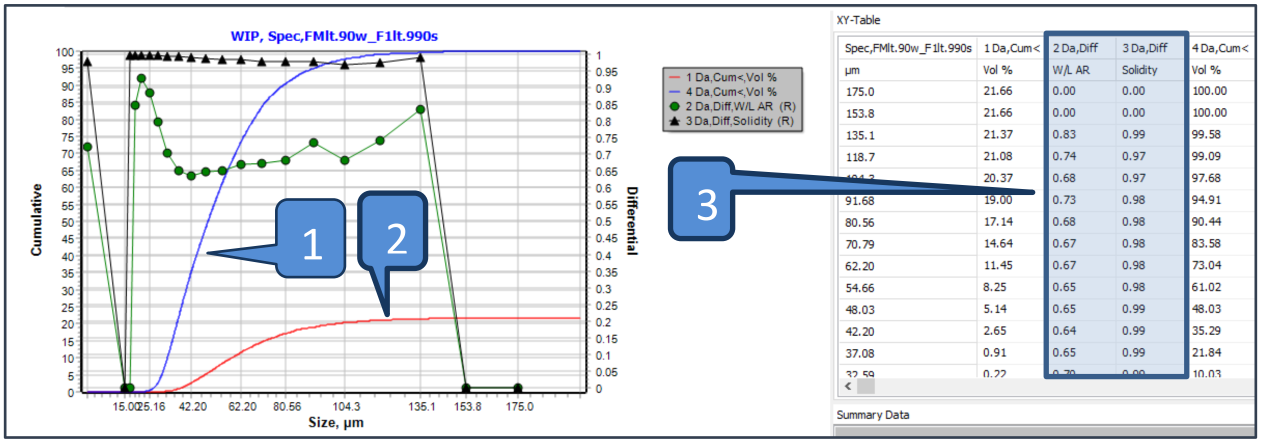


**Figure 13.** Graphical and Tabular results of W/L <0.900 vs Da size distribution and Solidity <0.99. Da size distribution for all particles as Cum % finer plot in blue (1) and only <0.9 W/L in red (2). W/L and Solidity plots and Table (3) show value of each shape for each size interval.

As a result of applying that SOP then, we can report this X-Y graph and Table. The blue curve (1) is a cumulative % finer curve of the area equivalent diameter, Da, for the entire sample. The red curve (2) is cum % finer for only those particles which meet the search criterion, or 21.66%, as was shown in the View Particles search result. The actual values of W/L and Solidity are shown as differential distributions, for each size fraction. And those values are listed in the Table (3).

## W/L Aspect Ratio < 0.900

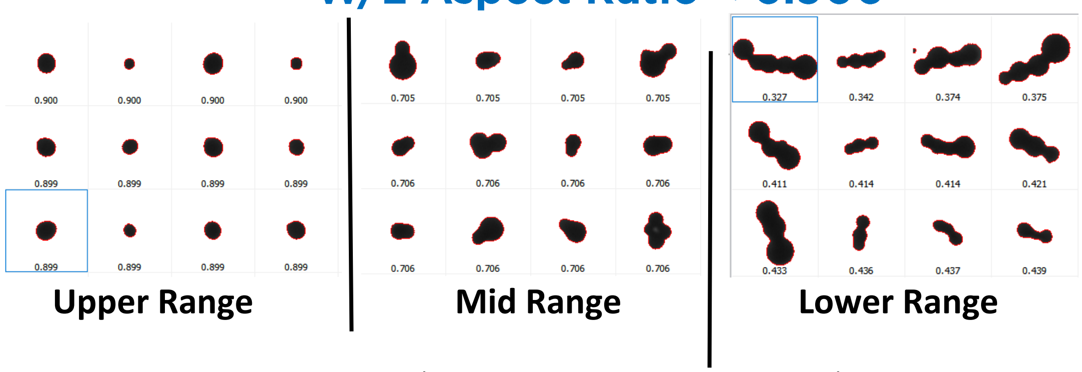


**Figure 14.** Selected images in W/L range less than 0.900. As W/L decreases, images show increasingly fused individual powder particles. QC departments can use images as guides for setting their specifications on shapes.

When those search results are reported, the image file in View Particles display shows only the particle images which match the search criterion. Here portions of that image file from that search result are displayed. From left to right, images in the upper range, then mid range, and finally the lower range of the elongated particles, where we see the particles becoming more and more elongated as the W/L ratio decreases. Once this analysis is complete, the QC manager has all the information needed to choose the specification limit for W/L parameter for this material. That limit is typically set by the customer using the powder to manufacture PM parts.

## Graphing Historical Results

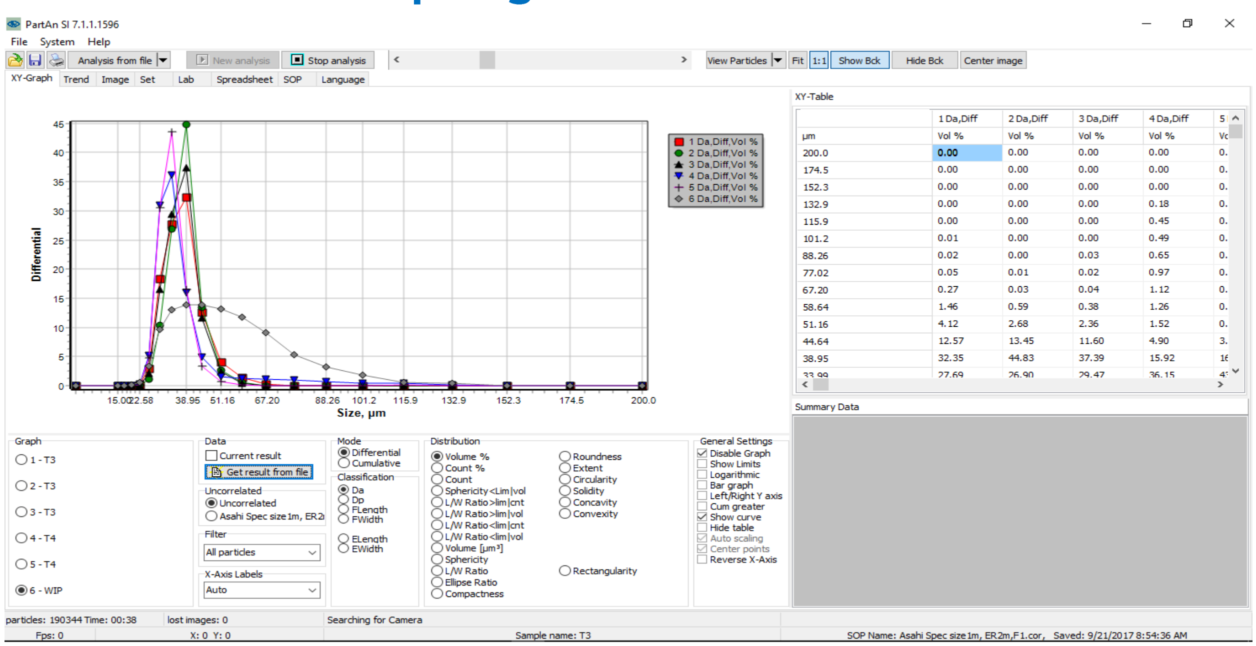


**Figure 15.** Six different results of previous measurements as Da differential Vol %. Any parameter can be chosen, sizes or shapes.

The results of historical records for one parameter can also be reported in the graph and table in the DIA software. Here six records reporting Da differential % by volume for metal powder samples are reported. Any parameter can be chosen, sizes or shapes.

## DIA Parameters Trend Charts


**Figure 16.** Four separate trend charts track any parameters. Multiple parameters on any charts, sizes or shapes.

Trend charts are a feature in the DIA software which allows the easy convenient tracking of any parameters from consecutively run samples. It's the perfect way for tracking key changes in sizes and shapes in the recycled streams in Laser Melting. Both size and shape parameters can be tracked here to indicate when any key parameter goes out of spec, and the stream can no longer be recycled for use. Control limits for the specs can be set on the charts for ease in decision making.

## On-Line Dynamic Image Analyzer

**Figure 17.** Measure recycle powder stream in real time on its way back to laser melter. Avoid recycling powder that has gone out-of-spec. Custom designed sampling system.


Pictured above is the only on-line Dynamic Image Analyzer commercially available for measuring metal powder process streams in real time. This analyzer can be used on the recycle stream to laser melting to report when it has gone out of spec or is contaminated with other particles.

**Summary**

- Sizes and Shapes (Morphology) of Metal Powders need to be measured:
  - To meet suppliers and users QC requirements
  - To identify/quantify contaminants and off-spec powder for all processes
  - To monitor recycle streams in Selective Laser Melting (SLM)
- Laser Diffraction (LD) is the size technology used in the industry for QC
- Dynamic Image Analysis (DIA) is the technology used for morphology
- A new instrument from Microtrac can now be used to make both measurements simultaneously on the same sample in a matter of minutes
- 30 Different Size and Shape Parameters are reported, and key powder parameters can be identified and quantified
- Real time on-line monitoring of the recycle stream to SLM can immediately detect off-spec feed.