

Comparing metal powders from different atomization processes using automated image analysis

Image analysis is an ideal tool for characterizing the particle size and shape distributions of atomized metal powders



PARTICLE SHAPE



PARTICLE SIZE

Introduction

The manufacture of complex shaped metal parts by Metal Injection Molding (MIM) is a growing industry. The shape and size of metal powders play an important role in determining process efficiency and properties of the final MIM components. Spherical powder particles are typically favored by MIM manufacturers looking to achieve best tolerances and properties in final components. Therefore, it is important to monitor atomized powder to ensure that particles of the desired shape and size are produced.

The metal injection molding process (MIM) is used in the manufacture of complex-shaped, high volume, low weight parts where intricate detail may be required along with accurate tolerance control. It involves 4 crucial steps which are outlined below:

1. Atomization of molten metal to form metal powders which are further processed by sieving and/or gas classification to obtain the appropriate particle size distribution. The powder is then mixed with thermoplastic binders to form pellets of feedstock ready for the next step.
2. Feedstock is injected into a mold or die to form 'green' metal injection molded parts.
3. The binder is removed from the 'green' part by solvent and/or thermal processes to leave a 'brown' metal part.
4. The 'brown' part undergoes a sintering process in a high temperature furnace where the metal particles fuse together. Particle size is important during this stage, but so is particle shape since spherical powders will have a higher packing density. This means more touching surfaces, faster sintering times and reduced shrinkage resulting in better dimensional control. Therefore, the

size and the shape of the original metal particles produced in step 1 will affect the final product and must be carefully controlled.

This application note describes how atomized powders with similar particle size distributions produced by two different atomizing processes, can have very different shape properties and how such parameters can be assessed using automated image analysis.

Materials and Methods

Two powder batches of stainless steel alloy 17-4PH (Sandvik Osprey Ltd, -32mm grade) produced by different atomization techniques were analysed using a Morphologi® (Malvern Panalytical) to determine both size and shape. The Morphologi is an automated particle characterization instrument which disperses the powder particles on a glass substrate using an integrated dry powder disperser. The instrument then captures images of individual particles by scanning the sample underneath the microscope optics and then performs image analysis to determine size and shape parameters. Sample A was prepared by a process of gas atomization whereas sample B was prepared by water atomization. An aliquot of each sample was automatically dispersed and analysed according to a standard operating procedure (SOP) which contained all the hardware and software variables for the measurement. Dispersion using the Morphologi's integrated sample dispersion unit (SDU) involved the application of a pulse of positive pressure on the sample at 4 bar for 8 ms then allowing the sample to settle for 240 s. A 20x objective was used for analysis combined with a 3 plane z-stack. Images of touching particles were excluded from the results. More than 55,000 particles were characterized for each sample. The samples dispersed well as can be seen from the field of view images shown in Figure 1.

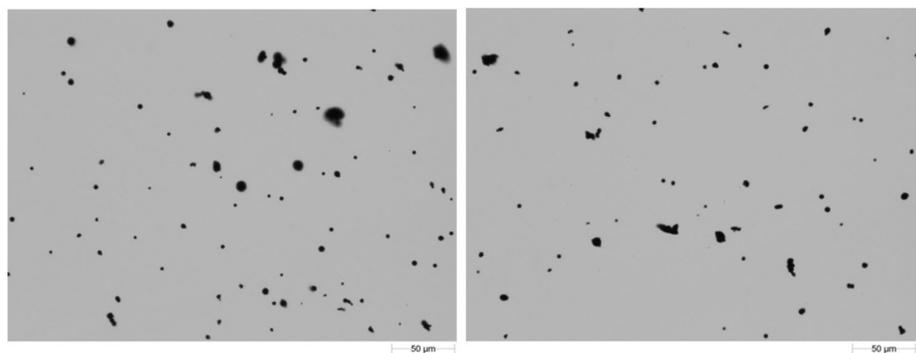


Figure 1: Field of view images of the two dispersed atomized metal samples with Sample A (left) and Sample B (right)

Results and Discussion

The results were compared using the Morphologi software's comparison tool. The parameter variability chart (Figure 2) shows how each of the morphological parameters assessed varies between the two samples.

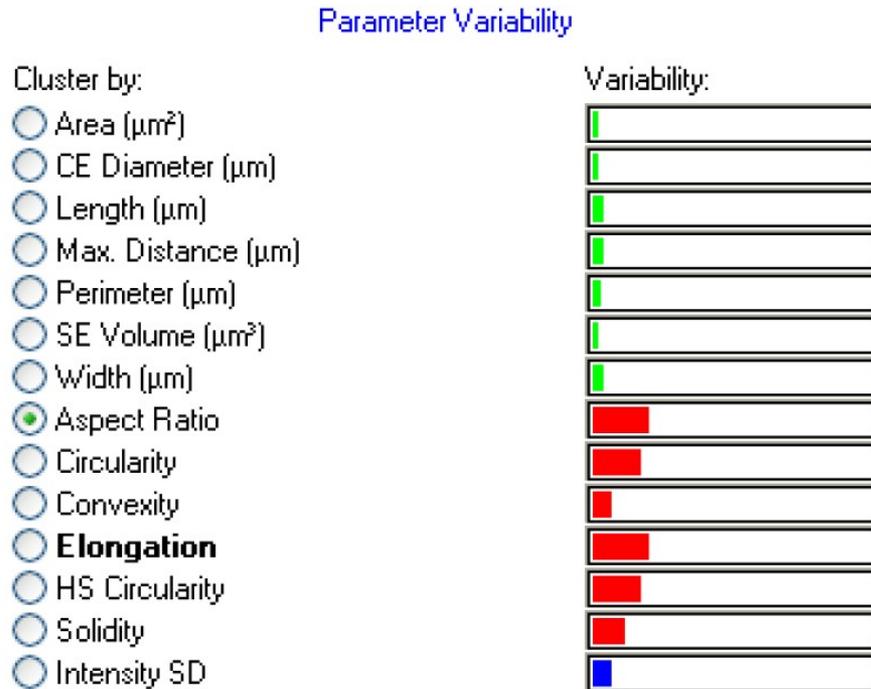


Figure 2: Parameter variability plot showing the degree of difference between the two metallic powder samples for each parameter analyzed

The green bars are related to particle size and the red bars to particle shape. The length of each bar indicates the degree of difference between the samples for each parameter. Clearly there are minimal differences between the two samples in terms of the size parameters, but there are differences in terms of some of the shape parameters, in particular elongation and aspect ratio.

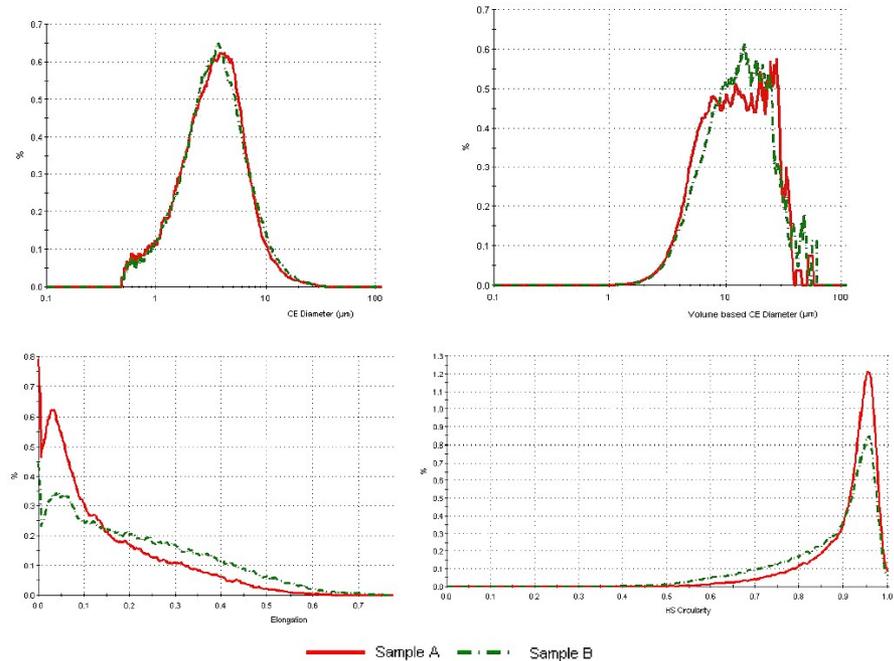


Figure 3: Overlays of some size and shape parameters. Samples A and B are very similar in terms of CE Diameter (both in number and volume weighting) but differ in terms of Elongation and HS Circularity

These observations are confirmed by the distributions shown in Figure 3. The samples are very similar in terms of circular equivalent (CE) diameter on both a number-weighted basis and a volume-weighted basis. CE diameter is the diameter of a circle with the same area as the particle image. However, sample A, produced by the gas atomization process, is less elongated and more circular than sample B, produced by the water atomization process. Elongation is a measure of the width to length relationship of the particle. A more needle like particle will present a higher elongation. High Sensitivity (HS) circularity is a measure of how close the 2D particle image is to a perfect circle. A perfect circle has a HS circularity of 1, and any deviations from a circle will reduce the HS circularity value. Particles produced by water atomization show a more irregular shape than those produced by gas atomization.

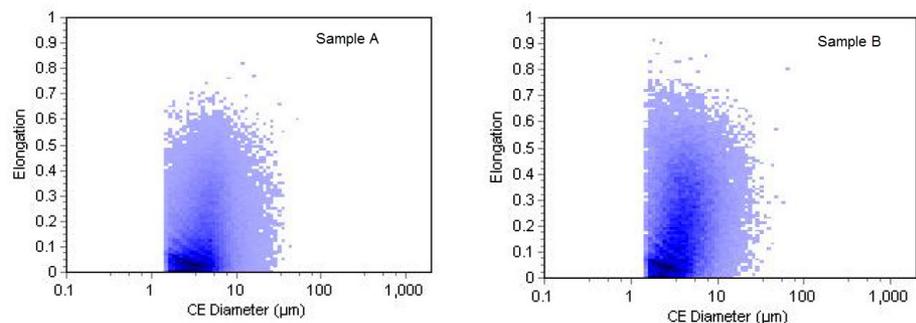


Figure 4: Scattergrams of CE Diameter vs. Elongation

Figure 4 shows the scattergrams of CE Diameter vs. Elongation for the two samples. Comparing the two scattergrams, differences in Elongation are apparent across the particle size range which may indicate a fundamental difference in the particle shape produced by the two atomization techniques rather than simply an agglomeration or fusing effect causing the difference in particle shape. During an analysis an image of every particle measured is captured and retained. The software allows regions of interest to be selected in the scattergram and the images of the particles fitting the region are displayed.

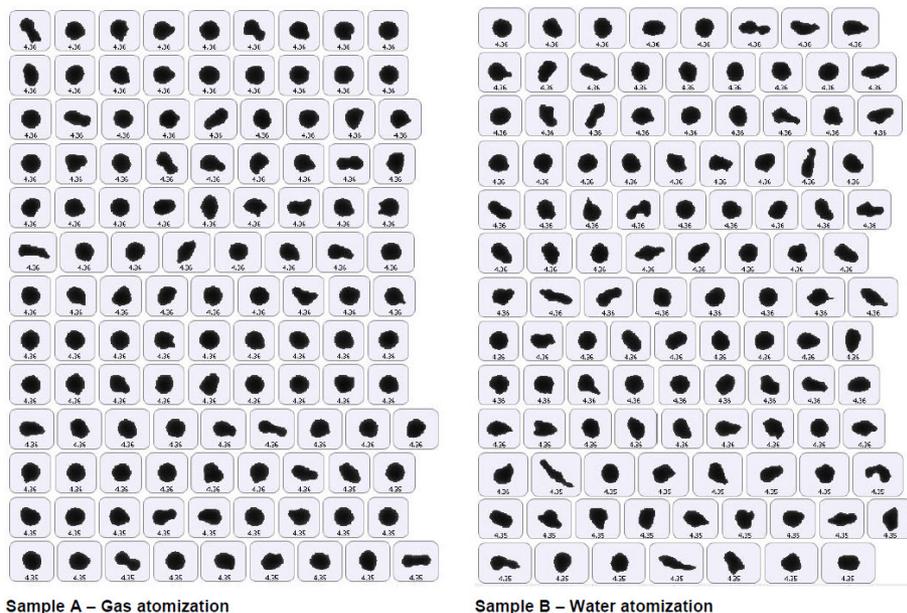


Figure 5: Example images of particles from around the modal size value (individual CE Diameter (μm)) indicated

Figure 5 shows examples of particle images from around the modal size for both samples. A visual inspection of the images confirms the view that there appears to be more irregular particles present in the water atomized sample than in the gas atomized powder. Despite the two samples having the same particle size, differences in particle shape were identified between the samples. The more spherical powders will have higher packing density which will typically improve sintering, reduce shrinkage and enhance physical properties of the final MIM component.

Summary

In a metal injection molding process the size and shape of the particles produced during the atomization step of the process can affect the properties of the final part. Automated image analysis provides an effective analysis of the powder so that manufacturers and users can monitor powder properties and thereby optimize their processes.

Acknowledgements

Malvern Panalytical wish to thank Sandvik Osprey Ltd for providing atomized powder samples for analysis.

References

1. ISO 13322-1 (2014) Particle size analysis - Image analysis methods - Part 1: Static image analysis methods
2. Willen U (2010) Automation in Image Analysis for Particle Size and Shape Measurement - Application Whitepaper, *GIT Laboratory Journal*

**Malvern Instruments
Limited**

Groveswood Road, Malvern,
Worcestershire, UK. WR14
1XZ

Malvern Instruments is part of Spectris plc, the Precision Instrumentation and Controls Company.

Spectris and the Spectris logo are Trade Marks of Spectris plc.

All information supplied within is correct at time of publication.

Malvern Instruments pursues a policy of continual improvement due to technical development. We therefore reserve the right to deviate from information, descriptions, and specifications in this publication without notice. Malvern Instruments shall not be liable for errors contained herein or for incidental or consequential damages in connection with the furnishing, performance or use of this material.

Malvern Instruments owns the following registered trademarks: Bohlin, FIPA, Insitec, ISYS, Kinexus, Malvern, Malvern 'Hills' logo, Mastersizer, MicroCal, Morphologi, Rosand, 'SEC-MALS', Viscosizer, Viscotek, Viscogel and Zetasizer.