

Optimizing metal powders for additive manufacturing

Exploring the impact of particle morphology and powder flowability



PARTICLE SHAPE



POWDER FLOW



PARTICLE SIZE

Introduction

In recent years, additive manufacturing (AM) has transitioned successfully from a prototyping tool to a still new, but established and economically viable choice, for component production. Annual sales of AM machines have risen from less than 200 in 2012 to more than 500 in 2014ⁱ as the aerospace, energy, automotive, medical, and tooling industries have embraced the technology, and this trend is expected to continue. The use of AM in manufacturing is increasing the proportion of the market dedicated to metal materials – which is expected to account for a quarter of the whole market by 2023ⁱⁱ.

AM offers certain specific advantages relative to alternative powder metallurgy methods, ranging from design flexibility to the potential for high material use efficiency, and is particularly suitable for the production of small to medium volumes of relatively small components, as well as enabling the creation of totally new complex parts that were previously unachievable. The development of AM machines is an important area of focus as the technology is adapted to produce larger components, and deliver higher throughputs. However, there is now equal emphasis on the properties of the powders used.

Up to one third of the production cost of an AM component is the cost of the powder used, with commercial viability resting on establishing a robust supply chain and effective powder recycling strategies. Identifying analytical tools that can be used to reliably set specifications for AM metal powders to validate quality and manage their use is vital. In this white paper, we review the key processes used in AM and how they determine the requirements of metal powders for this application. Case study data from the National Centre for Additive Manufacturing, part of the UK's Manufacturing Technology Centre (MTC, Coventry, UK), highlights the value of particle size and shape, powder flowability

and bulk density measurements in the selection, optimization and management of AM powders.

A process like any other

Additive manufacturing is 'the process of joining materials to make objects from 3D model data, usually layer upon layer, as opposed to subtractive manufacturing methodologies such as machining'ⁱⁱ. A tool-less manufacturing technique, it offers superior design freedom to any other and, uniquely, similar scalability for making one part versus many. Other benefits include: the possibility to create light weight structures and to build multicomponent parts in one step; reduced material consumption versus machining; and short production cycle times. To fully exploit these potential benefits, manufacturers need to understand the process, just as they would any other, the properties of material inputs and interactions between the two, so as to exert effective control.

There are a number of alternative technologies used within AM machines each of which subjects a metal powder to different flow, stress and processing regimes. Matching powder characteristics to any specific application/machine is therefore crucial. The most common commercial technologies can be classified as either powder bed or blown powder. A brief overview of how these processes work is useful in setting powder requirements in context.

Powder bed AM

Powder bed AM processes involve construction of the component on a progressively retracting platform, with a fresh layer of powder spread across the bed following the selective fusing of specified areas. With laser Powder Bed Fusion (PBF), a laser beam is used to locally melt the upper layer of the spread powder. PBF machines vary in terms of, for example, build volume and the number of lasers used, and are suitable for a wide range of materials including titanium, nickel and aluminium alloys, stainless and tool steels, and cobalt chrome. That said, build times are slow – in the order of just 25g per hour – so a primary aim is to reduce processing times.

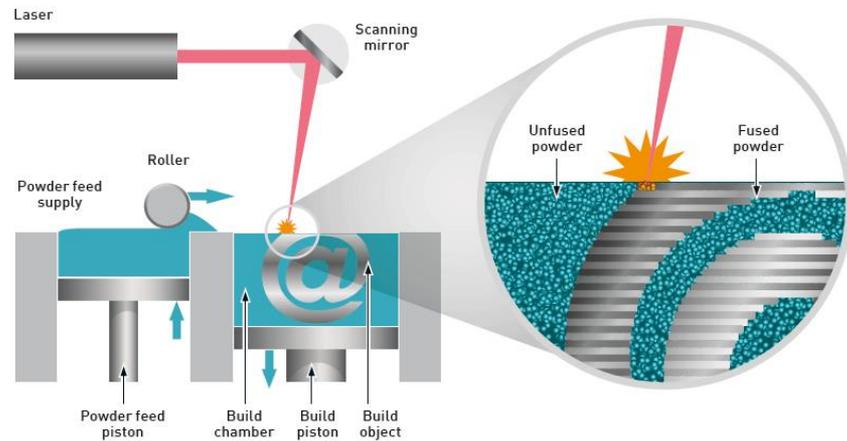


Figure 1: Powder bed AM processes such as PBF call for rapid, even powder spreading and effective recycling of the excess powder

A schematic of a typical PBF machine is shown in Figure 1. The metal powder is stored in a hopper and progressively exposed to the spreading or recoater roller by a rising piston. The roller spreads the exposed powder across the bed to create a thin, uniform layer around 20 to 50 microns in depth, with excess captured in a secondary container for re-use/recycling. A cycle of spreading, melting and fractional platform retraction is repeated, up to thousands of times, to build the finished component, layer-by-layer.

With Electron Beam Melting (EBM) the metal powder is fused using a high energy (3kw) electron beam which means that processing must take place in a vacuum chamber. This chamber is typically maintained at an elevated temperature (~700°C) which has the advantage of making the resulting parts almost free from residual stresses, an important gain in terms of product quality. On the other hand, the use of an electron beam has the potential to charge the metal particles, causing them to repel and form a cloud or 'smoke' around the working area. This undesirable effect is prevented by forming a pre-sintered cake in which the component is constructed. Powder recycling with an EBM process is therefore complicated by the additional need to break up the cake to return the metal powder to a usable form. Commercially, EBM is less widely used than PBF; there are fewer machines available and the range of materials that can be used is more limited.

Binder jetting

Binder jetting processes can be considered as a 'sub-set' of powder bed technology since they operate in a closely similar way. However, in binder jetting, a liquid binding agent is used to join the metal powder particles rather than them being melted or fused together through the application of heat. This results in the formation of a 'green' part that is removed from the printer. Metal solidification is then achieved in a second debinding/sintering step.

While EBM reduces residual stresses in the finished part by heating the component during construction, binder jetting processes similarly eliminate the thermal gradients that give rise to such stresses by not employing heat at all. Finished components are therefore largely free of residual stresses. Binder jetting can also be more cost-effective than other AM technologies. However, the materials available are more limited than for PBF, as are the mechanical properties achievable in the finished component.

Blown powder AM

In blown powder processes, such as Directed Energy Deposition (DED) (or Laser Metal Deposition), powder is blown through a nozzle at relatively high pressure in a carrier gas stream, into a melt pool on the specified surface. A laser beam forms the melt pool and is automatically moved across the substrate as required. DED processes offer higher productivity relative to PBF/EBM and enable the construction of larger scale components, but are unsuitable for the construction of features such as internal channelling and lattice structures.ⁱⁱⁱ These processes can also be used to make repairs and to augment the functionality of existing components, with controlled precision.

What makes a good AM metal powder?

All AM processes are typically operated with essentially 'fixed' parameters for a specific application, with current machines offering little opportunity for any form of responsive control. This means that inconsistent input material properties will translate directly into inconsistent finished component properties. Poor powder quality can produce defects in the end part including pores, cracks, inclusions, residual stresses and sub-optimal surface roughness, as well as compromising throughput. Understanding the correlations between material properties, processing performance and end component properties is therefore essential, both to select the best powder for an application and to ensure the consistency of that powder – from build-to-build, layer-to-layer, and through recycling. This raises the question of which properties are important in terms of defining a robust powder specification.

Chemistry is paramount. A powder needs to comply with the alloy composition of the material specified, and grade must be carefully selected so as to control the interstitial elements present – such as oxygen or nitrogen – which can impact the properties of the finished part. In addition, AM powders must be free from foreign particulate contamination – from other material batches at the powder production plant, the AM facility, or debris in processing/recycling equipment. Contaminant levels of just a few parts per million can be significant in terms of component quality.

Beyond chemistry, it is the physical characteristics of a metal powder that define AM performance. These characteristics include both bulk properties of the powder and properties of the individual metal particles. Key bulk properties are packing density and flowability. Powders that pack consistently well to give a high density are associated with the production of components with fewer flaws and consistent quality. Flowability, on the other hand, is arguably more closely associated with process efficiency. The ability to spread evenly and smoothly across a bed, to form a uniform layer with no air voids is essential for PBF processes, for example, while consistent flowability under very different conditions, as an aerated powder stream, is required for DED. These requirements intensify as processing speeds are increased.

Both bulk density and flowability are directly, though not exclusively, influenced by particle size and shape. The range of particle characteristics known to influence flowability, for example, includes stiffness, porosity, surface texture, density and electrostatic charge.^{iv} Figure 2 illustrates the relationship between aspects of particle shape and powder flowability. Generally speaking, smooth, regular-shaped particles flow more easily than those with a rough surface and/or irregular shape. Rougher surfaces result in increased interparticulate friction while irregularly shaped particles are more prone to mechanical interlocking; both of these effects decrease flowability.

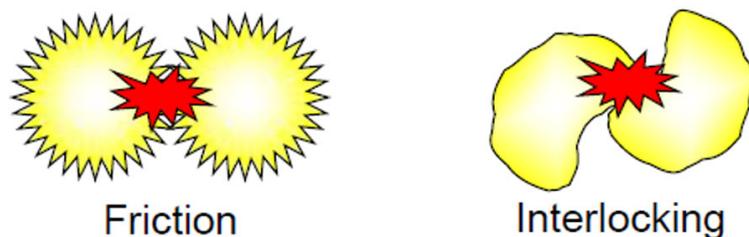


Figure 2: Smooth, regularly-shaped particles tend to flow more easily than those that are irregular and/or rougher, because of reduced friction and a lower risk of mechanical interlocking.

Similarly, spherical particles tend to pack more efficiently than those that are irregular giving rise to higher bulk densities.^v The bulk powder property

requirements for AM therefore suggest that sphericity is likely to be highly prized, a conclusion widely recognized within the industry.

When it comes to particle size, AM metal powders are necessarily fine to, for example, meet the requirement to form a powder bed just tens of microns thick. However, fines can be problematic from a health and safety perspective, and in terms of flowability. Because the forces of attraction between particles increase with decreasing particle size, finer powders are usually less free-flowing than coarser analogues, though optimising particle shape can help to mitigate this effect. In terms of packing, Figure 3 shows how both particle size and particle size distribution are influential. Maximum packing density is achieved with a distribution that includes both coarse and fine particles, with finer particles increasing density by filling the interstices left by larger ones.

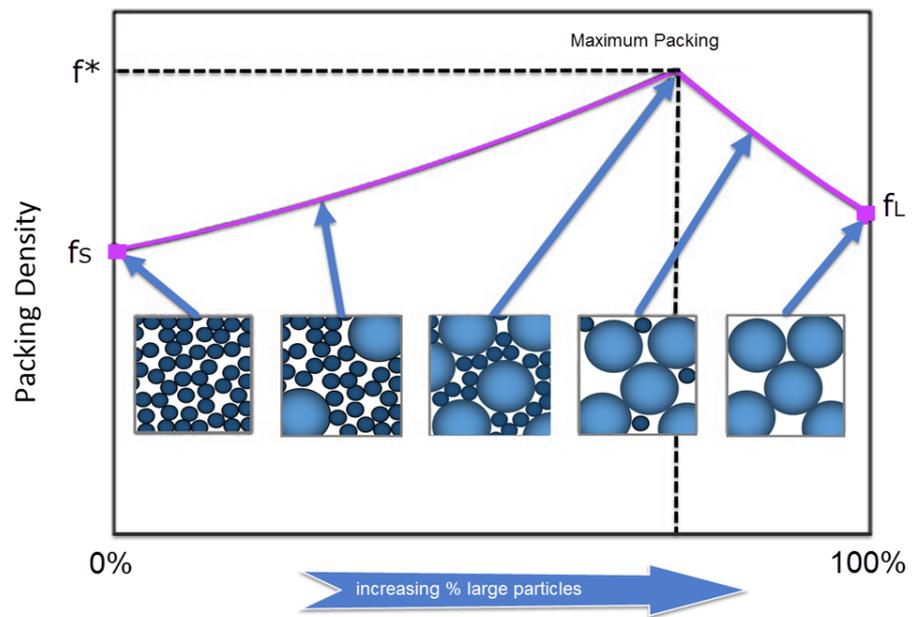


Figure 3: Packing density reaches a maximum when the particle size distribution includes both fine and coarse particles.

Metal powder manufacture substantially predates AM and many chemically consistent products are available on the market, the majority of which are manufactured via atomization processes. Particle size fraction can therefore be closely specified, as can particle shape, but often at a price. In particular, the cost of highly spherical metal powders is substantially higher than those containing particles that are more irregularly shaped. Measuring powders and particles so as to determine exactly what is required for a given process is the key to achieving beneficial performance at a competitive cost.

Making metal powders

The majority of metal powders used in AM are produced by gas atomization. In this process, a feedstock is melted in a crucible and then ejected through a nozzle into a high pressure gas stream (usually argon or nitrogen), breaking the molten stream into droplets.

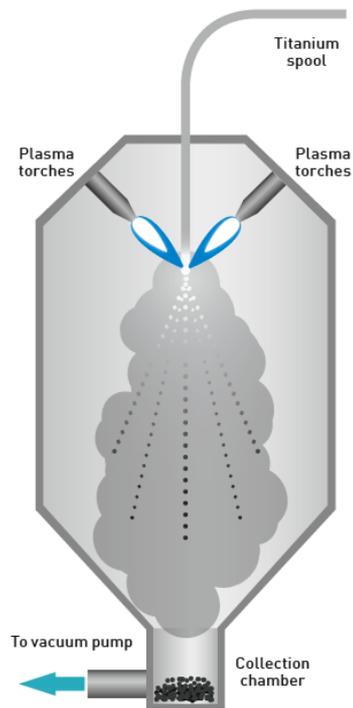


Figure 4: Schematic of a gas atomization process with metal spool feed and melting via plasma torches

The size of particles produced by gas atomization can be controlled by varying process parameters such as: gas pressure; melt properties; nozzle design and gas:metal ratio. However, the resulting product is not ideal for AM processes, which optimally require a narrower particle size distribution as shown in Figure 5. Various post atomization processes including 'Scalping' to remove the oversize particles followed by either air classification or sieving are applied to obtain the required size fraction. The lower atomization yields that result from the narrow size distributions required for AM is one of the factors that increases the cost of AM powders.

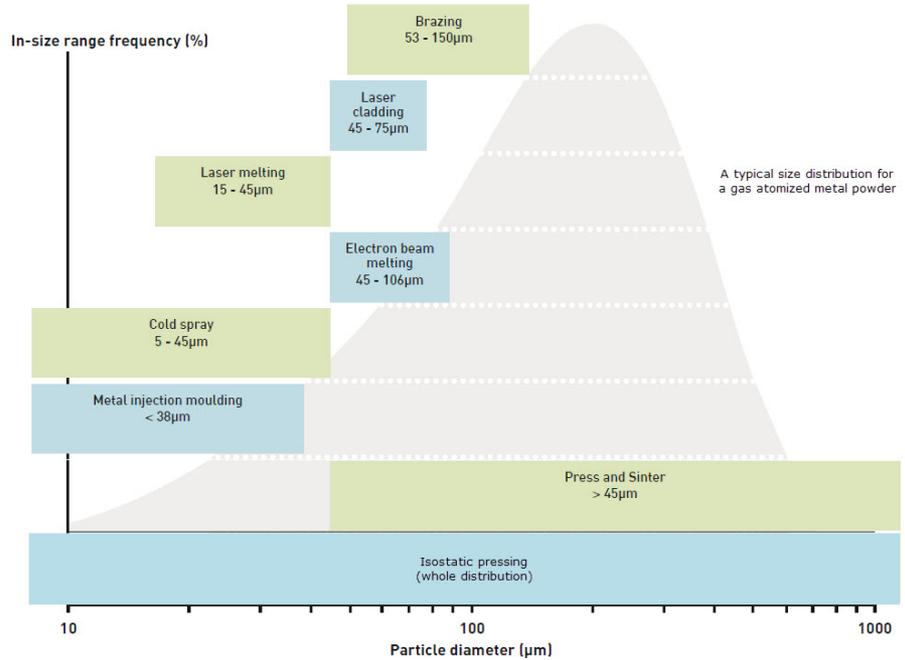


Figure 5: Typical as-atomized particle size distribution of gas atomized powders including required size distributions for various advanced powder metallurgy manufacturing technologies.

With regards to shape, gas atomized particles are relatively spherical but may exhibit any of the features shown in Figure 6. In particular, satellited particles are a problem – not only for flowability and packing, but because the satellite particles are so small (usually 1-10 microns), that if detached, can become an airborne health and safety risk. More spherical particles can be produced by Plasma Atomization or the Plasma Rotating Electrode Process (PREP), but at a higher price.

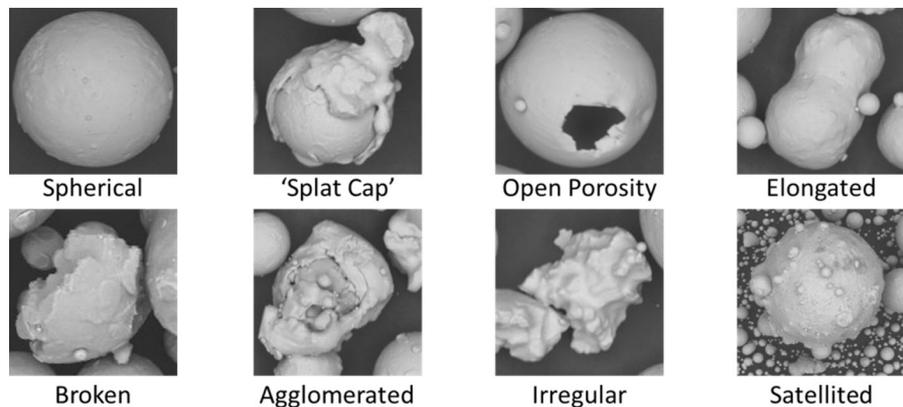
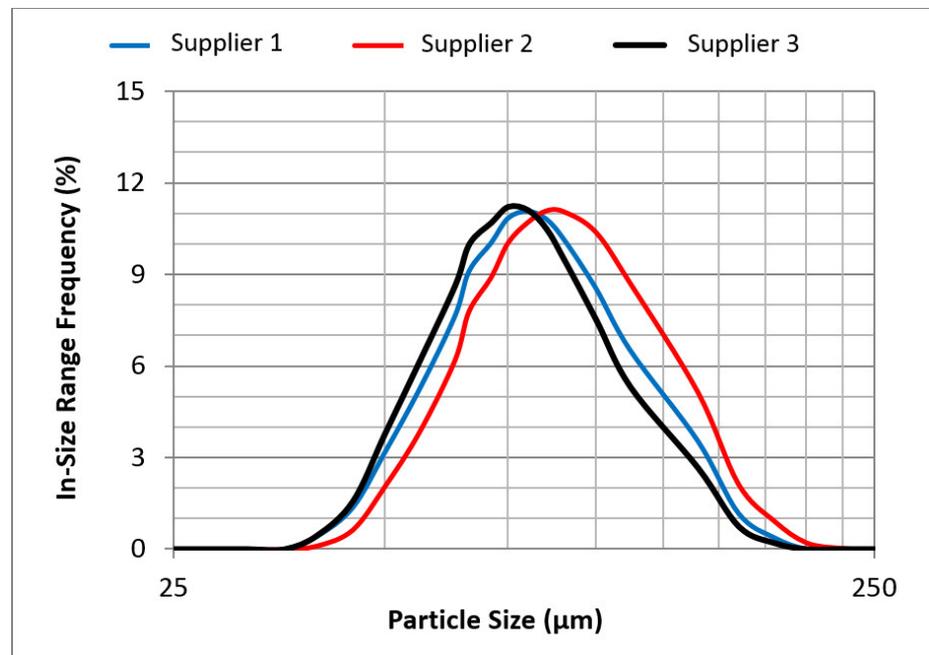


Figure 6: Images of individual metal particles, produced using gas atomization, illustrate the many different shapes of particle that may result from the process.

Case study: Evaluating metal powder supplies for AM applications

Researchers at MTC assessed three chemically identical Ti6/4 metal powder samples from different suppliers to assess their suitability for AM applications. All three powders met a specification for EBM, with a nominal particle size distribution of 45-106µm; two were produced via the same process but supplied by different vendors, while the third was produced using a different process. Details of the manufacturing processes applied were not supplied.

Particle size distribution data were measured for each of the powders using the technique of laser diffraction (Mastersizer 3000, Malvern Instruments, UK). The results are shown in Figure 7 and indicate that all three have a desirable monomodal particle size distribution. However, there are clear differences in the Dv50 (the particle size below which 50% of the particles lie, on the basis of volume) most significantly in the coarser end of the distribution. The material from supplier 2 has a higher level of coarse material than either of the other two, suggesting that the process used to narrow down the particle size distribution of the powder has been less successful.



	Supplier 1	Supplier 2	Supplier 3
D10	52.9 (1.62%)	57.0 (0.93%)	51.5 (%)
D50	77.7 (1.66%)	84.6 (0.96%)	74.3 (%)
D90	116.8 (1.65%)	128.4 (1.44%)	109.8 (%)

Figure 7: Particle size distribution data for samples of Ti6/4 from three different suppliers show marked differences, most especially in the amount of coarse material present.

Samples of each supply were subsequently characterized using a Morphologi G3 (Malvern Instruments, UK), a fully automated image analysis system. Automated imaging systems capture tens of thousands of particle images in just a few minutes and, from these, generate statistically valid size and shape distributions which can be used to characterize particle morphology in a more precise, objective and robust way than is achievable with, for example, Scanning Electron Microscopy. Using well-defined parameters, such as elongation and circularity, which define overall shape, and convexity, which quantifies the regularity of the outline of the particle, the particles in each sample were classified according to the following descriptions: rough particles; highly spherical particles; elongated particles; smooth non-spherical particles; and slightly spherical (see Figure 8).

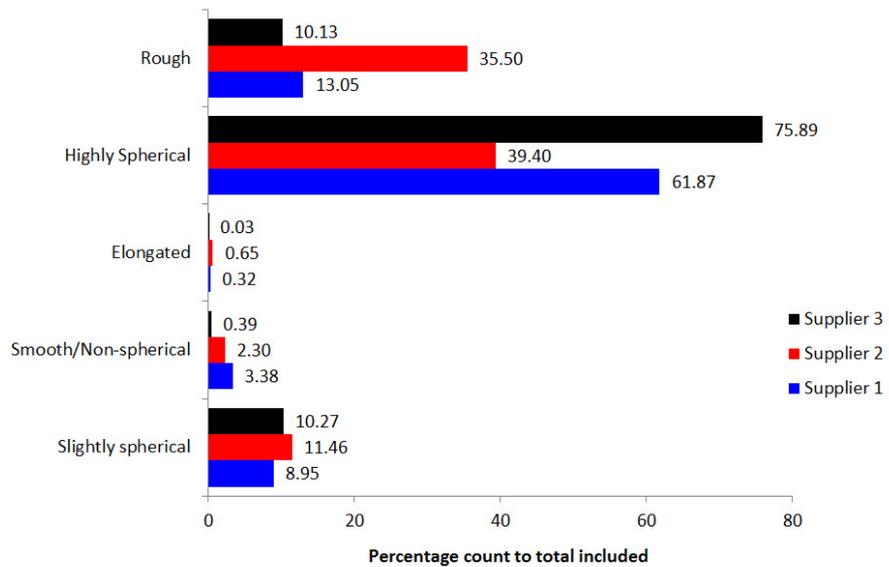
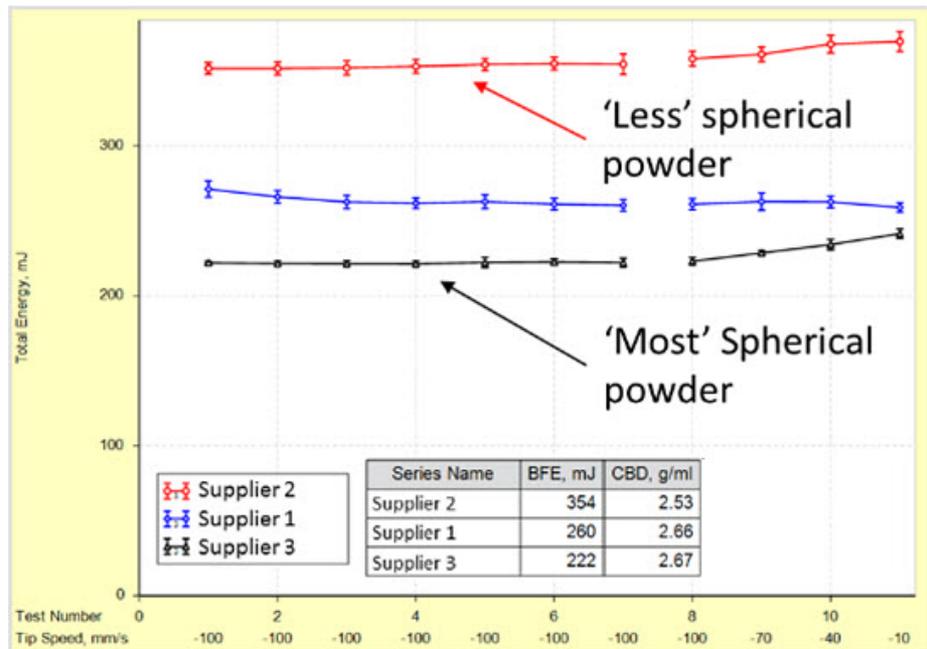


Figure 8: Automated imaging data reveals that the sample from supplier 3 contains a greater proportion of highly spherical particles than either of the other two.

The identified differences in the particle morphology of the three supplies suggest that the powder from supplier 1 will perform most effectively in AM processes. However, to understand securely the potential impact of these differences, and/or detect further differences that may influence process performance, we need to measure relevant bulk powder properties – flowability and bulk density.

Dynamic powder flow measurements were made for all three powders (FT4 Powder Rheometer, Freeman Technology, UK). Dynamic powder testing measures powders in motion, rather than in a static state, generating flow energy values from precise measurements of the axial and rotational forces acting on a blade as it is precisely rotated through a powder sample. Basic Flow Energy (BFE), a primary dynamic property, has been shown to be highly relevant to the performance of metal powders in AM processes (and indeed many others), far more so than alternative flow measurement techniques such as Hall's Index, which is based on the measurement of flow through an orifice^{vii}.



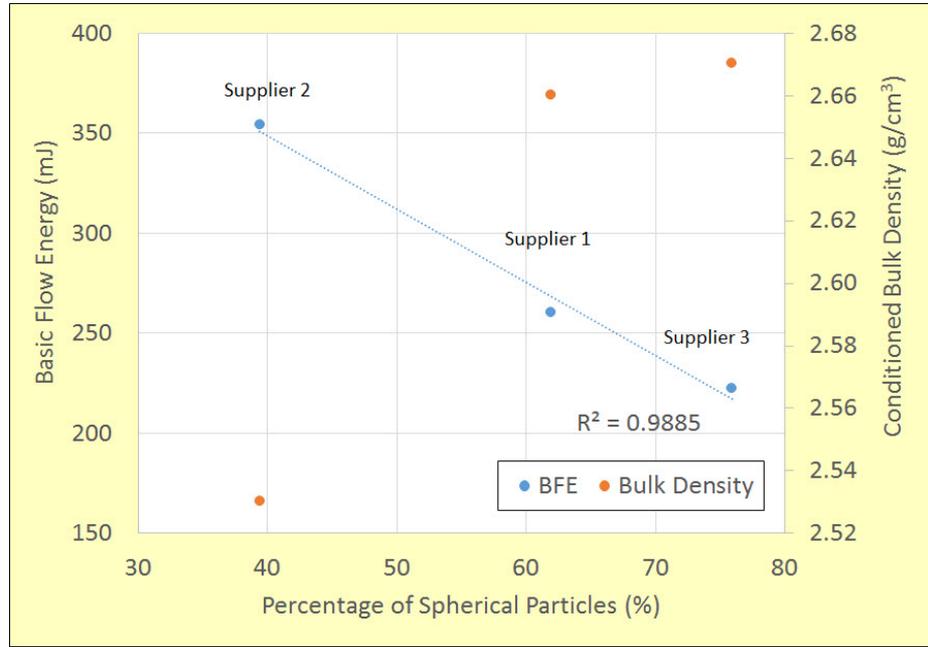


Figure 9: The flowability of the powder samples, as quantified by BFE, correlates directly with the percentage of spherical particles they contain.

Figure 9 shows BFE measurements for the each of the powders. There is a direct and strong correlation between flowability and the percentage of spherical particles in the sample with powders that contained more spherical particles flowing more freely than those with fewer. For these powders this correlation between shape and flow appears to be largely independent of the small differences in size between the samples. The bulk density (CBD) of each of the samples was also measured using the same instrument to assess packing density. Here, the correlation with shape is less strong, possibly as a result of the influence of other factors, such as particle size, but there is a clear trend associating more spherical particles with enhanced packing.

These results show that chemical consistency is no guarantee that physical properties, and consequently AM performance, will be identical, and illustrate the value of applying size, shape, flowability and bulk density measurement to robustly assess the performance of an AM performance. Particle size and shape are the parameters that are most easily specified and controlled in the metal powder manufacturing process, while bulk density and flowability allows closer, more relevant interpretation of the likely impact of changes in these variables on the performance of the AM process. In this instance, these techniques identify the powder from supplier 1 as superior to the alternatives for AM applications, a conclusion that is robustly supported by all four parameters.

The next industrial revolution

The extent to which AM becomes a mainstream manufacturing option depends equally on the development of AM technology, and the identification of powders that will work efficiently with it, to produce high quality components. Using complementary techniques, such as advanced automated image analysis and dynamic powder flow measurement, helps manufacturers to identify and specify suitable metal powders, optimize AM processes, monitor batch consistency, implement effective powder recycling strategies and achieve consistently high quality parts. These same technologies can also help metal powder suppliers to optimize their processes to meet the requirements associated with supplying growing and lucrative AM markets. Ensuring a supply chain of consistent and appropriate quality is an essential step towards fully exploiting the considerable potential of AM across all the industries where it can bring benefit and commercial value.

References

ⁱ Wohlers Report 2015. 3D Printing and Additive Manufacturing State of the Industry. Annual Worldwide Progress Report.

ⁱⁱ SmarTech Publishing 'The Top Three additive Manufacturing Predictions for 3D printing in 2017' available to view at: <https://www.smarttechpublishing.com/news/smartech-publishing-the-top-three-additive-manufacturing-predictions-for-3d>

ⁱⁱⁱ European Powder Metallurgy Association

^{iv} T Freeman 'An introduction to powders' e-book available for download at: http://www.freemantech.co.uk/styling/images/eBooks/intro_to_powders.jpg

^v 'Optimizing powder packing behaviour by controlling particle size and shape'. A Malvern WP available for download at: <http://www.malvern.com/en/support/resource-center/Whitepapers/WP160610OptimizingParticlePacking.aspx>

^{vi} <http://www.lpwtechnology.com/technical-library/powder-production/>

^{vii} J. Clayton and R. Deffley 'Optimising metal powders for additive manufacturing' Metal Powder Report July/August 2014

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