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SPECIAL FEATURE

Measuring the critical attributes of AM powders

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Metal powder additive manufacturing (AM) continues to be the subject of intense research activity driven by widespread recognition of its considerable value as a manufacturing technology. With an array of design and supply chain benefits from unique microstructures and part consolidation to agile, distributed production with minimal lead times, metal AM also offers potential for reduced waste and products with a lighter environmental footprint; it is now routinely applied for finished part production. The development of new metal powders, optimally specified for AM applications, is crucial for the further exploitation of this exciting technology, so is ensuring raw material consistency. Industries leading the way in AM, such as the biomedical and aerospace sectors, require certified feedstocks with robustly controlled characteristics.

In this article we consider an analytical toolkit for AM metal powders by examining their critical attributes, the properties that define in-process performance and/or the quality of the finished product. Case study data illustrate the application of key techniques.

Understanding the demands of AM

An understanding of metal AM processes is a useful starting point when it comes to the consideration of critical attributes of the associated powder feedstocks. The three processes used for the vast majority of metal AM are: powder bed fusion (PBF); binder jetting (BJ); and directed energy deposition (DED) [1,2].

PBF involves the use of laser or electron beam to selectively fuse regions of a powder bed. A key step is the formation of a uniform, dense layer of powder just tens of micrometers thick across the build platform via rapid powder spreading. Precise melting of the metal powder, and subsequent cooling, fuses one layer to the next, progressively forming the complete component (see Figure 1).

The spreading of powder to form uniform layers is similarly critical in BJ but here 'a liquid bonding agent is selectively deposited to join powder materials' [1]. Curing and sintering steps are required to produce the finished component. In both PBF and BJ, only a small proportion of the powder in any given layer is fused with the component with the vast majority recycled. Effective recycling strategies are therefore integral to the economic application of these processes, which account for the vast majority of metal AM.

Finally, in a DED process powders (or metal wires) are fused by a laser or electron beam as they are deposited. The capability to simultaneously deposit more than one metal and for processing away from an x-y plane makes DED particularly useful for repairs or feature addition (Figure 2).

Characterizing AM metal powders

The physical characteristics of AM powders influence packing behavior – formation of the powder bed – and the kinetics of melting and sintering, thereby impacting the density, porosity, dimensional integrity and surface finish of the finished component. Excellent flowability is a prerequisite for efficiency in all three processes. Measuring particle properties including particle size and morphology, density and specific surface area, along with bulk powder properties such as flowability robustly quantifies these behaviors. Techniques that are sufficiently sensitive to

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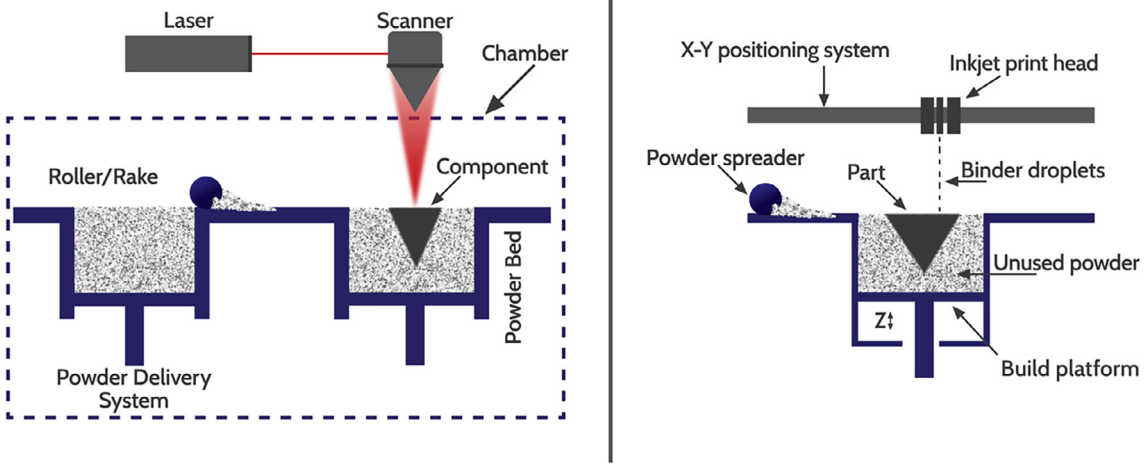

FIGURE 1

A schematic of a PBF process (left) and a BJ process (right).

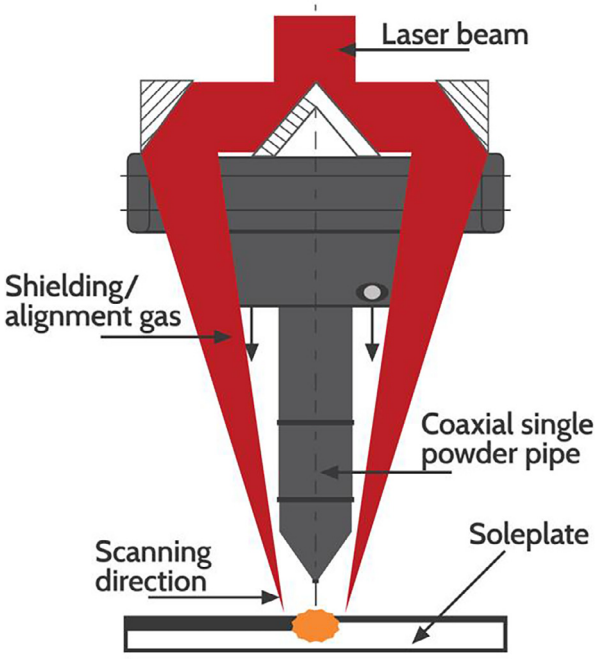

FIGURE 2

A schematic of a DED process.

detect the potentially subtle changes induced by processing are a prerequisite for the development of effective recycling strategies.

Particle size

Particle size strongly influences both packing behavior and flowability. The need for close particle packing calls for the use of finer powders though these tend to be associated with poor flowability. Particle size also influences the rate at which the powder melts and/or sinters. As a result of these correlations there are well-defined particle size ranges in place for metal AM powders, typically 15–45 μm for PBF for example [2].

Laser diffraction is the most common technique for sizing metal AM powders, but air permeability and X-ray sedimentation, both of which work well for relatively dense powders, are also used.

Laser diffraction

Particles illuminated by a monochromatic light source scatter light, with intensity varying as a function of angle to the incident beam depending on the size of particles present. A laser diffraction system detects the light scattering pattern produced by a sample and back-calculates particle size via the application of a mathematical model (typically Mie theory). Particle size distribution data are reported on a volumetric basis, on the assumption of a particle population that is spherical and isotropic. Laser diffraction reports metrics associated with the diameter of a sphere that produces the observed scattering pattern, with data typically summarized in terms of a weighted mean volume diameter ($D_{4,3}$) [3].

Traditionally, photo diode arrays are used as detectors, but the most powerfully discriminating systems use a charge coupled device (CCD) to significantly boost resolution and sensitivity. Such systems bring better definition to the extremes of a particle size distribution – the fines and tails – and can be extremely useful for AM powder characterization. Coarse particles, in a virgin or recycled material, for example, can disrupt the smooth plane of the powder bed, directly impacting powder fusion.

Air permeability

Air permeability is a classic ISO/ASTM standard technique for sizing particles that lie below the lower limit for dry sieving, made easier to apply by modern, automated instrumentation. In an air permeability measurement, the pressure drop across a powder bed is measured as air flows up through the sample at known flow rate. The average specific surface area of the bed is determined from these data, via the Carman-Kozeny equation, and used to generate an average particle size, typically a weighted mean surface diameter ($D_{3,2}$) [3].

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X-ray sedimentation

X-ray sedimentation measures particle size data on the basis of mass (which is equivalent to volume for samples of consistent density) and is underpinned by two well-understood physical phenomena – sedimentation and photon absorption. Sample dispersed in a suitable liquid medium is pumped through the sample cell of the instrument and agitation is then ceased. X-ray intensity is used to monitor the subsequent settling process, with progressively finer particles falling through the measurement zone. Settling velocity is determined from measurements of the time taken for particles to pass through the measurement zone with particle size calculated from settling velocity, through the application of Stokes' law. The relative mass concentration for each size class determined from X-ray absorption data via the Beer-Lambert-Bouguer law. X-ray sedimentation is particularly fast and efficient for regularly shaped, relatively dense particles and has a dynamic range (0.1–300 μm) that comfortably covers AM materials.

Surface area

The specific surface area of an AM metal powder defines the extent to which material is exposed to interact with other components and the localized environment. Surface area data can therefore elucidate sintering behavior and, in the case of BJ, binder interactions. Gas adsorption is the classical technique for detailed surface area characterization.

A gas adsorption apparatus determines the amount of gas adsorbed onto the surface – both internal and external – of a decontaminated sample, as a function of pressure. Measurements are made at progressively increasing pressure to produce sequential points on an adsorption isotherm; the reverse process characterizes desorption behavior. This isotherm is a fingerprint for the material, defined by surface area and any accessible porosity. Surface area is calculated from the isotherm by applying an appropriate mathematical model, most commonly the Brunauer, Emmett and Teller (BET) [4] theory.

Density

Multiple density parameters are relevant in characterizing an AM metal powder. Bulk density is simply mass divided by volume occupied and can be determined from putting a sample of known mass into a graduated cylinder. The volume measured includes the interstitial space between particles, so values are strongly influenced by particle interactions and packing behavior. They are relevant to bed formation and powder flowability, as well as routinely forming part of a material specification.

True (or absolute) density, in contrast, is an inherent material property calculated from the volume of material present which can be accurately and reliably measured by gas pycnometry, a displacement technique. Gas is used to charge a sample chamber of known volume, containing a sample of known mass, to a defined pressure. Discharging the gas into a secondary chamber of known volume enables calculation of the volume of the sample from pressure measurements, via the gas law.

For particles with no, or inaccessible porosity, pycnometry will measure the volume of all the individual particles. However, if there is accessible porosity that the displacement gas can penetrate then the measured volume will be lower; the resulting density is referred to as skeletal (or apparent) density. Generally, the requirement in AM is for fully dense powders that fuse to form a finished component with well-controlled porosity. True density is often used as a measure of purity and can be compared with theoretical density to gain insight into particle porosity.

Particle morphology – shape and surface topology

Packing behavior and flowability are influenced not just by particle size but also by particle shape and surface topography. Highly spherical particles are prized for their excellent flowability and efficient packing behavior. However, poor surface quality, including the incorporation of satellites can compromise the performance of otherwise regular particles.

Dynamic image analysis

Dynamic image analysis quantifies particle size and shape from images of individual particles captured by a high-resolution camera. These images are recorded at a rate of thousands per second as particles suspended in a carrier liquid flow through a measurement cell. Number based size and shape distributions are constructed from analyses of each image. The technique quantifies both the form and surface characteristics of particles via shape parameters such as circularity, convexity (a measure of particle edge roughness) and smoothness.

Scanning electron microscopy (SEM)

In SEM, a focused electron beam scanning across the sample provides detailed, high resolution surface characterization. With some materials electrons also penetrate relatively deeply into the sample inducing the production of X-rays which can be analyzed to provide elemental analysis. The resolution of SEM can be increased by focusing on a smaller scanning area making the technique a powerful and flexible tool for investigating surface topography at the sub-micron level.

Powder flowability

All metal AM processes rely on efficient powder distribution making flowability critical, particularly within the context of driving toward faster build rates. While particle size and shape are widely recognized as influencing flowability it is not feasible to predict flow properties from particle characterization data. Flowability measurements, of both virgin and recycled powder, therefore, have an important role to play in identifying materials that will perform efficiently.

Dynamic powder testing enables the quantification of flowability under low stress conditions, thereby generating data that are more relevant to AM processes than those produced by techniques such as shear cell testing in which moderate to high stress conditions are applied. In dynamic testing, flow properties are generated from measurements of the axial and rotational forces acting on an impeller as it is rotated precisely through a powder sample. Dynamic properties include Basic Flowability Energy (BFE) which is generated by applying a downward traverse of the blade, pushing the powder against the confining base of the sample vessel, and Specific Energy (SE), which is measured with an upward traverse. These parameters quantify confined/-

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forced flow and unconfined flow properties in a low stress powder, respectively.

Dynamic test methods offer extremely sensitive powder differentiation and are valued for detecting subtle, but relevant differences between powders to support effective AM feedstock selection and the development of efficient recycling strategies.

Case study 1: Comparing the particle size, morphology and surface area of stainless steel, tungsten and tungsten carbide metal powders.

Particle size, specific surface area and density data were measured for three different metal powders: 316 stainless steel (316L), tungsten carbide (WC) and tungsten (W). SEM was also applied to study particle morphology. The samples were subject to degassing ahead of gas adsorption analyses, but no other form of pretreatment.

Table 1 shows density values for each of the powders measured by gas (helium) pycnometry (AccuPyc II 1340*), alongside theoretical density values for each material. These results indicate that all three powders essentially consist of high purity particles with minimal closed porosity; differences between measured and theoretical values are attributable to unremoved surface contamination.

Table 2 (a) shows specific surface area data measured by gas adsorption (BET – TriStar*) and generated from particle size data from laser diffraction (Saturn Digisizer® II*) and air permeability analyses (Sub-Sieve AutoSizer*) (Table 2b). SEM images (Phenom*) for each of the powders are shown in Figure 3.

TABLE 1

Measurements of density, by gas pycnometry, indicate that all three metal powders are essentially pure and non-porous.

	Density measured by gas pycnometry (g/cm^3)	Theoretical density (g/cm^3)
316L Stainless Steel (316L)	7.886	8.0
Tungsten carbide (WC)	15.303	15.6
Tungsten (W)	18.952	19.3

*Micromeritics Instrument Corporation, Norcross, Georgia, USA.

The surface area data measured for the stainless steel are relatively similar with each technique. SEM reveals the particles to be essentially spherical with surface cracks and pores accounting for the slightly greater surface area recorded in BET measurements.

The surface area measurements for WC vary substantially from method to method, as do the reported particle size data. This material is highly aggregated, giving rise to particles that are far from spherical (an assumption in both laser diffraction and air permeability measurements). BET values for surface area are significantly higher because gas penetrates the complex aggregated shapes. These effects are also evident in the results for W though the particles are somewhat more regular and less aggregated.

These results highlight the important point that analytical techniques report different numerical values depending on the measurement principles employed and underlying assumptions. Virgin metal AM powders tend to be spherical but may become less regularly shaped once recycled, aggregating as a result of partial fusing, for example. SEM is helpful in identifying such changes which can directly impact measured particle size values.

Case study 2 – Comparing the properties of alternative metal AM powder supplies.

Tests were carried out to compare three different metal alloy powders being considered as feedstocks for an existing process. Supplier 1 provided two samples for assessment, one manufactured by plasma atomization (PA – Method 1), the other by gas atomization (GA – Method 2). Supplier 2 provided a single sample manufactured by a GA process. All three samples were manufactured to the same particle size distribution. The powders were characterized using dynamic, shear and bulk powder testing methods (FT4 Powder Rheometer, Freeman Technology, UK) applying the standard test methodologies for the instrument [5].

Shear cell data clearly differentiate the PA sample from the two GA samples, highlighting it as more free-flowing – lower Shear Stress values leading to lower Cohesion and Unconfined Yield Strength (UYS) – under moderate to high stress conditions.

TABLE 2

Surface area (m^2/g) (a) and particle size (b) (μm) data measured by different techniques, show clear differences arising from the principle of measurement. [*Note:* $D_{3,2}$ were calculated from laser diffraction data for comparison purposes.]

(a)

	Laser diffraction	BET	Air permeability
316L Stainless Steel (316L)	0.109	0.169	0.106
Tungsten Carbide (WC)	0.167	1.67	0.509
Tungsten (W)	0.093	0.609	0.223

(b)

	Laser diffraction (Weighted Mean Volume Diameter $D_{4,3}$)	Laser diffraction (Weighted Mean Surface Diameter $D_{3,2}$)	Air permeability (Weighted Mean Surface Diameter $D_{3,2}$)
316L Stainless Steel (316L)	12.60	6.99	7.18
Tungsten Carbide (WC)	3.18	2.36	0.77
Tungsten (W)	4.63	3.36	1.42

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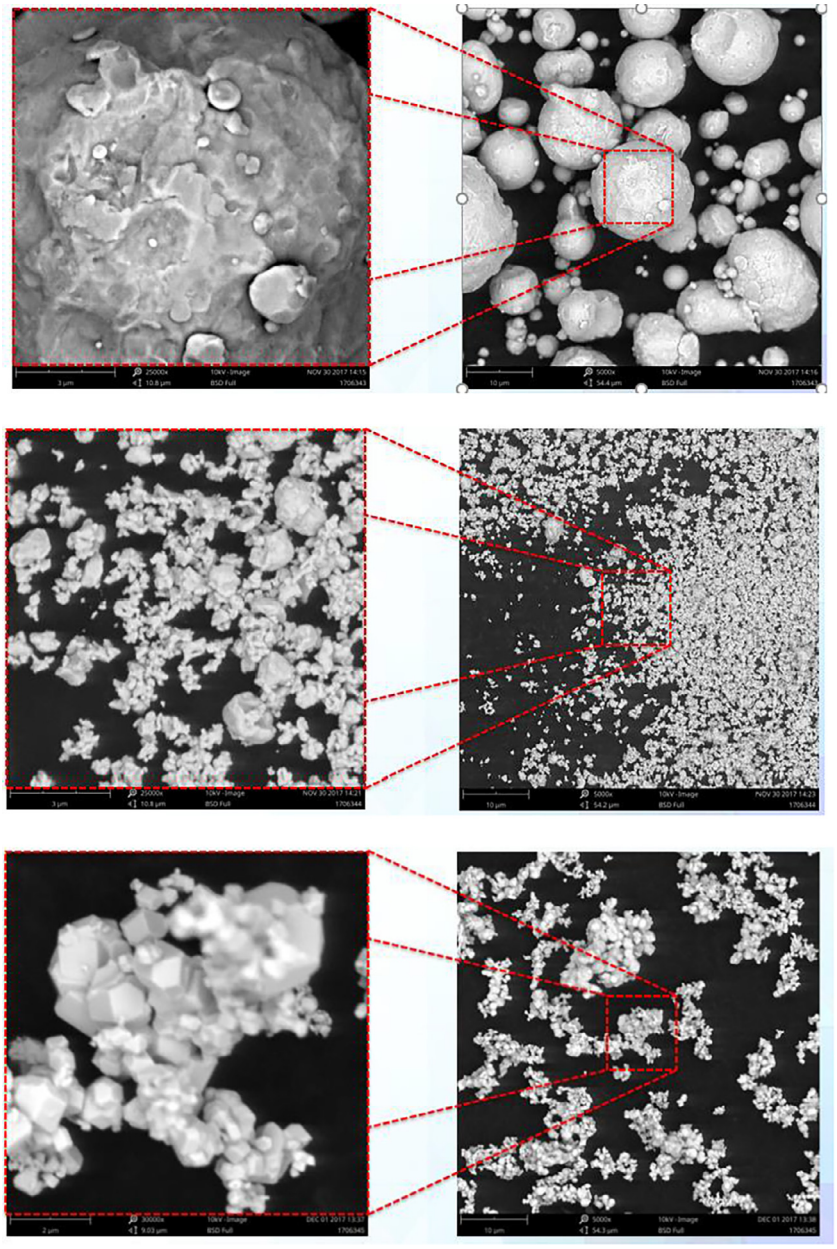

FIGURE 3

SEM images for a) 316L, b) WC and c) W help to rationalize observed differences in the particle size and surface area data.

However, these data do not differentiate the two GA samples (Figure 4).

Dynamic test data for the three samples confirm the PA sample as the most free-flowing via lower BFE and SE values and differentiate the two GA samples. The sample from Supplier 1 has a lower BFE and lower SE than that from Supplier 2 and is therefore likely to be more free-flowing under the low stress conditions applied during dynamic testing (Figure 5). Permeability data (not shown) also detect difference between the two GA samples, providing further confirmation that they will exhibit dissimilar process performance in certain circumstances.

These data illustrate the value of multi-faceted bulk powder characterization and the ability of dynamic and bulk powder properties to sensitively differentiate metal powders with the same particle size. Dynamic flow parameters reflect the behavior the powder will exhibit under the low stress environment applied during spreading and/or in a DED process and are therefore highly relevant for assessing the performance of both fresh and recycled AM powders.

In conclusion

Developing and manufacturing optimized AM powders relies on controlling characteristics such as packing and flow behavior, melting and sintering performance. Particle size and morphology, density and specific surface area along with bulk powder properties such as flowability define these characteristics. Techniques that enable the sensitive measurement of these variables therefore constitute a powerful toolkit that can be used to

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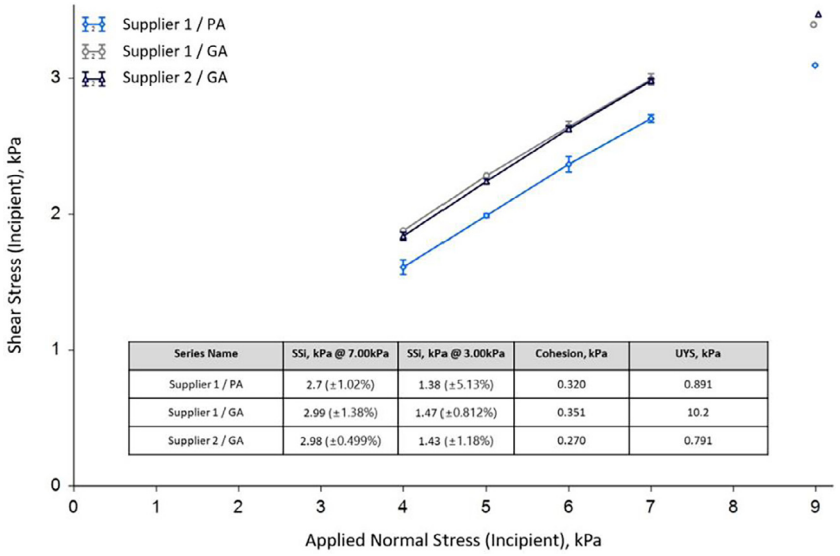
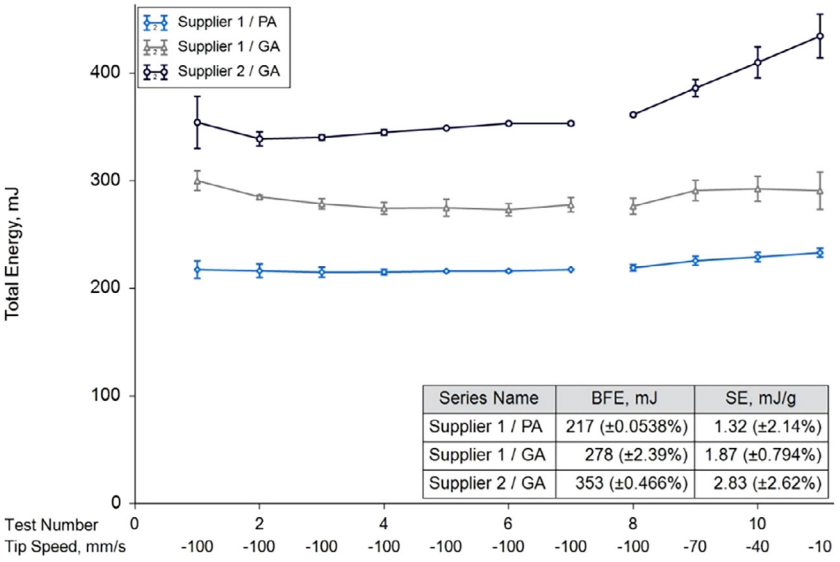


Series Name	SSi, kPa @ 7.00kPa	SSi, kPa @ 3.00kPa	Cohesion, kPa	UYS, kPa
Supplier 1 / PA	2.7 (±1.02%)	1.38 (±5.13%)	0.320	0.891
Supplier 1 / GA	2.99 (±1.38%)	1.47 (±0.812%)	0.351	10.2
Supplier 2 / GA	2.98 (±0.499%)	1.43 (±1.18%)	0.270	0.791

FIGURE 4

Shear cell data for alternative metal alloy supplies indicate that the PA sample is more free-flowing in this relatively high stress regime than those that are gas atomized samples.



Series Name	BFE, mJ	SE, mJ/g
Supplier 1 / PA	217 (±0.0538%)	1.32 (±2.14%)
Supplier 1 / GA	278 (±2.39%)	1.87 (±0.794%)
Supplier 2 / GA	353 (±0.466%)	2.83 (±2.62%)

FIGURE 5

Dynamic test data clearly differentiate all three potential feedstocks indicating that they will perform differently in a low stress environment.

develop competitively priced feedstocks and robust powder management strategies that support the ongoing exploitation of AM as a production technique.

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