The Application of Powder Rheology in Additive Manufacturing

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Original Source: Journal of Materials

The final publication is available at Springer via http://link.springer.com/article/10.1007/s11837-015-1293-z [10.1007/s11837-015-1293-z]

Abstract

Additive Manufacturing (AM) is sensitive to powder variability when applying fine layers in a uniform manner. This demands a high degree of consistency and repeatability in the feedstock. Particle size is often used as a critical quality attribute (CQA), but this is not sufficient to fully qualify a feedstock. Indeed, it is inadequate to suggest that any parameter from a single test, e.g. Hall Flowmeter or Hausner Ratio, can comprehensively describe a powder's characteristics. This paper uses four case studies to demonstrate the limitations of single parameter characterisation and how the rheological properties of several metal powders used in AM applications are used to establish in-process performance. In the first study, the significantly reduced permeability and increased specific energy of a one batch of powder demonstrate a clear link to poor layer uniformity. The second study investigates the impact of metal powder manufacturing methods and suppliers and shows how shear properties alone cannot be relied on to identify properties that influence the process. The effect of additives on the processability of polymer blends used in AM is also evaluated and the results show that even small quantities can have a significant effect on the permeability and basic flowability energy of feedstocks. The final study demonstrates the how rheological measurements can be used to identify the optimum blend of fresh and used material when reusing metal powders to manufacture components. These case studies illustrate the ability of a modern powder rheometer to detect minor variations in powders that are directly relevant to performance in AM processes in a way that traditional characterisation methods cannot.

Introduction

Additive manufacturing, also known as 3D printing, is a potentially transformative manufacturing technique [1-4]. It involves 'printing', often intricate, components to a tight specification by gradually building up powder layers which are then selectively fused together. Controlling the performance of the powders is critical for both process efficiency and final product quality. How the powder flows and packs, as the layers are formed, are defining aspects of this performance. Variability in feedstock can lead to inconsistent bulk density, non-uniform layering, low tensile strength and poor surface finish.

The extent to which AM will shape the industrial landscape depends on the development of high-speed, precision machinery and on the identification and consistent supply of powders that are able to meet the exacting demands of these machines [5, 6]. Increasingly the focus is turning to the powders themselves and how they can be optimised in an intelligent and reliable way. Powder characterisation has a vital role to play in supporting this process and testing techniques that can reliably measure properties that correlate directly with AM performance are essential. Identifying which powder properties lead to uniform, repeatable performance allows new formulations to be optimised, without the significant financial and time implications associated with running samples through the process to assess suitability, and helps reduce the occurrence of final products that are out of specification.

Existing techniques such as Angle of Repose, Flow through a Funnel, and Bulk Density tests are well-documented, but these methods were developed without the benefits of modern technology, and are often too insensitive to accurately characterise subtle differences between powders that behave differently in process [7, 8]. Indeed these techniques were historically employed in these case studies but did not provide differentiation between powders that were successfully processed and those that gave rise to poor product quality and/or process interruptions.

Powder rheology provides automated, reliable and comprehensive measurements of bulk material characteristics [9]. This information can be correlated with process experience to improve processing efficiency and aid quality control. Modern powder rheometers allow the measurement of dynamic flow and shear properties as well as providing the capability to quantify bulk properties such as density, compressibility and permeability.

Case Study 1: Quantifying Batch-to-Batch Variation in Feedstocks

The tight tolerances within which AM machines operate mean that differences between batches of feedstocks can lead to significant variability in the properties and quality of the final product. A method of screening batches before they enter a unit operation can help ensure consistency in performance. However, many traditional powder characterisation techniques are not able to identify subtle but important differences.

This study evaluated three examples of stainless steel powders from the same supplier but which demonstrated variable performance in an AM process; Metal Powder A and Metal Powder B both exhibited acceptable behaviour but Metal Powder C regularly caused blockages and poor deposition, resulting in sub-standard final products. All three samples had virtually identical particle size distributions, and produced similar results in Angle of Repose and Hall Flowmeter tests.

Tests using a powder rheometer highlighted several differences between the samples that correlated with the performance observed in process. Basic Flowability Energy, (BFE) quantifies the energy required to displace a powder during non-gravitational forced flow, for example, flow in a screw conveyer or mixer – the powder's resistance to flow in a constrained environment. It is measured as a blade traverses downwards through a test sample, from the top surface to the bottom of the test vessel. Specific Energy, (SE) uses the upward motion of the blade to evaluate a powder's resistance to flow in an unconstrained environment and is normalised against the mass of the powder.

In this instance, SE clearly differentiates Metal Powder C, with the higher SE being indicative of increased mechanical interlocking and friction which can contribute to blockages and other flow problems, (Figure 1).

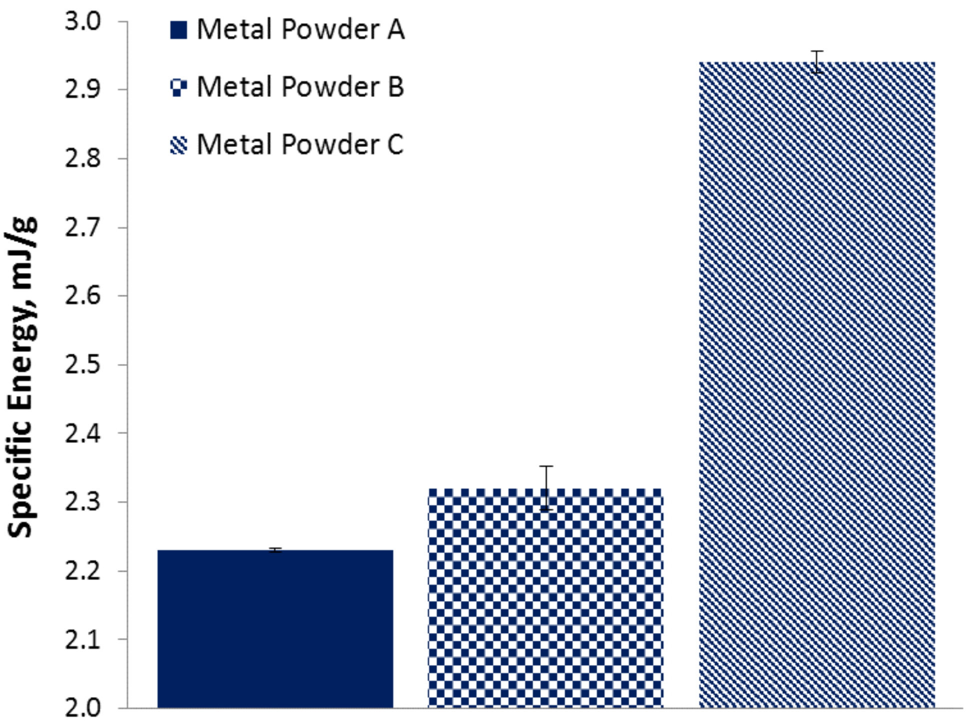


Fig. 1. Variation in SE with respect to batch.

Metal Powder C also generates a significantly higher Pressure Drop than A and B indicating that it is considerably less permeable, (Figure 2). Permeability is highly influential in any operation in which powder is required to move from one position to another, particularly when the motivating force is gravity. Air has to replace the space vacated by the particles, thus the more easily air can pass through the bulk, the more likely it is to flow freely. Low permeability also causes air to be retained in the bulk so, when required to deposit consistent quantities of powder during AM applications, low permeability can result in poor layer uniformity, leading to imperfections in the final product.

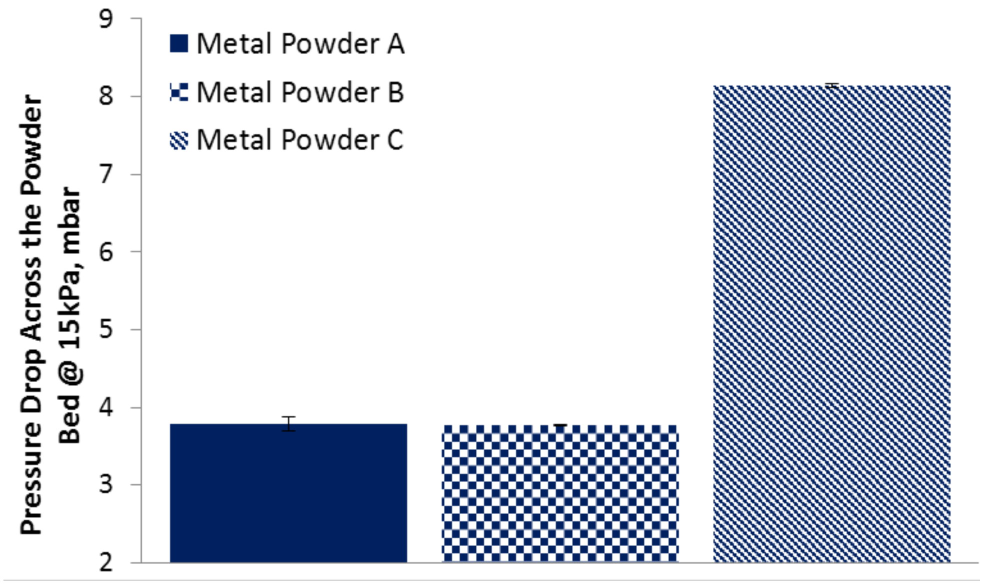


Fig. 2. Variation in Permeability (presented as Pressure Drop) with respect to batch.

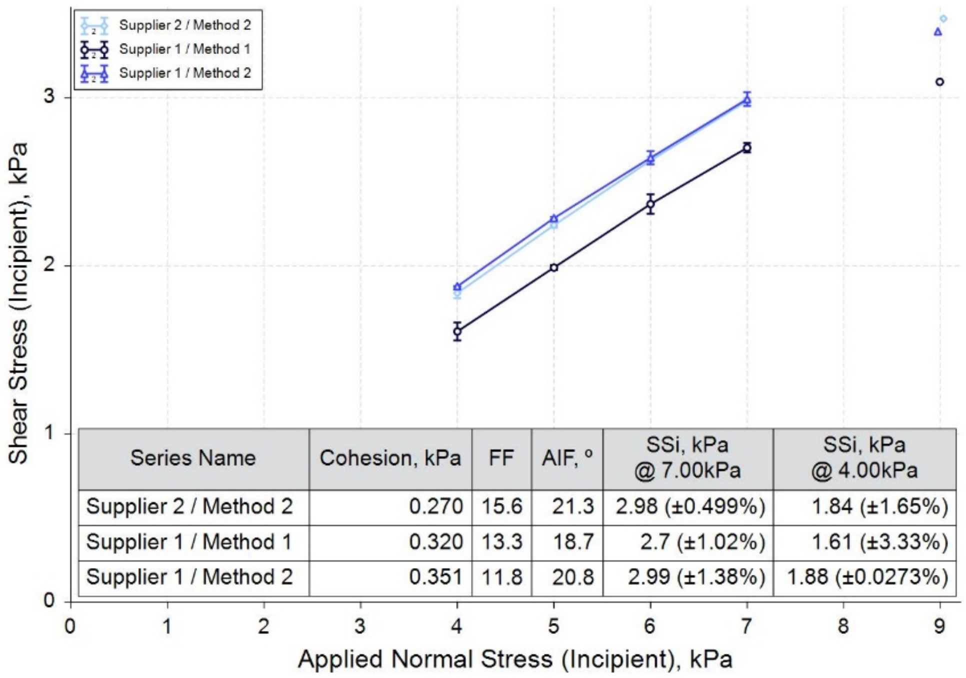
Case Study 2: The Influence of Different Suppliers and Manufacturing Methods

Feedstocks for additive manufacturing can be produced via different methods, each of which can generate powders with similar particle size and size distribution, and each manufacturer will have their own grades and acceptance criteria. However, the manufacturing method may alter other properties of the powder that aren't identified by these acceptance tests but can lead to varying performance in an AM process.

Three feedstock powders were evaluated using a powder rheometer. Two were produced using the same method, gas atomisation, but sourced from different suppliers (Supplier 1 and Supplier 2) and two from the same supplier but produced using different methods; plasma (Method 1) and gas (Method 2) atomisation. The materials all had similar d_{50} values and comparable particle size distributions.

Shear cell tests identified differences caused by the change in manufacturing method, with Method 1 generating lower shear stress values than Method 2 (Figure 3). This illustrates the impact of variables potentially outside of a customer's control and demonstrates the need for sensitive and regular evaluation of raw materials.

The samples produced by the different suppliers but using the same method, were categorised as identical by shear cell tests.



Series Name	Cohesion, kPa	FF	AIF, º	SSi, kPa @ 7.00kPa	SSi, kPa @ 4.00kPa
Supplier 2 / Method 2	0.270	15.6	21.3	2.98 (±0.499%)	1.84 (±1.65%)
Supplier 1 / Method 1	0.320	13.3	18.7	2.7 (±1.02%)	1.61 (±3.33%)
Supplier 1 / Method 2	0.351	11.8	20.8	2.99 (±1.38%)	1.88 (±0.0273%)

Fig. 3. Inability of shear cell tests to differentiate between suppliers.

However, dynamic tests not only reinforce the variation caused by the change in manufacturing method, but also identify differences in the samples from the two different suppliers using the same method. The Supplier 2/Method 2 sample has a higher BFE and SE than the Supplier 1/Method 2 sample (Figure 4), suggesting more cohesive behaviour in dynamic applications, such as filling and spreading. The variation in properties suggests that changing suppliers may have a significant influence on process performance which must be considered alongside the financial or logistical benefits of making such a change.

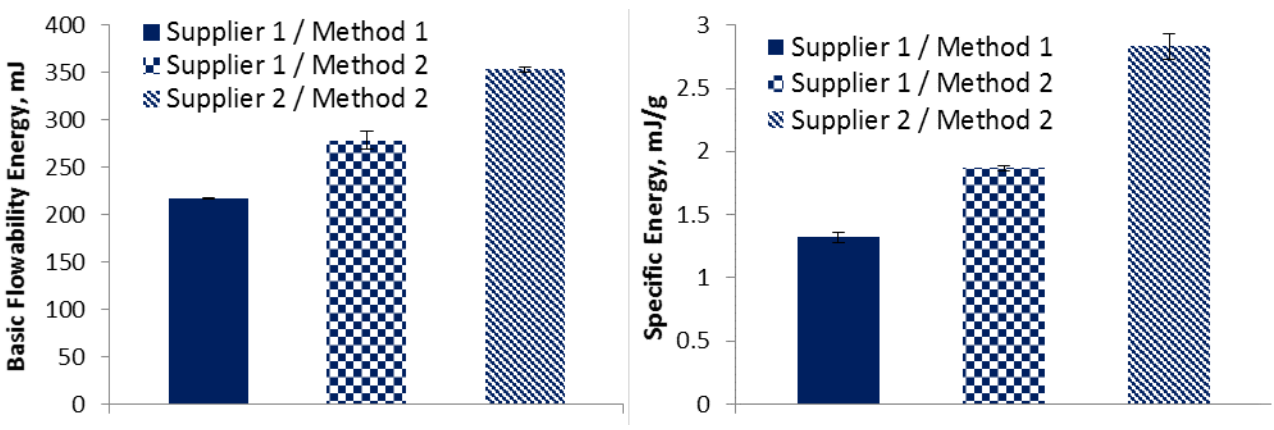


Fig. 4. Changes in BFE and SE with respect to supplier and manufacturing method.

Case Study 3: The Effect of Additives on Feedstock Properties

Feedstocks may be treated with additives to enhance functionality of the final product. However, these additives will also influence the behaviour of the feedstock and how it performs in a given unit operations. Being able to quantify the extent to which different additives will affect the properties of the mixture will allow both the mixture itself and the process to be optimised so that the benefits of the additive are maintained without compromising process performance.

Three samples of polyoxymethylene (POM), two of which contained different additives (a pigment and a flow additive) were used in a selective laser sintering (SLS) operation. It was observed that the three formulations flowed differently from the storage hopper into the sintering machine resulting in variations in the properties and quality of the final product. A range of traditional powder testing techniques did not provide differentiation between the samples so each was further tested using a powder rheometer.

The sample containing flow additive generated a higher BFE than the other two samples, requiring more energy to move the blade of the powder rheometer through the sample, (Figure 5). In this case, high BFE indicates more efficient packing within the bulk, and suggests that the addition of the flow additive has resulted in a more free-flowing material. This sample also generated the highest Pressure Drop value at a low consolidating stress, indicating reduced permeability and further reinforcing the denser packing state of this more free-flowing sample. However, while the Pressure Drop for all three samples increased with increasing consolidation stress, that of the pure POM and the sample containing pigment changed to a far greater degree than the sample containing flow additive, (Figure 6).

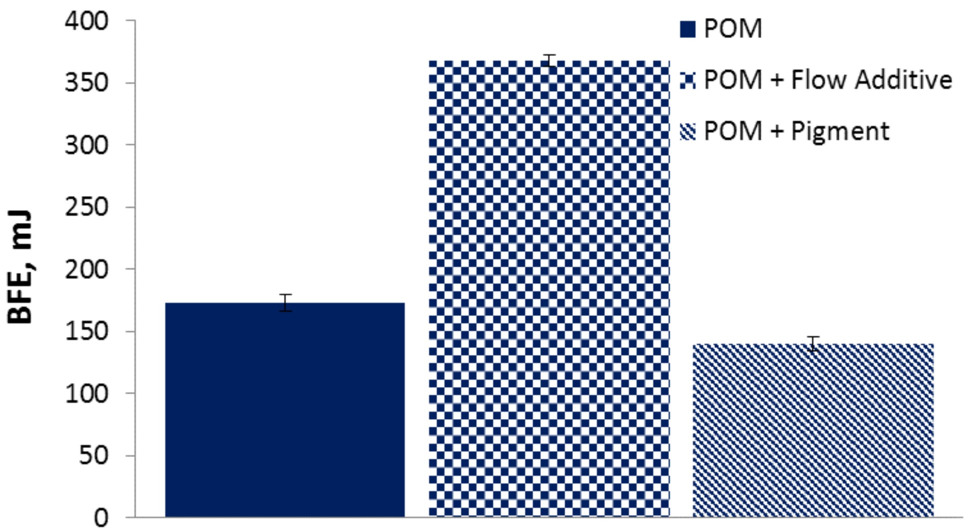


Fig. 5. The influence of additives on the BFE of POM.

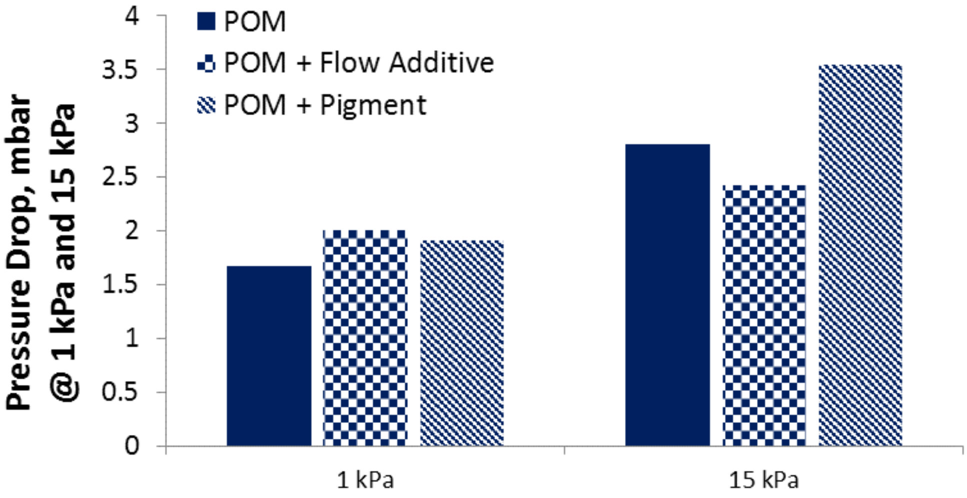


Fig. 6. The influence of additives on the Permeability (presented as Pressure Drop) of POM.

Low sensitivity to changes in consolidation stress is a further indicator of a more efficiently packed bulk, due to there being fewer air voids for the particles to collapse into when compressed. The Pressure Drop for the sample containing pigment changed to the greatest extent, consistent with it containing greater quantities of entrained air, a further indicator of high cohesivity.

Case Study 4: Process-Relevant Differences Between Fresh and Used Feedstocks

AM processes can consume significant amounts of powder, not all of which becomes part of the finished component. Powder re-use offers the potential to significantly reduce both raw material costs and overall levels of waste. However, re-use requires careful assessment of the extent to which powders are altered by an AM machine and whether further processing is possible without compromising the quality of the finished component.

A range of different feedstocks containing differing proportions of virgin and used powder were evaluated using a powder rheometer to determine whether critical characteristics of the used powder differed from those of the virgin material, and if so, what strategies might be employed to return the used powder to a condition that would enable successful re-use.

The results for the virgin and used powders show that processing has significantly increased the basic flow energy of the used powder, (Figure 7). This suggests that the used powder would not flow as freely as the virgin material and consequently is less likely to perform as well in the process. Powder exiting an AM machine may contain splatter from the melt pool in the form of larger particles, or the particles may have acquired surface contaminants for example. Experiments were therefore undertaken to determine whether sieving the used powder would return it to a state where the flow energy was again acceptable however, this ultimately had little impact.

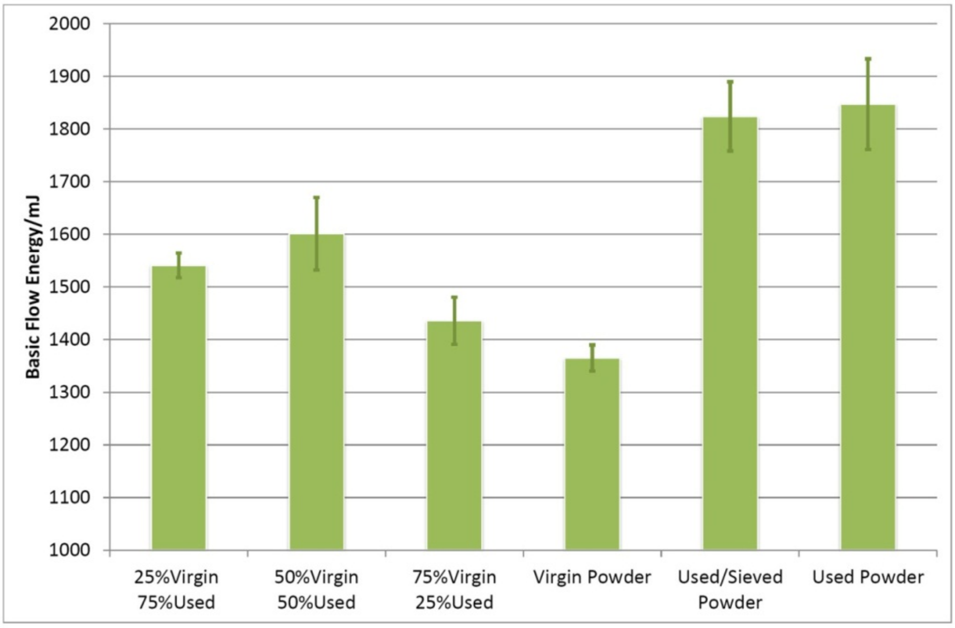


Fig. 7. Using powder rheology to optimise the use of recycled materials.

Further experiments were then conducted to ascertain whether used and virgin powder could be blended to form an acceptable feedstock for subsequent processing. A ratio of 75% virgin to 25% used powder produced a basic flow energy value most similar to that of the fresh powder. Interestingly, the 50:50 blend had the highest basic flow energy of all the blended samples, demonstrating that flowability does not change linearly with respect to the quantity of virgin fresh powder present and therefore cannot be predicted from knowledge of the blend constituents alone.

Conclusions

These case studies illustrate the ability of a modern powder rheometer to detect subtle changes in powders that are directly relevant to performance in AM processes. Powder rheology can therefore be applied to support successful optimisation and lifecycle management of powders for AM. It also demonstrates how a multivariate approach is required to fully identify powders that are compatible with a given process.

Powder flowability is not an inherent material property, but is more about the ability of powder to flow in a desired manner in a specific piece of equipment. Successful processing demands that the powder and the process are well-matched. It is not uncommon for the same powder to perform well in one unit operation but poorly in another. This means that multiple characterisation methods are required, the results from which can be correlated with process ranking to produce a design space of parameters that correspond to acceptable process behaviour.

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