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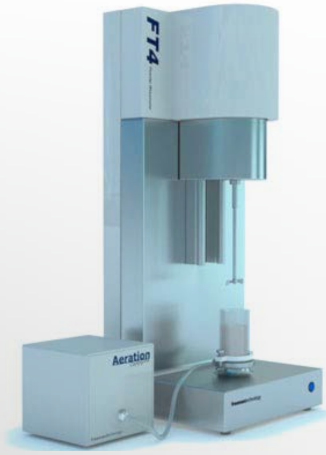
APPLICATIONS OF THE FT4 POWDER RHEOMETER® IN ADDITIVE MANUFACTURING - PART 1

POWDER FLOW

Additive manufacturing, also known as 3D printing, is a potentially transformative, highly efficient manufacturing technique. 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 process efficiency and end 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.



FT4 Powder Rheometer

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 able to meet the exacting demands of these machines. 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 of powder 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 testing, Flow through a Funnel, and Bulk Density measurements are well-documented. However, these methods were developed without the benefits of modern technology, and can sometimes be too insensitive to accurately characterise subtle differences between powders that behave differently in process.

The FT4 Powder Rheometer is a universal powder tester that provides automated, reliable and comprehensive measurements of bulk material characteristics. This information can be correlated with process experience to improve processing efficiency and aid quality control. Specialising in the measurement of dynamic flow properties, the FT4 also incorporates a Shear cell, and the ability to measure bulk properties like density, compressibility and permeability.

Quantifying Batch-to-Batch Variation in Feedstocks

The tight tolerances within which AM machines operate mean that differences between different batches of feedstocks can lead to significant variability in the properties and quality of the final product. A means of screening each batch before it enters the process can ensure that variation in performancewill be avoided. However, traditional powder characterisation techniques are often unable to identify the sometimes very subtle differences in properties that can lead to differences in performance.

Three examples of stainless steel powder from the same supplier demonstrated significantly 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 demonstrated a similar response in Angle of Repose and Hall Flow tests.

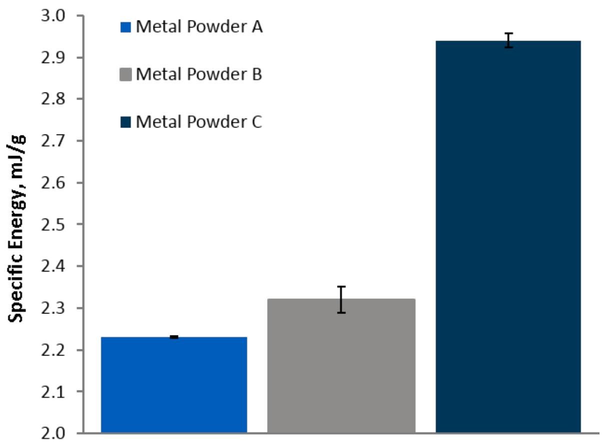


Figure 1

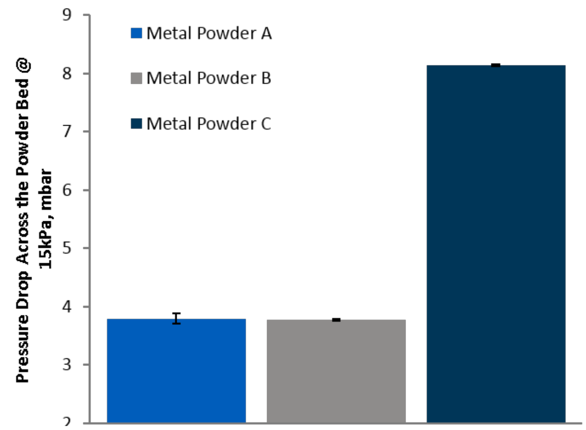


Figure 2

Evaluating the samples with the FT4, however, illustrated several differences between the samples that correlated well with the process performance. During Dynamic testing (**Figure 1**), the Specific Energy of the samples clearly differentiated Metal Powder C, with the higher value being indicative of increased mechanical interlocking and particle-particle friction. This increased resistance to flowing over itself is a common cause of blockages and other flow problems in low stress environments.

During Bulk testing (**Figure 2**), an even more differentiating result was generated by the Permeability test. Metal Powder C generates a significantly higher Pressure Drop across the powder bed than the other samples, indicating that Metal Powder C is considerably less permeable than Powders A and B.

Permeability is highly influential in any operation in which powder is moved from one position to another, particularly when the motivating force is gravity. Gas has to replace the space vacated by the particles, and the more easily the powder can transmit this gas through the bulk, the more freely it is likely to pour, and also to release any air entrained during the pouring process. Low Permeability causing an increase in the amount of air retained in the bulk when it is deposited, and when attempting to fill or deposit very consistent densities of powder during AM applications, will often cause poor uniformity in the layers, leading to imperfections in the final product that may require the product to be scrapped.

Process-Relevant Differences between Fresh and Used Feedstocks

Powder bed and laser deposition technology both require the use of 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 materials costs and overall levels of waste. However, re-use requires careful assessment of the extent to which powders are altered by passing through AM machines, and whether further processing is possible without compromising the quality of the finished component.

A range of different feedstocks containing differing proportions of fresh and used feedstock were evaluated with the FT4's Dynamic methodology, in an attempt to determine if critical characteristics of the used powder differed from those of the virgin material, and if so, what strategies might be successful in returning the powder to a condition that would enable its re-use.

Comparing the results for the virgin and used powders shows that processing has significantly increased the flow energy of the powder (**Figure 3**). This indicates 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 may have changed chemically, for example picking up contaminants on the powder surface. Experiments were therefore undertaken to determine whether sieving the used powder would return it to a state where its flow energy was acceptable. Here sieving improved powder flowability but did not return it to the original flow energy values measured for the virgin material.

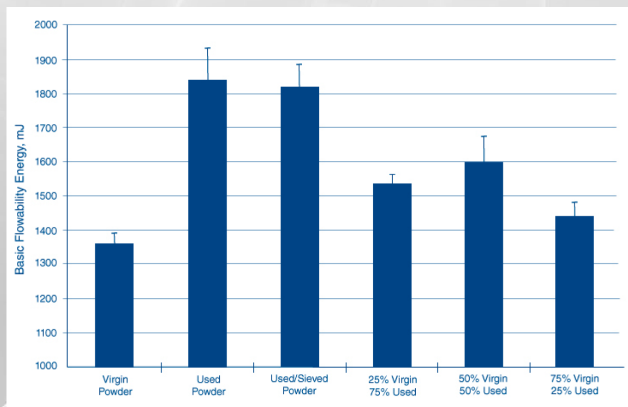


Figure 3

Further experiments were then conducted to see if the used and virgin powders could be blended together to form an acceptable feed for subsequent processing. A ratio of 75% virgin to 25% used powder produced a flowability most similar to that of the fresh powder, and this blend also exhibited relatively good performance. The 50:50 blend had the highest BFE of all the blended samples, which indicates that flowability does not change linearly with respect to the volume of fresh powder present.

These results highlight the ability of dynamic testing to detect subtle changes in powders that are of direct relevance to their performance in AM machines. As a result dynamic testing can support successful optimisation and lifecycle management of metal powders for AM, in a way that other powder flow testers cannot.

The Infuence of Different Suppliers and Manufacturing Methods

Powder feedstocks for additive manufacturing can be manufactured by different methods, each of which can generate powders with similar D50 and PSD, and each manufacturer will have their own grades and acceptance criteria. However, the manufacturing method can also influence other properties of the powder, which will lead to different performance in the overall process that the manufacturer's own acceptance testing may not be able to identify.

Three feedstock powders which had the same D50 and PSD were used for this study. Two powders from Supplier 1, one made using Gas Atomisation (GA) and the other by Plasma Atomisation (PA), and one powder from Supplier 2 made using GA. All three samples evaluated using the FT4, in an attempt to evaluate whether the different suppliers or manufacturing methods were likely to influence the performance of the powder in process.

Shear Cell tests (**Figure 4**) identified differences caused by the change in manufacturing method, with PA generating lower Shear Stress values than GA, demonstrating the impact of variables potentially outside of a customer's control, and highlighting the need for close, regular evaluation of raw materials. However, the samples produced by two different suppliers using GA were categorised as identical by Shear Cell tests.

Dynamic tests (**Figure 5**) not only reinforce the variation caused by changing the manufacturing method, but also identify differences in the samples from the two suppliers. The sample from Supplier 2 has a higher BFE and higher SE than the sample from Supplier 1, and this indicates more cohesive behaviour in dynamic applications such as filling and layering.

The variation in properties suggests that changing suppliers may have a significant influence on process performance, and this has to be considered alongside financial or logistical benefits to making the change.

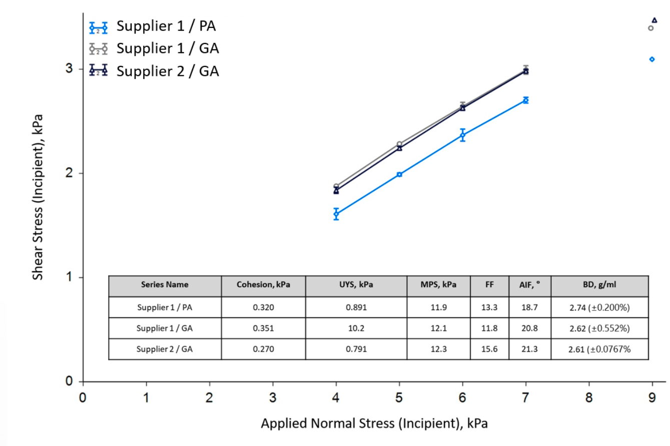


Figure 4

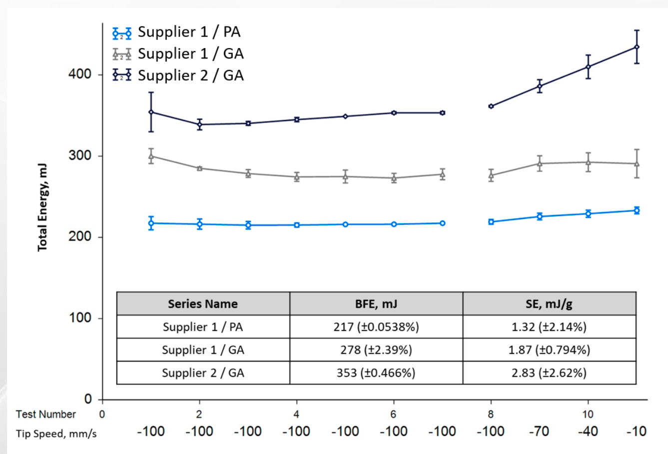


Figure 5

The Effect of Additives on Feedstock Properties

Feedstocks are often treated with different additives to provide useful properties, such as pigmentation, improved flowability or specific functionality in the final product. However, these different additives will each have a different influence on the properties of the feedstock, and over its eventual performance in the application. Being able to quantify the extent to which different additives will affect the properties of the formulation will allow both the formulation and the process to be optimised to accommodate the features of the additive and maintain an acceptable level of performance.

Three samples of Polyoxymethylene (POM), two of which contained different additives (a pigment and a flow additive) were used in an SLS operation. It was observed that the three formulations flowed differently from the storage hopper into the sintering machine resulting in variation in the properties and quality of the final product. A range of traditional characterisation techniques had been employed, but did not provide differentiation between the samples.

The sample containing the flow additive generated a higher Basic Flowability Energy (BFE) than the other two samples (**Figure 6**), requiring more energy to move the FT4 blade through the powder bulk. In this case, higher BFE is typically associated with the uniform structure of a more efficiently packed bulk, causing more particles to be displaced by the movement of the blade than in a more poorly packed powder.

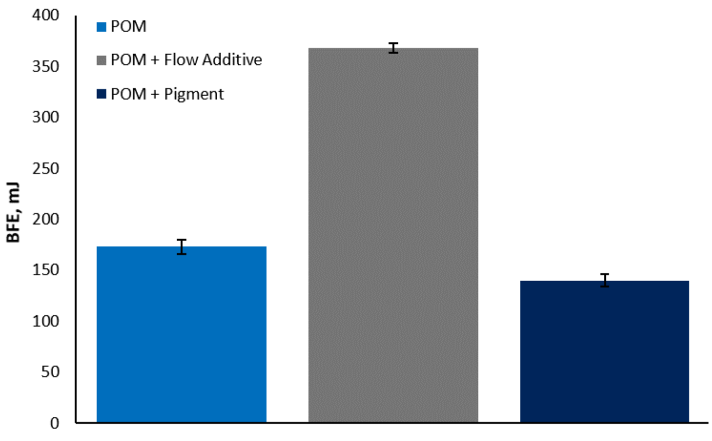


Figure 6

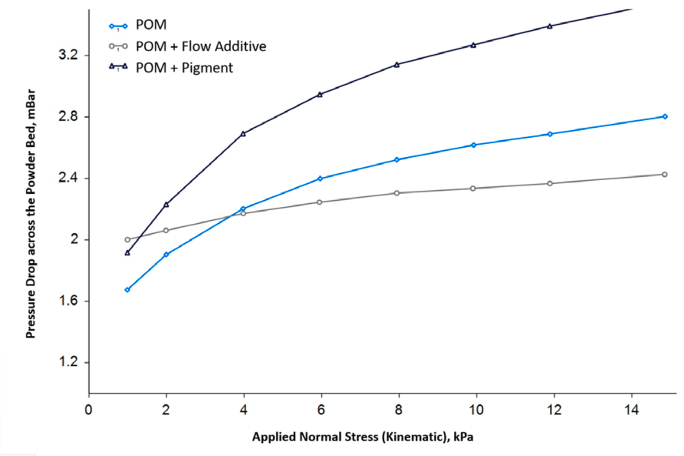


Figure 7

The sample containing the flow additive generated the highest Pressure Drop Across the Powder Bed at a low consolidating stress, indicating reduced Permeability and reflecting the denser packing state of this sample (**Figure 7**). However, as consolidating stress increased, while the Pressure Drop for all of the samples increased, that of the pure sample and the sample containing pigment changed to a far greater degree than the sample containing flow additive.

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 permeability of the sample containing the pigment changed to the greatest extent, consistent with it having the greatest volume of entrained air within the bulk, which is an indicator of high cohesivity in this mode of flow.

Conclusion

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, and it is not uncommon for the same powder to perform well in one unit operation within an AM process, but poorly in another. This means that several characterisation methodologies are required, the results from which can be correlated with process ranking to identify which parameters are most influential on performance, and produce a design space that corresponds to acceptable process behaviour.

These various studies highlight the ability of the FT4's multivariate approach to detect subtle changes in powders that are of direct relevance to their performance in AM machines. The FT4 can therefore support successful optimisation and lifecycle management of metal powders for AM, in a way that other measurement techniques cannot. It also demonstrates how even more modern techniques such as particle size analysis and Shear cell testing may not always be able to consistently characterise process-relevant differences between these types of samples, reinforcing how more than one technique is required in order to fully describe a powder's properties for a given process.

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