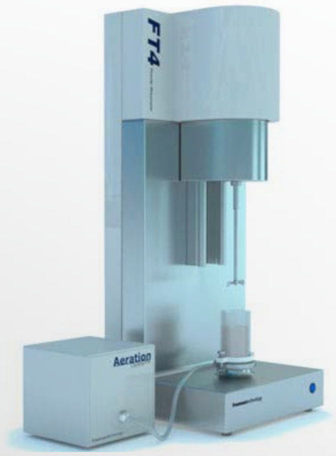
APPLICATIONS OF THE FT4 POWDER RHEOMETER® IN ADDITIVE MANUFACTURING - PART 2

POWDER FLOW

Optimising Powder Feedstocks for AM

Powder manufacturers, powder reprocessors and users of additive manufacturing (AM) all share the need to understand how to optimise powders for superior print performance. Strategies for improving performance range from changing gas atomisation conditions to the application of proprietary rejuvenation processes but successful application relies on being able to robustly evaluate impact. Powder testing can be extremely cost-efficient, relative to a print trial, when it comes to addressing the critical issue of whether a proposed processing step or strategy delivers a worthwhile gain. This application note draws together studies that illustrate application of the FT4 Powder Rheometer (Freeman Technology) within this context. Experimental data show how the instrument can relevantly quantify the impact of processing strategies to support the optimisation of AM powders.



FT4 Powder Rheometer

Case Study 1: Assessing the Impact of Gas Atomisation Conditions

Table 1/Figure 1 show data from a study by Chu et al[1] of two AlSi10Mg powder samples for laser powder bed fusion (LPBF) produced by gas atomisation. Batch 1 was made using a melt superheat of 180°C and an atomising gas pressure of 1.8 x 10^6 Pa, while Batch 2 was produced at a cooler temperature, 110°C melt superheat, and higher atomisation pressure, 3.5 x 10^6 Pa.

Powder Properties	Batch 1 Powder	Batch 2 Powder
HF (50g/s)	No-flow	No-flow
RA (˚)	39 ± 0.9	41 ± 1.0
BFE (mJ)	201.7 ± 11.0	269.3 ± 23.7
SE (mJ/g)	3.3 ± 0.2	3.7 ± 0.2
AE (mJ)	5.2 ± 1.7	15.3 ± 8.3
CE (mJ)	349.7 ± 3.5	540 ± 84.7
CI (%)	5.0 ± 0.2	6.4 ± 0.4
CBD (g/cm³)	1.5 ± 0.1	1.3 ± 0.1
CS (kPa)	0.3 ± 0.1	0.4 ± 0.2

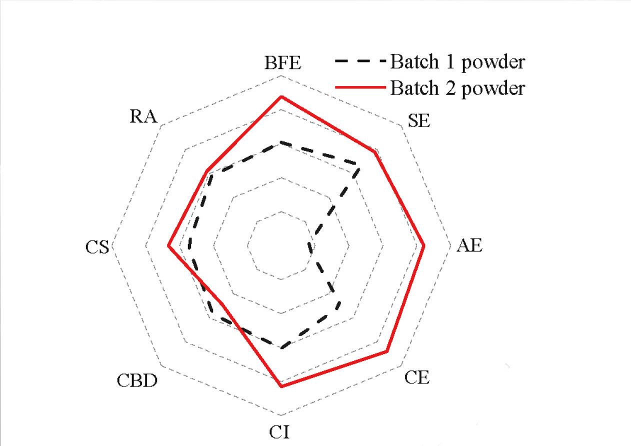


Table 1/Figure 1: Dynamic, shear and bulk property data identify Batch 1 as less cohesive and less resistant to flow than Batch 2. It also offers higher packing density (left). The lower bounded area of Batch 1 (CBD results not withstanding) identifies a powder with superior print performance.

Hall flowability (HF) data, generated using a flow through an orifice apparatus, fail to differentiate the two samples with a no flow result reported for both. Repose angle (RA), a second traditional powder testing technique, also provides no differentiation when measurement variability is taken into account. Dynamic (Basic Flowability Energy (BFE), Specific Energy (SE), Aerated Energy (AE) and Consolidated Energy (CE)), shear (Cohesion (CS)), and bulk (Compressibility Index (CI) and Conditioned Bulk Density (CBD)) powder properties were measured for both samples using an FT4 Powder Rheometer. These properties consistently identify Batch 1 as less cohesive and less resistant to flow. CBD data suggest that Batch 1 may also exhibit higher packing density though there is less robust differentiation between the two samples with this metric.

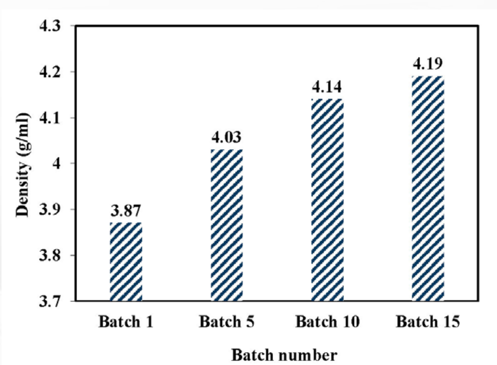
Since powders are required to flow easily in AM processes, to spread readily across the build platform to generate a uniform, densely packed powder layer, these data suggest that Batch 1 will deliver superior print performance. The radar plot is a useful way of combining these variables into an assessment of suitability since with the exception of CBD lower values are advantageous in each case. Powders with a lower bounded area on the radar plot are therefore preferable.

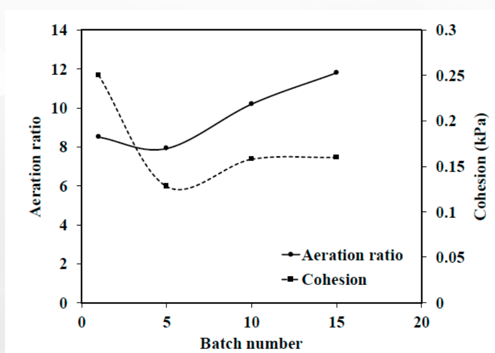
Print trials in a Laser Powder Bed Fusion (LPBF) printer (EOS, M280) confirm this correlation. Batch 1 produced samples of lower porosity, with fewer and smaller defects, notably with less lack of fusion defects.

Particle morphology provides a rationale for the poor performance of Batch 2 which consisted of coarser and less regularly shaped particles. Bulk powder testing translates the differences in particle size and shape data into quantifiable differences in process-relevant properties such as flowability. While traditional test methods - HF and RA - suggest that the powders will exhibit almost identical performance, sensitive, multi-faceted bulk powder characterisation robustly identifies the gas atomisation conditions associated with superior print performance.

Case Study 2: Assessing the Impact of Powder Re-use

Figure 2/Table 2 show data from a study carried out by researchers at the National Center for Additive Manufacturing Excellence, Auburn University using a commercial 17-4 PH SS powder (LPW Technology, UK)[2]. Print trials were carried out using a LPBF system (EOS, M290) starting with 80kg of virgin powder, Batch 1. After each print run residual, unmelted, powder was collected, sieved (80 µm screen) and mixed into the feed material which was then sampled ahead of a further run. Batch 5 is the batch of powder used for the fifth print run, similarly Batch 10 for the tenth print run, and batch 15 for the fifteenth and final run.





Print Number	1st	5th	10th	15th
RA%	29.50	35.40	34.70	33.0
ε_f	0.35	0.43	0.42	0.40

Figure 2/Table 2: CBD (left), AR and Cohesion (right) values quantify the impact of powder recycling. AR values correlate with trends in ductility observed in uniaxial tensile testing (RA% and ε_f).

Dynamic (Aeration Ratio (AR)), bulk (CBD) and shear (Cohesion) properties were measured for the samples using the FT4 Powder Rheometer. Density data indicate a progressive, though non-linear increase in particle packing efficiency as the powder is re-used, a beneficial trend. AR which is strongly influenced by inter-particular forces such as cohesion suggest a more complex change in behaviour, as recycling proceeds. Cohesion values measured by shear cell analysis provide no differentiation between more heavily used powders.

The mechanical testing of printed samples reveals that the trend in AR reflects that observed in ductility, as quantified via uniaxial tensile testing, where RA% is reduction in area and ε_f is true strain at fracture. In additional testing[3] Batch 15 was also found to exhibit superior fatigue strength at high cycle frequency, a result attributed to lower porosity which in turn can be linked with the higher packing efficiency evidenced by density values. Here then, dynamic and bulk properties both proved relevant to print quality supporting the robust assessment of the impact of powder re-use.

Case Study 3: Assessing Options for Powder Rejuvenation

Figure 3 shows data from studies to optimise the application of a rejuvenation process (Relay 3D, Eindhoven, The Netherlands) for polyamide powders for Selective Laser Sintering (SLS). The rejuvenation process restores reused powders that no longer perform well in the printer, boosting sustainability and process economics. However, it is not suitable for all powders. The aim of the study was to determine the feasibility of testing powders ahead of processing, to identify suitable candidates for rejuvenation. Traditional flow though a funnel and tapped density techniques had both proven unsuccessful for this task.

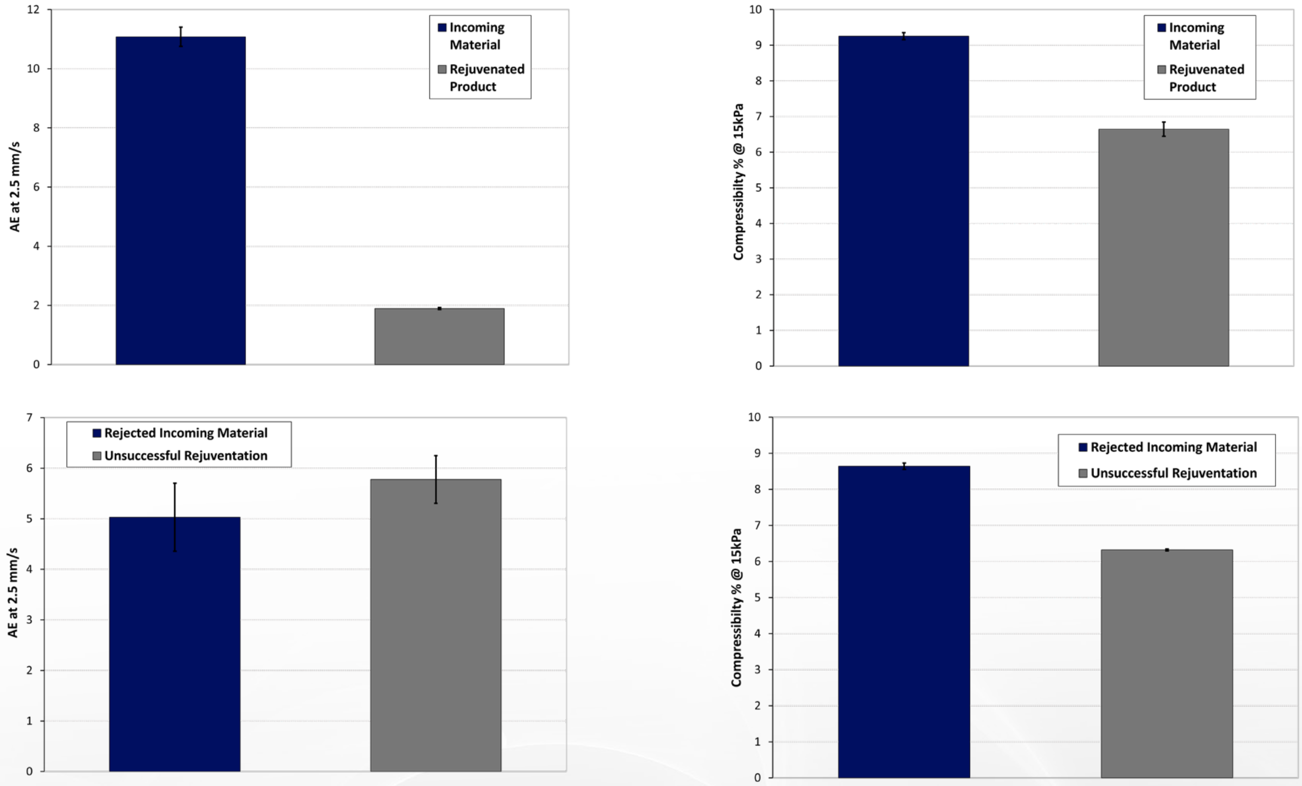


Figure 3: When successful, rejuvenation results in a significant reduction in both AE and compressibility (upper image). Failed application of the rejuvenation process is associated with an increase in AE.

Dynamic (AE) and bulk (compressibility) powder properties were measured for samples of 'as received' (by Relay 3D) and rejuvenated powders using an FT4 Powder Rheometer. Data for a successfully rejuvenated powder shows that the process substantially reduces both AE, measured with an upward air velocity of 2.5 mm/s, and compressibility. This suggests that rejuvenation lowers the strength of interparticular forces, making the powder less cohesive, more free-flowing and more efficient with respect to packing. In contrast, a failed application of rejuvenation results in an increase in AE though change in compressibility is comparable. Details of the rejuvenation process were not disclosed so the reasons for this alternative and unusual pattern of behaviour are unclear. However, observed changes were reliably associated with print performance.

Figure 4 shows data for three powder candidates: a fresh incoming sample (blue), an aged sample for which rejuvenation proved successful (orange), and an aged sample that failed to rejuvenate (grey). Aerated energy values are expressed in the form Normalised Aeration Sensitivity (NAS), a parameter that allows an easy comparison of a powder's response to aeration, independent of air velocity. Comparing the properties of powders that successfully rejuvenated with those that did not provides a method foridentifying suitable candidates. Incoming samples with a higher NAS, those that are more sensitive to the effects of aeration, especially in combination with relatively low compressibility, are unlikely to rejuvenate successfully. Samples that are more cohesive as sensitively detected by a lower NAS, are more suitable candidates.

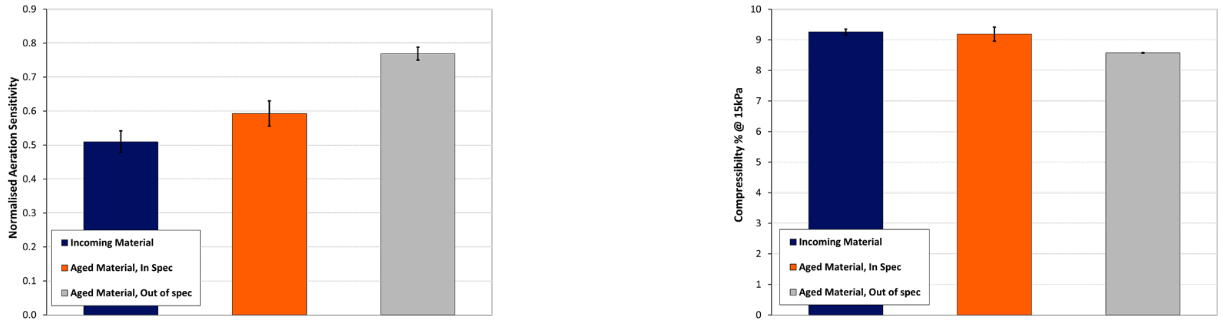


Figure 4: Suitable candidate powders for rejuvenation combine a low NAS value, with marginally higher compressibility.

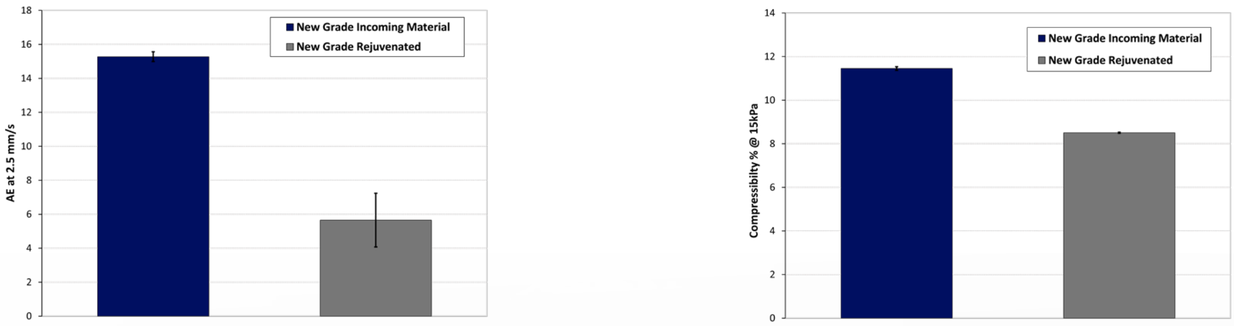


Figure 5: Data for a new grade of polyamide powder incorporating titanium dioxide demonstrates the applicability of the proposed method for identifying suitable candidates for rejuvenation.

To further test the validity of the proposed method it was applied to a new grade of polyamide powder incorporating titanium dioxide. Once again the successful candidate for rejuvenation was identified by aeration characteristics (see **Figure 5**).

Here, dynamic properties primarily proved a robust indicator of suitability for rejuvenation, with compressibility also providing complementary insight. This specification is extremely valuable since it prevents the application of rejuvenation to powders that will not benefit, preserving throughput for those that will.

Conclusion

When working to improve the print performance of AM powders, whether by modifying gas atomisation conditions or optimising recycling or rejuvenation strategies, it is valuable to be able to assess the impact of change without carrying out a print trial. The example studies presented here demonstrate how multifaceted powder testing with an FT4 Powder Rheometer meets this goal. Such testing differentiates powders sensitively and relevantly, robustly identifying changes with a positive effect on print performance. This advanced approach to powder testing is therefore a cost-effective tool for AM powder manufacturers, processors and users working to optimise powder performance.

References

[1] F. Chu et al 'Influence of Satellite and Agglomeration of Powder on the Processability of AlSi10Mg Powder in Laser Powder Bed Fusion' Journal of Material Research and Technology, https://doi.org/10.1016/j.jmrt.2021.02.015

[2] P. D. Nezhadfar et al 'The Effects of Powder Recycling on the Mechanical Properties of Additively Manufactured 17-4 PH Stainless Steel' Solid Freeform Fabrication 2018: Proceedings of the 29th Annual International Solid Freeform Fabrication Symposium – An Additive Manufacturing Conference.

[3] A. Soltani-Tehrani et al 'Fatigue behavior of additively manufactured 17-4 PH stainless steel: The effects of part location and powder re-use.' Additive Manufacturing, 36, 101398.

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