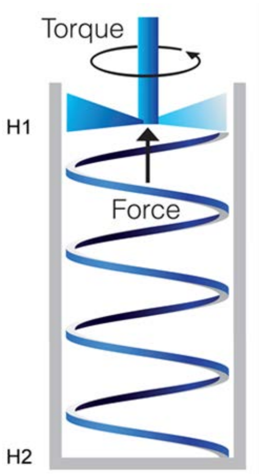
Additive manufacturing is rapidly gaining acceptance in a wide range of industries due to the ability to manufacture complex components quickly and precisely. As industrial implementation of the technology increases, so does the diversity of environmental and storage conditions which feedstocks are subjected to. This can significantly impact process performance and product quality. This study[1] investigates the relationships between the flow properties of two batches of a stainless steel powder, with varying particle sizes, and their preparation process and subsequent storage conditions. The results demonstrate how different baking processes can influence flow properties that are known to affect AM performance and also highlight how powders with different particle sizes respond differently to the same preparation and storage conditions.

THE FT4 POWDER RHEOMETER®

The FT4 Powder Rheometer® is a universal powder tester that provides automated, reliable and comprehensive measurement 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 such as density, compressibility and permeability, enabling a comprehensive characterisation of the powder in a process relevant context.

Dynamic testing employs a unique measurement technique to determine a powder's resistance to flow. A specially shaped blade traverses along a prescribed path through a precise volume of the powder. The force and the torque acting on the blade, as it moves axially and rotationally, are combined to generate a value for flow energy[2].



MATERIALS AND METHODS

Samples of twelve stainless steel powders were characterized using an FT4 Powder Rheometer® (Freeman Technology, UK). Two different particle size distributions (PSDs), PSD1 (d_{50} = 12 µm) and PSD2 (d_{50} = 15 µm), were divided into three sub batches. One batch was maintained in its virgin state, and the other two were baked in air or nitrogen (N_2) atmospheres at 200°C for 12 hours. Each batch was then split with half stored in ambient conditions and half with a desiccant, (16.5g calcium oxide desiccant stored in a pouch per 1600g of metal powder).

Particle Size	Baking Conditions	Storage
PSD1	N/A (Virgin)	Desiccant
PSD1	N/A (Virgin)	Ambient
PSD1	Air baked	Desiccant
PSD1	Air baked	Ambient
PSD1	N_2 baked	Desiccant
PSD1	N_2 baked	Ambient
PSD2	N/A (Virgin)	Desiccant
PSD2	N/A (Virgin)	Ambient
PSD2	Air baked	Desiccant
PSD2	Air baked	Ambient
PSD2	N_2 baked	Desiccant
PSD2	N_2 baked	Ambient

Table 1. Summary of samples measured via powder rheology

INFLUENCE OF BAKING CONDITIONS

The flow properties of samples subjected to different baking processes, then stored in ambient conditions were compared (Figure 1).

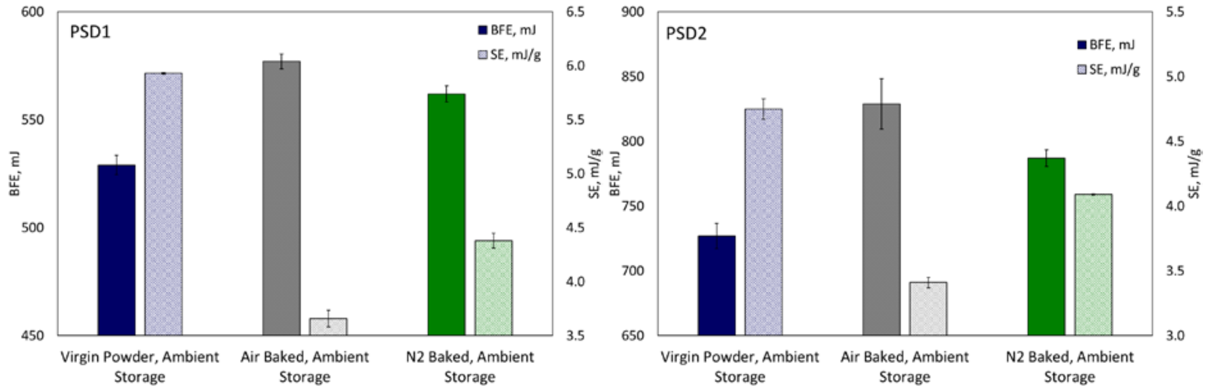


Figure 1. Dynamic flow measurements of PSD1 (left) and PSD2 (right) stored in ambient conditions, virgin, baked in an air or nitrogen (N_2) atmosphere.

For both PSD1 and PSD2, an increase in Basic Flowability Energy (BFE) and decrease in Specific Energy (SE) were observed when the virgin powder was baked, in both air and nitrogen. This suggests that baking the virgin material decreases the level of inter-particle friction, resulting in a more efficiently packed powder bed. These trends were most pronounced following baking in air. The variations in flowability are likely to result from changes in the surface properties.

INFLUENCE OF STORAGE CONDITIONS

This part of the investigation focused on how storing virgin and baked samples under ambient conditions or with a desiccant can influence flow properties. As shown in Figure 2, for both virgin PSD1 and PSD2, the materials stored in ambient conditions generated lower BFE and SE values compared to the desiccated samples. This suggests that moisture is likely being retained which subsequently decreases the resistance of the powder to the traversing blade.

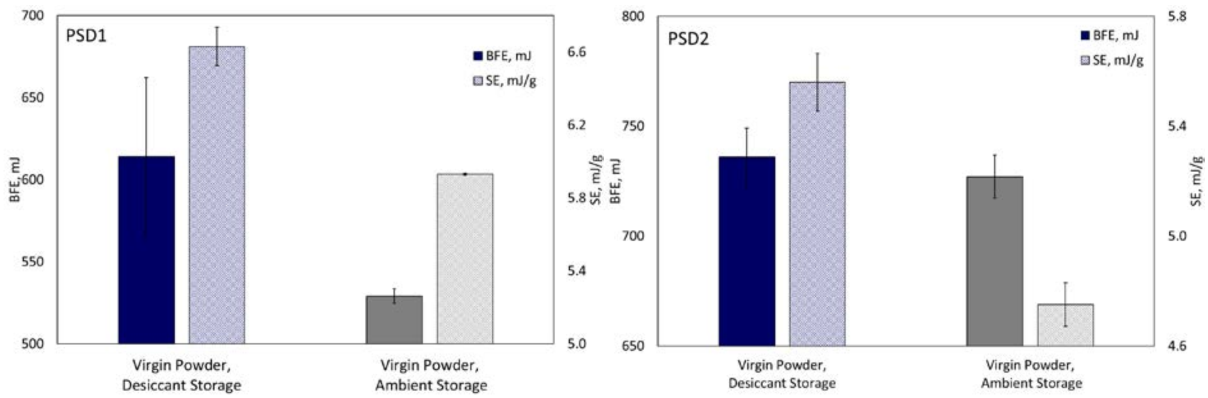


Figure 2. Dynamic flow measurements of virgin powder stored with a desiccant or in ambient conditions, for PSD1 (left) and PSD2 (right).

The PSD1 air baked samples displayed a comparable trend to the virgin samples as a function of storage conditions. The PSD1 samples stored with a desiccant generated the highest BFE and SE values (Figure 3), highlighting that these materials were more sensitive to storage conditions. Similarly, for PSD2, there was an increase in the SE value generated when the sample was stored with a desiccant. This suggests that powder baked in air and then stored under ambient conditions will exhibit a lower degree of friction under low stress conditions. It is possible that the moisture present in ambient conditions is acting as a lubricant decreasing friction between particles.

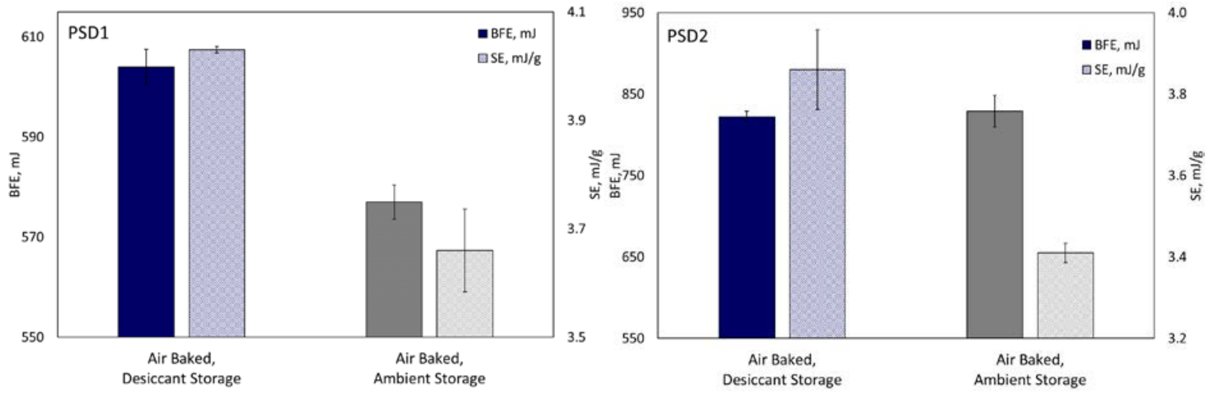


Figure 3. Dynamic flow measurements of air baked PSD1 (left) and PSD2 (right) stored with a desiccant or in ambient conditions.

The PSD1 nitrogen baked samples also displayed lower BFE and SE values when stored in ambient conditions. Whereas PSD2 under the same baking conditions has a lower SE and marginally higher BFE following ambient storage. This suggests that, as with powders baked in air, storing in ambient conditions results in a lower degree of particle-particle friction and potentially lower mechanical inter-locking. Further suggesting that the moisture present may act as a lubricant.

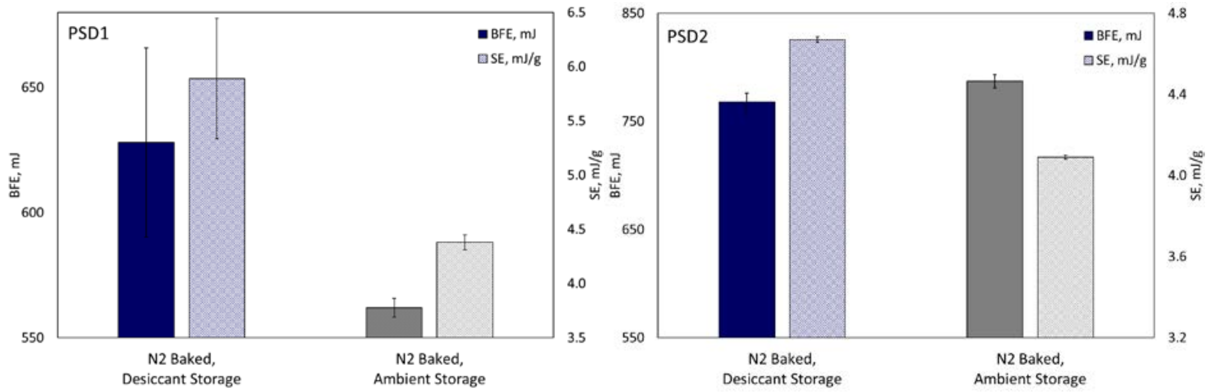


Figure 4. Dynamic flow measurements of N_2 baked PSD1 (left) and PSD2 (right) stored with a desiccant or in ambient conditions.

Despite large differences in BFE and SE being observed as a result of changes in baking conditions, the subsequent storage conditions were shown to impact the flow properties so significantly that changes in the ranking of the materials occurred. Figures 2 and 6 demonstrate that a comparable material ranking is observed for PSD2 following storage both under ambient conditions and with a desiccant, indicating that storage conditions did not have a significant impact on powder behaviour.

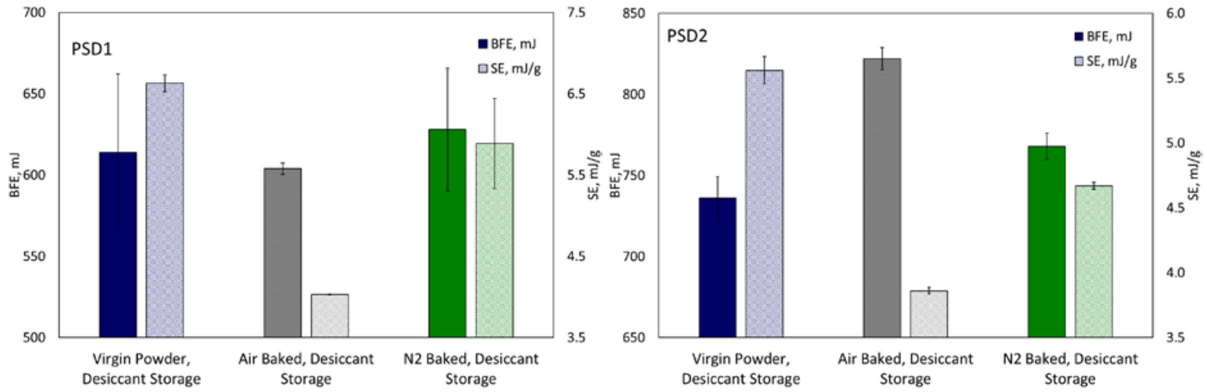


Figure 5. Dynamic flow measurements of PSD1 (left) and PSD2 (right) stored with a desiccant, virgin, baked in an air or nitrogen (N_2) atmosphere.

However, for PSD1, the significant increase in BFE observed for the baked, ambient samples did not occur when samples were stored with desiccant. This implies that additional considerations should be made into post-baking storage conditions. The resources committed to improving flowability through baking may be ineffective if subsequent storage controls are not maintained.

INFLUENCE OF PARTICLE SIZE

Given the two different PSDs, the differences in the measured flow properties between PSD1 and PSD2 are unsurprising. In general, PSD2 generated higher BFE values and has a lower SE. The varying responses of PSD1 and PSD2 to changing conditions (baking and storage) demonstrate the influence of particle size distribution on these processes.

CONCLUSIONS

In most cases, baking the powders resulted in a material that was more resistant to forced dynamic flow, but would also exhibit less resistance under unconfined flow conditions. This is highly likely to result from changes in the surface properties of the particles which lower the degree of inter-particular friction. Whereas, virgin powder and powder stored with desiccant was more likely to show increased resistance in unconfined flow processes. By storing the samples in ambient conditions, it is possible that moisture present on the surface of the particles is retained and acts as a lubricant.

This study demonstrates how powder rheology can be employed to identify and quantify minor differences in powders that directly impact performance in AM processes. Even for powders that may have the same chemical composition, variations in PSD can result in changes to how the powder responds to environmental variables. By studying different PSDs, it is clear that the effects of baking and storage is size distribution dependent, and conclusions or predictions cannot be made based on the performance of only one batch, tested under a single set of conditions.

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 process but poorly in another. Multi-faceted characterisation provides an essential foundation for understanding the variable behaviour of powders, enabling the properties that are most relevant to in-process performance in any unit operation to be identified and quantified.

For further information, please contact the Applications team on +44 (0)1684 851 551 or via support@freemantech.co.uk.

[1] *Dattani R. et al., The Effect of Preparation Techniques and Storage Conditions on the Flow Properties of Additive Manufacturing (AM) Feedstocks. EuroPM Proceedings, 2019*

[2] *Freeman R., Measuring the flow properties of consolidated, conditioned and aerated powders – A comparative study using a powder rheometer and a rotational shear cell. Powder Technology, 25-33, 174, 1-2, 2007*