LEARNING TO PREDICT PRINT QUALITY FROM AM POWDER PROPERTIES

The additive manufacturing (AM) industry continues to seek ways of reliably identifying powders that will deliver printed components with the required mechanical properties. There is widespread understanding that such specifications necessarily include multiple chemical and physical parameters. Physical specifications can be further sub-divided into particle variables, notably particle size and shape, and bulk powder properties such as density. Determining the bulk powder properties that are most relevant with respect to quantifying the spreading and packing behaviours that define AM performance remains an ongoing focus.

A new study from De Montfort University adds further weight to the case for including dynamic and bulk properties in AM powder specifications. In this work an advanced powder tester (FT4 Powder Rheometer®, Freeman Technology) was used to establish robust correlations between powder properties and the mechanical characteristics of polyamide (PA12) samples printed by Selective Laser Sintering (SLS). The results demonstrate how with the application of appropriate techniques it is feasible to predict print quality from test results and move away from a trial-and-error approach that is over-reliant on print trials.

Characterising powders for SLS

PA12 Smooth Print Ready (PR) and PA12 Smooth Fresh (Fresh) are closely similar commercially available grades of polyamide specified for SLS (Sinterit, Poland). Information from the manufacturer indicates comparable particle size distribution and suggests that the two products should be used in combination. PR is recommended for a first print run followed by subsequent recycling in a 70:30 ratio with Fresh to minimise waste and optimise cost efficiency. Fresh is known to have a smaller shrinking ratio than PR.

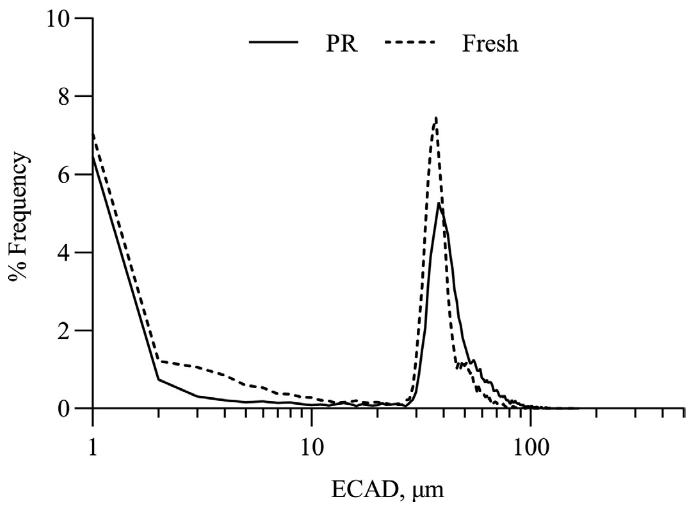


Figure 1: Image analysis data confirm the close similarity of PR and Fresh with respect to particle size distribution.

Figure 1 shows particle size data for PR and Fresh measured by image analysis (Particle Insight Dynamic Image Analyzer, Particulate Systems, USA). Particles predominantly lie in the 20 – 100µm size range for both powders with an appreciable level of dust/fines (<2µm) present in the as received state. PR has a median particle size of 42µm (equivalent circular area diameter) while that of Fresh is 37µm. Figure 2 shows scanning electron microscopy (SEM – EVO LS 15, Carl Zeiss, Cambridge, UK) images for the powders. These provide further evidence of comparable morphology though close inspection reveals nano-scale features on the surface of PR particles that are absent from Fresh.

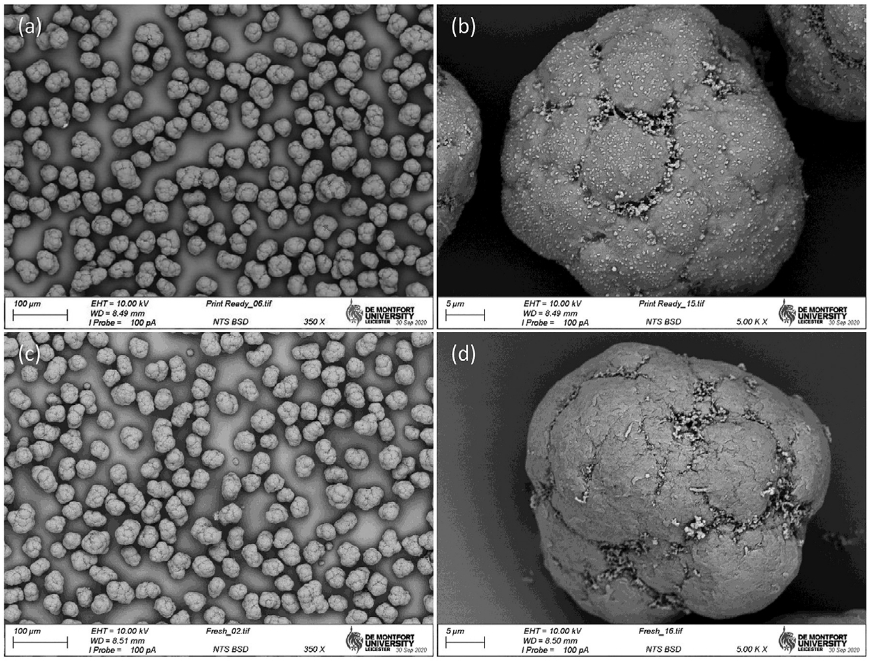
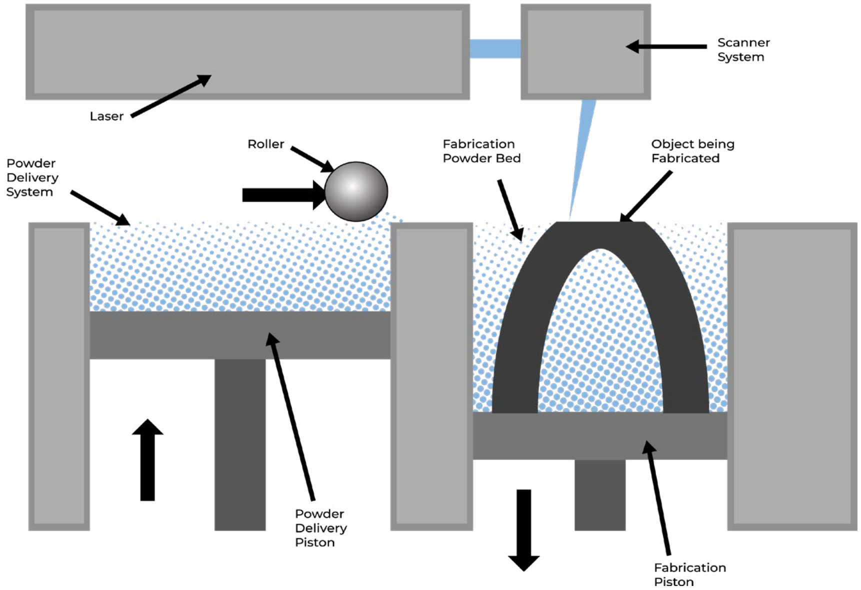


Figure 2: SEM images show that PR (a and b) and Fresh (c and d) particles have comparable morphology though there is evidence of nanoscale features on the surface of PR particles that are absent from Fresh.

These data provide useful insight into subtle differences between the two as received powders but do not enable the prediction of differences in print quality. Bulk powder properties were therefore measured to provide a more directly relevant assessment.

The SLS process environment



SLS is a prime example of the powder bed fusion processes that dominate the industrial AM landscape. Such processes are reliant on the progressive dispersion and spreading of fine, even layers of powder which are then thermally fused to fabricate a solid structure (see figure below).

Key elements of SLS technology include the powder delivery system, from which stored powders are consistently discharged on to the build platform, and the roller which spreads powder into uniform, well packed layers. Powder flowability is crucial to behaviour in this part of the process since intermittent or inconsistent flow can give rise to non-uniform deposition, porosity in the bed, and, by extension flaws or defects in the printed component.

The laser is required to impact just the upper powder layer forming a highly localised molten pool that equates to precise tolerances in the finished item. As only relatively small amounts of powder are incorporated into the emerging object powder recycling is essential for economic application. Partial melting and /or splatter can compromise the quality of unused powders and at the same time adversely affect surface finish and/or geometric accuracy.

Focusing on bulk powder testing

Samples of five powder blends were characterised to provide a basis for the correlation of powder properties and print quality (see table 1). The aim was to relevantly differentiate PR and Fresh and to develop an understanding of how the two behave in combination, in blends.

Powder Blend	Volume % of PR	Volume % of Fresh
PR	100	0
PR70	70	30
PR50	50	50
PR30	30	70
Fresh	0	100

Table 1: Blends of PR and Fresh were made up to provide samples for characterisation and printing.

Dynamic properties for the five blends are shown in Table 2 (FT4 Powder Rheometer, Freeman Technology, Tewkesbury, UK). Values of Basic Flowability Energy (BFE) are generated from measurements of the torque and axial force acting on the blade of the powder rheometer as it rotates down through a conditioned powder sample. The downward traverse of the blade pushes powder against the confining base of the test vessel subjecting it to a forced flow regime somewhat analogous to the spreading action of the roller across an SLS build platform. BFE measurements automatically generate values of Conditioned Bulk Density (CBD) a highly repeatable measure of bulk density due to the conditioning cycle that is integral to the measurement method. Conditioning involves gentle and reproducible agitation of the powder bed to ensure measurement in a homogeneous, uniformly packed state. CBD values therefore provide insight into inherent particle packing efficiency a critical behaviour with respect to the porosity of the layer laid down during the powder spreading process.

Aeration Ratio (AR) quantifies the impact of aeration on a powder sample and is determined by measuring flowability as air flows upwards through the sample, in this case at a velocity of 2.5 mm/s. The resulting values are ratioed to BFE to generate values of AR. AR values are sensitive to the strength of inter-particular cohesive bonds, with higher cohesivity giving rise to lower AR values.

Powder Blend	CBD, g/ml	BFE, mJ	$AR_{2.5}$
PR	0.463 (±1.57%)	74.4 (±3.44%)	10.4 (±9.51%)
PR70	0.457 (±0.92%)	74.3 (±0.90%)	10.4 (±25.50%)
PR50	0.492 (±1.87%)	83.6 (±9.84%)	13.5 ± (21.2%)
PR30	0.499 (±0.95%)	87.9 (±8.59%)	16.2 (±14.80%)
Fresh	0.517 (±0.53%)	102 (±3.48%)	31.2 (±28.80%)

Table 2: Dynamic properties clearly differentiate the five powder samples.

Given that particle density is equivalent for all the powders, the higher CBD of Fresh indicates greater particle packing efficiency than in PR; in general, data for the blends trend between these two extremes. Particle packing is directly relevant to print performance with porosity in the bed potentially translating into flaws and defects in the finished component. It also influences BFE values since efficiently packed powder beds, by definition, have fewer voids and by extension limited space for particle rearrangement. Such beds therefore present significant resistance to blade movement which consequently creates a large flow zone and a correspondingly high BFE value. This effect is evident in the BFE data with Fresh exhibiting a higher value than PR.

In aeration testing, there is potential for the upward air flow to individually lubricate each particle, substantially reduced flow energy. This effect is evident in the AR2.5 value for Fresh which is high suggesting the powder is readily fluidised. In contrast, the AR2.5 value for PR is considerably lower indicating stronger cohesive bonds and a level of particle-particle interaction that inhibits uniform aeration.

Overall, the observed trends in CBD, BFE and AR values are consistent and can be rationalised with reference to the nanoscale features on the surface of PR particles. Higher amounts of PR in the blend increase the likelihood of mechanical interlocking between particles and the strength of particle-particle interactions, creating structure and porosity in the powder bed and reducing flowability.

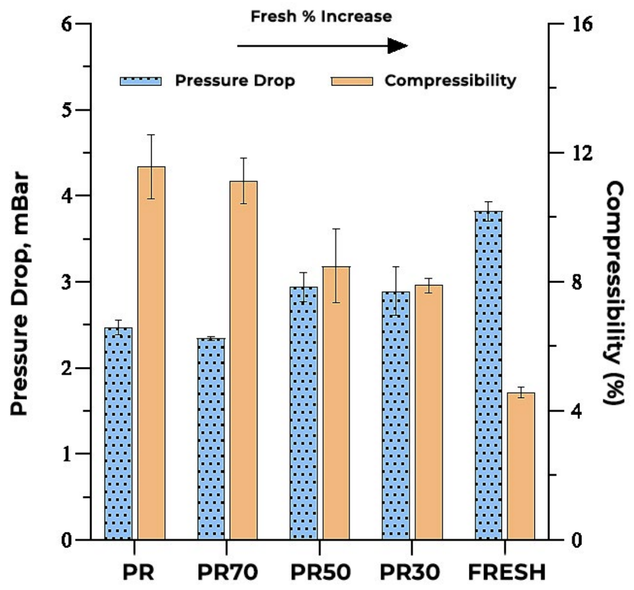


Figure 3: Permeability (pressure drop) and compressibility data provided further insight into the behaviour of the powder blends.

Bulk powder property measurements reinforce these observations. **Figure 3** shows permeability (pressure drop) and compressibility values for the powders. Permeability is determined from measurements of the pressure drop associated with the passage of air through a powder sample subject to an applied normal stress. Higher pressure drop values therefore equate to lower permeability and a greater resistance to the passage of air. Compressibility values are derived from measurements of the change in volume as a function of applied compressive load.

As the amount of Fresh powder in the blend increases the powder bed becomes progressively more densely packed creating greater resistance to the passage of air – pressure drop across the bed increases, permeability decreases – and reducing compressibility, due to lower bed porosity and a corresponding reduction in the scope for particle rearrangement.

Taken together, all these data suggest that Fresh will produce better quality print components than PR as a result of better flowability and improved packing efficiency.

Printing and characterizing test specimens

Test specimens were fabricated by SLS (Sinterit Lisa SLS 3D Printer, Sinterit, Poland) with each of the five powder samples, using a layer height of 0.125 mm and a laser power ratio of 1.0; the chamber temperature of the printer was set at 180°C.

- Nine 2 cm cubes were printed for volume and density assessment with density measured by gas displacement pycnometry (AccuPyc II 1340, Micromeritics Instrument Corporation, Norcross, USA).
- Five specimens for each of the five powders were printed for tensile testing with geometries in line with the ASTM D 638 03 Type IV standard and tensile properties was then quantified up to the point of specimen failure (Instron 3360, Norwood, USA).
- Print samples of dimensions 200*20*6 mm were printed for surface hardness testing in accordance with the ASTM D785 03 standard and used to measure HRL values (ZHR Rockwell Hardness Tester, Zwick Roell, Leominster, UK).

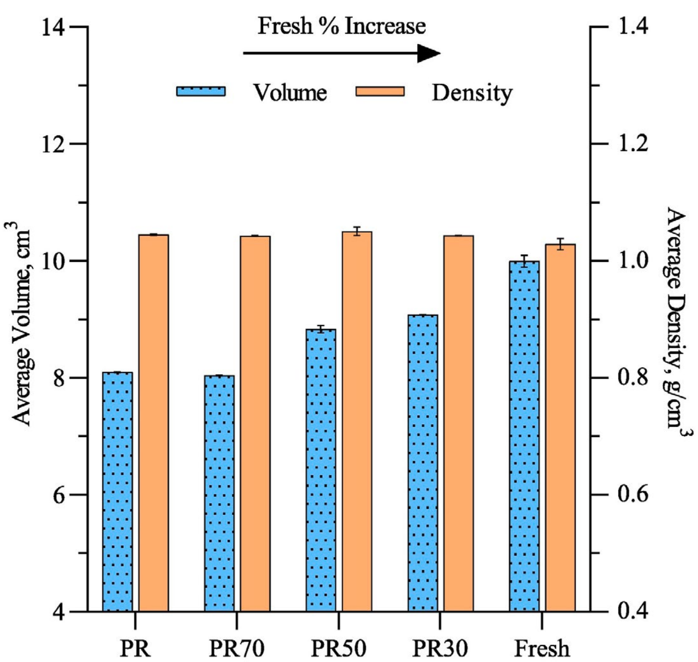


Figure 4: The five blends vary with respect to print precision, as evidenced by volume measurements, though density values are comparable for all powder samples.

Figure 4 shows volume and density data for the nine cubes. Repeatability for each blend is high (<1% RSD) indicating consistent powder distribution prior to sintering. However, as the level of Fresh in the blend increases geometric tolerance becomes poorer, the volume of the printed cubes increases beyond the target volume of 8 cm^3 (2*2*2 cm cube) with the Fresh powder producing cubes almost 10 cm^3 in size. Poor dimensional accuracy is a recognized issue in SLS and in this instance can be directly attributed to improvements in particle packing density. In more densely packed beds conductive heat transfer can sinter particles in proximity to the laser path eroding the precision of the fabrication process. The smaller shrinking ratio of Fresh is also likely to be a contributing factor.

On the other hand, printed sample density is similar across the powders. Though the higher packing efficiency associated with higher levels of Fresh might be expected to produce higher print densities the results suggest that this effect is offset by the lower shrinking ratio. The density of printed samples is therefore consistent despite the trend in bulk density.

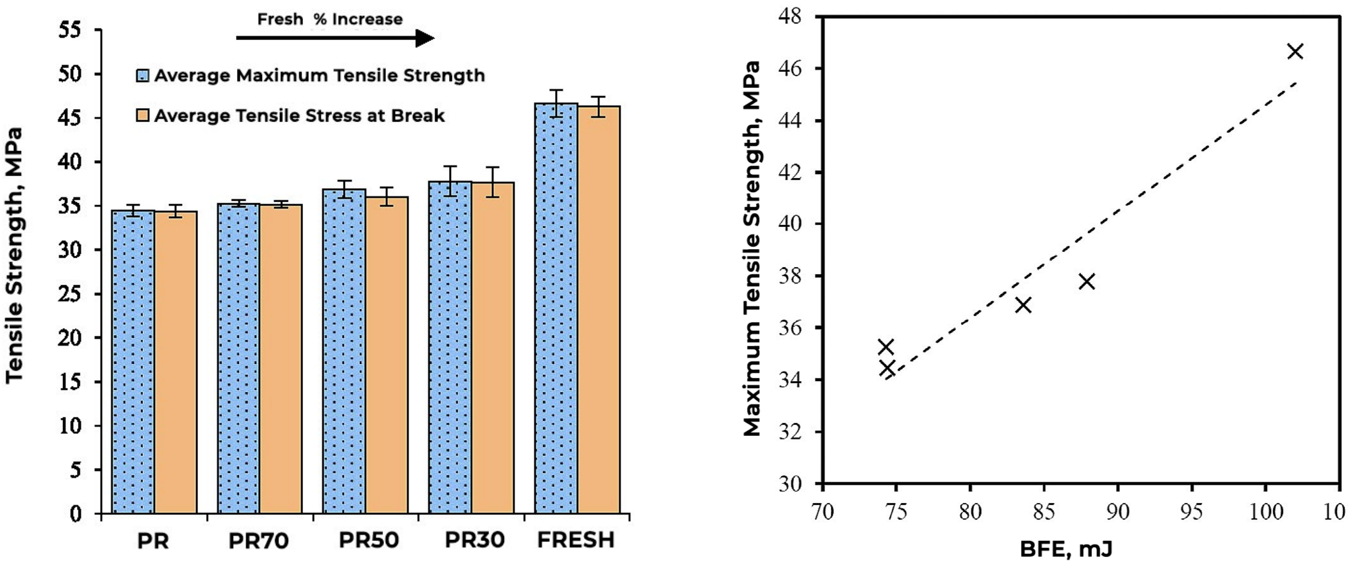
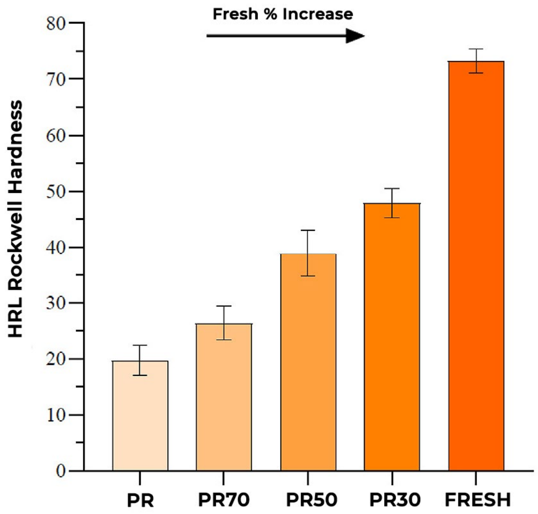


Figure 5: Tensile strength data show evidence of the superior mechanical properties associated with higher levels of Fresh.

Figure 5 shows tensile strength data for the powders and average tensile stress at the point of breakage. Here, higher ratios of Fresh, i.e. blends with higher CBD and BFE, produce enhanced mechanical properties with samples exhibiting higher strength at both peak tensile strain and immediately prior to breakage. The maximum tensile strength with Fresh is around 37% than higher than with PR. These results can be rationalised in terms of both powder flowability and bulk density. Better flowing powders efficiently produce densely packed beds that promote the formation of strong bonds during sintering and uniformly distributed pores in the finished component. In contrast to Fresh, PR flows less easily due to the nanoscale features on the particle surface which increase mechanical interlocking and reduce the contact area between particles. These effects disrupt the uniformity of the powder bed, giving rise to weaker sintered bonds that compromise mechanical properties. The robust correlation between maximum tensile strength and BFE (see figure 4 right) captures this relationship and illustrates the value of BFE measurements with respect to predicting tensile strength for a specific blend.



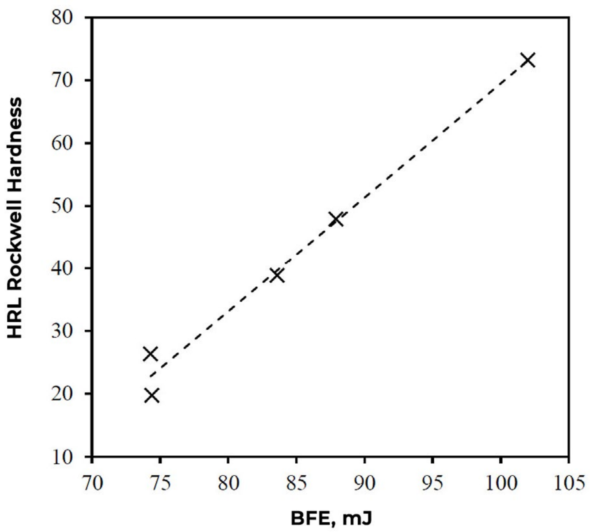


Figure 6: Surface hardness data show further evidence of the superior print quality associated with higher Fresh content.

Figure 6 shows HRL surface hardness values for the five powders which increase exponentially with Fresh content; the hardness of as received Fresh powder is around four times that of PR. Though this improvement is more marked than the differences observed in tensile behaviour it can be similarly attributed to improved particle packing and better powder flowablity, as evidenced by the direct correlation with BFE data (see figure 5 – right). This robust correlation provides further evidence of the value of the BFE measurements for the prediction of mechanical properties.

In conclusion

The ability to identify powders that will deliver target print quality and mechanical properties is extremely valuable for the efficient application of AM processes such as SLS. In contrast, an over-reliance on print trials increases costs and waste while at the same time slowing progress. The data presented here show how multi-faceted powder characterisation with an advanced powder tester can provide the detailed insight needed to differentiate powders with respect to print performance, even when those powders are closely similar. Powders with better flowability rapidly form efficiently packed powder layers providing a foundation for superior print quality, though the associated risk of poor geometrical accuracy also requires consideration. By providing such insights powder testing can make it easier, quicker, and more cost-efficient to identify and develop optimized powders for specific applications.

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