Powder pre-conditioning for the LS process

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Abstract

It has previously been shown that the use of recycled Nylon-12 powder leads to increased Elongation at Break (EaB) of Laser Sintered (LS) parts, possibly due to elevated powder temperature in the part bed causing increases in molecular weight. However, this increase in EaB often corresponds to a decrease in repeatability, likely to be through variations in powder history.

The research presented here has shown that thermal pre-conditioning of virgin powder can increase EaB of LS parts, with no significant loss of repeatability. Materials analysis of the conditioned powder batches is also presented in order to quantify the differences in powder properties and to help explain the mechanisms involved.

1.0 Introduction

Due to its relatively high mechanical properties, and the geometric complexity that it can produce, LS is one of most promising freeform fabrication processes for Additive Manufacturing. However, it is well-documented that, whilst tensile strength and modulus compare relatively well with injection moulded properties, the Elongation at Break of LS parts is substantially inferior.

Additionally, the inherent lack of repeatability within the process is acceptable for prototyping situations, where a part is required for purely visual, or limited fit/function purposes, but for manufacturing of end use parts it is essential to maintain high levels of repeatability.

Whilst some variations in properties occur as a result of part bed temperature variations or disparity between powder batches, a substantial decrease in repeatability occurs when producing parts from recycled powder.

1.1 Powder recycling

Standard practice is to mix specified refresh rates of virgin and recycled powder for LS builds, in order to maintain part quality whilst minimising material costs. It has previously been shown that the use of recycled or refreshed material produces LS Nylon-12 parts with higher Elongation at Break than virgin powder, but with a decrease in repeatability.

It has previously been reported [1] that powder undergoes an increase in molecular weight with re-use in the LS process, and that beyond a certain point deterioration occurs. Some work [2] has investigated the segregation of part cake into different ‘classes’, depending on proximity to parts themselves, and its molecular weight. This would then allow different refresh rates, depending on the quality of the powder to be recycled, and therefore higher quality and mechanical properties of parts.

However, in practise the history of recycled powder is often unknown, and these variations in history are likely to be the over-riding reason for the lack of repeatability observed when using recycled powder.

1.2 Thermal conditioning of powder

The major difference between virgin and recycled powder is that the recycled has been heated to close to melt temperature. During time at elevated temperature, chain segments within the Nylon-12 powder become more mobile, and are able to attach to one another, leading to an increase in molecular weight. and therefore it was proposed that heating virgin powder under controlled conditions would allow increased EaB, whilst maintaining the level of repeatability gained through the use of virgin powder. Some other research has been carried out in this area, investigating the melt flow rate of artificially aged LS powder [3], but the research presented here is focussed largely on the improvement of Elongation at Break of parts produced using thermally treated powders.

2.0 Methodology

In order to test the hypothesis stated above, LS parts were produced from several batches of pre-conditioned powder.

2.1 Conditioning procedure

Varying batches of Nylon-12 (PA2200) powder, from EOS GmbH, were conditioned by pre-heating in an oven, in an air atmosphere, at a temperature of 170°C, to simulate the temperature un-sintered powder is exposed to in the part bed of a Laser Sintering machine.

The powder was held for different times at this temperature, as shown in Table 1. Virgin powder was included as a condition, in order to provide a benchmark

	Batch				
	1	**2**	**3**	**4**	**5**
Hold time @ 170°C (hrs)	Virgin powder	0	15	30	45

Table 1 - powder conditioning times

Once the required hold time had been achieved, the parts were left to cool to ambient temperature, with no forced cooling.

2.2 Part production

All parts were built according to BS 2782 for tensile and BS 4370 for flexural specimens, and produced on an EOS Formiga P100.

Figure 1 demonstrates a diagrammatical representation of the cross-section of the build profile used in the initial experiments. It consists a warm-up powder deposition phase, followed by a further 4 mm of powder deposited at the final part bed temperature to provide further insulation to the part, as a shallow build volume was adopted. Both sets if parts were 4 mm in height, with 4mm of powder between the two sets.

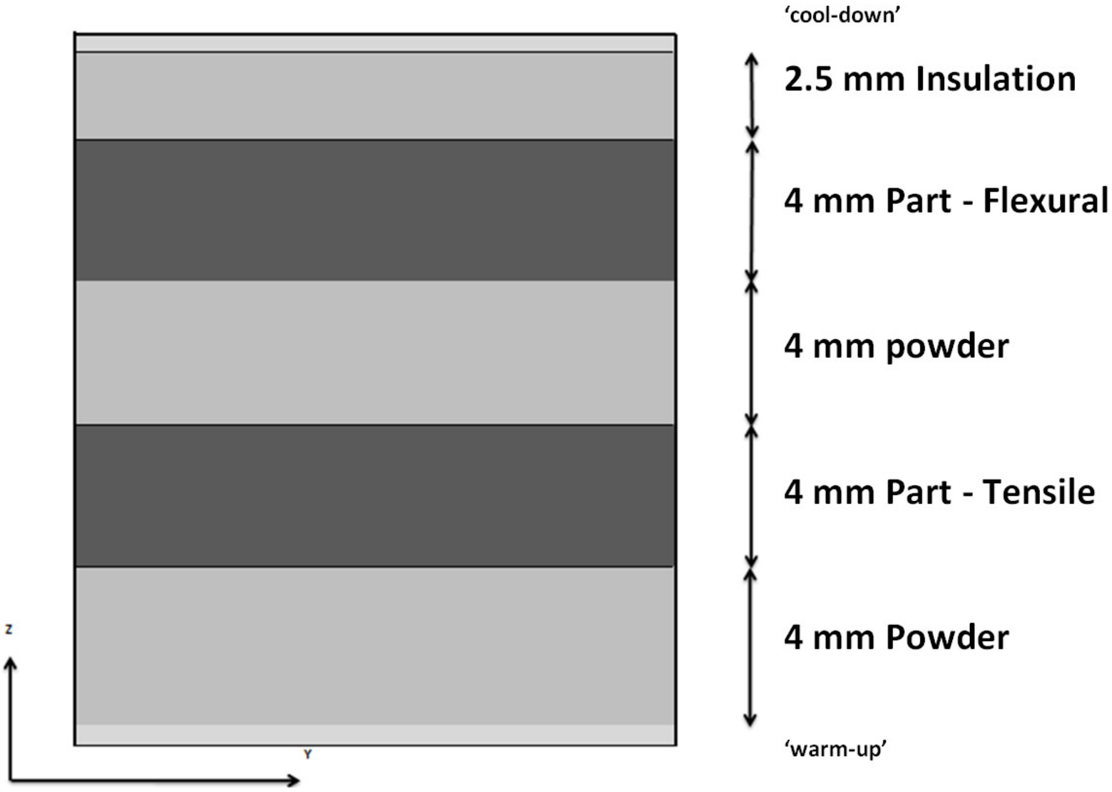

Figure 1 - Build profile (z-direction)

Figure 2 shows the layout of the tensile and flexural specimens within the appropriate layer of the build. Six of each specimen were produced, and a sacrificial strip built in each layer for later material testing if required.

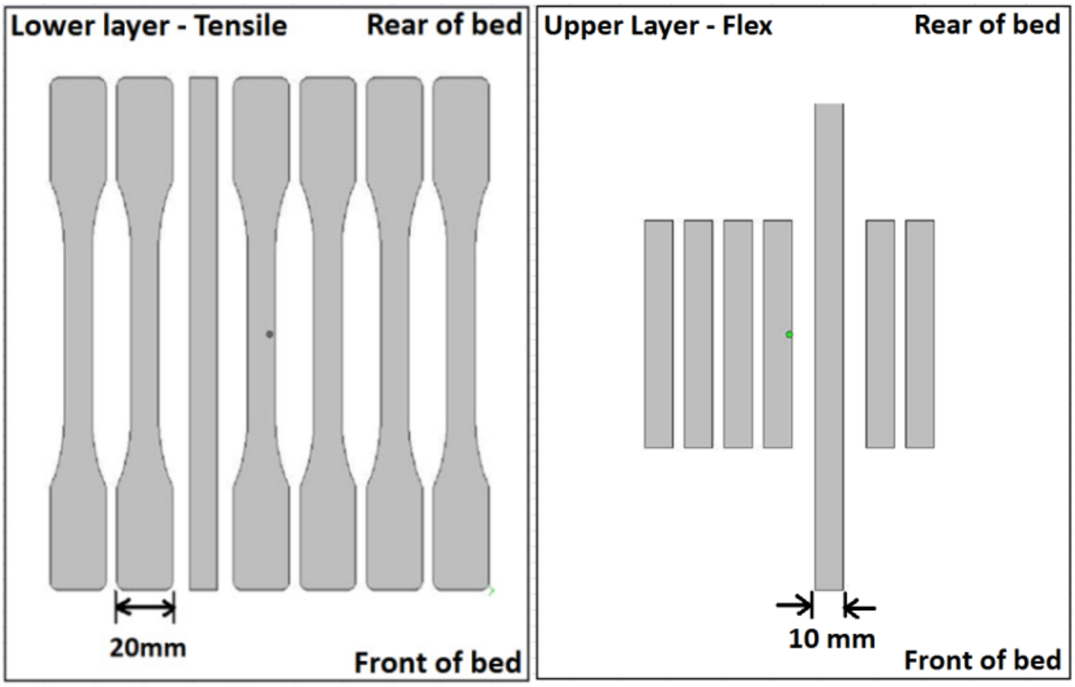

Figure 2 - Build profile (x-y plane)

Due to limited powder and LS machine availability, only one build could be completed for each powder condition. For this reason build parameters were optimised to allow building at the maximum (45 hour) hold time, and maintained constant for each powder condition. The main parameters are detailed in Table 2.

Parameter	Value
Fill laser power (W)	15 watts
Outline laser power (W)	16 watts
Scan spacing (mm)	0.25 mm
Part bed temperature (°C)	172 °C
Piston heater (°C)	150 °C
Laser scan speed ($m.s^{-1}$)	2500 $m.s^{-1}$

Table 2 - LS build parameters

3.0 Results

3.1 Tensile

All specimens were conditioned at 20 ◦C (+/−1 ◦C) and 50% (+/−5%) relative humidity. Tensile tests were performed on the specimens using a Zwick Z030 tensile testing machine fitted with an extensometer. The Zwick software, TestXpert, was used to enable the calculation of the Tensile Strength, Young's Modulus and Elongation at Break of each of the parts.

Figures 3, 4 and 5 show the Elongation at Break, Tensile Strength, and Young's Modulus respectively of the parts produced under the different conditioning procedures. Range bars have been included to indicate the maximum and minimum values recorded at each condition.

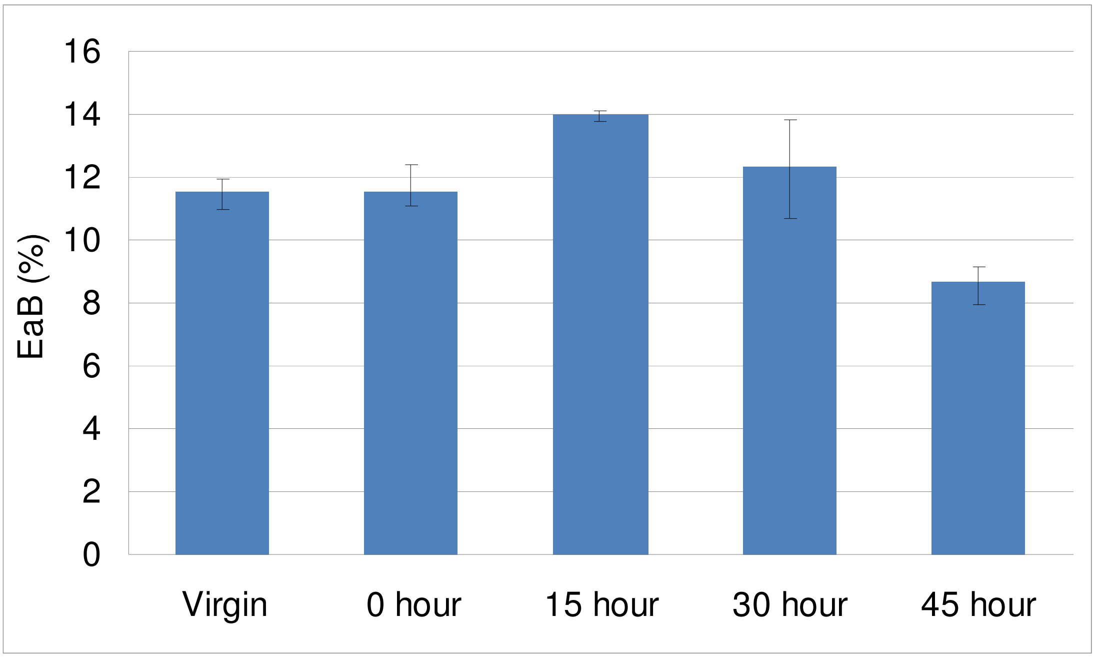

Figure 3 - Elongation at Break

It can be seen from Figure 3 that there is an increase in Elongation at Break (from 11.2% to 14.1%) between parts produced from virgin powder and those produced from powder conditioned for 15 hours. Beyond this point a decrease in ductility is observed, reducing to below that of virgin powder at the 45 hour condition. Crucially, the spread of the results shows no deterioration from that of parts produced with virgin powder, and in fact shows a slight improvement.

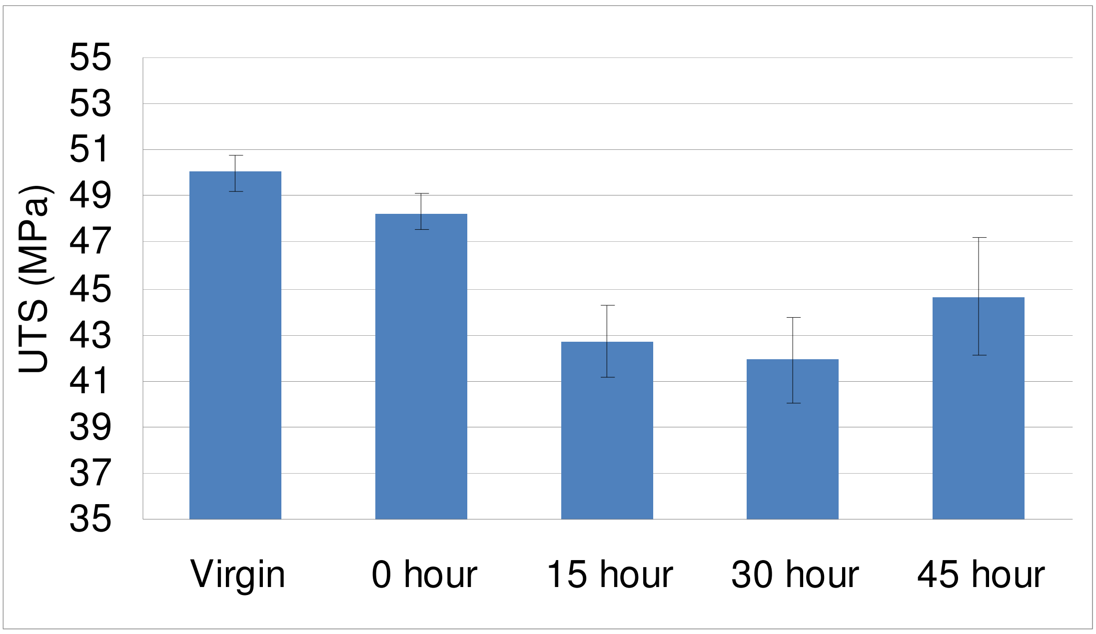

Figure 4 - Ultimate Tensile Strength

Figure 4 indicates that, as the hold time for the conditioned powder increased, a reduction in Tensile Strength (from 20Mpa down to 42.8MPa) occurred. Beyond this point a slight increase was observed, but with values remaining below those for parts built with virgin powder. There was a slight increase in the spread of results of part properties with thermal pre-conditioning.

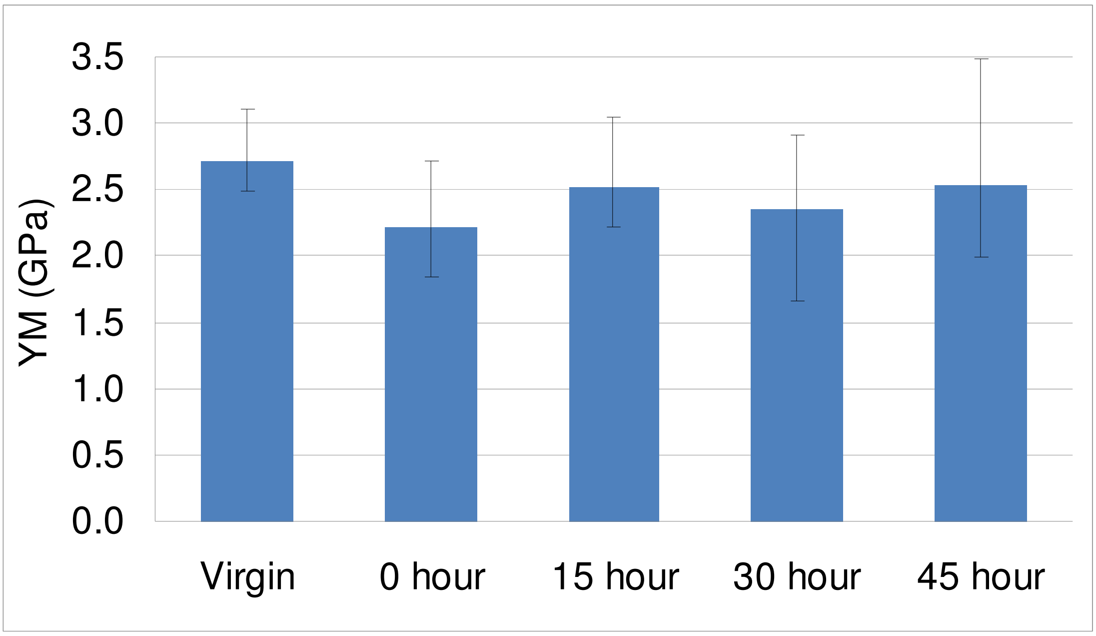

Figure 5 - Young's Modulus

The lack of identifiable trend, and the overlapping ranges of Young's Modulus at each powder condition, seen in Figure 5, allow for no definitive conclusions as to the effect of powder conditioning.

3.2 Flexural

All tests were carried out on a Zwick Z030 testing machine, according to BS 2782. Figures 6 and 7 show the Flexural Strength and Flexural Modulus respectively for each powder condition. Again, range bars have been included.

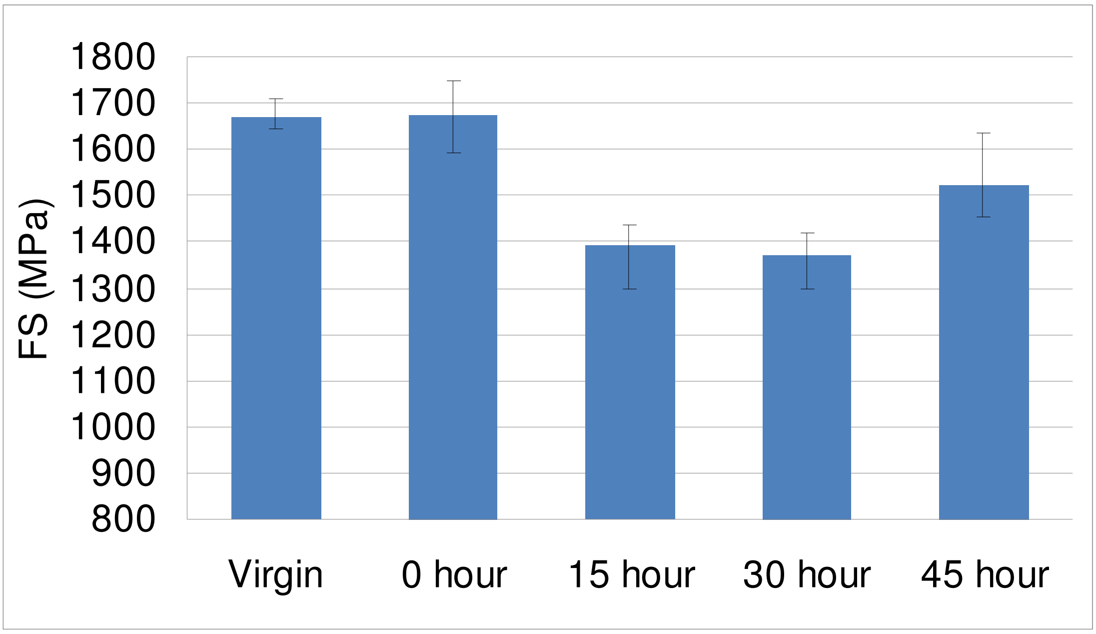

Figure 6 - Flexural Strength

Figure 6 indicates that, as with Tensile Strength, there is a decrease with hold time (1670 MPa to 1390 MPa) until around 15 – 30 hours, after which a slight increase is observed. The range of results at each level of powder conditioning is broadly consistent for each condition.

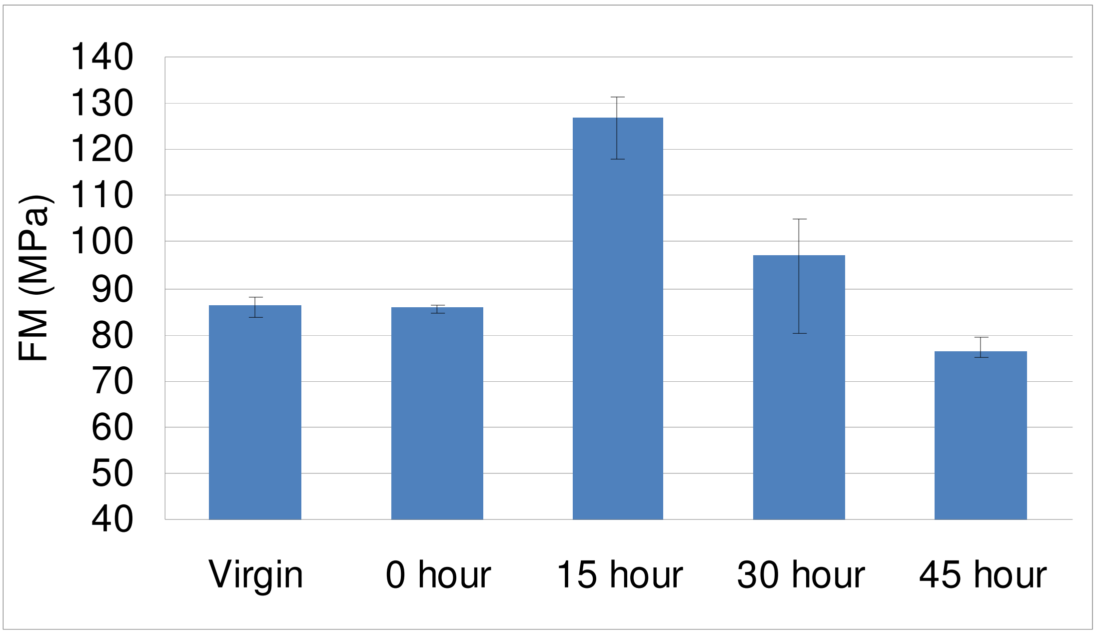

Figure 7 - Flexural Modulus

Figure 7 indicates a substantial increase (85 MPa to 125 MPa) between parts produced with virgin powder and those produced with powder held at elevated temperature for 15 hours. Beyond this point a decrease is observed, to below that of virgin powder at 45 hours of conditioning. However, an increased spread of results can be seen at the higher levels of conditioning when compared with virgin powder.

3.3 Melt Flow Rate

The Melt Flow Rate (MFR) indicates the viscosity of a material and is recognised as an indirect measure of a material's molecular weight, whereby an increase in viscosity indicates an increase in molecular weight. All MFI testing conformed to BS EN ISO 1133: Determination of Mass Flow Rate, and was carried out in the Department of Materials at Loughborough University. A weight of 2.16Kg and extruder temperature of 190°C were selected, based on prior experience with Nylon-12 LS powders within the department.

Figure 8 shows the flow rate recorded at each powder condition.

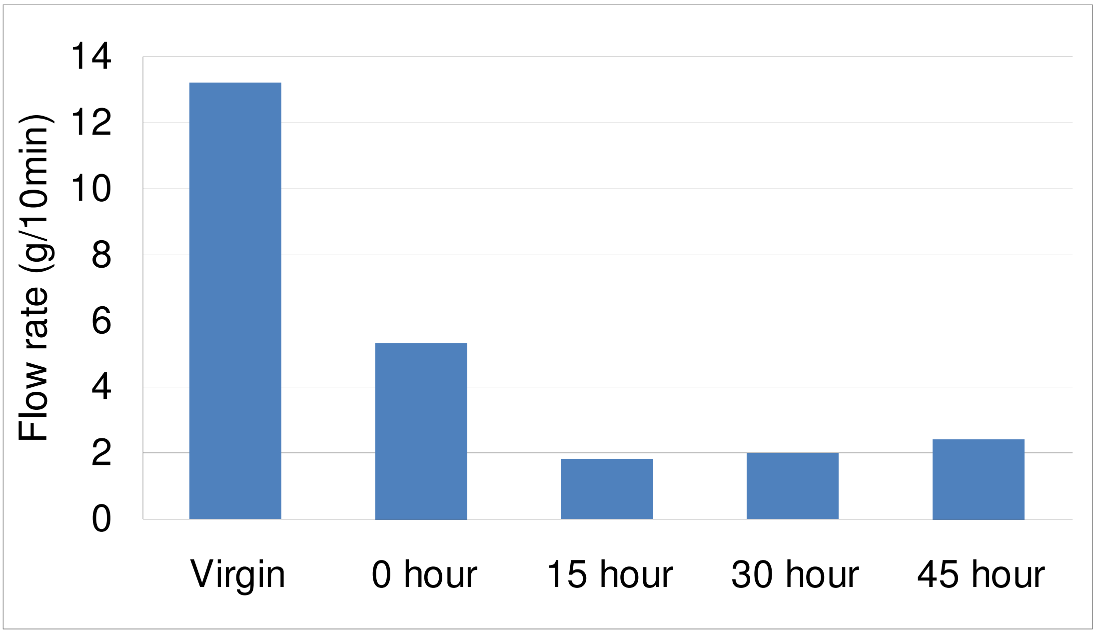

Figure 8 - Melt Flow Rate

Results show a significant decrease in MFR with pre-heating, appearing to reach a minimum at approximately a 15 hour hold time. This indicates increased viscosity, and therefore increased molecular weight, up to this level of thermal treatment. Beyond this point a slight increase in MFR was recorded, but substantially less pronounced.

4.0 Discussion

The initial investigations presented here have shown that thermal pre-treatment of powder for the LS process leads to a higher Elongation at Break of parts, which is consistent with lower crystallinity within these parts [4]. This corresponds to a slight decrease in Tensile Strength, as would also be expected from parts with lower crystallinity. For each property measured, a peak or trough was observed in the results, at around 15 hours of thermal conditioning, indicating that beyond this point the observed trends are reversed. This is consistent with previous observations [1,3] that after a certain level of recycling powder deterioration will occur.

Changes in molecular weight (indicated by changes in melt flow rate) have been observed as a result of thermal pre-treatment of the powder, which is consistent with effects observed when using recycled powder. The time held at elevated temperature has been shown to be crucial in affecting these changes in molecular weight, with a maximum reached at approximately 15 hours, after which a small increase is observed. These results tie in with other research indicating that powder recycled from a build of less than 20 hours is of relatively good quality, whereas beyond this point deterioration of part quality (and presumably of mechanical properties) will occur [3].

It is also possible that changes in the crystallinity of the powder itself, as a result of thermal treatment, may lead to changes in the crystallinity of parts produced, as a result of the incomplete melting of larger powder particles within the LS process reported elsewhere [5,6]. This is to form part of a further related investigation.

Previous research [7] has shown that Young's Modulus is generally the tensile property least affected by changes in processing conditions, and the same has been found here, with thermal conditioning showing no significant effect on this property.

It was also found with the heavily aged powder that the build parameters required were substantially lower than for virgin powder. It is possible that careful optimisation of these parameters for a given powder condition could allow for even greater increases in ductility.

It is thought that the absence of a nitrogen atmosphere within the processing oven may have lead to some oxidation of the powder during the pre-heating process. It is known [8] that nylons are susceptible to oxidation in air above ~85°C, leading to lower molecular weight and a subsequent decrease in mechanical properties (e.g. Elongation at Break). The results presented here could suggest that oxidation beyond ~ 15 hours conditioning has lead to the decrease in EaB recorded for parts produced from powder conditioned beyond this time period.

5.0 Conclusions and Recommendations

This research has shown that thermal pre-treatment of virgin powder has the potential to improve the Elongation at Break and Flexural Modulus of Laser Sintered parts, whilst at the slight detriment of the Tensile and Flexural Strength, and that at least some of these effects can be attributed to changes in molecular weight of the powdered polymer during the heating process. Crucially it has shown that the increased ductility is achieved with no loss in repeatability, suggesting that the pre-treatment is more suitable than the use of recycled powder.

The results also indicate that, above a certain hold time at elevated temperature, the trend of increasing ductility is reversed. Further work, for example FTIR, is planned to identify what, if any, chemical changes have occurred to the powder during the pre-heating process, possibly due to oxidation. If this proves to be the case, further experiments will be carried out using a nitrogen atmosphere during the thermal treatment, to more accurately replicate the LS process.

Once the conditioning process has been optimised, it is expected to have applications in the production of LS parts, in any areas where increased ductility is required.

6.0 References

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