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The Influence of Powder Ageing Characteristics on 316L Stainless Steel processed by Laser based Powder Bed Fusion

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ABSTRACT

The past 3-4 years has seen a transition in the use of Additive Manufacturing techniques from prototyping to series production. There remain a number of technical challenges that need to be resolved in order to increase adoption of the technology across a wider range of markets, one of which is improved part-to-part consistency. It is recognised that one factor that can influence the consistency of the laser based powder bed fusion process is the ageing of the metal powder used to manufacture parts, that is to say how the properties of the powder change following repeated use in repeated build cycles. Whilst commercial powder producers can exercise tight control over the 'as received' powder, changes that can subsequently occur following exposure to repeated process cycles in the laser beam melting machine warrant separate investigation.

In this paper we examine the changes that occur to the characteristics of gas atomised 316L stainless steel powder following multiple build cycles. We also relate these changes to the build quality of the processed parts in terms of part density, mechanical properties and surface finish.

INTRODUCTION

Stainless steels are one of the more widely used class of materials used in Additive Manufacturing (AM). The subject of this study, 316L (DIN 1.4404), combines good corrosion resistance with good formability and ductility and consequently finds use in applications where a polished surface finish is required. A number of studies on the laser based powder bed fusion of 316L have already been published, a summary of which is provided by Lavery et al⁽¹⁾; these studies demonstrate that high density levels can be readily achieved on a number of the commercial laser based systems currently available

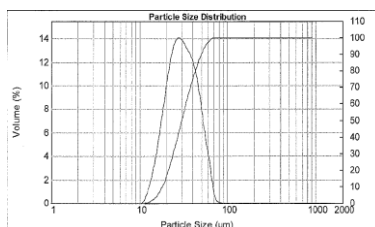
In contrast there is relatively little literature available which addresses the topic of powder ageing in relation to AM. Jarosinski's⁽²⁾ study of nickel based Alloy X observed no degradation in powder characteristics and consistent part properties over 10 consecutive builds whilst an extended ageing study of Ti6Al4V⁽³⁾ showed a gradual increase in oxygen and nitrogen levels over more than 20 build cycles. The objectives of this study were (1) to optimize the process parameters for the laser based powder bed fusion processing of 316L stainless steel and (2) to characterise the change in powder characteristics through repeated use over multiple build cycles. As well as evaluating the change powder properties over extended use aspects of build quality for the processed components, namely density, mechanical properties and surface roughness were also characterised.

EXPERIMENTAL PROCEDURE

A batch of 316L stainless steel powder was manufactured by Sandvik Osprey's proprietary inert gas atomisation process using nitrogen gas. The 'as-atomised' powder was sieved and air classified to produce a particle size distribution of -45+15µm. The chemistry and particle size distribution of the 316L powder batch are shown below.

	Fe	Cr	Ni	Mo	Si	Mn	C	S	P
Actual	Bal	16.8	12.4	2.3	0.7	0.7	0.02	0.01	0.02
Spec Min	-	16.0	10.0	2.0	-	-	-	-	-
Spec Max	-	18.0	14.0	3.0	1.0	2.0	0.03	0.03	0.04

Table 1: Chemical analysis of 316L powder used in this study (wt%)



Particle Size Data (µm)		
D ₁₀	D ₅₀	D ₉₀
18.2	30.2	50.4

Figure 1: Powder Size Distribution data for 316L powder (measured by laser diffraction)

The 316L powder was then processed on a Renishaw AM250 machine using argon as the shielding gas. Initially three build cycles were performed in order to identify the optimum machine parameters: an L9 and L25 array comprised of 10mm x 10mm x 10mm ‘density’ cubes were produced using different combinations of laser power (PW), Point Distance (PD), Hatch Spacing (HS) and Exposure Time (ET) as outlined in Figure 2.

Parameter	Range
Laser Power (LP)	160-200 W
Point Distance (PD)	25-105 μm
Hatch Spacing (HS)	70-175 μm
Exposure Time (ET)	70-150 μs

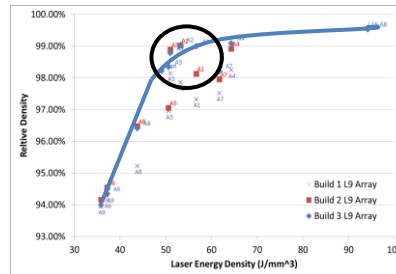


Figure 2: Plot of Density vs Energy Density for initial L9 and L25 arrays

The density of each cube was measured using the Archimedes method and the results plotted against the corresponding energy density as can be seen in Figure 2. From the fourth build onwards each build plate included an L9 array, located on the same position on the build plate, produced the same set of conditions highlighted. For comparison purposes the array also included one cube processed with the highest energy density of approx. 90 J/mm³

Details of the components produced on each subsequent build plate are described in table 2 below, in total 31 consecutive builds were performed. In addition to the L9 array vertical and horizontal test samples were produced for mechanical property testing on a number of the build plates. On a number of builds 90 density cubes were produced in order to assess the level of variability across the full area of the build plate. In some instances prototype parts were manufactured for other research purposes.

No.	Description	No.	Description
4	Single line plates	18	L9 Reference array + vertical & horizontal tensile bars
5	Build failed due to insufficient powder dosing	19	L9 Reference array + vertical test bars
6	L9 Reference array, vertical & horizontal tensile bars	20	Crucibles
7	L9 Reference array + horizontal tensile bars	21	L9 Reference array + vertical test bars
8	90 density cubes at optimal parameter settings	22	L9 Reference array + prototypes
9	L9 Reference array + horizontal tensile bars	23	Prototypes
10	90 density cubes at optimal parameter settings	24	Crucibles
11	90 density cubes at optimal parameter settings	25	L9 Reference array + vertical & horizontal tensile bars
12	L9 Reference array + prototypes	26	Prototypes
13	Crucibles	27	L9 Reference array + vertical & horizontal tensile bars
14	L9 Reference array + horizontal tensile bars	28	Prototypes
15	90 density cubes at optimal parameter settings	29	L9 Reference array + vertical & horizontal tensile bars
16	L9 Reference array + vertical test bars	30	L9 Reference array + vertical & horizontal tensile bars
17	L9 Reference array + vertical test bars	31	L9 Reference array + horizontal tensile bars

Table 2: Details of consecutive build cycles

Mechanical property testing was performed using Tinius Olsen equipment using BS EN ISO 6892-1:2009 standard test pieces whilst surface roughness measurements were carried out on 3 density cubes from each L9 array using a Mitutoyo hand held sensor probe. Samples of powder were taken from each build cycle at 4 locations in the build chamber (see Figure 3); the chemical composition of each was measured using a combination of ICP-OES, fusion and combustion techniques whilst the particle size distribution was measured by

laser diffraction using a Malvern Mastersizer. Flow properties of the sample from position 1 was also characterised using an FT4 Rheometer from Freeman Technology.

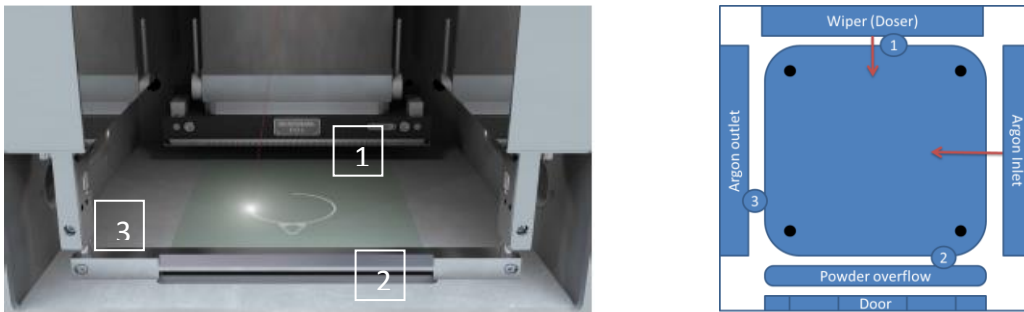


Figure 3: Location of sampling positions in the build chamber (note: position 4 relates to powder taken from the overflow chamber)

Following each build the unused powder was removed from the chamber and sieved under argon through a -63µm sieve mesh in order to remove any process ‘artefacts’ i.e. partially fused particles, before being added back into the powder hopper. As the number of builds progressed and powder was consumed fresh 316L powder, from the same parent production batch, was re-blended as outlined in table 3 below

Sequence	‘Fresh’ Powder (kg)	Used Powder (kg)	Total Quantity (kg)	Fresh/Used	Build Cycles
A	60	0	60	100%	1-14
B	35	17.5	52.5	66.67%	15-24
C	12	26.6	38.6	31.09%	25-31

Table 3: Details of Powder Blending strategy employed

RESULTS

Powder samples from multiple locations in each build were analysed for particle size and chemical composition. Figure 4 shows the particle size results for samples taken at positions 1 and 2 in the chamber. The results for position 1 show that the particle size distribution remains consistent across the 31 builds performed and in line with the ‘as received’ powder characteristics. Although not presented in the paper the data from position 3 followed the same trend. In contrast to this the data from position 2 indicates not only that the variability in particle size distribution has increased significantly but also that the measured D₅₀ and D₉₀ values have increased.

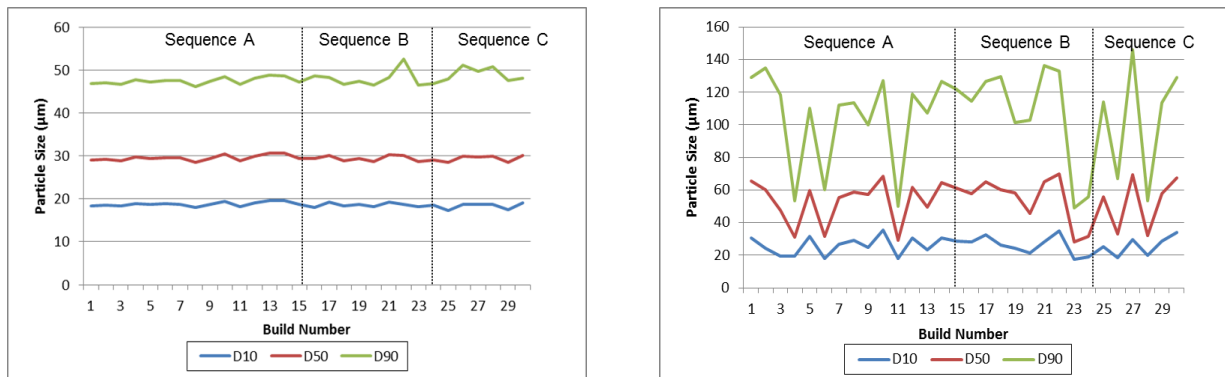


Figure 4: Particle size data samples for Position 1 (Left) and Position 2 (Right)

These observations are perhaps to be expected given that powder collected from position 2 will have come into contact with fused layers of the solid metal as it travels across the build chamber and therefore is likely to have both dislodged partially fused particles collected other agglomerated artefacts expelled from the melt pool during processing. The trend for powder taken from position 4 in the overflows shows the same trend as position 2. Comparing the data between positions 1 and 2 indicates that sieving the processed powder at 63µm after each build appears to be an effective method for reconditioning the powder back to the ‘as received’ state from the perspective of particle size

Figure 5 shows the measured oxygen and nitrogen levels for samples taken at position 1. The data indicates that an increase in nitrogen levels of approximately 50ppm has occurred over the 31 builds whereas oxygen levels have increased by approximately 80ppm across the same period.

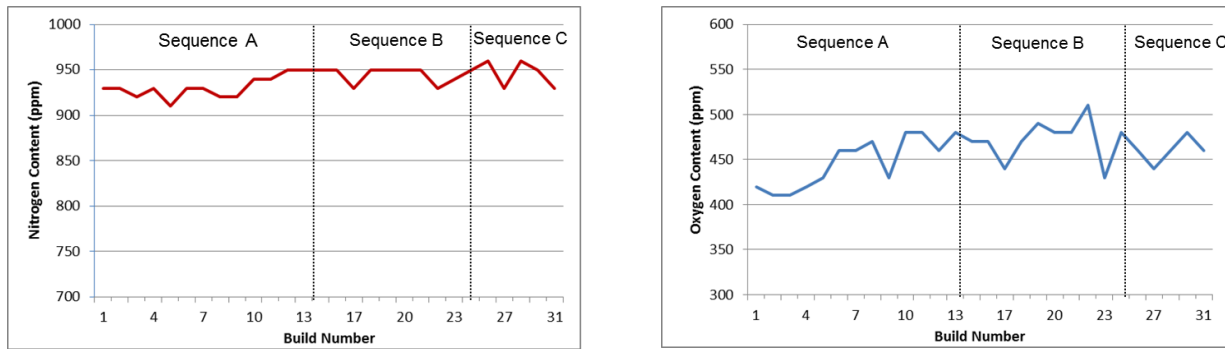


Figure 5: Particle size data samples for Position 1 (Left) and Position 2 (Right)

The other alloying elements outlined in table 1 were also analysed. Although the data is not presented it was observed that the measured levels were in line with the 'as received' levels and showed a high level of consistency across the full range of completed builds.

Figure 6 shows the results of the rheological analysis performed on samples taken at position 1, in particular the Basic Flow Energy (BFE) measurements.

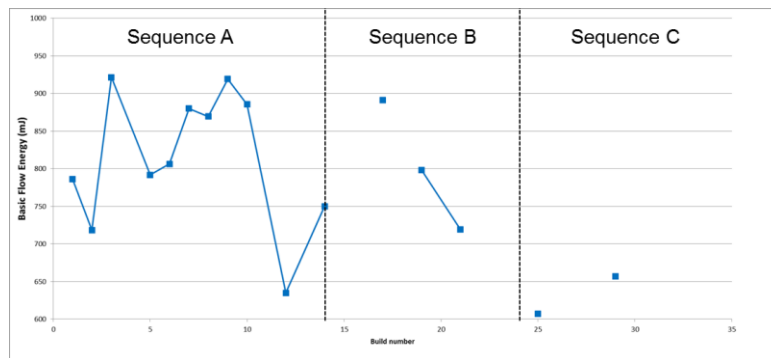


Figure 6: Basic Flow Energy (BFE) measurements for samples taking at position 1

Data for builds 1 - 10 shows a gradual increase in BFE of 100mJ after which the level reduced back to the 'as received' condition. The data available for later builds suggests that the re-blending of fresh, un-processed powder, combined with re-sieving the powder after every build, has the effect of reducing the BFE levels suggesting that the flow behaviour has improved. The significance and reliability of this observation is tempered somewhat by the limited number of test results available, this was due to insufficient sample weights in a number of cases. No observations of problems with poor powder flow were made during the builds.

In addition to profiling the powder ageing characteristics aspects of the build quality were also benchmarked. Figure 7 shows the variation in measured density for cubes processed using 3 different parameter sets in the reference L9 array. Parameter sets 1 and 2 reflect the optimised set of parameters highlighted previously in Figure 2 with energy densities in the region of 50 - 60 J/mm³ whereas parameter set 3 in red reflects the highest energy density used at approx. 90 J/mm³.

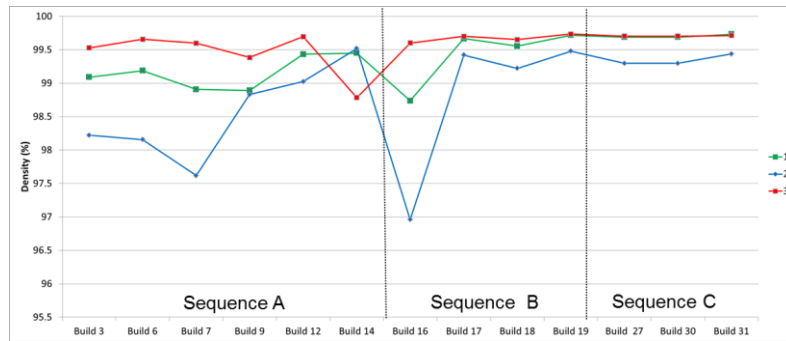


Figure 7: Measured density level for selected L9 Reference Array cubes (density measured by Archimedes method)

A review of the data shows that the majority of cubes tested had a density above 98%, indeed a significant proportion showed density levels in the range of 99 – 99.5%. A general trend of improving density with increasing build number was observed for parameter sets 1 and 2. The density levels achieved on build 16 appear to be lower than for the other builds; at the time of writing the report the reasons for this variation are still being investigated.

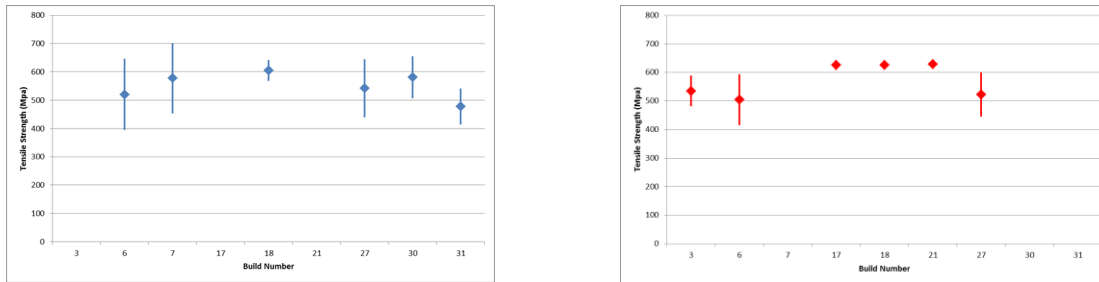


Figure 8: Tensile Strength data for Vertical (Left) and Horizontal (Right) test directions

Figure 8 shows the tensile strength measured in both the vertical and horizontal build directions. Tensile strength levels of 500 MPa were achieved across the full range of builds in both test directions. Variability in strength levels in the earlier builds would appear to be greater than in later builds; looking at this data in more detail reveals that the variability in tensile strength is related to the energy density used in producing the test samples. Figure 9 shows the relationship between tensile strength and energy density for the vertical samples produced during build 7. The data reveals a trend of increasing strength as energy density up to level greater than 60 J/mm³ after which strength levels plateau at approx. 600 MPa.

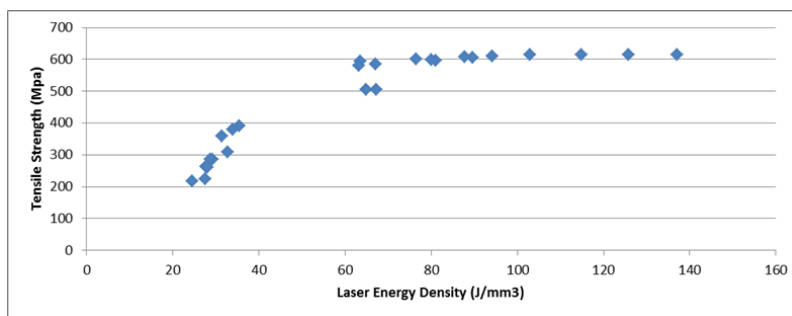


Figure 9: Chart of Tensile Strength vs Energy Density for vertical test samples from build 7

Finally surface roughness measurements were performed on the same 3 density cubes within the L9 array corresponding to parameter set 2 (see Figure 7). 3 measurements were taken on the back and right hand vertical faces (looking into the build chamber) and the data is presented in Figure 10 below.

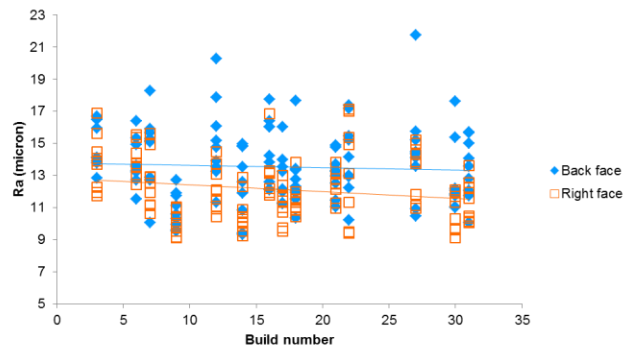


Figure 10: Surface Roughness Data for cubes processed using Parameter Set 2

A review of the data shows a typical variance in Ra values of 6 - 8 μ m on both the back and right hand face. Given this level of scatter the roughness levels appears broadly consistent, a linear regression analysis performed on the data does suggest a weak trend of reducing surface roughness with increasing build number.

DISCUSSION

Process optimisation trials on 316L, using Renishaw AM250 equipment, have demonstrated that high density levels can be achieved at energy density levels exceeding 50 J/mm³. Increasing energy density above this threshold does result in some further incremental improvement in density however this comes at the expense of both build rate i.e. productivity and also surface quality. For the purposes of this study the optimum process parameters were specified in a range of 50-60 J/mm³.

Characterisation of the powder properties at multiple locations in the build chamber shows a coarsening effect with the unused powder due to the presence of 'artefacts' generated as a result of the laser fusion process. The artefacts are effectively removed by sieving the powder at -63 μ m after each build cycle such that the particle size distribution and chemical composition of the re-sieved powder are in line with 'as received' levels. The consecutive re-use of the same batch does appear to result in a gradual rise in the powder's oxygen and nitrogen levels although in both cases the increase over 30 builds was limited to below 100ppm. Repeated powder re-use would also appear to have an effect on the powder's rheological characteristics however definitive conclusions are not possible due to limited test data. In planning future studies of this type greater attention should be paid when calculating the batch sizes required, allowing for anticipated powder consumption and handling/processing losses, to ensure that the samples of sufficient weight are available to enable a complete analysis across the total number of builds

SUMMARY AND CONCLUSIONS

The present study shows that 316L stainless steel can be effectively processed by laser based powder bed fusion to densities exceeding 99%. The process appears stable and consistent density levels can be replicated over multiple build cycles. The corresponding mechanical properties also appear to be consistent and repeatable. Multiple re-use, up to 30 builds, with periodic rejuvenation of the powder does not appear to have a significant effect on the powder characteristics, machine performance or the quality of the manufactured parts. There is some evidence of a gradual increase in oxygen and nitrogen levels however this does not appear to have a significant impact on the processed parts. At the same time there appears to be some changes in the powder's rheological behaviour over time however data for later builds was limited due to insufficient sample size. Sieving the powder after each build cycle appears to be an effective method for re-conditioning the powder to remove processing 'artefacts'.

ACKNOWLEDGEMENTS

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