

The Influence of Powder Reuse on the Properties of Laser Powder Bed-Fused Stainless Steel 316L: A Review

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There is a clear economic benefit for the recycling of metallic powder during additive manufacturing (AM). Laser powder bed fusion (L-PBF) is one such AM process and research has found that the properties of the powder feedstock can change when the powder is reused through mechanisms such as spatter generation and alterations in chemistry. Such changes to powder properties can accumulate and may lead to significant differences in the mechanical properties of the final component. Often the changes to part properties are reasonably small; however, there is not currently enough understanding of the specific links between powder properties and the characteristics of the end component for the effects of powder recycling to be discounted. Herein, the typical lifecycle of stainless steel 316L powder in L-PBF, the changes that occur to the powder feedstock, and the effects that this may have on the mechanical properties of components manufactured with recycled powder are reviewed.

machining, has multiple benefits. AM techniques allow designers more freedom with a high level of customization.^[2] Within AM techniques, L-PBF delivers the ability to create parts with high geometrical tolerances and complexity.^[2] The combination of customization and design flexibility is often used to create weight-saving structures such as meshes or lattices that otherwise would be expensive and time-consuming to create.^[2] This has typically meant that the sectors where AM has seen most use require high performance, complex, and potential weight-saving designs; this includes aerospace, biomedical, and energy.^[3] To compete on manufacturing cost with other traditional manufacturing methods, it is important that designers recognize the freedom that L-PBF can offer and redesign

1. Introduction


Additive manufacturing (AM) is an umbrella term to describe the manufacture of parts from a 3D computer aided design (CAD) model split into layers. The layers are usually sequentially built on top of one another by the addition of material.^[1] Depending on the AM method utilized, metallic, polymeric, ceramic, and some composite parts can be manufactured, of which some of these techniques may be referred to as 3D printing. Laser powder bed fusion (L-PBF) uses a metallic powder feedstock and a laser heat source to create fully homogenous solid metal parts. A simplified schematic drawing of the L-PBF process and the main stages in the formation of parts are shown in **Figure 1**. The method of adding material to form parts rather than traditional subtractive techniques, such as computer numerical control

and/or combine parts instead of rolling over existing designs.^[4] Even with this approach, L-PBF as a technique is most appropriate for the manufacture of low-to-medium production volumes of geometrically complex and mechanically sound parts, where traditional manufacturing routes such as high-pressure die casting can become economically less viable very quickly (**Figure 2**).^[4]

As each individual layer of a part is sequentially melted onto the build stage (**Figure 1**), there are large amounts of powder present on the build stage not required to form subsequent components. The exact amount of powder that is utilized on the build stage will depend on the part(s) produced. For a single part, the cost of material has been reported to be 31% of the manufacturing costs of L-PBF.^[8] This value is dependent on the material used, build plate utilization, machine running cost, and a myriad of other factors, but does not account for the reuse of powder. However, it is an indication that material costs can make up a significant proportion of the overall running costs for L-PBF. Therefore, it is economically preferable to reuse the excess powder that has not been fused on the build stage than to buy extra powder. This is classed as powder recycling. Excess powder is generally collected and sieved to remove larger spatter particles. This powder may then be mixed with additional virgin powder or powder that has undergone a different number of cycles and then reloaded in to the machine to be reused in a subsequent build. Powell et al. reviewed the effect of combining virgin and reused powder and suggested that some changes to powder properties from recycling were reduced by this practice.^[9] Different manufacturers and research institutions will have their own individual powder management strategies which may specify factors such as sieve size, amount of recycled powder that can be used, the level of permitted recycling in powder, and the addition of new metal powder to the powder chamber. All

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DOI: 10.1002/adem.202200596

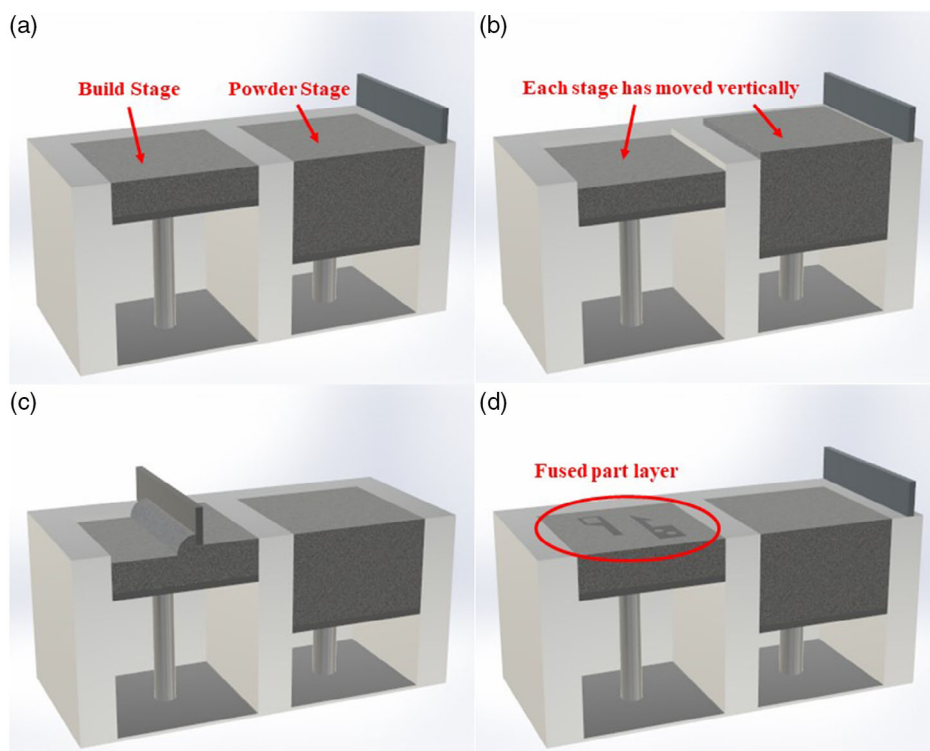


Figure 1. A simplified schematic diagram of the L-PBF process, a) the powder and build stage are flat, b) the build stage descends by one layer and the powder stage raises by one layer, This stage movement is dependent on machine set up but typical values are between 20 and 45 μm .^[5–7] c) powder is spread across the build stage, d) a pattern is melted in the powder on the build stage. The geometry of the melted pattern derives from 2D slices of a 3D CAD file which when combined, create the desired 3D geometry.

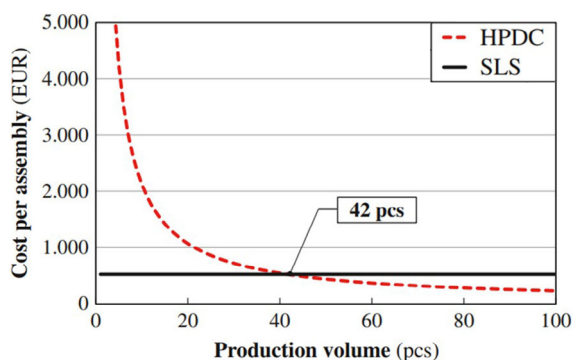


Figure 2. Breakeven analysis illustrating the cost per assembly for an aircraft landing gear manufactured via HPDC and SLS. The analysis shows that at relatively low production volumes, traditional manufacturing can become more economically viable than AM. This breakeven point is specific to this analysis and will vary significantly in different scenarios. Reproduced with permission.^[4] 2002, Springer.

these factors can complicate assessing how much powder in a batch is reused and what age it is. Powder age may be characterized by the number of reuses it has undergone or the exposure time of the powder in the build chamber.

A major concern for powder reuse is that the physical and chemical properties of the powder may change with an increasing number of recycles. Factors such as the chemical composition and morphology of a powder batch are carefully specified for

use in L-PBF and altering these could have a knock-on effect on part quality. The interaction of the melt pool with powder is one mechanism for powder to change. Powder particles ejected from the melt pool are classed as spatter. To minimize these spatter particles landing back on to the build stage, an inert gas is blown across the baseplate to remove them. The optimization of this gas flow reduces the amount of spatter redeposited back on to the powder surface.^[10] Wang et al. identified three distinct types of powder spatter when studying how it is generated (Figure 3).^[11] Some powder is fired up through the laser due to a recoil pressure. As it travels up through the laser, much of the powder is vaporized. The remaining spatter is remelted and generally has a highly spherical shape and smooth surface. Powder spatter is also generated from Marangoni convection currents in the melt pool and from the bow wave ahead of the melt pool.^[12] This powder is more likely to pick up satellite particles, especially the spatter generated ahead of the melt pool as this powder is hot and rolling over particles. Overall, spatter powder has a coarser powder size distribution (PSD), as the distribution of particle sizes tends toward larger sizes. The oxygen, silicon, and carbon content of powder spatter is increased and the tensile properties of parts manufactured with powder heavily contaminated with powder spatter are considerably reduced.^[11,13] In addition to the spatter generated at the melt pool, there is also a heat-affected zone (HAZ) around the melt track. The microstructure and powder morphology appears to be unaltered as a result of residing in this HAZ. However, the oxygen content

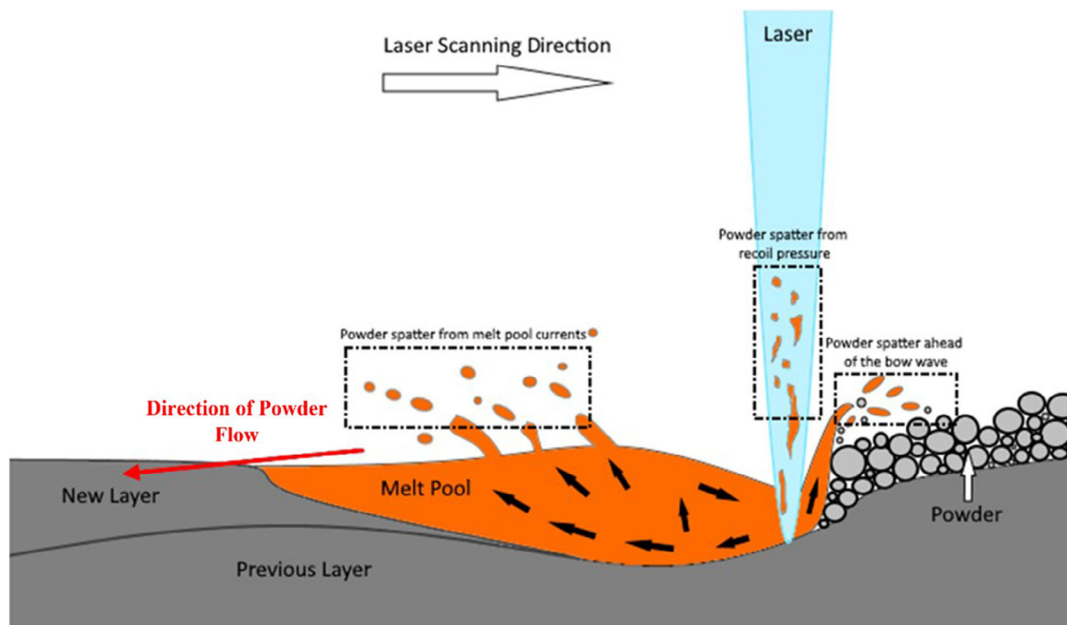


Figure 3. Laser spatter being generated during L-PBF, three types of spatter are illustrated. Reproduced with permission.^[11] 2017, Elsevier.

of the powder has been shown to increase in 316L in the particles located in close proximity to the melt pool.^[14]

As a manufacturing technique, L-PBF is not exempt from part defects that arise during processing. Comprehensive reviews of these defects already exist.^[12,15] For the purpose of this review, only defects that could arise as a result of powder recycling will be focused on. In their 2020 review paper, Snow et al. identified gas trapped within powder as a potential source for gas porosity within parts.^[15] This problem is exasperated by the fact that gas solubility in liquid metal increased at high temperatures.^[12] They reported that gas porosity within powders is a result of the gas atomization process and was not a major function of powder recycling. The other source of gas porosity in parts was due to the release of gases during rapid solidification, a process made worse by an increase in elemental impurities such as oxygen on powder. If recycled powder shows an increase in internal porosity or elemental contamination such as oxidation, both of these could lead to an increase in gas porosity, which would likely have a negative impact in the part's mechanical performance. The other source of defects directly linked to powder properties identified by Snow et al. was in poor or irregular packing of the powder on the base plate, which could lead to lack of fusion (LoF) defects. The PSD, powder flowability, and powder packing density are all dependent properties.^[16] Therefore, the altering of one of them could potentially lead to an increase in LoF defects.

For L-PBF to grow in use, the economics of the process must be optimized and that will include safely maximizing powder recycling. If there are significant changes to the properties of parts as a result of powder recycling, this generates risks for the production of parts manufactured from highly recycled powder. The severity of these risks and how best to manage and mitigate them needs to be evaluated. In this way, future testing and production control and monitoring of powder parts can be optimized. Therefore, a summary of the key changes to the powder feedstock as a result of recycling and the effects this imparts on

the mechanical properties of parts is required. This paper aims to complete a thorough review of the existing knowledge on the powder recycling of 316L to achieve this.

2. Changes to Powder Properties as a Result of Recycling

2.1. Powder Size Distribution and Flowability

Before use in L-PBF, a powder batch is usually defined by specific variables. These include the PSD and chemistry of the powder batch. As previously discussed, there are mechanisms present within the build chamber that allow for the alteration of these specified variables. The PSD of a powder batch describes the relative occurrence of powder sizes. For L-PBF, the PSD directly affects the packing factor and flowability of metallic powder.^[16] Higher packing values are desirable for L-PBF as porosity in packing on the build stage could be retained within the end product, which may reduce the mechanical performance.^[6,17] Instead of creating a narrow PSD of very small-diameter powders, to reduce pore size, a wider PSD is generally the optimal approach where fine particles can locate within the interstitial sites formed from larger particles.^[16] Changes to the PSD that affect flowability may also have a significant effect on defect formation if they are significant enough to prevent flow of powder particles in to homogenous layers. An adequate quantity of fine particles is necessary for good tensile strength in L-PBF 316L, while elongation can be improved through larger particles.^[5] The increase in tensile strength has been attributed to the easier melting of smaller particles and hence, finer grains. The PSD and the fine particle content of a powder batch needs to be controlled as they may have statistically significant effects on the mechanical properties of parts. Understanding the ways that the PSD could change by powder reuse is therefore important.

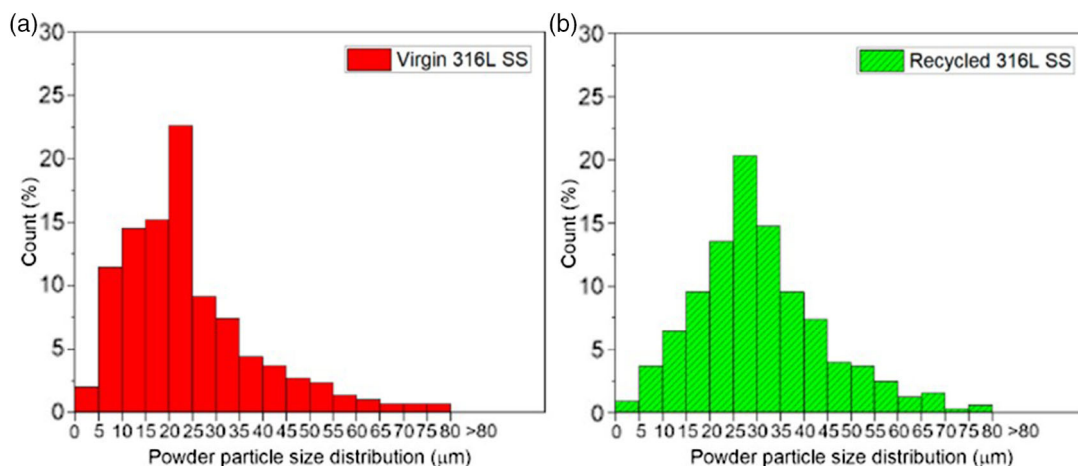


Figure 4. The changing powder size distribution of 316L powder feedstock, (a) virgin powder, and (b) 20 times recycled 316L powder, The percentage of particles measuring 20 μm decreases from the virgin state as the powder size distribution shifts towards coarser particles. Reproduced with permission.^[18] 2020, MDPI.

The PSD of 316L particles has often been shown to slightly coarsen with more powder reuse. This involves a reduction in fine particle content and an increase in larger particles, which shift the PSD Gaussian curve to a larger particle size, as illustrated by Yusuf et al.,^[18] in **Figure 4**. Coarsening was observed in 316L powder used 20 times, where the D_{50} (particle diameter at the 50th percentile) particle size increased from 22 to 29 μm.^[19] Reuse of 316L powder up to 30 cycles was investigated by Heiden et al.^[18] who demonstrated a slight coarsening of the PSD and a slightly wider distribution of powder particle sizes. The average particle diameter increased from 22.4 to 35.7 μm when tested by laser diffraction. The size of D_{10} , D_{50} , and D_{90} particles (particle diameter at the 10th, 50th, and 90th percentile, respectively) increased by 26%, 28%, and 81%, respectively, when measured via laser diffraction. Delacroix et al. recycled 316L powder 15 times using a 50 μm mesh to sieve powder between reuses.^[20] Their PSD was initially specified between 20 and 45 μm, a finer distribution than other studies. Again, some coarsening was observed in the D_{10} , D_{50} , and D_{90} particles sizes but this was only within the region of 2 and 3 μm for the D_{10} and D_{90} values, respectively. The study undertaken by Delacroix et al. involved the continuous use of metal powder where no topping up was completed with virgin powder. In this scenario, any

changes as a result of powder recycling would be expected to be exacerbated as no virgin powder is added to dilute the effects of powder recycling. This approach of no topping up of the powder batch with virgin powder was also adopted by Lu et al.,^[21] which also resulted in powder coarsening with recycling. After 30 recycles the D_{10} value increased 26% from 15.54 to 19.60 μm, while D_{50} and D_{90} also increased by 18% and 15%, respectively. The percentage increase in D_{10} after 30 recycles is almost identical to that observed by Heiden et al.^[18] after 30 recycles. However, Heiden observed a significantly larger increase in the D_{90} of powder particles. Lu et al. also observed a progression with the coarsening of the metal powder from 10 recycles to 30 recycles, as shown in **Figure 5**.^[21]

In contrast to these results, Gorji et al. saw very little change in the PSD of 316L as it was recycled over 10 cycles, with a decrease in the average diameter (D_{50}) of the recycled powder by just 1 μm, falling from 26 to 25 μm.^[22] When analyzing 316L powder recycled eight times, Sartin et al. saw changes in the percentage volume of powder for different sieve sizes.^[23] However, these changes were not consistent across the whole range of sieve sizes and therefore did not effectively illustrate a consistent reduction in fine particles with an increase in the volume of larger particles. ASPEx scanning electron microscope (SEM) was used for these measurements.

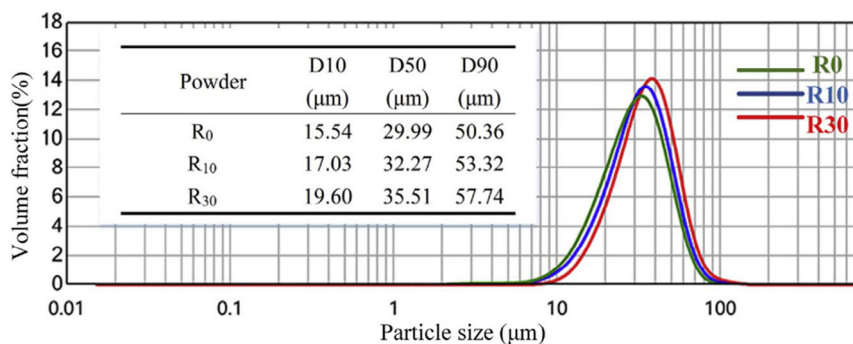


Figure 5. The PSD of virgin 316L powder compared to the same powder that has been recycled 10 and 30 times.^[21] The coarsening of the powder gradually shifts the peak of the PSD to the right. Sieving of the powder stops the right edge of the PSD curve moving significantly. Reproduced with permission.^[21] 2022, Elsevier.

Table 1. The change in powder morphology for 316L powder for L-PBF at different levels of recycling.

Powder reuses	Analysis method	Average particle size [μm]		D_{10} [μm]		D_{50} [μm]		D_{90} [μm]		Source
		Virgin	Recycled	Virgin	Recycled	Virgin	Recycled	Virgin	Recycled	
30	SEM image based	16.4	16.9	7.0	8.2	16.9	15.4	26.9	27.5	[18]
	Laser diffraction	22.4	35.7	13.6	17.2	21.1	27.1	32.7	59.3	[18]
10	XCT	26	25	–	–	–	–	–	–	[24]
≈ 20	SEM image based	–	–	≈ 8.5	≈ 14.7	22	29	≈ 43.2	≈ 48.9	[19]
15	Laser diffraction	32.7	35.4	23.2	25.2	31.5	33.8	44.3	47.3	[20]
10	Laser diffraction	–	–	15.54	17.03	29.99	32.27	50.36	53.32	[21]
30	Laser diffraction	–	–	15.54	19.60	29.99	35.51	50.36	57.75	[21]

The sources referenced in **Table 1** show differing recorded outcomes for the effect of powder recycling on PSD. Both analysis method and recycling level are expected to create variation between results. Heiden et al. used both laser diffraction and an SEM image-based method to calculate PSD and saw significant variation between the results.^[18] The powder sampling method and powder sample size are key when making assumptions about the powder population from an individual sample. Gorji et al. used X-ray-computed tomography (XCT) and saw very little change with a sample size of ≈ 1350 powder particles.^[24] Heiden et al. studied a sample of at least 50 000 powder particles for their SEM image-based method.^[18] Larger sample sizes give greater confidence to their result; however, variations between recycling level and analysis technique will also affect the outcome. Enough of the sources in Table 1 and in literature on other materials have identified coarsening to the PSD with recycling to record it as a likely phenomenon.^[25] More focused research is needed to understand the specific process/recycling parameters that increase or decrease the rate of powder coarsening. This indicates that the very finest particles are more significantly consumed by recycling than other powder sizes and as powder is reused the spatter generated can coalesce into large quantities of oversized powder particles.

Many sources^[18,19,26] agree that the PSD of metallic powder can be altered by the reuse of metal powder. This change is often coarsening of the PSD and increase in size for the finest particles in the distribution; both factors could potentially affect the mechanical properties of parts made by heavily reused powder.^[5] The level of evolution of the PSD is variable and is likely to be dependent on the specific L-PBF system employed. Therefore, it is advisable for individual L-PBF users to analyze their process until the evolution of metallic powder is better understood and standardized.

There are several mechanisms to consider the explanation of the coarsening of the PSD. Fine powder particles are preferentially used within the powder build as they filter down the body of powder, filling spaces between larger powder particles on the build stage. This has been shown with 316L powder within a single build where fines were more prevalent both close to where the blade first sweeps powder on to the bed and within the initial lower layers within a build.^[27] The D_{10} of powder particles at the start of the build platform, where powder spreading had begun, was $\approx 5 \mu\text{m}$ lower than at the end of the build stage where powder spreading finished. Another factor is the difference in size between the layer thickness of parts and the size of the largest powder particles. Many operators run a layer thickness, which

is smaller than the size of the largest particles, and some have suggested that this may cause large powder particles to be preferentially swept across and be unable to rest on the build stage.^[28] The coarsening of powder may also occur from the fusion of particles, either from spatter or from the fusion of powder in the HAZ near the melt pool. However, when investigated by Galicki et al., coarsening of the PSD was found to be unrelated to the proximity of powder to the HAZ.^[14] This could suggest that the fusion of particles in the HAZ is not a major contributing mechanism of the coarsening of metallic powder. Gas flow across the powder bed to remove spatter could also be responsible for blowing off very fine particles from the powder stage. As all these processes are occurring simultaneously, it is difficult to determine what the most significant driving force for powder coarsening is.

One of the effects of PSD can be on the flowability of metallic powder. Flowability can be a difficult value to quantify as there is no single standardized test measurement and multiple approaches can be used to arrive at a flowability value. In addition, as the methodology for each flowability test is different, it is not always appropriate to compare directly between various testing methods.^[29] For best practice, flowability tests should mimic the real-life situation as much as possible.^[30] Existing methods for measuring flowability of AM powders include gravity-based flow methods, where powder is made to flow down a funnel through an orifice of specific size.^[29] Angle measuring-related methods where the angle of the powder rests in a pile, or the angle of the powder slope in a rotating drum, are alternative measures that can be quantified.^[31,32] Different approaches to measuring an angle of repose (AoR) are summarized in **Figure 6**. Other novel techniques include approaches that aim to measure the resistance to flow with stirring.^[29,33] In each case, it is important to realize what approach most accurately replicates the desired use. In an L-PBF build chamber, powder flows by the movement of a blade sweeping powder across a build stage. A study undertaken to identify the most appropriate testing for this scenario found that the AoR tests, as well as some more novel testing methods measuring avalanche angle or basic flowability energy (BFE), were adequate to provide an indication of powder spreadability on the build plate.^[34]

Generally, the reviews of recycling on the flowability of powder particles have been inconclusive, with some sources finding an increase in powder flowability with reuse, whereas others found that no significant change occurs. Delacroix et al. studied the flowability of 316L powder recycled through 15 cycles and observed a consistent increase in flowability with each recycle,

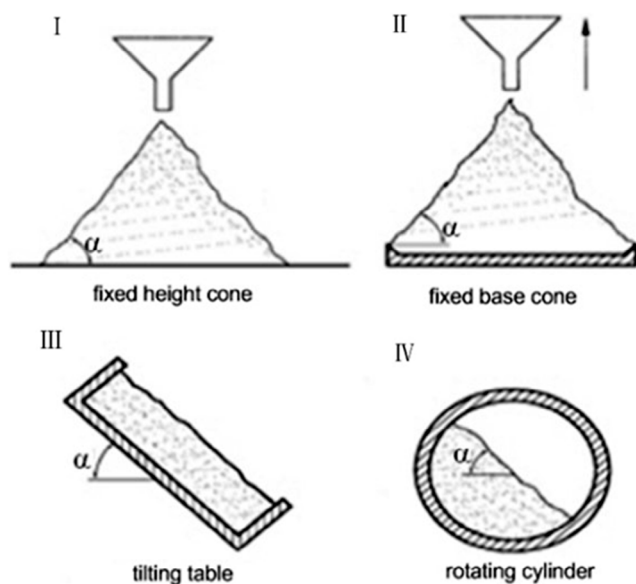


Figure 6. The different approaches to obtain an AoR measurement from powder. Reproduced with permission.^[31] 2006, Elsevier.

as shown in **Figure 7**.^[20] Their recycling strategy involved no virgin powder being added between cycles so the effects of powder recycling on flowability were not interrupted and hence the consistent trend. The PSD consistently shifted to coarser particles while the powder increased in flowability. Other specific reports on the flowability of 316L L-PBF powder in virgin and recycled states are not currently available in the wider literature. However, the increase in powder flowability has been reported on the very similar 304L metallic alloy powder for virgin and seven times reused powder.^[26] Sutton et al. analyzed both powder batches using the Hausner ratio, a ratio of compressed and loose powder, and imaging of the avalanche angle of powder using a revolution powder analyzer. Although the Hausner ratio indicated a small increase in the flowability of powder, the reduction in ratio was 0.01 to 1.08 and not significant. However, there was a reduction

of 14% in the avalanche energy when comparing virgin and recycled powder, which is significant. This change was attributed to an increase in particle sphericity and a reduction in fine particle size.^[35]

For other metallic alloy powders, the same trend of increasing flowability with powder reuse has also been observed. For titanium-based alloys, both Carrion et al.^[33] and Strondl et al.^[28] investigated the flowability of powder for L-PBF. Both studies used a powder rheometer to observe the changes in flowability. In both cases, the flow properties of the recycled powder were superior to that of its virgin counterpart. Interestingly, Strondl et al. also took the same powder from an alternative AM powder bed process, electron beam melting (EBM), where the heat source is provided by an electron beam instead of a laser. The starting PSD of the EBM powder was significantly coarser than the L-PBF powder. The majority of EBM powder is typically over 40 μm in particle diameter while the majority of powder for L-PBF usually has a particle diameter under 50 μm . Here the recycled powder performed worse during flowability testing with a powder rheometer, which was used to generate values for BFE as well as bulk and tapped density.^[29] With nickel-based alloys, Mellin et al. observed an increase in flowability in the Hall Flow and Gustavsson funnel methods, where powder is placed in a funnel and made to flow through an orifice.^[29] The Gustavsson funnel was more sensitive to the changes between virgin and used powder. Differences in flow time between new and used powder increased from 1.5% (using Hall Flow) to 14% (when using the Gustavsson funnel). With 17-4PH stainless steel metal powder, flowability has also been seen to increase when analyzed using Hall Flow. Flow time decreased from ≈ 13.3 to 11.9 s from 3 recycles to after 11 cycles respectively. No flows were recorded for powder samples from builds 1 and 2, which show that this powder had even poorer flowability.^[35]

The change in powder flowability is often attributed to the change in fine content of powder. Flowability has been seen to be adversely affected by an increased fine particle content since fine particles may agglomerate or clump together under the effects of Van der Waal forces.^[26,36] Although all particles will

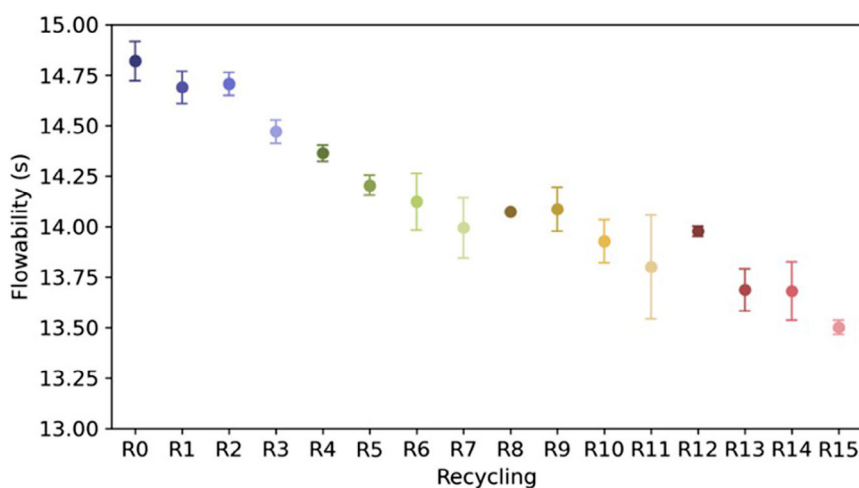


Figure 7. The Carney Flow time of 316L metallic powder with an increasing number of recycles. The consistent increase in powder flowability, as shown by the reduction in Carney flow time, can be correlated to the coarsening of the PSD. Reproduced with permission.^[20] 2022, Elsevier.

be affected slightly by Van der Waal forces, the effects on smaller particles will be proportionally greater and other forces will become dominant for larger particles. The cutoff point for this crossover is likely dependent on the specific powder but has been reported to be between 20 and 30 μm .^[30,36] The previous section showed that it is typical for the PSD to coarsen when recycling metallic powder. This shift to larger particle sizes with recycling will decrease the proportion of powder particles under the 20–30 μm size range, that can cause agglomeration and inhibit flowability. Therefore, the overall flow properties of a recycled powder batch may be expected to increase. However, PSD may not be the only factor affecting flowability, with sphericity/morphology and interparticle friction all having a role to play. Carrion^[33] observed no significant reduction to the D_{10} of Ti–6Al–4V powder particles yet saw a reduction in the BFE of recycled powder from ≈ 70 to 25 mJ when tested with no aeration, as illustrated in **Figure 8**. Adequate flowability is important to allow good coverage of powder on the build stage. Without this, parts will be fused with pores in the build stage, translating to pores within the finished parts. If this was compounded over multiple layers, the effects could be even more significant, potentially leading to the development of surface breaking defects and delaminations.

2.2. Chemistry

In addition to the PSD, the chemistry of metallic powders is also defined for a given powder batch in the preparation for L-PBF. The composition of the powder batch will likely be defined within a specific range. There are multiple points in the gas atomization process that could lead to contamination occurring to a powder feedstock.^[37] As L-PBF is a contained process, there are limited opportunities for powder to be contaminated. During L-PBF, the build chamber is filled with inert gas to eliminate the presence of oxygen and help to reduce the oxidation of parts and powder. Despite this, some oxidation can be expected to occur. Contamination and oxidation can occur when powders are stored

for extended periods of time, for example, inert gas fusion infrared absorption (IGF-IR) of Invar 36 powder stored poorly for over 1 year showed six times increase in oxygen content.^[38] The chemical composition of 316L for powder for L-PBF is defined in ASTM-F3184-16 and displayed in **Table 2**. An oxygen content of over 1000 ppm as a result of poor storage or recycling would put powder outside of these limits.

Spatter particles from the melt pool may also be highly oxidized. 316L spatter from five times used powder showed an increase in oxygen content from the formation of iron oxides.^[13] This was observed by the intensity of the oxygen peak through energy-dispersive X-ray spectroscopy (EDS) analysis, almost doubling when tested and comparing spatter to virgin powder. An increase in oxygen content in spatter particles for CoCr alloy metal powders has also been noted, where the oxygen content increased from 0.021 wt% to 0.046 wt% when comparing spatter with regular particles.^[11] In addition to the increase in the oxygen content within spatter particles, oxygen content may also increase in the HAZ next to the melt pool, as shown by Galicki et al.^[14]. Here the oxygen content of powder within the HAZ was seen to increase from 0.033 wt% in virgin powder to 0.043 wt% in heat-affected powder. With 316L powder, the amount of oxidation observed in powders was found to be related to the distance the powder was from the melt pool. As the distance from the melt pool increased, the oxygen content of powder was seen to decrease. The reasons for the increase in oxygen content were discussed by Galicki et al. and the thickening of the oxide layer under heating was deemed a more significant contributor than powder spatter resting back on to the area around the melt pool.^[18] Thicker Mn-rich oxide layers have also been observed on recycled 316L powder.^[20,22,23]

Due to the addition of oxidized spatter particles and oxidation of powder near the HAZ, recycled 316L powder would be expected to have an increased oxygen content compared with virgin material. This has been observed, but oxygen pickup does vary between studies. Yusef et al.^[19] saw an approximately three-fold increase in the oxygen content, from 1.42 to 4.68 wt% of 316L metallic powder in the virgin condition, as compared with that that had been reused ≈ 20 times, as confirmed via EDS. Subsequent XRD studies showed no variation in the phases

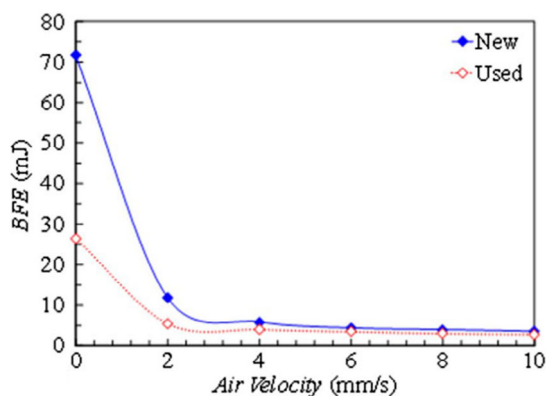


Figure 8. The basic flow energy (BFE) of virgin and 15 times reused Ti–6Al–4V powder. With no aeration of the powder, cohesive forces between the particles are still present. Virgin powder therefore has a higher basic flow energy as it contains more particles that are affected by interparticle forces. As the amount of aeration increases, interparticle cohesive forces are disrupted and the difference between the two samples become negligible. Reproduced with permission.^[32] 2019, Springer.

Table 2. The composition of 316L powder for L-PBF as defined by ASTM-F3184-16.

Element	Composition [wt%]
Cr	16.0–18.0
Ni	10.0–14.0
Mo	2.0–3.0
Mn	≤ 2.0
Si	≤ 1.0
N	≤ 0.1
O	≤ 0.1
Ph	≤ 0.045
C	≤ 0.03
S	≤ 0.03
Fe	Balance

being formed within the powder, indicating that any increase in oxygen content did not lead to major microstructural change. The threefold increase in oxygen as measured by EDS is a problematic result as EDS is generally unable to accurately provide information on the relative abundance of light elements, $Z < 11$.^[39] Other studies have also observed a much less significant increase in the oxygen content of recycled powder. Heiden et al. found an increase in the oxygen content of 316L powder reused 30 times, increasing from 0.067 to 0.090 wt%, despite the oxygen content in the L-PBF machine being kept below 1000 ppm during manufacture.^[17] Here, the number of reuse cycles was 10 times greater than the previous study, yet the oxygen pickup was much less and was reported to have remained within the ASTM F3184-16 standard range.^[40] 0.09 wt% is close to the limit however and greater recycling or virgin powder with a high oxygen content could have led to a further increased oxygen content. Other changes to the composition, such as an increase in chromium, nickel, and manganese, were not statistically significant. The average oxygen content of virgin 316L for two batches was 0.46 wt%, as reported by Keaton.^[41] The recycled powder oxygen content was 0.5975 and 0.905 wt% after 15 and 18 recycles, respectively, after use in SLM 280 machine. The increase in oxygen content in recycled 316L powder was found to be due to an increase in surface oxides by Gorji et al.^[42] In addition to changes in surface composition, the composition around internal porosity was also analyzed, finding that iron and chromium were distributed inside the pores, whereas manganese, nickel, and silicon diffused to the outer rim of the pore.^[24] Some level of diffusion could be expected with the heating associated with L-PBF. Small manganese-, silicon-, and oxygen-rich precipitates have been identified at the base of ductile dimples for tensile fractures of 316L AM material.^[43] Delacroix et al. observed a 200 ppm increase in the oxygen content of powder recycled 15 times.^[20] However, it is important to note that the initial oxygen content of this virgin powder was lower than for the majority of comparative sources shown in **Table 3**. As a result, the oxygen content did not come close to breaching 1000 ppm. Lu et al. recycled 316L powder for twice as long (30 recycles) as Delacroix et al. saw twice the increase in oxygen content of 400 ppm.^[21] The gathered sources show that oxygen pickup is highly likely in recycled 316L powder. Only Sartin et al. did not observe this trend.^[23] The scale of oxygen pickup registered varies significantly between sources

with the highest levels of oxygen being observed by Gorji et al. and Yusuf et al. Both researchers used a variation of EDS for their oxygen analysis. As already identified, there are significant limitations to the use of EDS for quantitative oxygen content analysis. All the studies that used inert gas fusion to calculate the oxygen content of their powder registered an oxygen content within the same order of magnitude between 0.01 and 0.1 wt%. This is a more accurate representation of the oxygen content of 316L powder. Despite the limitations of some methodologies, the majority of sources show increases to oxygen content for powder recycling. For all of the studies that used inert gas fusion to calculate oxygen content, the levels of oxygen were below the threshold of 1000 ppm set by ASTM F3184-16. However, recycling powder 30 times was shown to increase the oxygen content by up to 400 ppm.^[21] If virgin powder with an oxygen content above 700–800 ppm was heavily recycled, then this could be at risk of breaching the 1000 ppm limit. Further research is needed to confirm this but best practice should be to limit oxygen content in virgin powder.

The undesirable effects of large amounts of unintentional oxides within parts are widely understood. If the increased oxygen content of powder translates into an increase in the oxygen content of parts, then this would be detrimental to mechanical properties. However, it may not necessarily be true that all the oxygen in powder will all end up within a part. Yet, there is evidence that for Invar 36 metallic powder, an increase in oxygen content in the powder can change the melt pool dynamics, which in turn generates larger and further spreading and more irregular powder spatter, as observed in **Figure 9**.^[38] Evidence of this has yet to be recorded in 316L.

2.3. Other Changes

There are several other less-reported effects that powder recycling has on 316L metallic powder that warrants attention. Both 316L powder and parts have a predominantly austenitic microstructure. However, with recycled powder, an increased ferrite content has been observed when compared with virgin powder. Heiden et al. found that spatter particles for recycled 316L contained larger amounts of ferrite within their microstructure, potentially from the rapid cooling of the spatter.^[18] Ferritic metal powder in reused powder was also found to have a single-crystal microstructure, as shown in **Figure 10**. Ferrite is magnetic whereas austenite is not and it has been suggested that this could cause agglomeration of ferrite particles on the build plate and create voids.^[44] Magnetic ferritic powder particles were also observed by Delacroix et al., in recycled 316L.^[20] 15 times recycled powder contained 4 vol% ferrite compared with 0–1 vol% in virgin powder. These ferritic particles all consisted of a single-grain microstructure due to rapid cooling. The effect of ferrite powder on the characteristics of 316L powder has been investigated by Pinto et al.^[44] They found that when powder was reheated for metal fusion, the ferritic microstructure present in some recycled powder was lost and parts remained austenitic. This was true even with 6 vol% ferritic powder present in the feedstock. Therefore, phase changes in parts due to recycling are not a major concern. Instead, magnetism between particles was found to prevent good spreading of powder on the build

Table 3. The oxygen content of 316L powder in the virgin and recycled states.

Powder reuses	Analysis method	Oxygen content		Source
		Virgin	Recycled	
30	ICP-MS	0.067 wt%	0.095 wt%	[18]
30	Inert Gas Fusion	0.035 wt%	0.075%	[21]
≈20	EDS	1.47 wt%	4.68 wt%	[19]
15	Inert Gas Fusion	0.047 wt%	0.067 wt%	[20]
14	EDS	0.4575 wt%	0.5975 wt%	[41]
18	EDS	0.4575 wt%	0.905 wt%	[41]
10	XPS	27.04%	34.19%	[42]
12	Mass Spectrometry	No trend in oxygen content with reuse		[23]

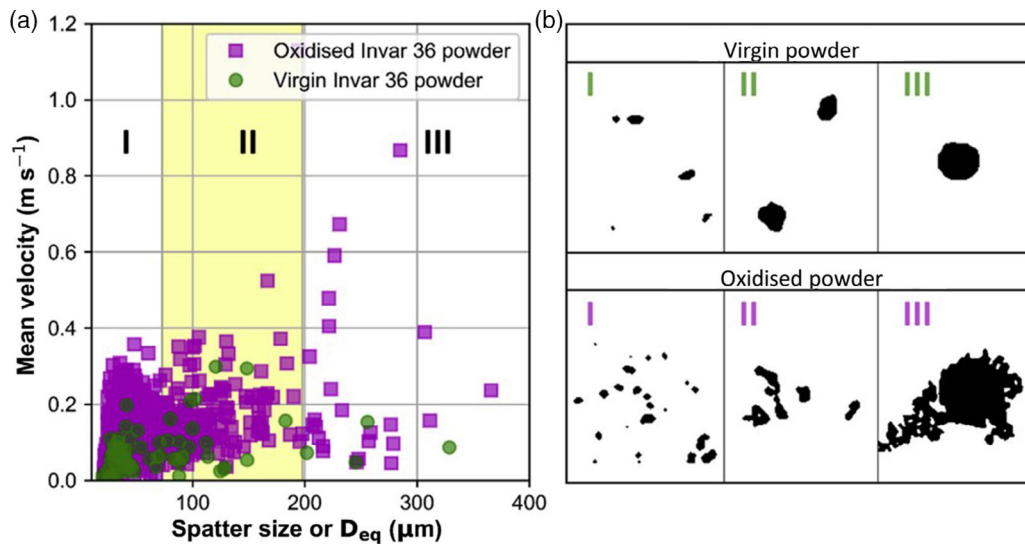


Figure 9. a) The size and velocity of spatter generated from oxidised and virgin powder, b) The 2D outlines of: I. powder spatter; II. powder spatter that has agglomerated and droplet spatter; III. droplet spatter. Oxidised powder has resulted in spatter that is larger, ejected faster and with a more irregular morphology. Reproduced with permission.^[37] 2019, Elsevier.

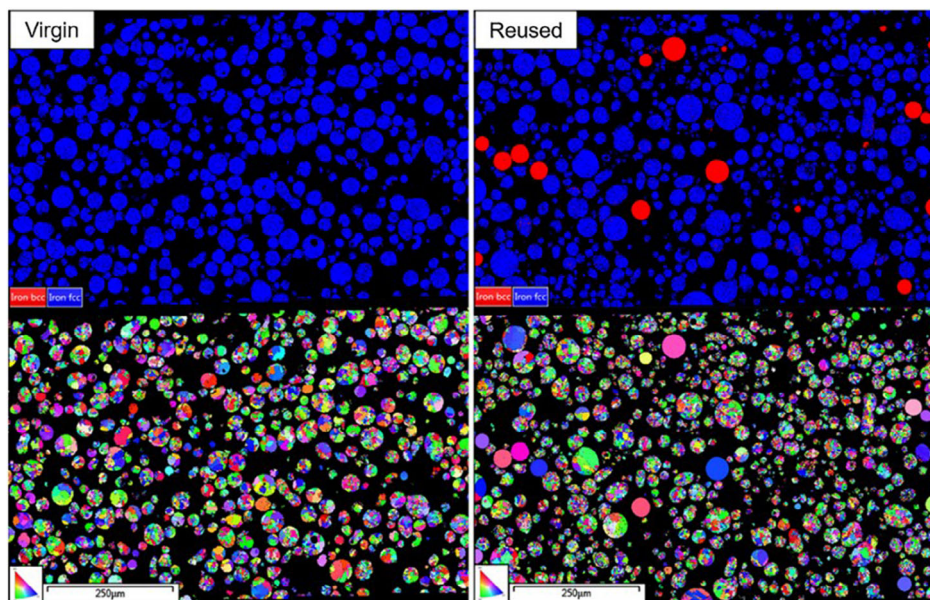


Figure 10. EBSD scans of virgin and reused 316L powder. IPF maps show that ferrite grains (red) have a single crystal structure and are only present in reused powder. Reproduced with permission.^[19] 2019, Elsevier.

stage, which could lead to voids on the build stage which are then retained in parts. Lu et al. quantified this increase in magnetism to show that the magnetization intensity increased from 0.0236 to 0.0967 emu g^{-1} for virgin and 30 times recycled powder, respectively.^[21]

Another factor is internal porosity, which may be present in gas-atomized powder before any changes could potentially occur due to the L-PBF process.^[45] Internal porosity has also been reported in recycled 304L and 316L metal powder.^[43] Heiden et al. saw a decrease in the average internal pore volume of

recycled powder, dropping from 241.3 μm^3 in virgin powder to 63.0 μm^3 .^[18] However, Keaton observed that the total pore volume of recycled 316L metal powder increased by 28% over 18 build cycles.

The morphology of powder will also influence the flowability and packing factor of the powder batch with irregular-shaped particles typically more difficult to pack, exhibiting inferior flowability. Spatter particles may either be highly spherical or extremely irregular, depending upon how they are generated and the type of spatter it is, as discussed previously.^[11] Therefore, there is at least

one mechanism for the average sphericity of metal powder to change as the level of powder recycling increases.

The aspect ratio of 316L in the population does not seem largely affected by powder recycling. Identical or very similar aspect ratios were present in 316L powder recycled 30^[18] and 20 times.^[19] However, in the same study, Yusef et al. saw a 2% reduction in the aspect ratio of 316L metallic powder after 20 recycles, while 17% reduction was seen for an aluminum and magnesium metal alloy, also recycled 20 times.^[18] Yet as reported by Strondl et al., in Ti-6Al-4V the aspect ratio was nearly identical for new and recycled powder.^[28]

3. Changes to Mechanical Properties as a Result of Powder Recycling

As summarized in the previous section, due to the nature of the L-PBF process, the changes that occur in 316L metallic powders in general are varied, yet are better understood than the changes that can occur in the resulting mechanical properties of components manufactured by L-PBF. The next section will cover some of the existing research on the changes to mechanical properties in 316L parts.

3.1. 316L Stainless Steel

Tensile testing of 316L parts with recycled powder has shown little deviation when compared to tests performed on samples containing virgin powder. Heiden et al. tested 316L high-throughput tensile samples with powder that had been reused 30 times using a 3D system ProX DMP 200 machine.^[18] Argon was used as an inert gas and the oxygen content was kept below 1000 ppm. There was a slight decrease in the ultimate tensile strength (UTS) of test specimens manufactured from reused powder, from 600–587 to 590–582 MPa for tensile samples from across the build plate. This minor decrease was reported to be within the standard deviation for these results and therefore it does not signify any significant change. This was also accompanied with a slightly larger increase in elongation to failure from 43–50% to 50–52%, respectively. As with the UTS, this change in elongation sits within the bands of standard deviation, which may suggest they are not statistically significant. Changes to the UTS, elongation to failure, and yield stress from recycling were similar to changes in these values when comparing samples from different regions of the base plate, suggesting that other potentially controllable factors such as build plate location may be as significant as any changes from powder recycling. This lack of variation in tensile properties with powder recycling

was also observed by Delacroix et al.^[20] and Sartin et al.^[23] As shown in **Table 4**, Delacroix et al. observed very little difference in the values of UTS, yield, or elongation when using powder that underwent 15 reuse cycles. In addition, there was no trend in the mechanical properties across the 15 reuses. The powder used showed strong correlations between oxygen content and powder reuse, as well as PSD and powder reuse. Evidently, these changes in powder properties were not significant enough to impart any significant change in the mechanical properties of parts.

Unlike Delacroix et al. and Heiden et al. who identified variation in their powder with reuse, Sartin et al. did not identify any trends in the powder characteristics, such as the PSD and oxygen content. This also resulted in no notable effect in the mechanical properties with powder reuse (**Figure 11**). Scatter in the results does appear to increase with powder recycling; however, this was reported to be a result of machine variation as opposed to powder variation.

The majority of studies of mechanical properties for recycled 316L do not observe a clear effect of powder recycling on the tensile properties. Liu et al. compared the properties of 316L parts manufactured with virgin and contaminated powder, which was reused five times, but not sieved to remove spatter, as illustrated in **Figure 12**.^[13] Parts were manufactured in a DiMetal-100 SLM machine with a maximum oxygen content of 500 ppm. The change in PSD for the powder analyzed by Liu et al. was far more significant than in other studies, such as that found by Heiden et al.^[18]. Heiden et al. started with a D_{10} value of virgin powder at 13.6 μm , which increased to 17.2 μm after 30+ recycles. Yet the powder used by Liu et al. had an original D_{10} of 22.5 μm , which increased to 46.2 μm after only five recycles. Both investigations used a mesh to sieve powders over 75 μm in diameter. For Liu et al., parts made from contaminated powder had significantly lower UTS and strain to failure values. The UTS of fresh powder samples was 664.7 MPa, while it was 499.8 MPa for contaminated powder. The strain to failure also fell from 29.3% to 13.7%. This was attributed to the inclusion of large spatter particles in the powder that were too large to be melted by the laser heat source and therefore retained in the microstructure. This large particle spatter, although created by powder recycling, would be removed by regulatory sieving procedures and therefore is not a major concern for powder recycling in other processes. The drift of PSD to coarser particles was identified previously as a potential impact of powder recycling, and the effect of PSD on the tensile properties of 316L L-PBF parts has been studied further by Spierings et al.^[5]. The mechanical properties of parts from three different batches of powder with three different PSDs were compared. The PSD of batch 1 and 3 started from the same point but batch 3 was skewed far more due to the presence of coarser particles. The D_{10} and D_{90} values of

Table 4. Mechanical properties for 316L parts manufactured with virgin and reused powder.

Powder Reuses	UTS [MPa]		Yield [MPa]		Strain [%]		Source
	Virgin	Recycled	Virgin	Recycled	Virgin	Recycled	
30	595 (average)	584 (average)	440 (average)	441 (average)	46.5 (average)	51 (average)	[18]
15	658	665	571	581	38.0	37.5	[20]
12	See Figure 7		–	–	See Figure 7		[23]
5 (No sieving)	664.7	499.8	488.9	436.3	29.3	13.7	[13]

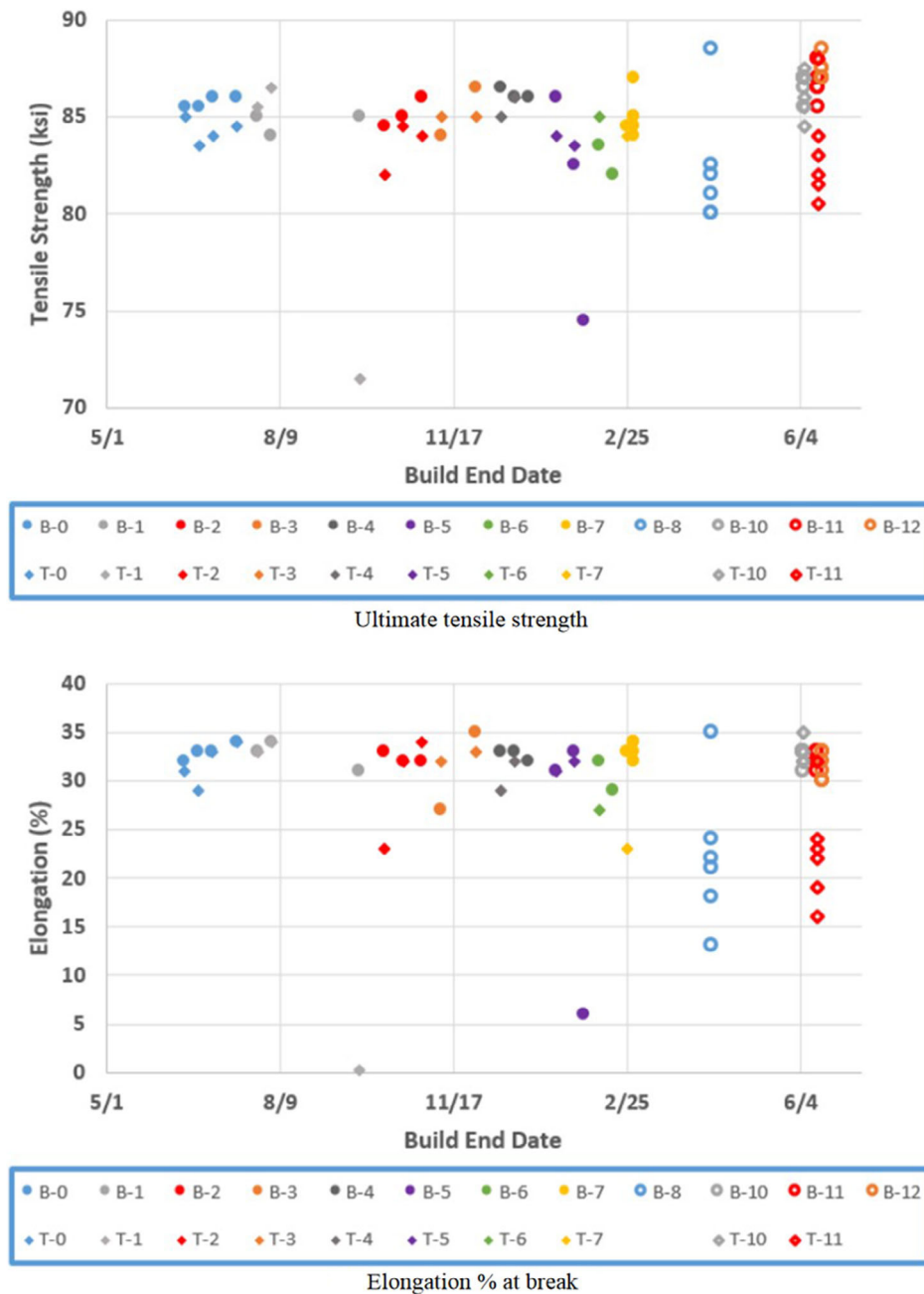


Figure 11. UTS and Elongation to failure behaviour for multiple tensile tests conducted on L-PBF 316L specimens manufactured with powder of different ages. Reproduced with permission.^[22] 2017, University of Texas at Austin.

batch 1 was 7.12 and 24.17 μm , compared with 15.26 and 55.54 μm for batch 3. In tensile testing, samples from batch 1 registered an approximate UTS of 760 MPa and 0.2% offset yield stress of 720 MPa, greater than the 605 and 590 MPa values observed for batch 3. Conversely, the elongation at fracture for batch 1 samples was 30%, compared with 40% for batch 3 samples.^[5] Here the D_{10} of powder, doubling from ≈ 7 to 15 μm , resulted in a noticeable effect on the mechanical properties with finer particles offering a higher UTS but lower elongation to fracture. Porosity in parts manufactured with the coarser powder was

increased, explaining the inferior UTS. The mechanism for this could potentially be a lack of fine particles to fill voids within the build plate or large volumes of coarser particles requiring more energy to remelt and therefore remelting between layers is reduced. This effect has not been observed in many recycling studies due to the limited change in D_{10} of particles with recycling, which is in the range of 3–4 μm when measured by laser diffraction.^[18,20]

Similarly to the changes in PSD, the changes in oxygen content from recycling were not significant enough to create

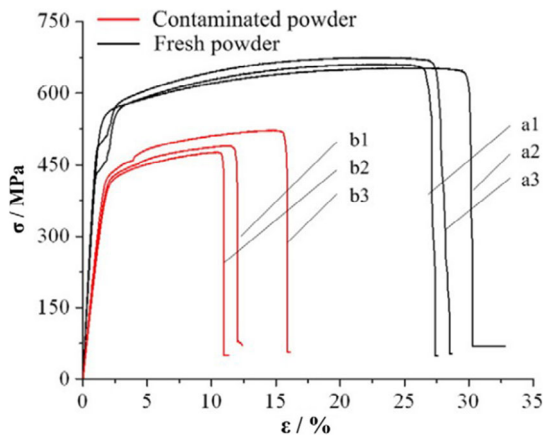


Figure 12. The difference in tensile properties as seen in virgin and contaminated (spatter from 5 times used powder). Reproduced with permission.^[13] 2015, Elsevier.

observable change in mechanical properties, despite oxides being retained in parts to form oxide inclusions if the oxide layer is thick enough.^[12] In other materials, powder oxidation resulted in increased porosity from the interaction of oxide films within the melt pool.^[38] Sutton et al. also observed 304L, where powder recycling increased oxygen content, which correlated with a decrease in the material's impact toughness.^[46]

4. Concluding Remarks

Powder recycling has been shown to affect the properties of L-PBF components manufactured with recycled/reused 316L powder. This in turn could also have a knock-on effect on the mechanical behavior of the parts manufactured with recycled powder, if the changes were significant or not managed effectively. However, with a suitable initial specification of powder and sieving during reuse, powder recycling has not been shown to significantly and, most importantly, adversely affect the mechanical properties of parts. A coarsening of the PSD with an increase in the diameter of fine particles has been observed for 316L recycled powder and similar stainless steel alloys. The level of powder coarsening is heavily dependent on the specifics of the process utilized and the number of reuses is not adequate as a method of comparison to capture the relative effects. As the PSD coarsens, there is generally a trend toward better flowability as the agglomeration of small particles is less prevalent. The method used to measure powder flowability also needs to be considered when comparing flowability as different techniques may not be analogous.

The oxygen content of powder is another factor that increases with continued powder recycling. Yet, the reported increases have remained within standard limits. Other reported changes to recycled 316L powder include changes to the microstructure. 316L has an austenitic microstructure but the recycling of metallic powder has been reported to increase the ferrite content from negligible quantities to 6–7 wt% with recycling.

When considering mechanical properties, the tensile behavior of 316L has been reported to show little change with powder

recycling and any reductions in UTS and yield have been small or insignificant. Other factors such as build plate location have often shown equal or greater significance. Large changes to the tensile properties of 316L samples are possible when large changes to the PSD occur. This suggests that the changes to PSD from recycling are generally not significant enough to influence major changes to mechanical properties but may be a contributing factor.

In summary, the changes in PSD, flowability, and oxygen content from powder recycling may have the potential to change the mechanical performance of parts. Generally, this is either positively for flowability or negatively for PSD and oxygen content. Further work needs to be undertaken in this field to draw clearer links between the changes in powder and part properties when using recycled powder. In particular, effort is needed to isolate specific changes to powder and monitor their effect on mechanical performance. Understanding the exact danger zones and cut-off points for powder properties affecting mechanical properties would be a powerful predictive tool when controlling quality during the production of parts.

Acknowledgements

The current research was funded under the EPSRC Industrial Case Award EP/T517537/1. The provision of a research bursary, materials, and supporting information from Rolls-Royce plc. is gratefully acknowledged.

Conflict of Interest

The authors declare no conflict of interest.

Keywords

additive manufacture, laser powder bed fusion, mechanical properties, metallic powders, powder recycle, stainless steel 316L

Received: April 26, 2022

Revised: June 15, 2022

Published online:

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