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Effects of powder recycling on laser-based powder bed fusion produced SS316L parts

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Abstract. Laser-based powder bed fusion (PBF-LB/M) is one of the extensively used additive manufacturing (AM) methods as the parts printed by PBF-LB/M have high resolution due to low layer thickness. Recycling and Reusing of the powder in this process has a significant impact on the surface roughness, mechanical properties such as hardness, elastic modulus and fracture strength etc. of the manufactured parts. The aim of the present study is to understand the effects of powder recycling on the properties of SS316L parts such as hardness and surface roughness. The developed method featured the use of SS316L powder five times over with weight percentage adjusted as required for the build job. The printed parts were checked for surface roughness across different surfaces with respect to the build direction along with Vickers hardness test. The surface roughness of the parts before polishing showed a steady increasing trend of about 40% with the recycling count while there was no significant effect on hardness as the values stayed in the range of 230+/-5 HV. Powder morphology studies with SEM displayed visible changes in terms of satellite formations, broken particles etc. with the increase in counts of recycling and particle size distribution showed a linear increase with the increase in recycling counts. This study was performed within the limited scope of a bigger study for which a detailed methodology of powder recycling will be developed.

Keywords: Additive Manufacturing, Laser powder bed fusion, powder recycling, characterization, hardness, surface roughness, digital manufacturing

1. Introduction

Laser-based Powder Bed Fusion (PBF-LB/M) is one of the extensively used additive manufacturing methods (AM) as the parts manufactured have high resolution due to low layer thickness [1]. In the industry, it is a common practice to reuse the metal powder used for manufacturing parts in the PBF-LB/M process for the purpose of cutting costs and ensuring sustainability [2]. Reusing and recycling metal powder has an effect on the part properties such as surface roughness, density, hardness, tensile strength, fatigue, fracture toughness etc. and powder properties such as particle size distribution (PSD), packing density and morphology [3][4]. The SS316L stainless steel material is often used in the medical and aerospace industries where corrosion resistant parts are required [5]. The surface characteristics of such parts are important in the said fields as the PBF-LB/M manufactured parts are often used as a part in assemblies [6].

In PBF-LB/M process, various parameters such as raw material parameters- particle size distribution (PSD), powder flowability, packing density, process parameters- powder spreading speed, powder layer thickness and build parameters- laser energy density, laser power, laser scan speed have an effect on the powder properties and the part properties [7]. When it comes to powder reuse, the research is focused on how the reusing and recycling phenomenon affects the part properties[8]. Emminghaus et al. have studied the effect of residual oxygen content and powder recycling on the part properties of PBF-LB/M produced Ti6Al4V [3]. The study was conducted by studying the effect of residual oxygen on virgin and recycled powders through understanding the part properties such as porosity, surface roughness and density. It was concluded from the study that the surface roughness of the parts increased steadily with the counts of recycling for the top surface and the side surface with respect to the build direction. A standard deviation of 4.01µm was observed for the top surface and a standard deviation of 2.79 µm. The reason was that there was an increase in the PSD of the powder particles with the counts of recycling. There was no significant change in relative density of the parts due to powder recycling.

Ahmed et al. studied the effect of powder recycling of 17-4PH Stainless steel powder on the part properties without adding any virgin powder in the recycling iterations. The powder was recycled ten times over after starting with the virgin powder. In each recycling stage, the powder characteristics were investigated by performing powder rheology, morphology, microstructure and chemical compositions. The morphology study showed that there was an increase in the powder particle size by 3% with each iteration of recycling. This is due to the spatter and partially molten particles getting attached to the spherical particles of the virgin powder and the subsequent powders from there. The difference in tensile strength was also studied between the recycled and the virgin powders where the parts manufactured with recycled powder showed no significant difference. The reason for the observation was suggested to be the lack of austenite phase present in the samples [9]. Gorji et al. conducted a synchrotron x-ray study on SS316L powder to understand the effect of recycling on the oxidation of powders. This study revealed that there was an increase in the concentration of metallic oxides on the surface of the powder particles as the recycling iterations increased. This was supported by the phenomenon seen in the SEM images of powder particles where there were indications of cracks and porosity within the powder particles as it is an effect of oxygen weakening the bonds existing in the powder particles [2].

Quinn et al. studied the effect of recycling SS316L powder on the parts manufactured using an EOS M280 machine. The design of experiments for the research included the measurement of hardness, surface roughness, and porosity for SS316L samples for each iteration of recycling. It was concluded from the study that the surface roughness of the parts manufactured increased by 50% by the time the number of recycling runs were finished while no significant difference in the hardness was observed. The increase in the surface roughness was attributed to the increase in the PSD of powder particles with the number of recycling runs. An increased PSD means that there is less than ideal fusion of the powder particles, which causes the formation of rough surfaces [10].

Different studies have been conducted to understand the effect of powder recycling on powder degradation and how the powder properties change throughout the recycling process [11]–[13]. Research was also carried out on how the additively manufactured part properties are affected by powder recycling as well. The scope of the present work is on how the number of recycling iterations affects the PSD and powder morphology of the powder that is used for printing. In addition, an investigation of the change in part properties such as surface roughness and hardness is also conducted. The scope of the present work is part of a bigger study where the aim is to develop a methodology to qualify metal powder for part production where recycling of powder is the key point of consideration. The study will include simulation from the point of view of powder bed and the effects of recycling will be simulated. The idea to include simulation is motivated by the need to reduce the dependency on the experimental data and procedures.

2. Materials and Methods

2.1. Laser Powder Bed Fusion
Stainless steel powder of grade SS316L has been used in Aconity MIDI+ PBF-LB/M printer from Aconity3D GmbH for this study. The SS316L powder used for printing was provided by SLM Solutions GmbH and has a PSD of 10-45µm and consists of mostly spherical shaped particles. Figure 1 shows the set of samples printed using SS316L which belong to the template defined for the project which funds the research. These samples included the powder canisters, bending test samples, tensile test samples and 10x10x10mm cubes.

Figure 1. Print layout for the project, sample of interest highlighted

The scope of the study presented in this paper however only covers the cubic samples that can be seen in different locations across the build plate. The 10x10x10mm cubes were printed with a laser beam scan speed of 800mm/s, laser power of 150W and laser beam size of 80µm. Hatch distance was 80µm. Laser was 400W continuous wave single mode fiber laser with Gaussian beam intensity profile and wavelength of 1070nm. The used shielding gas was argon.

2.2. Powder Recycling
The powder recycling methodology for this study involved recycling the powder five times over after starting with the virgin SS316L powder. For each build, the virgin powder was added to the remaining unmelted powder after printing to match the required weight of the powder to carry out the printing process. For the given set of samples that were printed using the pre-defined layout, the ratio between the used powder and re-added virgin powder was 3:2. This method was repeated five times to understand the effect of recycling on the produced parts and also the powder properties. In this methodology, the unmelted powder was not sieved to completely understand the behaviour of untouched and unmelted powder.

2.3. Powder properties
The powder properties that were studied in the scope of this research are particle size distribution and powder morphology. Powder samples were collected from each build starting with the virgin powder build until the 5th recycling iteration. PSD was measured using a Malvern3000 laser diffraction particle size analyzer from Malvern Panalytical Ltd. The PSD measurements were performed in the dry powder analysis method for all the powder samples from the five recycling iterations. The experiments were repeated three times for each sample for repeatability and were conducted a total of 15 times. Scanning Electron Microscopy (SEM) was performed on all the powder samples from the first four builds with high resolution FE-SEM Thermo Scientific Apreo 5 from Thermo Fisher Scientific Ltd. For SEM, only one samples of powder from each build was taken and studied.

2.4. Surface Roughness
Surface Roughness measurements were performed on the as-printed samples on the top surface of the cubic samples with respect to the build direction and also on the diamond polished samples of the same surface. The equipment used to measure surface roughness is the Bruker Alicona Infinity G6 from Bruker Alicona, which measures the form and surface roughness of samples. The machine performs an optical 3D scanning of the sample surface and provides values of R_a. The measurement for R_a was performed parallel to the hatch direction and perpendicular to the build direction. For the surface roughness measurement of the as-printed parts, a magnification of 800WD17 was used which indicates the working distance of 17mm and a resolution of 50nm. For the measurement of surface roughness of the same surfaces which have been diamond polished, a magnification of 400WD19 has been used which indicates a working distance of 19mm and a resolution of 30nm. The roughness measurements were conducted for a distance of 15µm perpendicular to the build direction on the sample surface. This procedure was repeated for both polished and unpolished samples.

2.5. Hardness
Vickers Hardness test was performed on all of the samples of interest which were diamond polished. The equipment used to perform the tests is the Innovatest Falcon 600G2 from Innovatest Europe. For all the samples, HV10 scale was used to measure the Vickers hardness, which means that the dwell time for the indentor was 10 seconds for a force of 1kgf. For each sample 4 indents were made across the diagonal of the top surface with respect to the build direction. The spacing between each indent was 1mm.

3. Results and Discussion

3.1. Powder properties
Figure 2 indicates the powder morphology of the SS316L starting from the virgin powder until the 4th recycling iteration as measured with SEM . The back scattered electron images show that there is a visible change in the morphology of the 2nd build powder sample when compared with the sample from the 4th recycling iteration. Powder from the 5th recycling iteration was not characterized as the gas flow stoped in the chamber during build time, leading to the powder attaining a burnt texture.

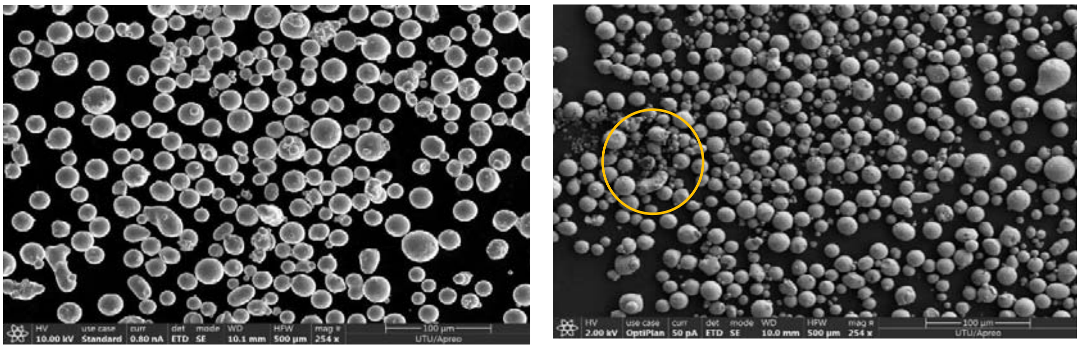


Figure 2. SEM images of powder samples. Build 2 (left) and Build 4 (right, particles with different morphologies)

The powder from the second build presents a morphology that is mostly spherical in nature for the powder particles present as the powder has not undergone significant changes within the build chamber of the PBF-LB/M machine. The particle size from the SEM images for build 2 is observed to be about 46µm while for the 4th build, it is observed to be at 49 µm. When the recycled powder is considered, it can be noticed from the image on the right that there are different types of morphologies present in the powder samples that belong to the recycled powders. The presence of satellite particles can be seen on the larger spherical particles of powder along with smaller nanosized particles. Other morphologies such as cracked and broken particles can also be observed. Partially fused particles, which occur due to the improper fusion of the melt pool can also be noticed in this highlighted area. The appearance of cracks and broken particles can be seen in figure 3.

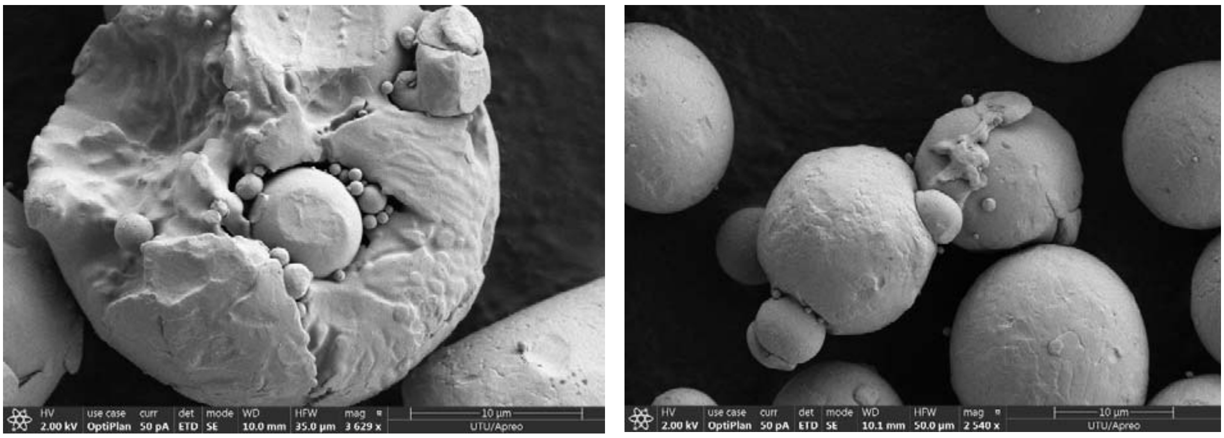


Figure 3. Broken powder particle (left) from build 4 and partially fused powder particles (right) from build 4

The cause for the formation of satellites on the larger powder particles is e.g. due to the fact that there is partially melted powder particles resulting from the laser beam and material interaction. These particles are formed inside the build chamber due to different phenomena such as spatter, particle breakage upon laser impact, improper melting of the particles etc. In this case, it is visible that the nano-size particles which are attached to the larger powder particles are spatter. The change in morphology is also supported by the PSD data accumulated for all the four powder samples.

The bar diagram in the figure 4 indicates a steady increase in PSD of the powder samples as the number of counts of recycling is increased.

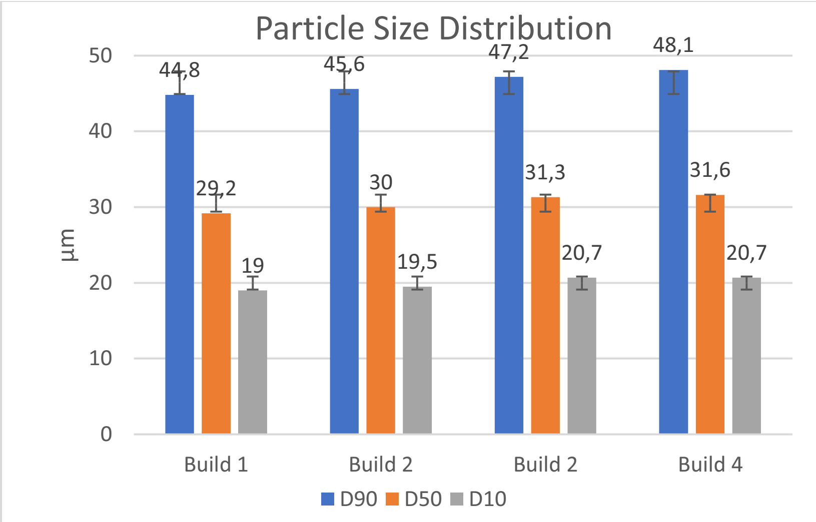
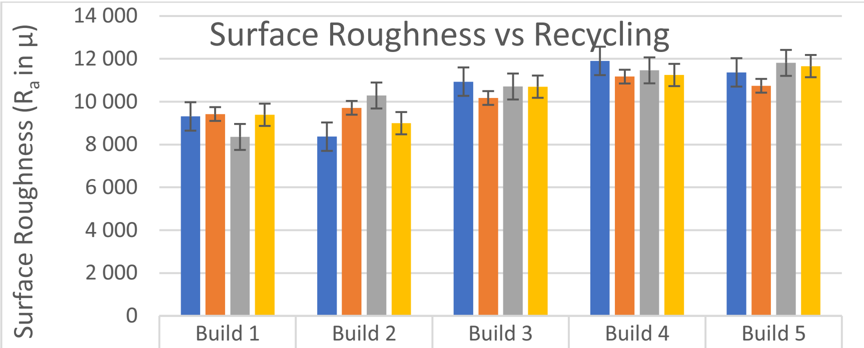


Figure 4. PSD of powder samples from each build

The D90 for Build 1 in the figure 4 shows that 90% of the powder particles in the given samples are of the minimum size of 45µm or above. D50 is an indication that 50% of the particles in the sample are of the minimum size of 29,2 µm and D10 shows that 10% of the samples in the build are of the minimum size of 19 µm. The increase in the PSD with the increase in the count of recycling is due to the formation of satellites on most of the powder particles within the sample and also due to lack of fusion between the two regular-sized powder particles which ultimately form a bigger particle.

3.2. Surface Roughness

The graph in the figure 5 depicts the average surface roughness of the samples for each build that has been measured on the top surface of the samples, perpendicular to the build direction. The surface roughness here corresponds to samples which were in the as built condition, meaning without polishing or any other surface treatment performed on the sample surface.



	Build 1	Build 2	Build 3	Build 4	Build 5
Series1	9 312	8 368	10 938	11 906	11 369
Series2	9 425	9 715	10 174	11 171	10 744
Series3	8 355	10 290	10 710	11 464	11 813
Series4	9 389	8 997	10 702	11 248	11 663

Figure 5. Surface Roughness of the pre polished samples

A trend of steady increase in the surface roughness of these samples is observed to be a direct effect of powder recycling. In PBF-LB/M, the surface characteristics of the parts produced are of high interest as they have an effect on the functionality of the part and as seen in the figure, there is a significant difference between the surface roughness of the first build and the surface roughness of the 4th build. The significant difference comes down to the surface characteristics that are affected by the lack of fusion and partial fusion of the powder particles on the surface of the sample while printing. For the preset laser parameters, the increase in the particle size of the powder samples means that there is not enough fusion in the powder bed when the laser beam interacts with the material. The fusion of the powder particles progressively becomes worse as the counts of recycling are increased and it is visible in the increasing values of the surface roughness of the samples which is an increase of 20%.

Figure 6 is a representation of the surface roughness of the diamond polished samples across all the builds. When diamond polishing is done, all the irregularities in the surface are removed. This can be seen in the values of the surface roughness across all the builds. The average surface roughness (R_a) of all the samples in the four builds is about 0.25µm.

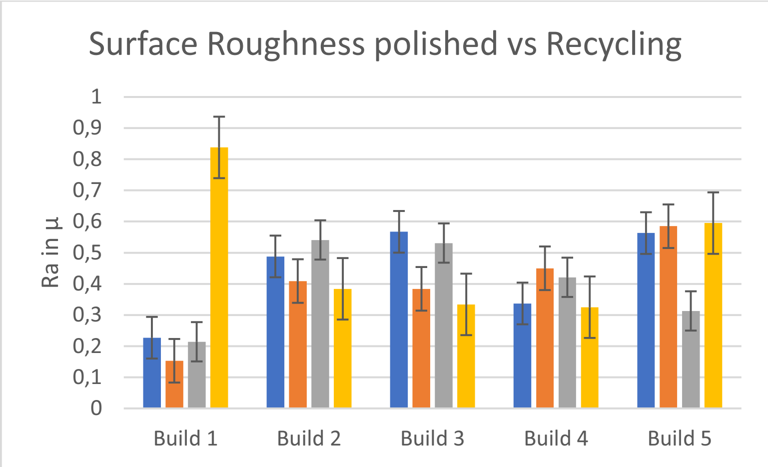


Figure 6. Surface Roughness of the polished samples

The similar values of surface roughness across all the builds suggest that the effect of recycling is not as significant after the polishing in comparison to before polishing. Once the removal of the irregularities and the contaminants from the surface occur, the recycling does not seem to have enough effect to alter the surface characteristics of the cubic samples printed. A polishing anomaly for the 4th sample in the 1st build incurred a really high value for surface roughness which is 300% higher than the average value of surface roughness. In both the cases of polishing and non-polished samples surface roughness is also affected by various factors such as packing density, powder flowability and melting behaviour. A uniform PSD means that the powder bed which is melted is smoother, meaning there is a uniform packing of the particles in a given layer. The nature of PSD for the powder obtained from the recycled builds is non-uniform, which means that the powder layer is irregular and causes improper melting of the powder particles and thereby promoting higher surface roughness.

3.3. Hardness

Figure 7 shows the Vickers Hardness values of the cubic samples. The values correspond to the average hardness of the build for which the measurements were done.

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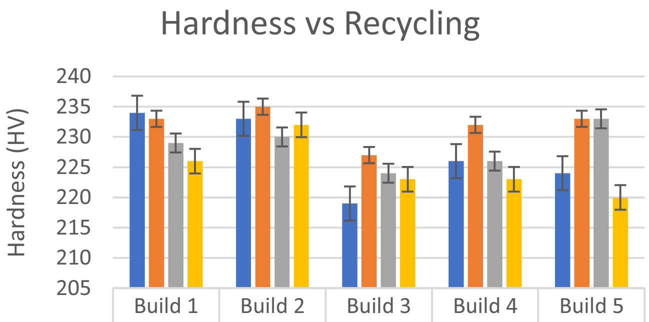

	Build 1	Build 2	Build 3	Build 4	Build 5
Series1	234	233	219	226	224
Series2	233	235	227	232	233
Series3	229	230	224	226	233
Series4	226	232	223	223	220

Figure 7. Hardness of the SS316L samples

Hardness values were measured to find out the existence of a possible correlation between them and powder recycling. As it can be noticed, the mean hardness of the samples falls in the category of 230+/-5 HV for all the 5 builds. This suggests that there is no significant effect of powder recycling on the hardness values. The recycled powder in the first and second iterations showed a higher hardness as compared to the cubic samples printed with the virgin powder. This is due to the oxidation of the powder which localizes the powder particles closer to the surface and influences the hardness. A similar phenomenon was studied and reported in the research of Gorji et al. where nano-indentation was performed to measure hardness [2]. The expected behaviour usually in the case of hardness of the samples is correlated to porosity present in the samples. The non-uniform PSD of the recycled powder causes the improper melting of the powder which leads to porosity and higher hardness. However, this phenomenon could not be seen in this case. The plausible reason for this could be that the laser parameters used to print the samples were at an optimum which would play down the effects of powder recycling.

4. Conclusion

The aim of this study was to understand the effect of powder recycling without sieving on part properties such as surface roughness and hardness has been studied with respect to the counts of recycling of powder. 5 builds have been printed according to the main project template out of which the cubic samples have been used for optical depth scanning to measure the surface roughness (for both as-printed and diamond polished samples) and Vickers hardness measurements. The powder samples collected only from the first four builds were analyzed because of handling issues. Powder morphology was analyzed by SEM and PSD was measured using laser diffraction method.

- The surface roughness of the as built samples showed an increasing trend of until 50% with the counts of recycling which suggests there was improper fusion of powder particles closer to the surface of the samples
- The trend in the hardness values for the samples is very uniform with the values being in the range of 230+/-5HV which suggests that there is no significant effect of recycling of the powder on hardness of the samples
- The trend in surface roughness of the samples shows that the packing density and PSD of the powder have a significant effect on melt behaviour of the powder, however, this effect is not visible in case of

hardness of the samples. This could be attributed to optimized laser parameters which offset the porosity effects.

5. Future work

Powder recycling has a significant effect on the powder properties and the parts produced by PBF-LB/M. The work in this direction will continue with the characterization of the samples outside the scope of this study. Tensile testing and Bending testing will be carried out on the samples that were printed along with the samples that were printed for this study to understand the effect of powder recycling on part properties such as tensile strength and fatigue.

In addition to the finish the remaining part property characterization, new materials will be printed and the parts will be characterized with a similar methodology. Simulation will also be introduced at a certain point to try and understand the changes in part properties.

6. Acknowledgement

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7. Conflict of interest

All the authors involved in the article report no conflict of interest.

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