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Characterization of metal powder reused multiple times for laser powder bed fusion

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Abstract

Laser Powder Bed Fusion (L-PBF) enables the manufacturing of highly complex parts with less material consumption for being used in various industrial applications. Metal powders are commonly used as feedstock material and the powder can be reused several times. Especially in aerospace applications, the reused powder characteristics are quite important in terms of traceability over the part quality since the aerospace industry requires high-quality and reliable end-use parts. In this study, Alloy 718 metal powder, which is generally utilized in high-temperature applications in gas turbine engines due to their good creep and corrosion resistance, were characterized over a 20 series of L-PBF build cycles. The powder characterization was performed in order to investigate the flow behavior of powder reused multiple times by using several methods such as powder rheology, particle size distribution (PSD), and quantitative morphology analysis along with apparent and tap density measurements. The results showed that no major and meaningful difference was seen among 20 times reused powder in terms of PSD and their morphology whereas there is a prominent difference seen by density measurements and with shear cell measurements by powder rheology. These methods revealed that reusing powder causes worse flowability trend.

Keywords: Laser powder bed fusion, powder reuse, powder characterization, powder rheology, particle size distribution and morphology.

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1. Introduction

Unlike the conventional manufacturing methods, Laser Powder Bed Fusion (L-PBF) allows parts to be manufactured with less material loss by reusing un-melted powder [1, 2].

Powder can be reused in several build cycles without significant change in chemistry or powder physical characteristics. However, after a number of reuse cycles, one of the main powder characteristics which is powder flowability starts to change with affecting the powder spreadability in L-PBF process. The final part quality decreases when the powder is not spreaded homogeneously throughout the build plate [1,3]. Since the aerospace industry requires high-quality and reliable end-use parts, the characteristics of powder that is used in the L-PBF process need to be traceable and interpretable well [4]. In aerospace industry, Alloy 718 is commonly used in high-temperature applications such as gas turbine engine because of its good creep and corrosion resistance [5]. Therefore, understanding and defining the Alloy 718 reused powder characteristics play an important role for this industry.

On the other hand, defining and understanding reused powder characteristics for L-PBF process is a complex phenomenon and also challenging. [3, 6, 7]. Recently, several studies on reusing powder in L-PBF for different materials have been investigated. For instance, Yi et al. studied on powder characteristics of reused Inconel 718 powder. They revealed that with increasing number in powder reusing the powder particle size increases [2]. Just as Yi et al., Ardila et al. studied on reusing Inconel 718 powder. They reused the powder up to 14 cycle and characterized the powder by doing particle size distribution analysis. Similar to Yi et al.'s results, Ardila et al. showed that particle size distribution after several production cycles was similar, with the exception of a small amount of particle aggregation in bigger particle sizes [5]. Moghimian et al. researched on the reusability of titanium, nickel and aluminum alloys in order to give some insights on reused powder characteristics [4]. Cordova et al. used different characterization methods such as particle size distribution, morphology, density, chemical composition and rheological analysis to reveal the effects of powder reuse for Inconel 718, Ti6Al4V, AlSi10Mg and Scalmalloy materials [7]. Cordova et al. reused AlSi10Mg powder 6 times, Ti6Al4V powder 11 times and Inconel 718 powder 38 times. However, contrary to other studies, in Cordova et. Al.'s study, Inconel 718 powder was rejuvenated with virgin powder for each cycle. Gruber et al. [8] also studied on the characterization of 20 times reused Inconel 718 powder by analyzing morphology, flowability, and physico-chemical behavior. One of the main outcomes of this study showed that virgin powder had worse flowability than reused powder. Nguyen et al. [9] reused Inconel 718 powder 10 times and showed that reused powder had lower flowability than virgin powder, as opposed to Gruber et al. [8] and Yi et al. [2]'s results. Since there are contradictory results in the literature and powder handling procedures vary from user to user, exploring of powder characteristics requires more attention.

In this study, Alloy 718 powder was featured over 20 series of L-PBF build cycles. The powder characterization was performed in order to investigate the flow behavior of multiple times reused powders applying several methods such as powder rheology analysis with both shear and powder cell, particle size distribution (PSD), quantitative morphology analysis and Hausner Ratio calculation. This study aimed to help users to explore the reused powder characteristics by using different methods with novel approaches.

2. Material and methods

2.1. Powder handling, sampling and manufacturing

As a feedstock material, gas atomized Alloy 718 (UNS N07718*)* powder from Oerlikon Metco was used. The Powder was characterized over 20 series of L-PBF built cycles. L-PBF process was performed in EOS M400 system equipped with single yttrium fiber laser under argon atmosphere. Each build cycle was produced using the same build job as given in Fig. 1. The 65,7 J/mm^3 volumetric energy density was used for the process. The direction of gas flow in process chamber was opposed to scanning direction of build parts. Powder handling and sampling was carried by the same operator with the same procedures. The virgin powder was loaded to start the first process and no rejuvenation was made on the reused powders for the rest of the 19 cycles. Powder batch samples, which is to be characterized, were collected from 63 µm mesh-sized sieving after each build process was completed. For each cycle, 200g of powder was collected for characterization purposes.

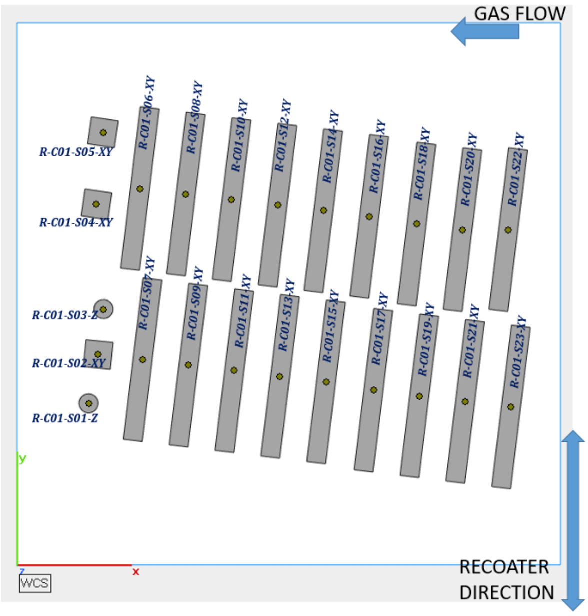

Fig 1. Build plate plan produced in each reuse job.

2.2. Powder Characterization

The powders were analysed using various powder characterization methods shown in Table 1.

Table 1. Characterization plan of the presented study.

Characterization Method	Characteristics	Standard
Particle Size Distribution (PSD): Laser Diffraction (LD) + Dynamic Image Analysis (DIA)	Particle Size	ASTM B822, ISO 13320
	Morphology	ISO 13322-2
Density measurements and Hausner Ratio calculation	Apparent Density & Bulk density	ASTM B212, ASTM B417
	Tap Density	ASTM B527
	Hausner Ratio	n/a
Powder Rheology: Powder cell + Shear Cell	Powder cohesion behavior	n/a
	Powder shear behavior	n/a

The particle size distribution analysis was carried out using Microtrac FlowSync and Malvern Mastersizer Hydro 2000G. Both of these devices used wet operation with water with 1.33 Refractive Index (RI) as a dispersant. Besides, 1.98 of RI was used for Alloy 718 particle in both devices. The main reason of using two different device was to validate the particle size results.

The powder morphology analysis was employed by using Microtrac FlowSync which has Dynamic Image Analysis (DIA) capability. Powder morphologies of each reuse cycles was compared with each other according to quantitative morphological analysis shown in Table 2. The morphological calculations are mathematically defined in the software of device. The particles were filtered by 0.5, 0.7 and 0.9 values and accordingly the percentage of particle volume were compared with each other.

The apparent density measurement was done according to ASTM B212 standard by using flow measurement kit supplied from LPW Technology. The tap density measurement was done in accordance with ASTM B527 standard by performing a tap density tester supplied from Torontech. The volume defined on a cup of 25 cm^3 was recorded after 3000 taps. According to apparent and tap density results of powders, hausner ratio was calculated as in (1).

$$Hausner\ ratio = \frac{Tap\ density}{Apparent\ density} \qquad (1)$$

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Table 2. Quantitative morphological analysis parameters.

Morphological Parameters		Description
Shape	Sphericity (4πarea/Perimeter)	changes from "0" to "1". "1" describes the perfectly spherical particle.
	W-L aspect ratio (Area/CHull Area)	changes from "0" to "1". "1" describes the perfectly spherical particle.
Surface Roughness	Solidity (Area/CHull Area)	changes from "0" to "1". "1" describes the perfectly smooth particle.

The rheological analysis of powders was carried out using Anton Paar MCR 702 MultiDrive system with the equipment of powder flow cell and shear cell.

The powder shear cell equipment was employed for the measurement of Coefficient of flowability (ffc) which is a function of major principal stress (σ1) and unconfined yield strength (σc) as defined in (2) [3].

$$ffc = \frac{\sigma 1}{\sigma c} \quad (2)$$

The measuring system was a large shear system of PSC43 having 18.9 ml volume. Shear cell measurement was started with gathering pre-shear stress measurements under multiple repetitions of 3 kPA, 6 kPA and 9 kPa normal stresses and their reduced stresses. After that, yield focus analysis was carried out using Mohr-Coulomb model by the rheometer device and flow function was obtained accordingly. 1^{st}, 5^{th}, 10^{th}, 15^{th} and 20^{th} reuse cycles were measured three times in order to make a clear assessment on the results.

The powder cohesion strength was analyzed using powder flow cell. Two-blade stirrer ST36-2V-10/PFC was used as a measuring stirrer. For the powder measurement preparation, the powder memory was erased by the pressure drop method applying fluidization process at 2,5 l/min at the first stage. Then, powder cohesion strengths were calculated by recording torque signals. Cohesion strength results were defined from average of the last 20 data points. The same amount of material which is 170g was analyzed for 1^{st}, 5^{th}, 10^{th}, 15^{th} and 20^{th} reuse cycles.

3. Results and discussion

3.1. Particle Size Distribution and Powder Morphology

Fig. 2(a) and (b) present the PSD curves of different reuse cycles using Microtrac Sync and Malvern Mastersizer 2000G, respectively. The results describe that reusing powder causes shifting PSD curves both left and right side by decreasing the peak of curve. This shows that not only agglomerated particles increase but also a small size of particles caused by spattering or soot increases.

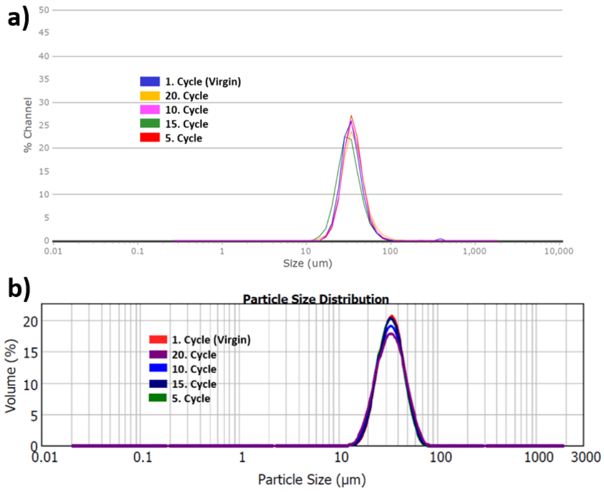


Fig 2. PSD curves of reuse cycles: a) acquired by Microtrac Sync, and b) acquired by Malvern Mastersizer 2000G.

Table 3 shows the median particle size (D50) and span of the same cycles using different PSD devices. It can be seen that the width and span tendency of PSD increase with reusing numbers. This also points out that the agglomerated and small sized particles increase due to spattering or sooting in the powder bed.

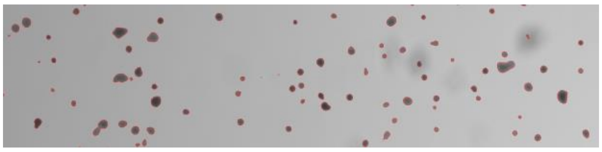
Table 3. Median particle size (D50) and span of the same cycles using different PSD devices.

Particle Size Distribution				
Reuse Cycle	D_{50} (Microtrac Sync)	D_{50} (Malvern Mastersizer 2000G)	Width (Microtrac Sync)=(d84-d16)	Span (Malvern Mastersizer 2000G) =(d90-d10)/d50)
1.	33,51	34,35	18,14	0,746
5.	34,91	33,62	18	0,759
10.	34,82	33,82	19,28	0,811
15.	31,30	34,14	19,22	0,761
20.	35,34	33,93	21,44	0,865

In quantitative morphology analysis, all reuse cycles were measured via Microtrac Sync. However, it was seen that the particle count was an important parameter that effects the results. For this reason, only the reuse cycles with roughly the same particle count were considered for measurements. Table 4 shows the different filtration of particle sphericity for the 9^{th} and 20^{th} time reused of powders. This describes that by reusing powder, the sphericity of particles tends to decrease from 86.18% to 83.82% when considering the sphericity value bigger than 0.9. The trend was the same all the other morphological parameters given in Table 2. However, there was also seen that the controversial results exist in comparison to the reuse of 3rd and 17th. Therefore, quantitative powder morphology analysis by dynamic image analyzer (DIA) needs to be investigated in detail by comparing and validating via scanning electron microscope analysis.

Table 4. Quantitative sphericity analysis of powder batches by dynamic image analyzer method.

Sphericity



Reuse Cycle	Particle Count	Filter	Passing Volume (%)
3.	61451	>0.5	100
		>0.7	99.06
		>0.9	**84.25**
9.	57747	>0.5	100
		>0.7	99.06
		>0.9	**86.18**
17.	61551	>0.5	100
		>0.7	98.71
		>0.9	**85.39**
20.	55344	>0.5	100
		>0.7	98.91
		>0.9	**83.82**

3.2. Powder density measurements and Hausner Ratio

The apparent and tap density measurements were carried out for over 18 cycles. With the increase in reuse times, there was no significant and meaningful changes in apparent densities whereas the tap densities increased from 5.07 g/cm^3 (virgin) to 5.26 g/cm^3 (18th cycle). This increasing trend in tap densities of reused powder can be seen in Gruber et al and Yi et al.'s studies [7,9] that point out changes in powder packing and powder morphology.

Hauser ratio calculation was done and accordingly flow behaviour was determined based upon Kaleem et al.'s study [10] for the cycles as shown in Fig 3. It is clearly seen that the trend of hausner ratio increases with powder reusing number and thus, the flowability of powders becomes worse.

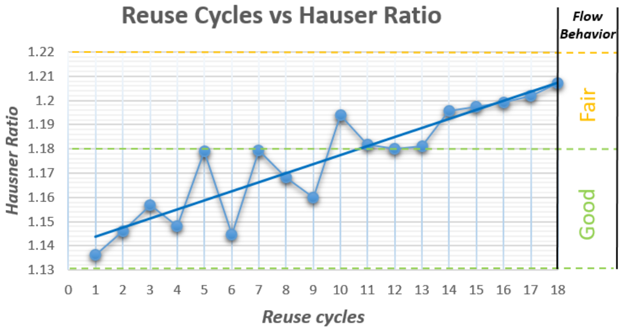


Fig 3. Hausner ratio and flow behavior of reuse cycles.

3.3. Powder rheology

Rheological tests were carried out to determine the coefficient of flowability (ffc) and powder cohesion strengths for 1st, 5th, 10th, 15th and 20th reusing cycles.

Fig. 4 demonstrates the ffc and flow behavior under 9 kPA normal stress, which was determined according to the Mohr-coulomb principle by the rheometer device. The relationship between ffc and powder flowability can be classified as "free-flowing" (ffc >10), "easy-flowing" (4<ffc<10), "cohesive" (2 < ffc<4) and "very cohesive" and "not-flowing" (ffc < 2) [11]. The results in shear test measurement show that powder flowability trend decreases with powder reusing increases.

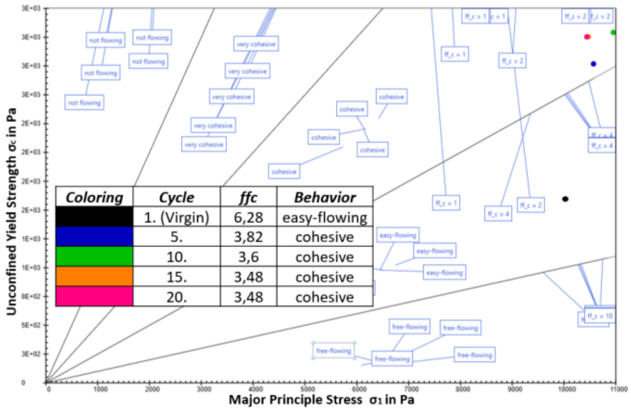


Fig 4. Shear cell measurements and flow function behaviors of Cohesion strength measurements of 1st, 5th, 10th, 15th and 20th reuse cycles.

Fig. 5 points out the results of cohesion strength tests performed for 1st, 5th, 10th, 15th and 20th reuse cycles by powder cell equipment of reometer device. The correlation between the reuse cycles was not clearly and meaningly seen in these measurements. The main reason of this can be the measured powder amount which is 170g equals to below 40 ml in powder cell. This powder amount may not be enough to predict the powder cohesion strength reliably since the blade stirrer surfaces need to be fully covered with powder before measurement.

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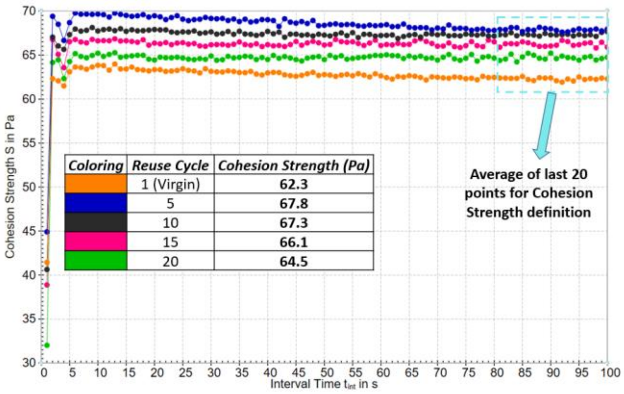

Fig 5. Cohesion strength measurements of 1st, 5th, 10th, 15th and 20th reuse cycles.

4. Conclusions

In the present paper, we explored the effect of powder reuse for Alloy 718 produced with the L-PBF method by characterizing powder reuse cycles via PSD and morphology analysis, powder density measurements and powder rheology. The following conclusions can be drawn from the present study:

1. Powder reuse causes shifting PSD curves both left and right side by decreasing the median size of the curve. This shows that not only agglomerated particles increase but also a small size of particles caused by spattering or soot increases. The trend of width or span of the PSD curve is also increasing due to the abovementioned phenomenon.
2. Quantitative morphology analysis by DIA requires detailing investigation in order to define the powder characteristics. Furthermore, the particle count plays an important role to compare the reuse cycles.
3. Tap and apparent powder density measurements revealed that the trend of hausner ratio increases with reusing powder. This result shows that the flowability of powders becomes worse with reusing number of the powder increases.
4. Powder shear cell measurements by rheometer show that the powder flowability trend via defining flow functions decreases with increasing reusing of powder.
5. In the powder cohesion strength measurement by rheometer, there was not seen a meaningful and clear correlation between the reuse cycles. Additional tests with different amounts of materials are required to evaluate the reused powder characteristics.

The future work will continue with performing additional build cycles with the same powder batch. Furthermore, different powder characterization methods will be used such as a scanning electron microscope for the evaluation on morphological analysis for a better understanding of reused powder characteristics. Moreover, the influence of reused powder on chemical composition, microstructure, and mechanical properties including fatigue behavior, is an ongoing study.

Acknowledgments

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Author's statement

Conflict of interest: Authors state no conflict of interest. Informed consent: Informed consent has been obtained from all individuals included in this study. Ethical approval: The research related to human use complies with all the relevant national regulations, institutional policies and was performed in accordance with the tenets of the Helsinki Declaration, and has been approved by the authors' institutional review board or equivalent committee.

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