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Reviewed Paper

Powder Reuse Effects on the Tensile Behavior of Additively Manufactured Inconel 718 Parts

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Abstract

Inconel 718 (IN718), with a wide range of applications in aerospace industries and good weldability, is a popular powder feedstock in the laser beam powder bed fusion (LB-PBF) additive manufacturing (AM) process. Due to fabrication, handling, and storage costs, powder feedstock is commonly reused several times. Therefore, it is important to understand how the mechanical properties of LB-PBF parts can be affected by powder reuse given that powder characteristics may change after repeated recycling. This study aims to investigate the effect of powder reuse on the tensile properties of LB-PBF IN718 parts. Powder characteristics such as cohesion and compressibility will be quantified in order to shed light on the variations observed in the part performances. In addition, by correlating the state of the reused powder with tensile properties, the most critical metrics for quality aspects in powder reuse will be determined.

Keywords: Additive manufacturing, LB-PBF (L-PBF), Tensile properties, Inconel 718, Powder recycling, Powder characterization

Introduction

Metallic powder, as the material feedstock in the laser beam powder bed fusion (LB-PBF) method, is commonly reused/recycled after each fabrication. While this is a common practice and can remarkably affect the powder characteristics such as particle size distribution (PSD) and morphology, there is still no standardized procedure on how to recycle the powder. Therefore, understanding the effects of powder reuse on powder characteristics, and in turn, the mechanical properties of additively manufactured (AMed) parts is important. In addition, each material system (e.g. nickel-based and titanium alloys and stainless steels) can behave distinctly as the powder packing and flow behaviors are dependent on many factors such as PSD, particle shape and microstructure, etc [1–3].

In this study, Inconel 718 (IN718), a nickel-based superalloy, was selected as the powder feedstock which is extensively used in aerospace industries due to its great high-temperature strength, creep, and corrosion resistance [4–6]. In addition, the feasibility of fabricating net-shaped parts with additive manufacturing (AM) has made it an appropriate processing method for IN718 due to its poor machinability, making it difficult for post-fabrication surface treatments. Therefore, it is important to fully understand the feedstock-structure-part performance relationships in AM of IN718.

Some studies have been conducted in an attempt to shed light on the effects of powder characteristics and its reuse on the powder and the performance of AMed IN718 parts. For instance, Nandwana et al. [7] studied the effects of reusing IN718 powder in electron beam powder bed fusion (EB-PBF) on its characteristics. It was reported that IN718 can be chemically stable with powder reuse. In addition, negligible changes in powder flow behavior were observed as a result of reusing the powder six times. Cordova et al. [8] also examined the effects of powder reuse on the characteristics of gas-atomized IN718 powder with an initial particle size distribution (PSD) of 20-63 µm in LB-PBF.

In their study, the powder was reused 38 times with the "top-up" practice. In the top-up method, unlike continuously reusing the powder, some virgin powder is mixed with the reused one to rejuvenize it [9]. In the 38th cycle, the PSD became narrower, which was attributed to the reduction of fine particles in the batch. Furthermore, the used powder particles were found to be less spherical as compared to the virgin powder which was correlated with the number of times that the powder was reused.

It was also reported that the flowability of IN718 may increase with continuously reusing the powder. This behavior was ascribed to the existence of fewer fine particles in the reused batch. The reason was explained by the higher cohesion between fine particles and their tendency to agglomerate, which in turn, decreases the powder flowability [10,11]. Ardila et al. [12] analyzed the effects of 14-times reusing IN718 powder in LB-PBF. In this study, the powder was filtered via a 63-µm sieve. Afterward, it was dried in an oven with air circulation and reused for the next fabrication. A slight decrease in the amount of finer particles was observed due to reusing the powder 14 times. No significant trend in the fracture toughness, obtained from Charpy impact tests, as a function of powder reuse was reported.

The effect of powder reuse on the surface chemistry of IN718 powder particles was studied by Gruber et al. [13]. A plasma-atomized IN718 with an initial PSD of 45-105 µm was reused in an EB-PBF system. On the surface of virgin powder particles, a thin oxide layer was observed. However, this layer was seen to coarsen with continuously reusing the powder as the aluminum (Al) was exposed to the environment in the EB-PBF process. This increase in the oxide layer was in accordance with the increased oxygen content within the batch as a result of powder reuse.

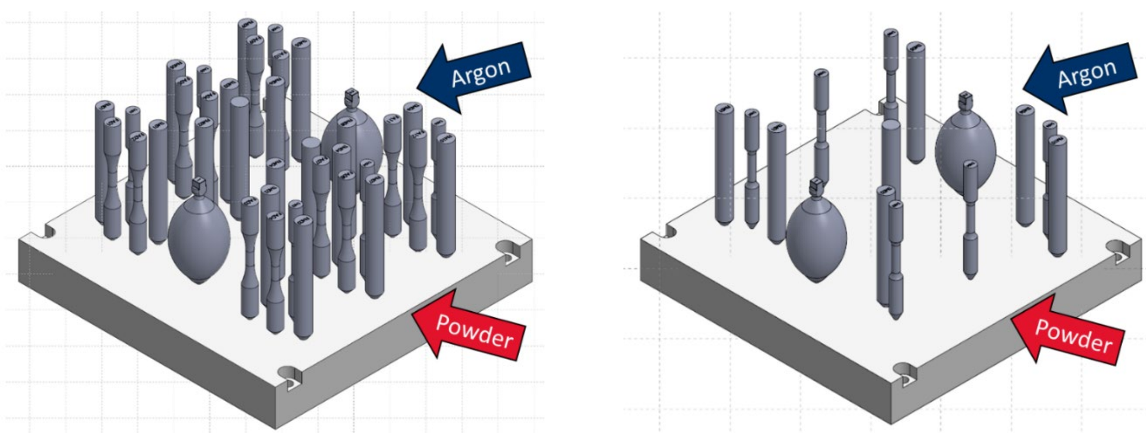
In the aforementioned studies, more attention was paid to how powder characteristics evolve as a function of powder reuse with less focus on mechanical properties. Some research studies on other material systems (e.g. Ti-6Al-4V and 17-4 PH SS) have reported that these changes in powder characteristics can govern the defect formation and ultimately the mechanical performance of additively manufactured parts [2,10,14]. Therefore, this study aims to investigate the effects of reusing IN718 powder on the tensile properties of parts fabricated via LB-PBF. Additionally, any variation in tensile properties will be explained by the observed differences between powder batches as a result of the top-up reuse practice. It needs to be also specified that the number of times that powder is reused may not be as critical; instead, the changes in powder characteristics need to be focused on as they may also change depending on the geometry and number of parts on the build plate [9]. Finally, some conclusions will be made based on the findings of the present study.

Experimental Program

In this study, argon-atomized PRAXAIR TruForm IN718 powder was employed. The virgin powder had an initial PSD of 17-48 µm, which was measured with the Anton Paar PSA 1190 particle size analyzer based on laser diffraction technology. All parts were fabricated via an EOS M290, an LB-PBF AM machine in an argon environment. In addition, the grid nozzle was used to establish a laminar flow inside the chamber.

The major infill process parameters employed for fabrication consisted of 285-W laser power (P), scanning speed (V) of 960 mm/s, hatching distance (H) of 0.11 mm, and 40-µm layer thickness (t). As seen in Fig. 1, two different layouts including full and half were used. The number of specimens on the half layouts was less as compared to the full layouts in order to ensure the powder amount is sufficient to successively fabricate 18 sets of specimens.

The full layouts were used for the 1st, 6th, 12th, and 18th prints, and half layouts were used for the interval prints. In each layout, specimens were placed in different locations. In Fig. 1, the argon flow inlet and the powder delivery directions are also illustrated by dark blue and red arrows, respectively.



(a) (b)

Fig. 1 (a) The full and (b) half build layouts including six net-shaped tension test specimens.

Fabrication was started with an initial amount of 65 kg virgin powder. Powder samples were collected before starting each fabrication using a multi-level sampler according to ASTM B215 [15]. After each fabrication was completed, the used powder in the machine was collected and transferred to a sieving module, equipped with a 63-µm mesh sieve. After sieving, the virgin powder was added to the used powder to ensure the total weight is the same as the initial one and rejuvenate the reused powder. The powder was uniformly mixed by rotating the powder more than 20 times. Subsequently, the powder was transferred back to the machine for the fabrication of the next batch. This procedure was repeated 18 times; however, the results of tension tests are provided for the first 10 sets.

On the full and half layouts, they were six net-shaped tension test specimens following the ASTM E8 [16] with the geometry shown in Fig. 2. All the specimens were steam cleaned after fabrication and subjected to heat treatment. The heat treatment schedule included both solution annealing and precipitation hardening. The details regarding the heat treatment schedule cannot be disclosed. It needs to be specified that some other parts including cylindrical rods and net-shaped fatigue specimens were also placed on the build plates which were not used in the current study.

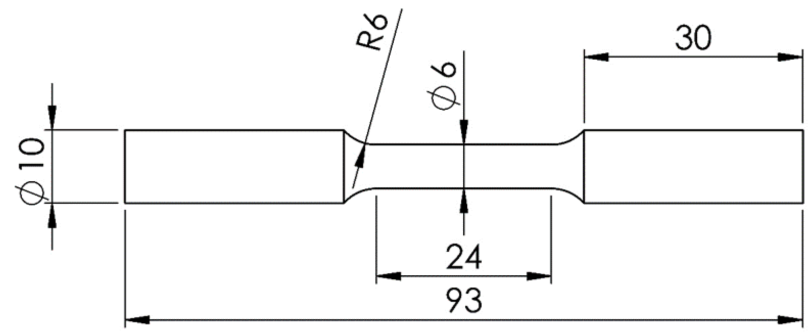


Fig. 2 The geometry of net-shaped tension test specimens according to ASTM E8. All dimensions are in mm.

Tensile tests were conducted using an MTS Landmark servo-hydraulic testing machine with a 100 kN load cell according to ASTM E8 [16] at room temperature. An MTS mechanical extensometer was attached to each specimen in the gage section to measure the strain. Before initiating the test, parallel lines were marked on the gage section to measure the percent elongation to failure (%EL) after the test is finished. All tensile tests were performed in displacement-control mode with a displacement rate of 0.0127 mm/s (i.e., 0.0005 in/s) to achieve a 0.001 mm/mm.s strain rate.

Additionally, each test was paused at a strain of 0.045 mm/mm to remove the extensometer to prevent any damage to it. The tests were subsequently continued until the final fracture. Finally, powder characteristics were quantified with a Freeman Technology (FT4) powder rheometer. Some rheological properties including powder compressibility, cohesion [17], and aeration energy (AE) were evaluated.

Results and Discussion

As seen in Fig. 3, the compressibility, and cohesion are illustrated. Compressibility is defined as the density change of the powder bulk as normal stress is applied [18]. Therefore, lower compressibility can show fewer void spaces within the powder bulk and a better packing ability [19]. As noted from Fig. 3(a), the compressibility has decreased by ~6% when reused 11 times. Therefore, it may be assumed that the reused powder consisted of a better and tighter particle arrangement, leaving smaller gas pockets among particles.

The lower compressibility of the 11x reused powder can be attributed to the reduction of very fine and coarse particles due to continuously reusing the powder [8]. Although finer particles can fill the empty spaces, they can also form agglomerates due to their higher interparticle frictional forces and leave empty spots in the powder bulk. Large particles also break down when they are passed through the filter. This observation was consistent with the particle sizes that they were measured. For instance, the mean particle size increased from 33.1 µm in the virgin powder to 34.7 µm in 6 times reused powder.

Overall, the variations in the PSD were negligible which can be attributed to the "top-up" method that was employed to reuse the powder, which was also manifested in the slight decrease in compressibility results. Cohesion was the other powder characteristic that was evaluated. Cohesion can indicate the resistance of powder to shearing, and less cohesion can represent a higher flowability [18].

Therefore, a smaller cohesion coefficient is desired in LB-PBF as it can lead to a more uniform powder bed with low mechanical interlocking (i.e., agglomeration) [19]. As seen in Fig. 3(b), cohesion slightly decreased when the powder was reused 5 times and then increased again after 11 times reuse, showing the presence of fewer fine particles and more uniformity of powder particles in the 5x reused powder. Upon investigation of the morphology of powder particles in Fig. 4, no particular change to particle morphology was seen in the 9x reused powder which was used for fabrication of the 10th batch (B10), In addition, the circularity was evaluated and there were negligible changes between the virgin (0.95 circularity) and 9x reused (0.94 circularity) powder batches.

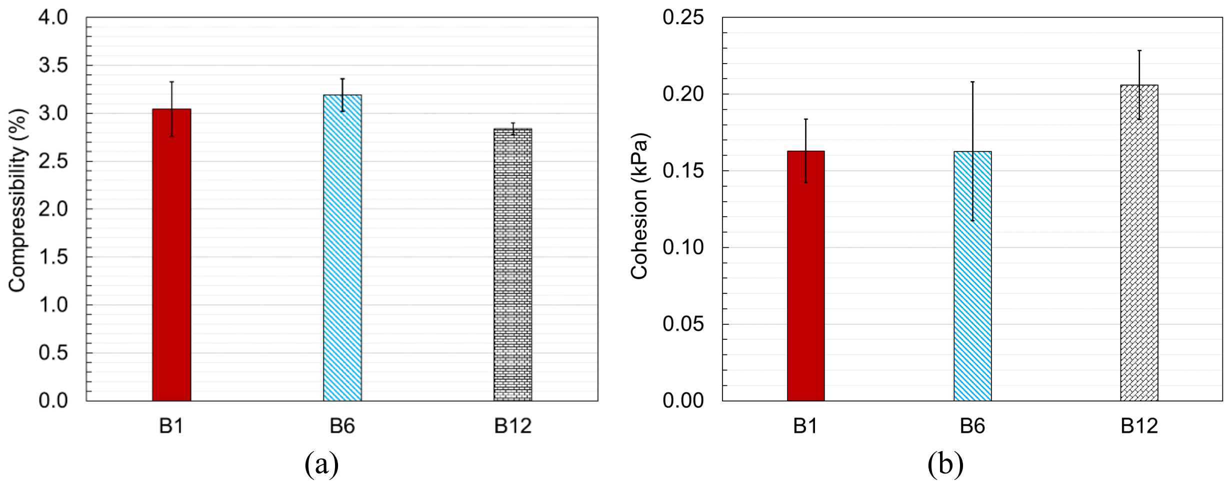
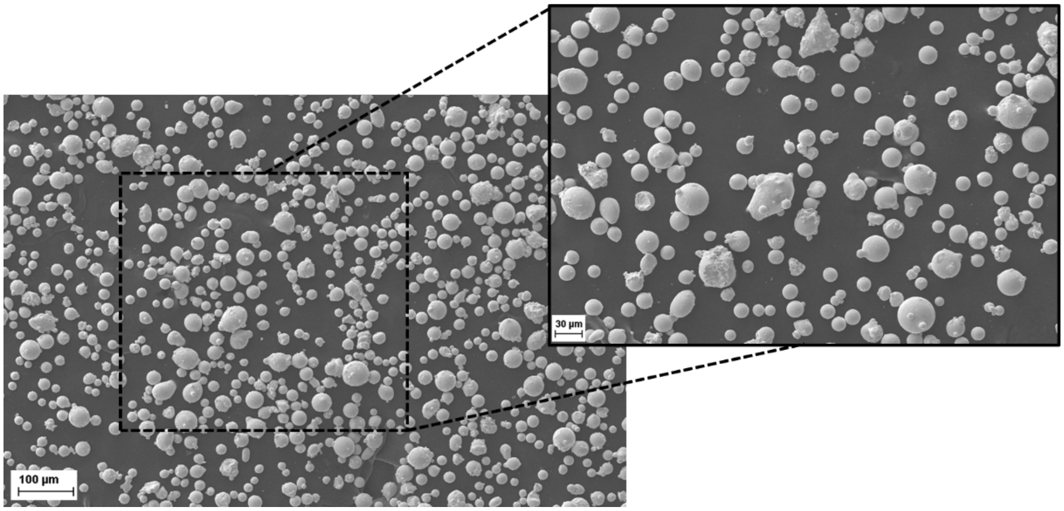
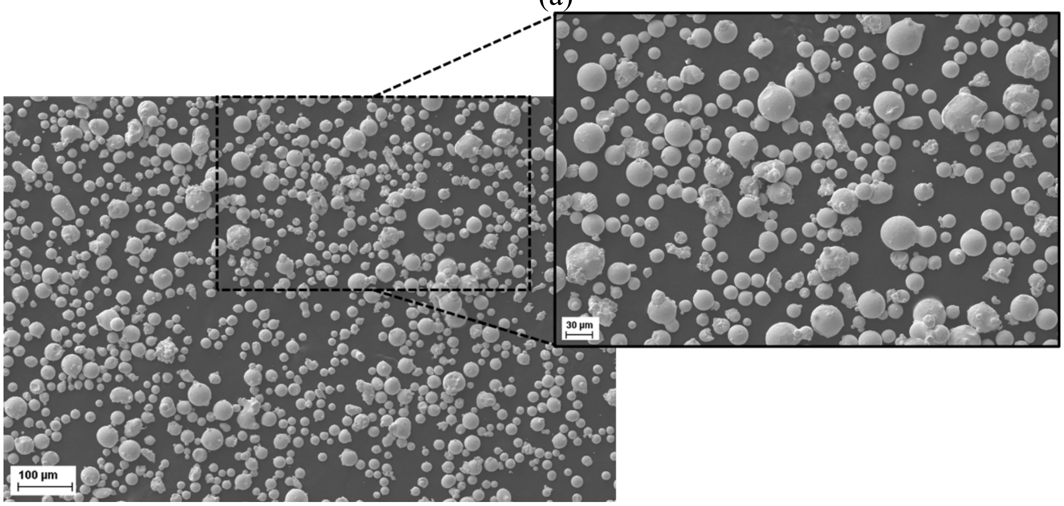


Fig. 3 IN718 powder rheological properties including (a) compressibility, and (b) cohesion for B1 (virgin powder), B6 (5x reused powder), and B12 (11x reused powder).

As seen in Fig. 5, tensile tests were performed and plotted for B1-B10 fabrication sets for the net-shaped tensile specimens (6 specimens per condition). No specific trend in yield strength (YS) and ultimate tensile strength (UTS) was found. The %EL, however, showed some improvement in the first few reuse iterations and it decreased afterward. The comparable tensile strength based on powder reuse can be attributed to the similar microstructure of tensile specimens as reported by Ref. [2,10,20] which is not usually affected by the powder reuse practice.



(a)



(b)

Fig. 4 Particle shape morphology for the (a) virgin (used for B1 fabrication) and (b) 9x reused powder (employed for B10 fabrication).

The differences in %EL, however, can be correlated with the defect content. It seems that there were fewer defects in the specimens fabricated with 5x reused powder (B6) as compared to B1 and B10. To verify these observations, the AE of the powder was evaluated and reported in Fig. 6. As seen in this figure, the AE is at the minimum for B6 as compared with B1 and B12. AE can represent the tendency of the powder to agglomerate. Typically, powder batches with lower AE values have less inclination to form agglomerates and particle interlocking [19].

Interestingly, these observations on AE conforms well with the results provided by Nandwana et al. in Ref. [7]. In their study, it was reported that IN718 is typically stable over a large number of cycles. The PSD was stated to slightly change which was also the case in the present article. In addition, negligible changes were reported in the chemical compositions with powder reuse. Although the results in Ref. [7] were only for 6 reuse cycles, it was seen that even after 18 times powder reuse, the compressibility of IN718 does not considerably change (see Fig. 3(a)).

It was also related that the only factor that might limit the powder reuse of IN718 can be the flowability [7]. Therefore, it is essential to ensure there is no severe mechanical interlocking nor agglomeration. It was noticed that the cohesion in B6 was lower than the B1 (manufactured from virgin powder) (see Fig. 3(b)). Consistent results were also noticed in Fig. 5 where a higher ductility was noted for the reused powder in B6 as compared to B1.

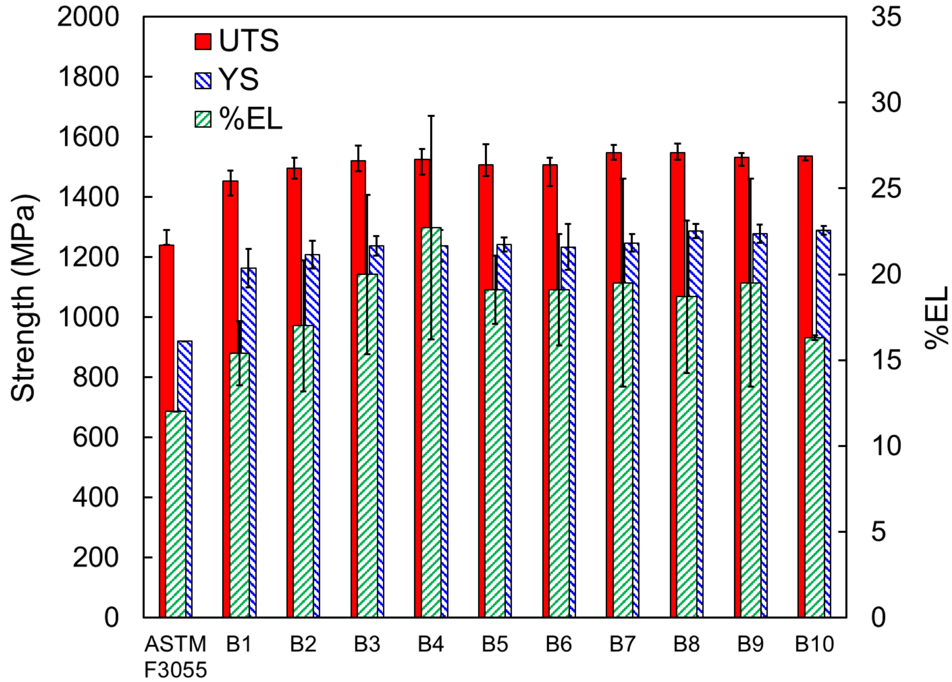


Fig. 5 Tensile properties including UTS, YS, and %EL for the LB-PBF IN718 specimens for different powder reuse iterations (B1-B10).

It needs to be specified that the present work will be continued to study fatigue performance. Generally, the fatigue failures in IN718 are less sensitive to the presence of defects [4], lessening the importance of some powder characteristics such as compressibility. As a result, it is important to understand which powder characteristics can play a major role to determine the recyclability of the material to be used in AM based on the load-bearing application.

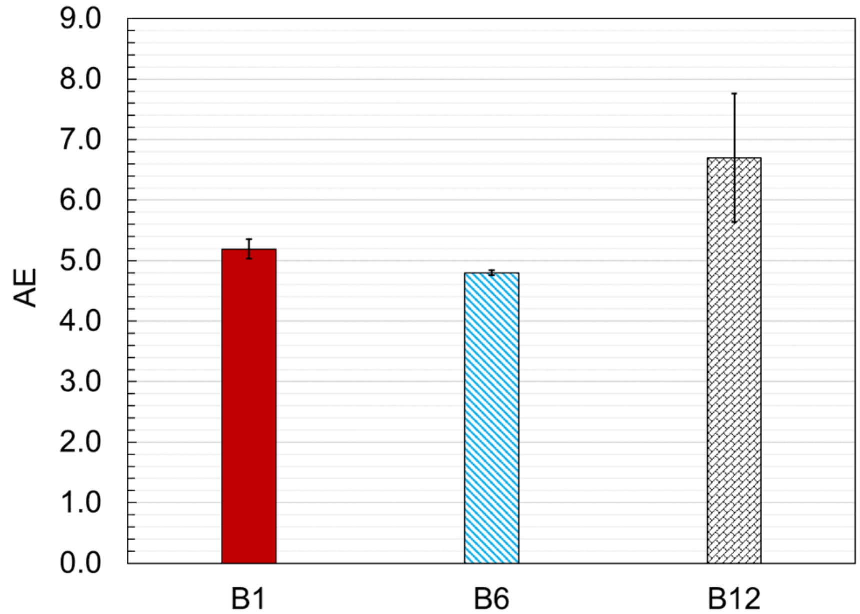


Fig. 6 AE values for IN718 powder in different reuse conditions. B1 implies virgin powder and B12 refers to the powder which was reused 11 times.

Conclusions

In this study, IN718 was reused 18 consecutive times in LB-PBF AM with the "top-up" procedure to investigate the effects of powder reuse on powder characteristics as well as tensile properties. Based on the experimental results and observations for the first 12 sets, it is concluded that:

- Based on the packing behavior obtained from compressibility results, 11 times reusing IN718 slightly improved the particle arrangement and lessened empty spaces within the powder bulk.
- Flowability was found to be an important parameter when IN718 is reused. Reusing IN718 improved the flowability at first, however, it decreased with further reusing the powder.
- No considerable difference was found in the morphology of the IN718 powder particles after reusing the powder multiple times and spherical particles remained almost intact.
- No difference in tensile strength including UTS and YS was observed as the microstructure was not affected by the powder reuse.
- Some differences were noted in the ductility of LB-PBF IN718 parts due to the powder reuse practice which was correlated with the defect content in parts affected by changes in powder flowability.

Acknowledgments

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