Solid Freeform Fabrication 2018: Proceedings of the 29th Annual International Solid Freeform Fabrication Symposium – An Additive Manufacturing Conference
Reviewed Paper

Effect of Powder Degradation on the Fatigue Behavior of Additively Manufactured As-Built Ti-6Al-4V

Patricio E. Carrion[1, 2], Arash Soltani-Tehrani[1, 2], Scott M. Thompson[1, 2], Nima Shamsaei[1, 2, *]

[1]*National Center for Additive Manufacturing Excellence (NCAME), Auburn University, Auburn, AL 36849, USA*

[2]*Department of Mechanical Engineering, Auburn University, Auburn, AL 36849, USA*

* Corresponding author: shamsaei@auburn.edu

Abstract

Additive manufacturing (AM) technology has enabled many industries to generate functional parts with an increased level of complexity via a layer-by-layer melting. In laser-powder bed fusion (L-PBF), the most commonly used AM process for metals, powder is often recycled due to its high cost. However, there is no comprehensive study on how recycling powder affects its rheological properties, and the mechanical and fatigue behavior of the final manufactured part. In this study, a comparison of new and used Ti-6Al-4V powder characteristics was made. The comparison includes morphology, size distribution, as well as monotonic tensile and fatigue behavior of fabricated specimens. Conclusions and suggestions on powder recycling are made. Results indicate that the powder particle size distribution (PSD) becomes narrower and the morphology of the particles change with recycling. However, no comparable effect was observed on the monotonic tensile and fatigue behavior of the AM as-built Ti-6Al-4V specimens.

KEYWORDS: Laser-Powder Bed Fusion, Ti-6Al-4V, Fatigue Life, Powder Degradation, Powder Recycling.

Introduction

Additive manufacturing (AM) has provided the opportunity to generate complex parts in an integrated unit. This has helped eliminate the need for processes to join parts including brazing and welding. The two most common AM methods for metals are direct energy deposition (DED) and powder bed fusion (PBF). In the PBF process, unlike subtractive conventional manufacturing, parts are fabricated in a layer-wise system via metallic powders. Laser powder bed fusion (L-PBF), a type of PBF process, uses a laser as the energy source. In this process, the metal powder is spread over the substrate, and the laser initiates a continuous, selective fusing process. After the desired part is fabricated, the remaining powder can either be discarded or recycled. If recycled, the powder from the collector bin is sieved to remove spatter or debris, and then mixed with any remaining or unused powder in the feed bin. Due to the high cost of metallic powders, specifically Ti-6Al-4V alloy, powder recycling can be a viable option for cost reduction. Few studies have been conducted on the effects of powder recycling on final mechanical properties, and there is no specific procedure on how to recycle nor quality control.

Powder is defined as a finely divided solid which is smaller than 1 mm in its maximum dimension [1]. A particle is the smallest unit of the powder which cannot be subdivided. Powder properties such as size distribution, flowability, and chemical content have been shown to affect the mechanical properties of the AM fabricated parts [2–6]. For instance, it has been observed that a 0.15% water amount added to a powder can result in agglomeration in metallic powders and consequently powder flow is affected [1].

Jacob *et al.* [2] studied the mechanical properties, including tensile and hardness, on 11 similar L-PBF builds using nitrogen gas atomized 17-4 PH stainless steel. It was observed that the recycled powder had a better flow time, increased powder bed density and apparent density. However, no comparable change was observed in powder size distribution (PSD), and shape. Also, the martensitic-ferritic phase was slightly increased due to fabrication and recycling in the nitrogen atmosphere. The parts fabricated from recycled powder showed the same mechanical properties as the parts from new powder.

Liu *et al*. [3] studied two stainless steel 316L batches from LPW Technology Ltd and Sandvik Osprey Ltd with PSDs of, respectively, 15-45 µm and 0-45 µm, to study the effects of PSD on the processing parameters optimization in L-PBF and final part properties. It was observed that powders with narrower range of PSD have better flowability – leading to parts with higher ultimate tensile strength (UTS) and increased hardness. It was seen that a wider PSD leads to higher densities since finer particles can fill in the vacant areas between larger particles. It was also found that the powder with a wider PSD had a smoother surface finish. However, wider PSDs were shown to adversely affect agglomeration due to the higher friction between particles.

Seyda *et al*. [4] investigated the effects of aging on Ti-6Al-4V powder. Part properties including density, porosity, hardness, and surface quality were studied to understand the effects of aging. It was found that the particles coarsened and flowability increased due to the apparent density increase and PSD enlargement after recycling multiple times. No significant effect was seen on the porosity, density, surface roughness, hardness and mechanical strength for the aged manufactured specimens. However, less pore locations were found in the bulk material of the aged powder with increased pore sizes. Also, there was an increasing trend in the surface roughness values with aging.

O'Leary *et al.* [5] observed that the surfaces of particles roughened after recycling Ti-6Al-4V powder five times. Also, it was seen that the number of finer particles (< 15 µm) decreased while larger particles (> 45 µm) increased. No significant change in amount of oxygen and nitrogen was seen. However, a comparable amount of oxygen absorption was observed in the parts while the fabrication atmosphere was argon.

Tang *et al.* [6] studied the effects of powder recycling on the Ti-6Al-4V powder quality and parts fabricated via selective electron beam melting (SEBM). It was observed that the oxygen content slightly increased after recycling 21 times while the Al and V contents were almost constant with a negligible decrease. It was also seen that the powder particles became less spherical. No significant effect was observed on tensile properties of produced specimens.

The aforementioned studies have demonstrated changes in powder characteristics due to recycling and the subsequent effects on the mechanical properties of fabricated parts. However, these parts are often subjected to dynamic loading; therefore, there is a further need to evaluate the fatigue performance of parts fabricated using recycled (used) powder. By incorporating unique powder/part characterization methods, this study quantifies the rheological properties of continually recycled Ti-6Al-4V powder while focusing on the PSD and shape. The AM specimens are subjected to quasi-static tensile and fatigue testing, the results are then correlated to the changing powder quality.

Experimental Program

Powder Characterization

The material selected for this study was gas-atomized Ti-6Al-4V Grade 23 powder with a particle size of 15-45 μm, manufactured by LPW Technology. The powder was in two conditions, new (i.e. virgin) and used (recycled more than 14 times). It is important to mention that the recycling procedure involved sieving the used powder with an 80-μm mesh size sieve after each print, and if needed, new powder was added in order to have a sufficient amount to complete the next part/component fabrication. Particle size distribution (PSD) analysis was conducted via a Malvern Mastersizer 3000 Hydro EV laser diffraction particle size analyzer and based on ASTM E2651-13 [7]. The powder used for size analysis was sampled according to ASTM B215-15 by taking equal increments from different depths of the container [8].

Material and Specimen Fabrication

L-PBF Ti6Al-4V specimens were fabricated using an EOS M290 system in two separate batches, one with new and one with used powder. EOS default process parameters were used, including 280 W laser power, 1200 mm/s travel speed, 100 mm stripe width, 140 μm hatch spacing, 67° hatch rotation, and a 30 μm layer thickness. The specimens were fabricated vertically (90° from the build plate) with argon as the shielding gas. The specimen geometry was based on ASTM E606 standard [9], which includes a uniform gage section with a 3.75 mm gage diameter, and a 15 mm gage length. After fabrication, specimens were annealed at 704 °C in an argon atmosphere for one hour, and then air cooled to the room temperature to remove residual stresses. Specimens were then tested without further post-processing and considered to be in the "as-built, annealed" condition. It is important to emphasize that the only variable was the Ti-6Al-4V powder (i.e. new and used), as great effort was employed to keep all other variables such as specimen layout on the build plate, shielding gas, etc., consistent; to only investigate the powder recycling effects on the mechanical properties and fatigue performance.

Testing Procedures

Strain-controlled quasi-static tensile and fatigue tests were conducted using an MTS 810 servohydraulic system, and an MTS extensometer with a 10 mm gage length was used to record strain. Tensile tests were performed at a 0.001 s^{-1} strain rate on two specimens fabricated with the new powder, and two with the used powder. Fully-reversed (R_ε = -1) strain-controlled fatigue tests were conducted according to ASTM E606 standard [9]. The strain amplitudes values selected for this study were 0.001, 0.003, and 0.005 mm/mm. The test frequency was adjusted for each strain amplitude to eliminate strain rate effects on the fatigue behavior. Experiments that reached over $4\text{x}10^6$ reversals were determined to be a run-out. Three fatigue tests for each strain amplitude were conducted for verification of results. Specimens from the same location on the build plate, but from different powder batches (new and used), were tested with same parameters.

Experimental Results and Analysis

Powder Recycling Comparison

The particle size distribution (PSD) of both powder samples are presented in Fig. 1. It may be seen that there is a difference in PSD between the new and used powders. The size distribution becomes narrower for the used powder. This is explained by the gap between the wiper/recoater and the substrate being smaller than 50 µm, hence finer particles are spread on the build plate and participate in the melting and solidification processes during manufacture. However, particles greater than 50 µm will be transferred to the collector bin. After completion of each print, the powder is sieved and mixed with the remainder of the powder in the feed bin. Repeating this procedure will lead to narrower PSDs because finer particles are melted and solidified, and larger particles get detached when passing through the 80 µm mesh size screen. The average diameter of used powder particles was slightly less than the new powder particles.

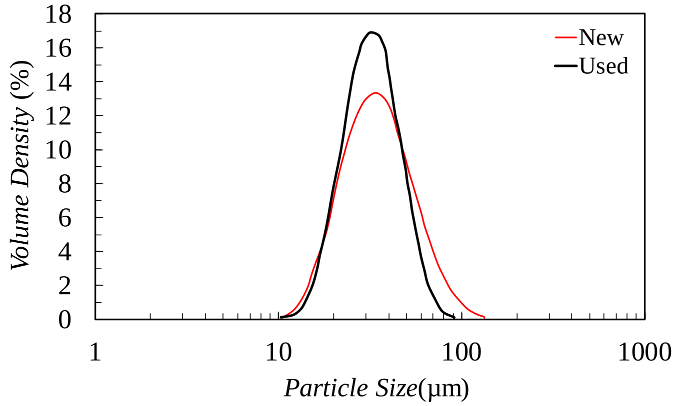
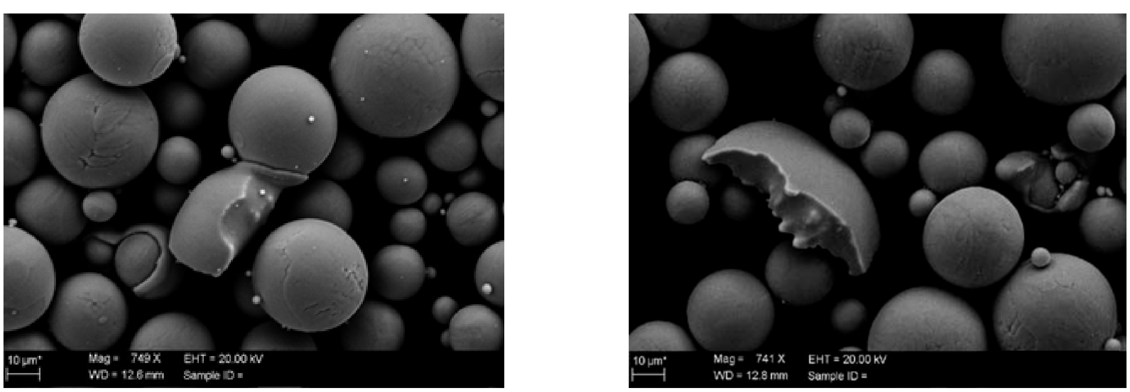


Fig. 1. Particle size distribution (PSD) comparison of the new and used Ti-6Al-4V powders.

Representative powder morphology, obtained via scanning electron microscopy (SEM), is shown in Fig. 2. Results indicate that fewer "satellites", smaller particles attached to the larger particles, were observed in the used powder when compared to the new one. These particles may contribute to defect or pore formation since they leave void spaces by spattering from the deposited layer when the laser scans the selected area. It was also observed that most particles are still spherical after recycling the powder; however, similar irregular shape particles were observed in both new and used powder.



(a) (b)

Fig. 2. Particle morphologies obtained via SEM for (a) new and (b) used Ti-6Al-4V powder.

Tensile Deformation

Quasi-static tensile stress-strain curves of L-PBF Ti-6Al-4V specimens fabricated with new and used powders are presented and compared to wrought Ti-6Al-4V [10] in Fig. 3. It may be seen that the yield, and ultimate tensile strength of L-PBF Ti-6Al-4V are higher (10%, and 5% respectively) than the wrought counterpart, but the elongation to failure is comparable. It is also shown that the ultimate tensile strength of the new powder specimens is 5% higher than the used powder, while the yield strength and elongation to failure differences were negligible. These results differ from other studies such as Tang *et al.* [6], where it was determined that for Ti-6Al-4V parts fabricated via SEBM, the yield and ultimate tensile strength increased with increased powder usage due to higher oxygen content. These results were also determined to be of negligible impact on the overall mechanical properties. Jacob *et al.* [2] however, presented findings for 17-4 PH stainless steel, where it was concluded that the effects of powder recycling on the mechanical properties are negligible. From the quasi-static tensile experiments, it can be concluded that effects of powder recycling do not have significant effects on the mechanical properties of L-PBF Ti-6Al-4V.

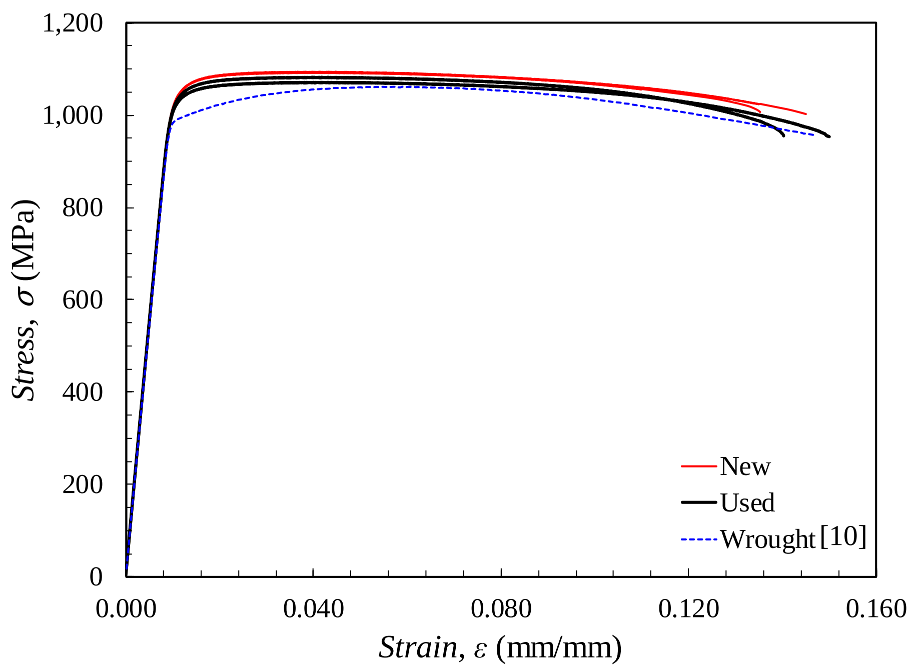


Fig. 3. Tensile deformation comparison of L-PBF specimens fabricated with new and used Ti-6Al-4V powder compared to wrought Ti-6Al-4V [10].

Fatigue Behavior

Figure 4 presents a semi-log plot of the strain amplitude versus reversals to failure of L-PBF Ti-6Al-4V specimens fabricated with new and used powder. It may be seen that there are no significant differences in fatigue life across the three strain amplitudes tested (0.001, 0.003, and 0.005 mm/mm). Horizontal arrows indicate run-outs (i.e. a fatigue life over $4x10^6$ reversals). At a strain amplitude of 0.001 mm/mm, one of the specimens fabricated with the new powder resulted in a run-out while for the used powder there were two. These data points are denoted by the “x3” in in Fig. 4. Minimum differences in fatigue lives could be attributed to the as-built surface roughness condition, which has been found to have significant influence on the fatigue performance of AM parts [11]. It provides the opportunity for failure to occur from surface defects which typically have higher stress concentration than internal defects. In conclusion, the fatigue performance of as-built L-PBF Ti-6Al-4V specimens is not affected by powder recycling as substantial differences or scatter in data were not observed.

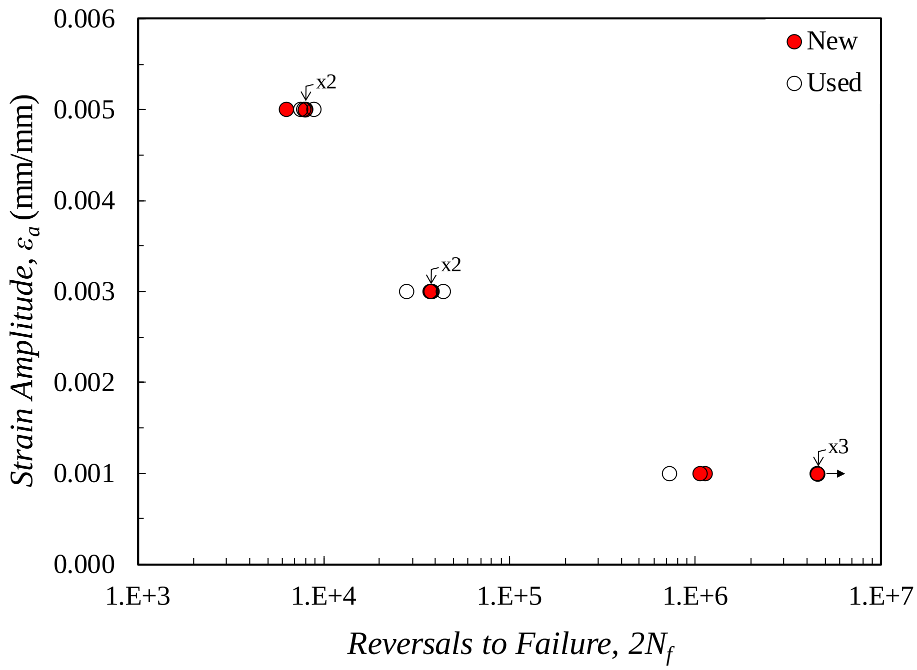


Fig. 4. Strain-life comparison of L-PBF specimens fabricated with new and used Ti-6Al-4V powder.

Conclusions

In this study, the change in powder particle size distribution due to recycling and its effects on the tensile deformation and fatigue behavior of L-PBF Ti-6Al-4V under strain-controlled fully-reversed (R_ε = -1) fatigue loading was investigated. The following is a summary of the main conclusions and novel findings:

1. Fewer satellite/fine particles were observed for the used powder and the particles were near-spherical with fewer irregularly-shaped particles after recycling.
2. The particle size distribution became narrower after recycling due to finer particles being melted and solidified while the larger particles are moved to the collector by the wiper.
3. Larger particles (>80 µm) available in the new powder get discarded due to the sieving process before each fabrication.
4. Powder recycling has negligible effects on the mechanical behavior and fatigue performance of the manufactured as-built L-PBF Ti-6Al-4V specimens. This is explained by the surface roughness having a greater effect in fatigue.

Acknowledgements

This research was partially funded by the National Science Foundation (NSF) under grant No. 1657195

References

[1] German RM. Powder metallurgy science. Met Powder Ind Fed 105 Coll Rd E Princet N J 08540 U 1984 279 1984.

[2] Jacob G, Brown CU, Donmez MA, Watson SS, Slotwinski J. Effects of powder recycling on stainless steel powder and built material properties in metal powder bed fusion processes. NIST Adv Manuf Ser 2017.

[3] Liu B, Wildman R, Tuck C, Ashcroft I, Hague R. Investigation the effect of particle size distribution on processing parameters optimisation in selective laser melting process. Int Solid Free Fabr Symp Addit Manuf Conf 2011:227–38.

[4] Seyda V, Kaufmann N, Emmelmann C. Investigation of Aging Processes of Ti-6Al-4 v Powder Material in Laser Melting. Phys Procedia 2012;39:425–31.

[5] O'Leary R, Setchi R, Prickett P, Hankins G, Jones N. An Investigation into the Recycling of Ti-6Al-4V Powder Used Within SLM to Improve Sustainability. SDM2015 2nd Int Conf Sustain Des Manuf 2015 2015:14–7.

[6] Tang HP, Qian M, Liu N, Zhang XZ, Yang GY, Wang J. Effect of Powder Reuse Times on Additive Manufacturing of Ti-6Al-4V by Selective Electron Beam Melting. JOM 2015;67:555–63.

[7] Powders M, Compounds R, Air U, Methods S, Media S, Characterization S, et al. Standard Guide for Powder Particle Size Analysis 2016:1–7.

[8] International A. Standard Practices for Sampling Metal Powders. ASTM Stand B215 2010;10:9–11.

[9] ASTM E606/E606M-12 Standard Test Method for Strain-Controlled Fatigue Testing, ASTM International, West Conshohocken, PA, 2012.

[10] Carrion PE, Shamsaei N, Daniewicz SR, Moser RD. Fatigue behavior of Ti-6Al-4V ELI including mean stress effects. Int J Fatigue 2017;99:87–100.

[11] Yadollahi A, Shamsaei N. Additive manufacturing of fatigue resistant materials: Challenges and opportunities. Int J Fatigue 2017;98:14–31.