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Reviewed Paper

Effect of Powder Reuse on Microstructural and Fatigue Properties of Ti-6Al-4V Fabricated via Directed Energy Deposition

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Abstract

In metal additive manufacturing (AM) processes, due to the high cost of metal powder, it is common to reuse the collected powder from the build envelope for future builds. Powder reuse may adversely affect the powder characteristics, including the flowability, size distribution, chemical composition, resultant microstructural, and consequently, mechanical properties of the fabricated parts. This study aims to investigate the effect of powder reuse on the microstructural features and fatigue performance of Ti-6Al-4V specimens fabricated using a directed energy deposition (DED) process. Characteristics of reused powder particles, such as the size distribution and chemical composition, were evaluated and compared with that of virgin powder. Microstructural features and characteristics of the process-induced defects were examined using scanning electron microscopy and x-ray computed tomography, respectively. Fatigue performance of the specimens fabricated using reused powder was evaluated and compared to their control counterparts, fabricated using virgin powder.

Keywords: Additive manufacturing; Powder recycling; Virgin powder; Powder flowability; Powder size distribution

1. Introduction

The domain of metal additive manufacturing (AM) contains different techniques that join materials in a layer-wise manner, providing an opportunity to produce complex-shaped objects directly from 3D model data [1,2]. Laser-direct energy deposition (LDED), also known as laser-engineered net shaping (LENS®), is a powder-based metal AM process commonly used for fabricating metallic parts [3]. In this technique, metal powder is blown into the melt pool and consecutive layers are fabricated along the build direction [4]. Theoretically, blown powder particles should be delivered to the melt pool zone, however, some of the dispensed powder particles, experiencing laser heating, may not get captured in the melt pool and hit the cold surface of the build [5]. Low deposition efficiency of the LDED process results in large amounts of unused powders, which is economically and environmentally expensive [1,6]. Therefore, the leftover powder is usually reused/recycled for future builds.

Experiencing laser heating, the leftover powder particles may experience changes in their characteristics, such as powder flowability, size distribution, chemical composition, etc. [7,8]. Changes in the characteristics of powder may affect the microstructural features and formation of process-induced defects, i.e., gas pores and lack-of-fusion (LOF) defects, leading to variations in the mechanical performance of the fabricated parts, particularly their fatigue performance. Furthermore, due to the high reactivity of some materials like titanium alloys with oxygen at high temperatures, oxygen contamination during the powder recycling process can be detrimental to the quality and structural integrity of the part [9]. Therefore, there is a need to understand and correlate the effects of powder reuse/recycling on the microstructural and mechanical performance of the fabricated part.

Studies on Inconel 718 [5,10], 316L stainless steel (SS) [11], and Ti-6Al-4V [9] alloys fabricated via LDED process showed that powder recycling causes little variations in flowability [5,11], discoloration in microstructures [11] or powder particles [10], but no significant changes in chemical compositions of powder [5,9–11], oxygen content [5,9], and microhardness values [5,10]. While some studies reported an increase in particle size distribution due to powder reuse [9–11], an opposite trend was reported for particle size distribution by Carroll et al. [5]. Carroll et al. reported a marginal decrease in particle size as a result of reusing the Inconel 718 powder [5]. Terrassa et al. reported that recycling of powder had no significant effect on compression yield stress and tensile properties of 316L stainless steel (SS) specimens [11].

A review of the published literature shows that powder recycling is a trending topic in powder bed fusion (PBF) AM systems. Several research efforts have been done aiming to understand how powder recycling influences the characteristics of PBF parts [12–20]. However, there are only a few research efforts focusing on how the reused powder feedstock influences the properties of parts fabricated via LDED process [5,9–11,21]. As a result, there is very limited knowledge on how reused powder may affect the structural integrity of LDED parts. Although results of current research on powder recycling in PBF processes may provide useful insights, observations and outcomes of these studies cannot be directly applied to the parts fabricated via DED process, considering the fact that the characteristics of the build process in PBF systems being different from that of DED systems. To fill this gap, the present work aims to investigate the effect of powder reuse on the characteristics of the recycled powder (i.e., oxygen content, chemical composition, particle size distribution, and powder flowability) as well as the microstructure and fatigue performance of LDED Ti-6Al-4V.

2. Experimental Procedure

2.1. Specimen fabrication and preparation

Plasma atomized Ti-6Al-4V powder with a particle size distribution of 45-90 μm (LPW technology) was utilized in this study to fabricate samples via an OPTOMEC LENS 750 system equipped with a 1 kW laser source (Nd:YAG). As-received (i.e., virgin) and recycled (i.e., un-melted powder particles recovered by previous depositions) powders were used as starting feedstock material to fabricate two groups of fatigue specimens: (i) virgin and (ii) recycled. The recycled powder was obtained through nine deposition cycles. To remove any under/over-sized particles after each deposition, the powder collected from the build stage was sieved, and the agglomerates larger than 150 μm, and particles smaller than 45 μm were removed. Before starting the deposition in each cycle, the build plate was prepared by sandblasting and cleaning with soap and alcohol. Figure 1 shows the flowchart of the procedure utilized for the collection of recycled powder.

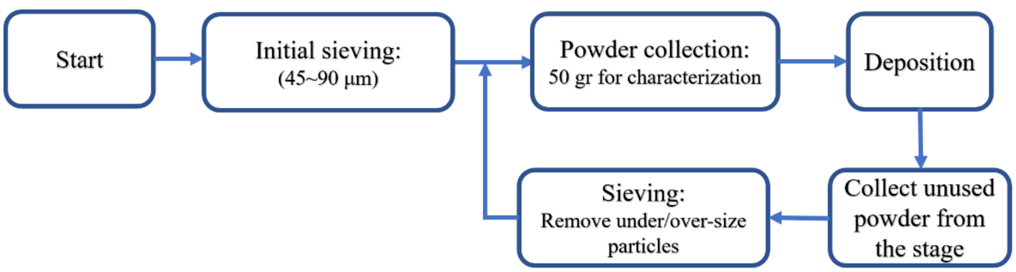
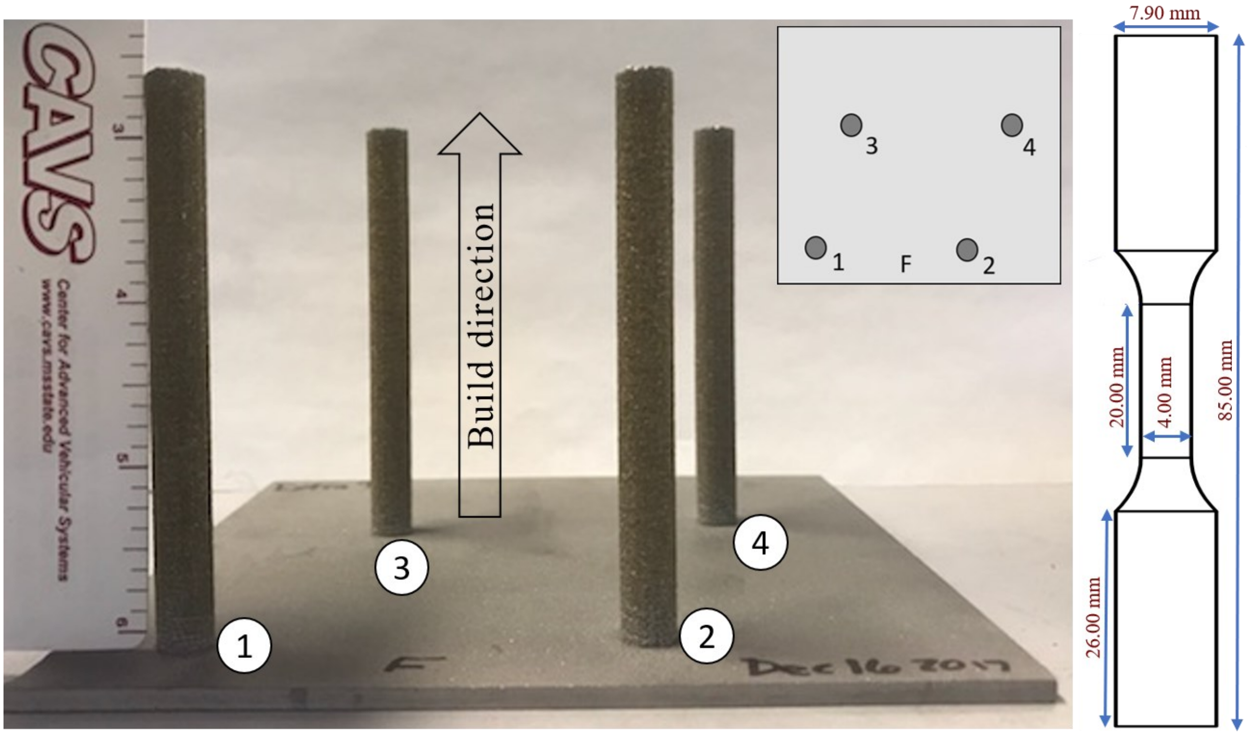


Figure 1. Flowchart of the building procedure and collecting recycled powder.

Process parameters utilized for the fabrication of Ti-6Al-4V samples, based on previous operating experience with the alloy [22,23], are given in Table 1. Vertically oriented cylindrical coupons with 85.0 mm in height and 8.0 mm in diameter were fabricated using virgin and recycled powders. Arrangement of the cylindrical coupons on the build plate is shown in Figure 2(a). All samples were annealed at 760 °C for 1 hour in an inert Argon atmosphere, followed by air cooling. This procedure allows for stress relief and decomposition of the martensitic phase [24]. Cylindrical rods were machined to the dogbone shape, with a reduced diameter of 4.0 mm at the gage section, as shown in Figure 2(b), for uniaxial fatigue testing in accordance with ASTM E466 [25].

Table 1. Process parameters utilized in this study for fabrication of the Ti-6Al-4V samples.

Process Parameters	
Laser power (W)	350
Travel speed (mm/min)	1016
Powder flow rate (rpm)	2.5
Hatch distance (μm)	508
Hatch rotation (degree)	120
Scan pattern	Zig-zag
Layer thickness (μm)	508



(a) (b)

Figure 2. Arrangement of the cylindrical coupons on the build plate.

2.2. Material characterization

In order to evaluate the powder characteristics, including the chemical composition, particle size distribution, and flowability, a 50 g sample of powder was collected from the virgin powder and recycled powder. Powder morphology, microstructural features, and process-induced defects were examined using scanning electron microscopy (SEM). Energy dispersive X-ray analysis (EDAX) was used to identify the elemental composition of powder samples. The oxygen content of the powder samples was analyzed using a LECO ONH836 elemental analyzer. Particle size distribution of powder samples was measured by a laser analyzer (Horiba LA 950). The flowability of powders was measured using a Hall flowmeter according to the ASTM B213 [26]. In order to evaluate the formation of any possible process-induced defects, fatigue specimens were scanned using an X-ray computed tomography (XCT) system. To improve scanning resolution, only the gage section of specimens and each specimen separately was scanned. Uniaxial fully-reversed (R = -1) fatigue tests were performed under the load-controlled mode using a servo-hydraulic load frame at room temperature. Fatigue tests were conducted using a sinusoidal loading waveform until failure occurred or 10^7 cycles were achieved, in which the test was considered to be a ‘run-out’.

3. Results and Discussion

3.1. Powder characterization

Results of oxygen content analysis revealed a slight increase in the concentration of the oxygen for the recycled powder as compared to their virgin counterparts. As shown in Figure 3, powder recycling caused 18% increase in the amount of oxygen, as the oxygen content from 0.076

wt.% in the virgin powder raised to 0.089 wt.% in the recycled powder. Although the increase in the amount of oxygen was noticeable, the overall oxygen content did not exceed the allowable oxygen content (i.e., 0.2 wt.%) for the Ti-6Al-4V, specified by the ASTM F2924-14 standard [27]. The EDAX analysis did not show significant differences in the elemental composition of titanium between virgin and recycled powders, as shown in Figure 4. However, due to powder recycling, a noticeable loss in aluminum (14%) and a rise in vanadium (21%) were observed. Our observation regarding the increase in vanadium concentration is in contrary to what was reported by Rousseau et al. [9], in which a loss of vanadium in the reuse powders was observed.

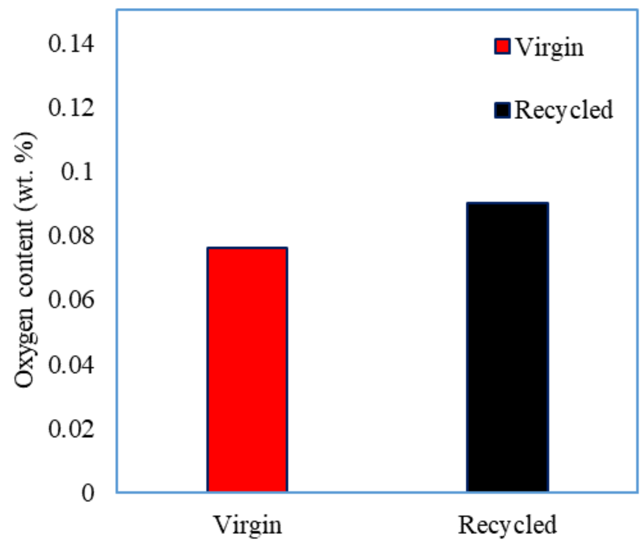


Figure 3. Oxygen content in the virgin and recycled powders.

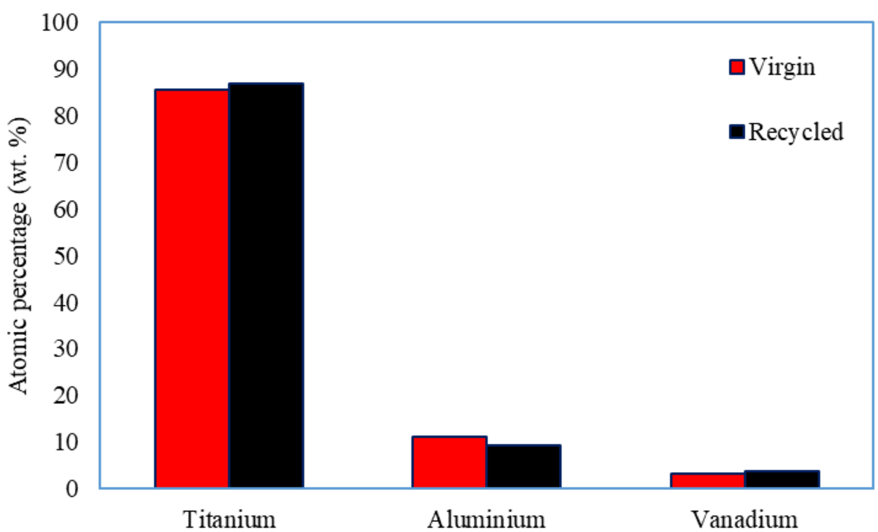


Figure 4. Elemental composition of the virgin and recycled powders (atomic percentage of titanium, aluminum, and vanadium) obtained by EDAX analysis.

In order to observe the changes in the powder particle morphology after recycling, the virgin and recycled powder samples were examined using SEM. The SEM images of particle morphology of the virgin powder and recycled powder are shown in Figures 5(a) and (b), respectively. Observations indicated that the recycling process caused deformation in the shape of the particles and increase in the number of satellite particles sticking to the surface of larger particles. The particle shape and the presence of satellite powder will seriously affect the flowability and other properties of the powder. The presence of satellite powder particles was noticeably increased after powder recycling, as seen by comparing Figure 5(a) with (b). The formation of satellite particles was most likely due to particle collisions during the deposition process. In addition, some powder particles were partially melted and stacked to other powder particles during the deposition process, forming larger agglomerated particles, as can be seen in Figure 5(b).

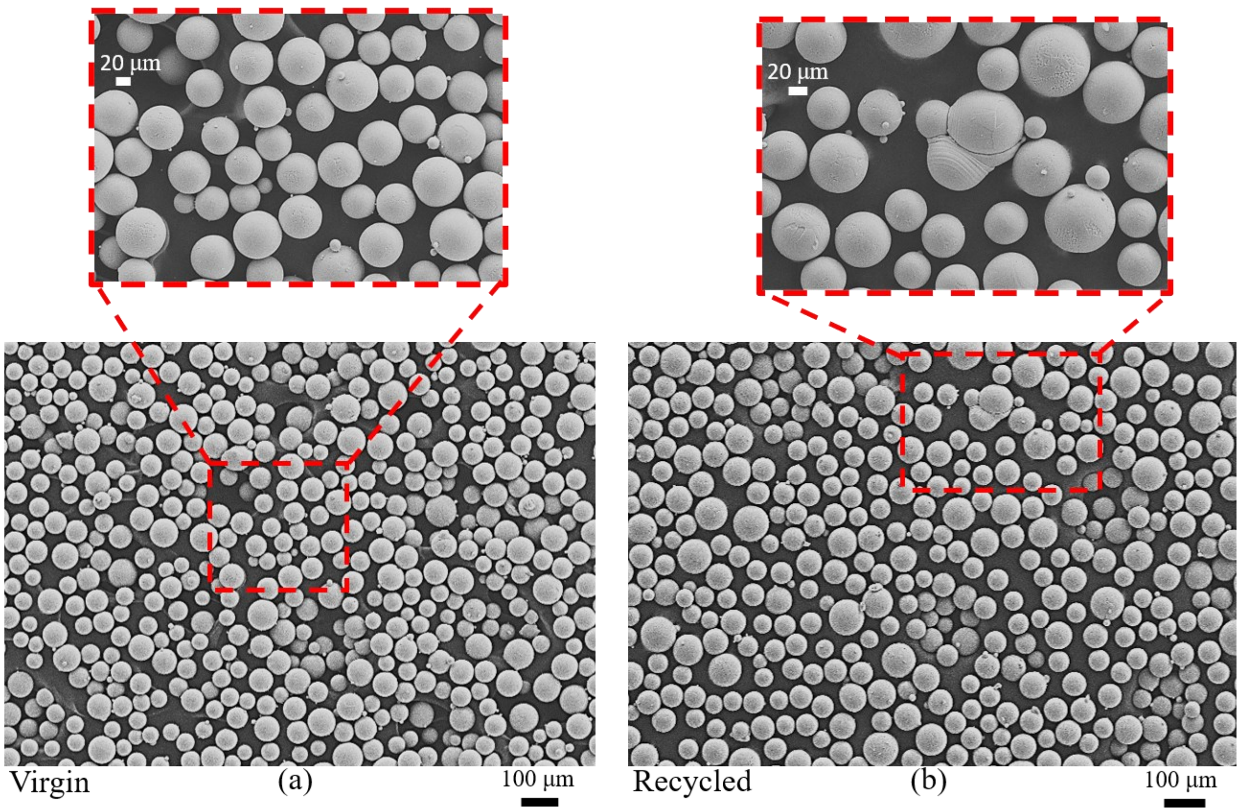


Figure 5. SEM images of the (a) virgin powder and (b) recycled powder.

The size distribution for the virgin and recycled powder is presented in Figure 6. As can be seen, size distribution of the recycled powder is skewed as compared to the virgin powder. In addition, the mean size of particles after recycling has noticeably increased, agreeing with the results reported by Renderos et al. [10]. Particle size distribution can influence the powder flow rate, which must be controlled for the quality of the fabricated parts [11]. Despite the noticeable differences observed in the particle size distribution and morphology of virgin and recycled powders, results from the flowability analysis revealed a slight difference in the flowability of recycled powders compared with virgin counterparts. Table 2 presents the flow rates of the virgin and recycled powders measured by the Hall flowmeter according to ASTM B213 [26]. As can be seen, the flow time of recycled powder is slightly higher as compared to virgin powder.

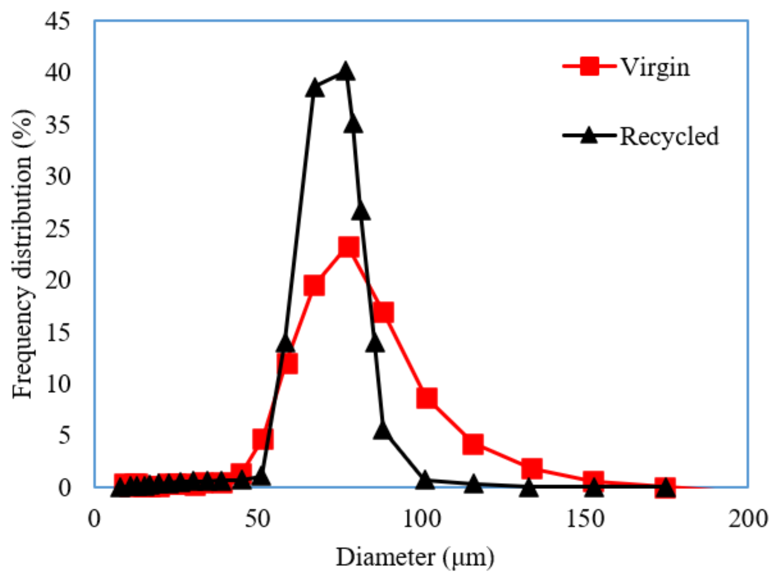


Figure 6. Particle size distribution of the virgin and recycled powders.

Table 2. Flow rates of the virgin and recycled powders, measured by the Hall flowmeter according to ASTM B213 [26].

	Virgin Powder	Recycled Powder
Elapsed time (sec)	20.23	21.88

3.2. Microstructural characterization

The SEM micrographs of the samples fabricated using virgin and recycled powders are presented in Figure 7. No significant differences in the grain size and morphology were observed, most likely due to the fact that both groups underwent the annealing treatment at 760 °C for 1 hour. A discoloration was observed on recycled samples, similar to the observation reported by Renderos et al. and Terrassa et al. [10,11].

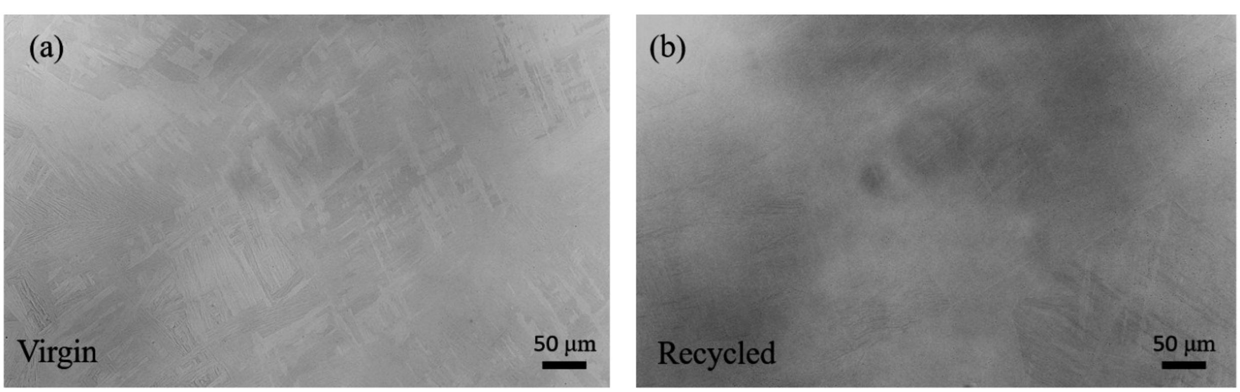


Figure 7. SEM micrographs of the samples fabricated using (a) virgin powder and (b) recycled powder.

Results from the XCT analysis, including the number of defects, largest volume of defects, average volume of defects and total volume of defects, are presented in Figure 8. Each value was obtained by getting average of all specimens in their category. For example, the largest defect volume for the virgin category represents the average of largest defect volume detected by XCT in the specimens fabricated via virgin powder. As can be seen in Figure 8(a), the number of defects in specimens fabricated using recycled powder is significantly higher as compared to their virgin counterparts, i.e., fabricated using virgin powder. This can be attributed to the formation of process-induced defects, i.e., gas pores and lack of fusion (LOF) defects, resulting from changes in characteristics of powder during recycling. As can be seen in Figures 8(b) and (c), the largest and average volume of detected defects in the specimens fabricated using recycled powder is almost doubled as compared to their virgin counterparts. Comparing the increase observed in the number of defects with the average volume of defects in the recycled specimens indicates that the huge rise in the total volume of defects should be more attributed to the increase in the number of defects. Although the microstructure observations showed no notable differences, the XCT results revealed a significant difference in the level of process-induced defects between the specimens fabricated using virgin powder and recycled powder.

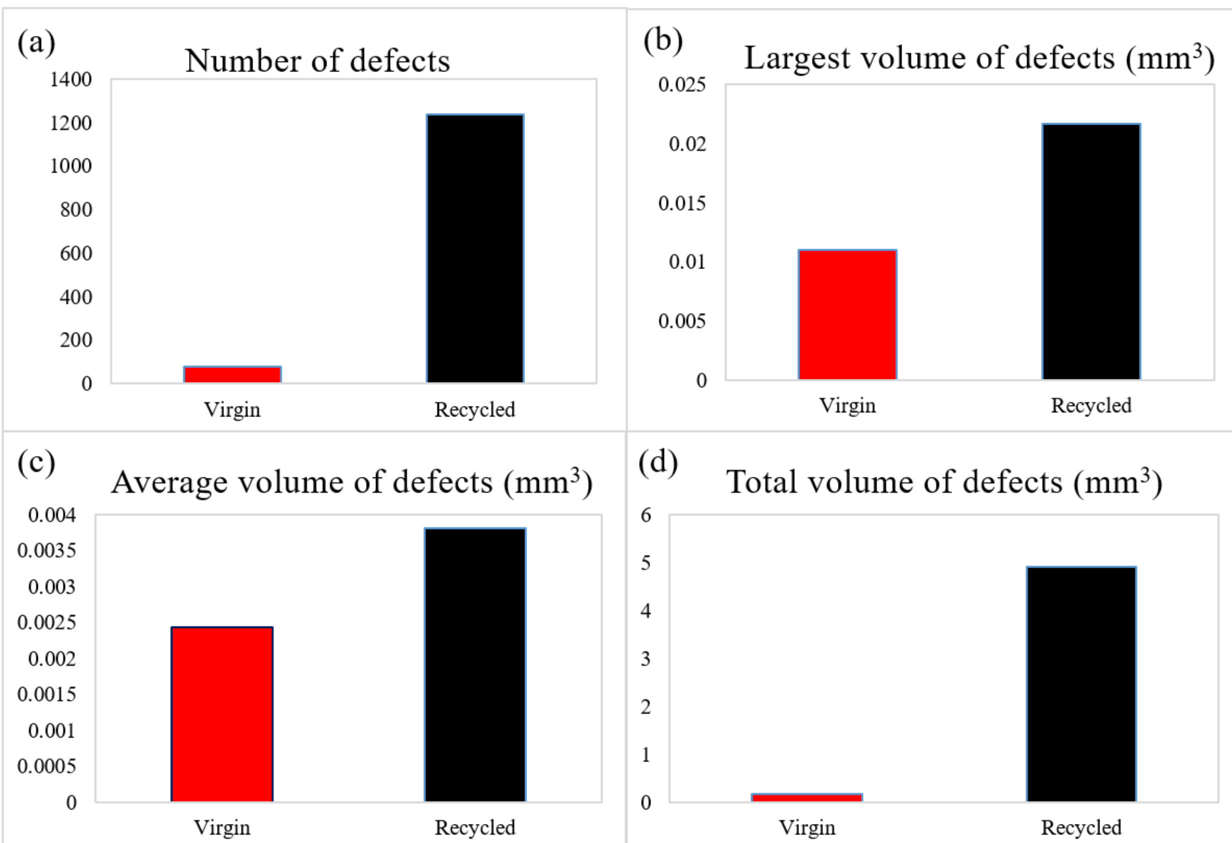


Figure 8. XCT analysis of defects for the specimens fabricated using virgin and recycled powders: (a) number of detected defects, (b) largest defect volume, (c) average defect volume, and (d) total volume of detected defects.

3.3. Fatigue behavior

Figure 9 presents the uniaxial fully-reversed (R = -1) fatigue test results for the LDED Ti-6Al-4V specimens fabricated using virgin and recycled powders. The specimens fabricated using virgin powder showed a higher fatigue resistance than those fabricated using recycled powder in both short-life and long-life regimes. The lower fatigue resistance of the recycled specimens as compared to their virgin counterparts is directly attributed to the higher level of defects in recycled specimens.

Fractography of fatigue fracture surfaces revealed that the process-induced LOF defects were the main source of crack initiation in both groups of fatigue specimens, i.e., fabricated using virgin powder and recycled powder, as shown in Figure 10. In general, defects that initiated cracks in the recycled specimens appeared to be larger, resulting in shorter fatigue lives of the recycled specimens as compared to their virgin counterparts. In most cases, LOF defects that served as the crack initiation sites were attached to the surface, as can be seen in Figure 10(b). However, in some cases, subsurface LOF defects were observed to be the crack initiation site. Due to cyclic loading, these defects extend to the surface, indicating that subsurface LOF defects still play a dominant role in the fatigue life of the specimens.

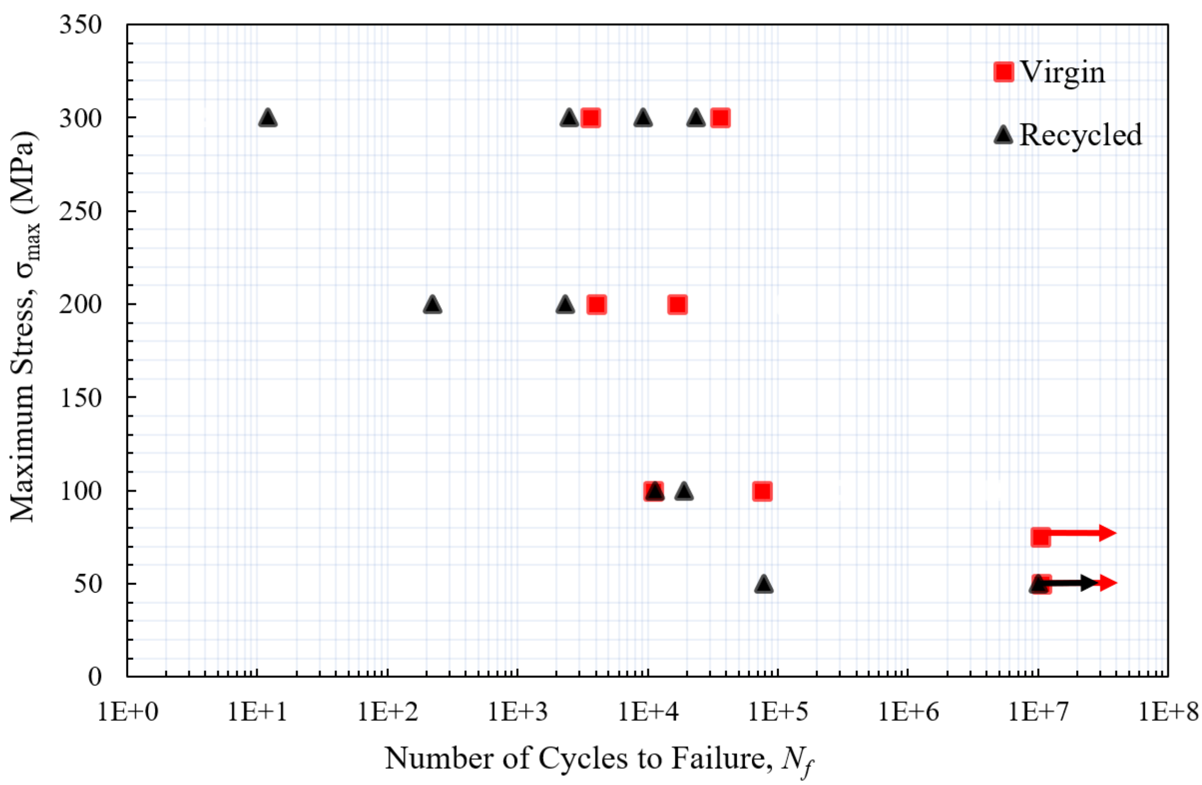


Figure 9. Stress-life fatigue data of the LDED Ti-6Al-4V specimens, fabricated using virgin and recycled powders.

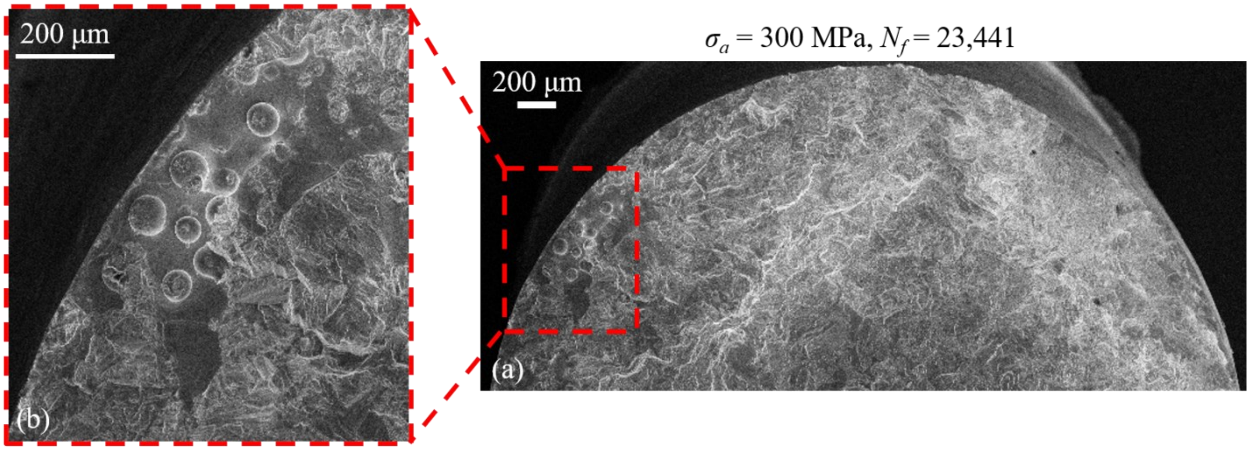


Figure 10. (a) An overall view of a fracture surface for fatigue specimens fabricated using recycled powder at high stress amplitude, i.e., 300 MPa, (N_f = 23,441 cycles), and (b) a magnified view of the LOF defect served as the crack initiation site.

4. Summary

In this study, the effects of powder reuse/recycling on the powder characteristics as well as the microstructure and fatigue behavior of LDED Ti-6Al-4V samples were investigated, and the results were compared with their counterparts fabricated using virgin powder. A slight increase was observed in the contents of oxygen, titanium, and vanadium for the powder after nine times of recycling. The recycled powder had a noticeably smaller particle mean size as compared to the virgin powder. The flowability of the recycled powder, measured using the Hall flowmeter, was slightly lower than that of the virgin powder. The XCT results revealed a significant increase in the number and size of process-induced defects for the specimens fabricated using recycled powders. Containing a higher level of porosity, the specimens fabricated using recycled powder showed a lower fatigue resistance compared to their counterparts fabricated using virgin powder. For both groups of fatigue specimens, i.e., fabricated using virgin powder and recycled powder, process-induced LOF defects served as the crack initiation site.

5. References

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