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Fatigue crack growth in Ti-6Al-4V EBMed samples: impact of powder recycling

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Abstract

Within the powder bed fusion electron beam (PBF-EB) category, due to the high cost of material feedstock, the unused powder can be reused in subsequent production cycles, thereby reducing the costs associated with powder production. However, since the impact of recycling processes on the metallurgical and mechanical properties has not been fully investigated in the scientific literature, this study aimed to analyze fatigue crack growth propagation by examining three types of compact tension (CT) specimens. The specimens were manufactured using the Electron Beam Melting (EBM) process with three different batches of Ti-6Al-4V powder particles: virgin, recycled five times, and recycled more than 100 times. The results reveal that the oxygen content increases with each cycle, indicating the potential effects of recycling. Furthermore, from a mechanical testing perspective, the obtained results did not indicate any significant effects of powder reuse on the investigated CT samples, as the obtained curves exhibited a high degree of similarity. This can be attributed to the presence of numerous internal defects, such as porosity and Lack of Fusion, and thus, it is believed that the effect of powder recycling has been overshadowed by the influence of these defects.

Keywords: Additive Manufacturing; fatigue crack growth; CT samples; crack growth propagation; Ti-6Al-4V; Electron Beam Melting; powder recycling; internal defects; microstructure.

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1. Introduction

The ability of Powder Bed Fusion (PBF) processes to produce complex components in a single production run from raw materials in powder form has led to significant growth in these additive manufacturing processes in recent years. When referring to "complex components," the discussion includes polymeric artifacts (Leirmo & Semeniuta, 2021), lattice structures (Bellini et al., 2021), jewelry (Klotz & König, 2022) and even a complex cross-section in the shape of the Mona Lisa (Plotkowski et al., 2021). Consequently, the range of materials that can be employed ranges from frequently utilized metal alloys such as Ti-6Al-4V (Bellini et al., 2023) or Inconel 718 (Zhao et al., 2020), to polymeric substances (El Magri et al., 2022) and even precious metals like gold (Klotz et al., 2017) and silver (Robinson et al., 2020). During a production cycle, it is important to note that only a portion of the initial powder placed in the production chamber is actually melted or sintered to create the final component (only 10-50% of the build volume (Santecchia et al., 2020)), while the remaining fraction is removed at the end of the manufacturing cycle, effectively becoming production waste (Faludi et al., 2017). Although there is currently a lack of standardized rules for powder recycling, the primary goal of the research is to reduce production costs and improve process efficiency by reusing waste powder as raw material for subsequent processing steps, tapping into user-experience-based recycling methods found in the literature (Powell et al., 2020). It is important to note, however, that the waste powder could differ from the initial virgin powder in a variety of ways, either due to thermal factors (such as the preheating of the production chamber and powder bed, as well as the heat resulting from the proximity of the powder to the melting pool (Price et al., 2017) (Soundarapandiyan et al., 2021)) or mechanical factors (such as the blasting process used to remove the part from the surrounding powder at the end of the production cycle (Petrovic & Niñerola, 2015) or the sieving operation applied to the waste powder to remove any large agglomerates or highly irregular particles (Harkin et al., 2021)). As a result, depending on the number of reuses, recycled powders may not have the same qualities as virgin powders, which might result in poor mechanical properties of components manufactured from these recycled powders. Consequently, understanding the alterations in powder properties is essential to minimizing the loss in the performance of the produced components. Several studies have been conducted to investigate the differences between virgin and reused powders, as well as the differences in the components manufactured starting with these powder sources, and it was found that recycled powders have a lower level of quality when compared to virgin powder counterparts. Specifically, according to the results provided by numerous studies (Tang et al., 2015) (Emminghaus et al., 2022) Ti-6Al-4V reused powders tend to contain higher levels of oxygen when compared to their unused counterparts. Other elements, such as V and Al, appear to remain at the same levels as the number of reuses increases. The increased oxygen content of reused powders can be attributed mostly to the powder's recurrent circulation in the processing zone, which leads to increased oxidation. Furthermore, once the powder has been removed from the Electron Beam Melting (EBM) equipment, it is exposed to moisture and the surrounding atmosphere, resulting in an increase in oxygen absorption (Shanbhag & Vlasea, 2021).

In terms of powder morphology, various researchers (Emminghaus et al., 2022) (Carrion et al., 2019) (Gatto et al., 2021) have demonstrated that reused powders have less satellite particles, which are small particles that cluster or attach to the surface of bigger particles, than their as-received counterparts. This reduction in satellite particles has been discovered to be beneficial since it considerably increases particle flowability. Furthermore, it has been found that reused powders have a narrower particle size distribution than virgin powders (Carrion et al., 2019)(Yusuf et al., 2020) (Nie et al., 2021). Particles combining to form larger clusters during the manufacturing process (Carrion et al., 2019), some small particles remaining suspended in the chamber and not being reused, thus not being counted (Seyda et al., 2012), or small particles adhering to larger ones, making them difficult to count (Sutton et al., 2016), are possible explanations for this change. In addition, studies has shown that there are no visible variations in the microstructure of virgin and reused Ti-6Al-4V powders, with both largely consisting of α' acicular martensite (Emminghaus et al., 2021) (Sun et al., 2018). Moreover, components manufactured from virgin or reused powders have no microstructure differences, with thin acicular grains within columnar prior-grains orientated in the same direction as the building process (Carrion et al., 2019) (Strondl et al., 2015). However, other research (Opatová et al., 2020) have discovered that because of slower cooling rates over production cycles, reused powders often have a coarser microstructure than virgin powders.

The mechanical characteristics of components produced from virgin and reused powders differed, indicating that the quality of the raw material only represents one of many factors that might influence the mechanical strength and features of the final components (Santecchia et al., 2020). In terms of tensile properties, an increase in mechanical strength was observed as the number of recycles increased (Seyda et al., 2012), and this was also observed when both types of components were subjected to the same hot isostatic pressing (HIP) treatment (Quintana et al., 2018). Other researchers, on the other hand, found no noticeable difference in tensile strength (Carrion et al., 2019) (Strondl et al., 2015), although the scatter of data was high in some cases (Denti et al., 2019). There were no apparent variations in fatigue performance between components manufactured from virgin and reused powders (Hann, 2016). In contrast, some researchers noticed a degradation in fatigue qualities, as demonstrated by a decrease in High Cycle Fatigue (HCF) strength as the number of reuses increased (Del Re et al., 2018). Other studies, on the other hand, found no noticeable variations in HCF, while they did highlight a slightly reduced fatigue strength in the Low Cycle Fatigue (LCF) regime for components made with reused powders (Foti et al., 2022).

The aim of this study was to investigate the effect of powder reuse on fatigue crack growth propagation in three different kinds of Ti-6-Al-4V alloy specimens produced via the Electron Beam Melting (EBM) technique.

2. Materials and Methods

To investigate the effect of powder reuse on crack propagation behavior, the EBM technique was used to build three samples from three different batches of powder. Advanced Powders and Coatings, Inc. (AP&C) supplied the Ti-6Al-4V powder, which included batch A consisting of grade 5 powder reused 100 times, batch B consisting of grade 5 powder reused 5 times with virgin grade 23 powder (ELI powder) with low oxygen content added at each recycle, followed by batch C consisting of as-received grade 5 powder. The chemical composition of the three types of powders is shown in Table 1.

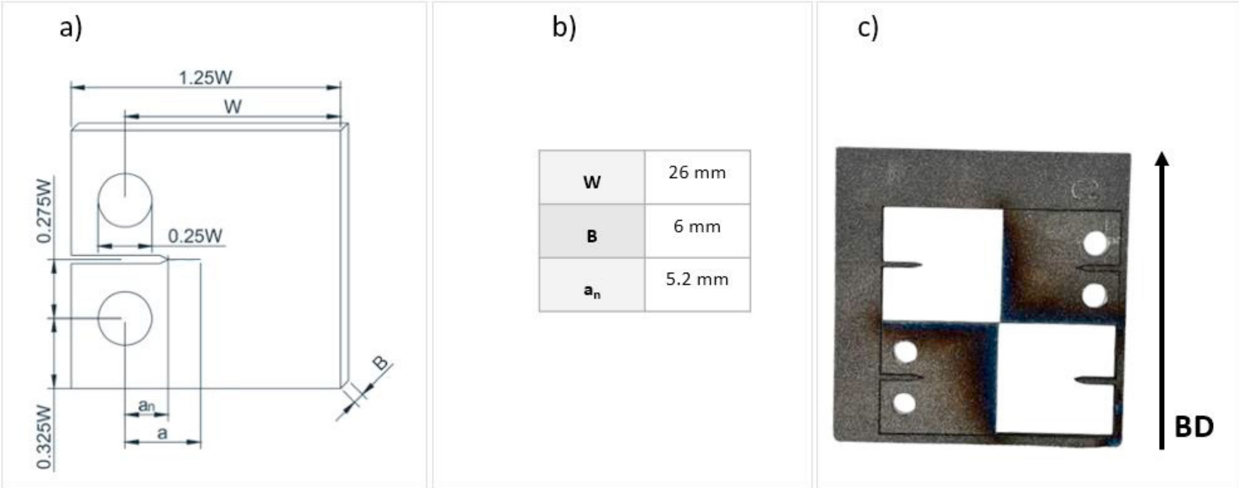
Table 1. Chemical composition of the Ti-6Al-4V powder particles

	% Al	% V	% C	% N	Fe %	% O	% Ti
Batch A – Reused powders 100 times	6.46	4.03	0.01	0.03	0.202	0.30	Remaining
Batch B - Reused 5 times	6.48	4.02	0.01	0.02	0.196	0.19	Remaining
Batch C - Virgin	6.50	4.03	0.01	0.02	0.205	0.11	Remaining

It is worth noting that ASTM F2924 (Ghods et al., 2020) indicates that the acceptable limit for oxygen content in aeronautical applications is 0.2%. The results in Table 1 show that using grade 23 Ti-6Al-4V powders at each reuse in Batch B keeps the oxygen content within the limit, whereas the Batch A powders results in an oxygen level of 0.30, which exceeds the limit.

The compact tension (CT) specimens used in the crack growth propagation tests were produced on the Arcam EBM A2X machine utilizing the Electron Beam Melting method by manufacturing parallelepiped-shaped specimens. The parallelepiped plate was sectioned appropriately, yielding four CT samples for each plate. Figure 1 illustrates the dimensions, geometry, and sectioning of the samples, as well as the building direction. After that, the samples are removed for polishing in order to form the pattern required for digital image correlation analysis (DIC). The pattern is made by painting with varying shades of grey to distinguish various areas that will be used to monitor crack propagation. The fatigue crack growth propagation test is carried out on the Instron 10000 machine, on which the CT specimens are set up. A camera perpendicular to the specimen surface and an illumination system are installed prior to the test to improve focus and avoid any reflection on the specimen surface, ensuring proper digital image correlation (DIC). The fatigue crack propagation test is started with a load ratio R of 0.05, with the minimum load application of P_{min}=75 N and maximum P_{max}=1500 N (ΔP=1425 N).

After the specimen had been fractured, the fracture surfaces were examined with the Hitachi S-2500 scanning electron microscopy (SEM) with a work distance of 20 mm and a voltage of 25 kV. Images were captured at magnifications of 1000x, 2500x, and 4000x, with 1 mm displacements from the fracture apex.



W	26 mm
B	6 mm
a_n	5.2 mm

Fig. 1. CT samples specifics: (a) sample geometry; (b) dimensions; (c) cut-out samples from the parallelepiped block and building direction (BD).

3. Results and Discussions

The results of the fatigue crack propagation test are shown in Fig. 2. As can be observed, the results of all three samples are comparable and overlap, demonstrating that powder reuse has no noticeable effect on fatigue crack propagation behavior. This data overlap was detected in both the da/dN vs ΔK plot, Fig. 2-a**,** and the a/W vs N plot, Fig. 2-b. Slightly greater resistance to crack propagation can be observed in the virgin powders of batch C, visible in Fig. 2-b, although this difference is minimal.

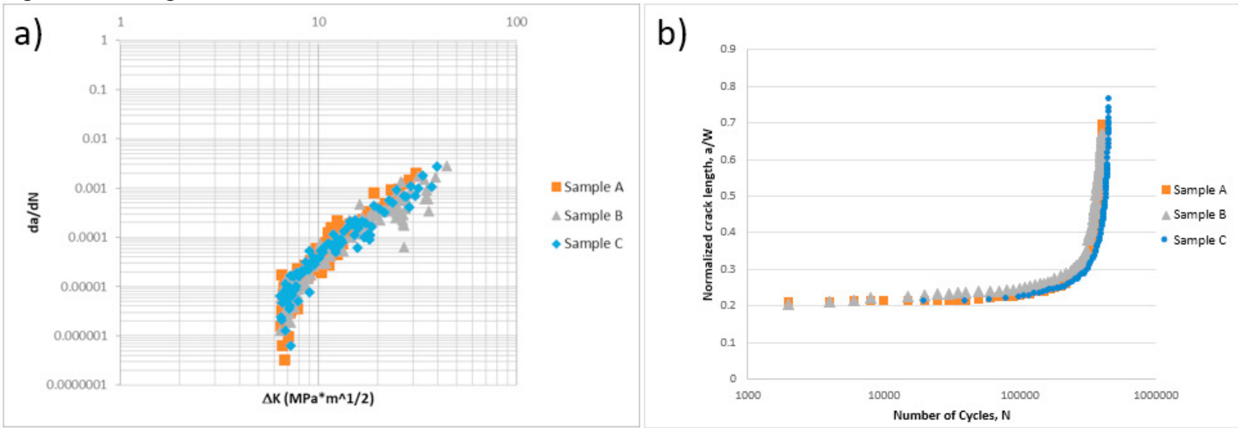


Fig. 2. Fatigue crack growth propagation results; (a) da/dN vs ΔK diagram; (b) normalized crack length a/W vs number of cycles N diagram.

There was not a noticeable distinction between the three different kinds of samples based on fracture surface analysis. All three samples had a significant level of internal porosity, which was related to the process parameters of the EBM machine. In addition, a transition of the fracture mode from ductile to a mixed mode of ductile-brittle fracture can be shown in Fig. 3. Indeed, area closer to the apex of the fracture, which fall into the stable crack propagation regime, are characterized by a more ductile mechanism. In contrast, when approaching the final fracture zone, the fracture mechanism increasingly becomes more brittle. This pattern was observed in all three types of CT samples examined.

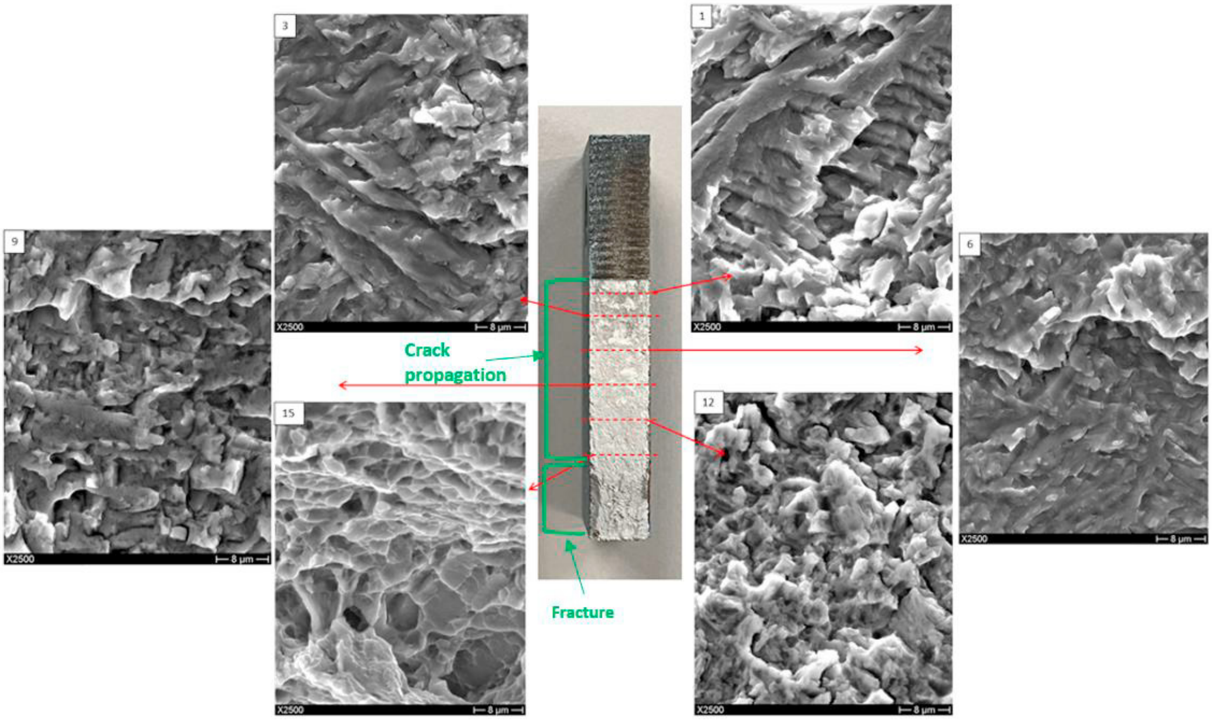


Fig. 3. SEM images of the fracture surfaces of the batch B sample; the tiny numbers in the upper left corner of the individual fractography indicate the distance to the crack apex in millimetres.

4. Conclusions

This study analysed the effect of powder reuse on the fatigue crack growth behavior on the Ti-6Al-4V components produced with the Electron Beam Melting (EBM) process, starting from three batches of powder: batch A consisting of grade 5 powder reused 100 times, batch B consisting of grade 5 powder reused 5 times with virgin grade 23 powder (ELI powder) with low oxygen content added at each recycle, followed by batch C consisting of as-received grade 5 powder.

The results showed that:

- The three samples exhibited similar behavior when subjected to the fatigue crack propagation test, with comparable results among them.
- A minor difference is noticeable in the Type C component, which shows slightly higher resistance, although the difference is minimal.
- The fracture surfaces of all three samples showed a high level of porosity, mainly due to the process parameters of the EBM machine.
- A transition of the fracture mode from ductile to a mixed mode of ductile-brittle fracture was observed in all three kinds of specimens.

Considering the apparent variation in results across various mechanical tests, as highlighted in the introductory section of this research, it is considered appropriate to carry out additional crack propagation tests while considering other variables, such as machine process parameters, in addition to evaluating the impact of the powder quality used as the raw material.

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