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Effects of recycling on defects and microstructure in Ti-6Al-4V powder particles and samples fabricated by Electron Beam Melting process

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Abstract

Electron Beam Melting (EBM) is an additive manufacturing process that forms part of the Powder Bed Fusion (PBF) category. All the PBF processes share the same basic printing steps and use as material feedstock the metal powder which is previously atomized according to several existing methods. After the printing cycle, the powder in excess may be reused following a suitable sieving procedure to lower the production costs, otherwise, a large percentage of unused powder would be lost. The waste powder may differ in several respects compared to the starting virgin ones, depending on how many times it has been reused. Moreover, degradations in powder properties and its chemical composition can lower the quality and the mechanical performances of the components fabricated by Electron Beam Melting. The aim of this work is to investigate the microstructure variations in two batches of Ti-6Al-4V powder: virgin powders produced using a plasma atomization process and powders recycled more than one hundred times. Printed with the two batches of powder feedstock, 6 EBMed cylindrical bars were analyzed both from the point of view of microstructure and internal defects using an Optical Microscope. The bars were firstly cut and then embedded in resin, polished, and etched with a Hydrofluoric acid (HF) solution. The experimental results show several types of internal imperfections including macropores, micro voids, Lack of Fusion (LOF) defects, which are directly dependent on process parameters and quality of the material feedstock. While for the microstructure few changes were found both in powder particles and in their printed bars.

Keywords: Additive Manufacturing; Ti-6Al-4V; Electron Beam Melting; powder recycling; internal porosity; microstructure; external defects.

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1. Introduction

The primary feature of the Powder Bed Fusion (PBF) technologies is the ability to create very complex shapes, such as lattice structures (Bellini, et al., 2021) (Bellini, et al., 2021), aerospace components (Gardan & Schneider, 2015), or even uniquely shaped jewels (Cooper, 2016) by adding material layer by layer, rather than removing it, in a single manufacturing step (Guo & Leu, 2013). The PBF processes, as the name implies, use powder as the raw material and typically require an amount of powder to be introduced into the construction chamber that is higher than what is necessary to produce the desired final component. Therefore, at the end of a production process, the excess powder that did not melt and did not contribute to the construction of the final product, can be collected, and sifted to remove any damaged particles. This excess sifted powder can then be mixed with virgin, unused powder and reintroduced into the system for future production cycles (Bellini et al., 2022). With these recycling approaches, it is feasible to reduce the costs associated with the material feedstock, which are known to be somewhat higher due to the atomization procedures (Kassym & Perveen, 2019). However, depending on the number of reuses, recycled powders do not have the same properties as virgin powders, and understanding the changes is crucial to minimize performance degradations in manufactured components. In fact, components made with recycled powders could manifest significant differences in their mechanical properties, such as hardness (Emminghaus et al., 2022), fatigue life (Foti et al., 2022), crack growth propagation and tensile properties (Tang et al., 2015) (Emminghaus et al., 2022). Several researchers have examined the variations between unused powders and powders that have undergone recycling, as well as the distinctions between the components fabricated using these powder types. In general, recycled powders tend to have lower quality than virgin powders. More in detail, in the Ti-6Al-4V alloy, recycled powders typically contain higher levels of oxygen compared to virgin powders, while the levels of other elements such as V and Al have been found to remain consistent (Tang et al., 2015) (Emminghaus et al., 2022). The increased oxygen content in recycled powders is mainly attributed to the powder being repeatedly circulated around the process zone, leading to higher levels of oxidation. Furthermore, the powder is exposed to moisture and the surrounding atmosphere when taken out of the EBM machine, which is another factor that contributes to the pickup of oxygen (Shanbhag & Vlasea, 2021). At the same time, the recycled powders are known for their lower amount of satellites compared to the as-received powders which is beneficial since it improves the flowability of the particles (Carrion et al., 2019) (Emminghaus et al., 2021) (Gatto et al., 2021). This occurs due to two main reasons: firstly, the temperature conditions during the EBM preheating and laser heating cause the melting of satellites present on the surface of larger particles (Popov et al., 2018); and secondly, the sieving process results in a reduction of the percentage of satellites by eliminating agglomerated particles or partially sintered ones (Yusuf et al., 2020). Additionally, when examining the size of particles, the recycled powders appear to have a narrower particle size distribution, than the unused powders (Strondl et al., 2015), (Carrion et al., 2019), (Nie et al., 2021), (Yusuf et al., 2020). There are a few possible reasons for this change. During the manufacturing process, some particles stick together and form larger clumps or droplets of melted material that are bigger than the individual powder particles (Carrion et al., 2019). Or this may occur because the tiny particles get lifted up and stay in the air inside the chamber, so they can't be reused (Seyda et al., 2012). Another reason could be that the small particles stick to the larger ones, which makes them hard to count (Sutton et al., 2016). However, other research has shown that sometimes the reused powders can have a broader range of sizes compared to virgin powders (Gatto et al., 2021).

In terms of the microstructure, some authors have discovered that there are no variations between the virgin and reused Ti-6Al-4V powders (Emminghaus et al., 2022), consisting mostly of acicular α' martensite (Sun et al., 2018a). At the same time, some authors discovered that there are no dissimilarities in the components produced from either new or reused powders. Specifically, they observed that both Ti-6Al-4V components displayed a microstructure comprised of a slender acicular α' within the columnar prior-β grains oriented in the same direction as the building process (Carrion et al., 2019). Other authors confirmed no differences regarding the microstructure in components produced starting with as-received and recycled powders (Strondl et al., 2015).

Conversely, it has been noted that recycled powders generally exhibit a more coarse microstructure than virgin ones. This is attributed to the fact that during manufacturing cycles, the cooling rate is slower that during the atomization process (Opatová et al., 2020). It was further noted that the microstructure is strongly dependent on the process parameters, particularly the laser power and scanning speed. More in detail, a microstructure with thin prior beta grains and a fine structure is observed when employing low laser power and high scan speed. Conversely, an increase in laser power and a decrease in scanning speed result in wider prior beta grains (Emminghaus et al., 2021).

With the findings mentioned earlier, this study seeks to explore how reusing powders affects the microstructure and defects present in both the powder particles and the resulting components made from these powders.

2. Experimental

The current research compared two batches of Ti-6Al-4V powder particles: virgin and recycled. Advanced Powders and Coatings, Inc. (AP&C) provided the virgin powder (grade 5) which was atomized using their proprietary Advanced Plasma Atomization process (APA™). APA™ is well known for its ability to produce highly spherical powders with precise size distributions, low oxygen content, and low internal porosity. Furthermore, to avoid internal pores caused by entrapped argon gas in the particles, APA™ powders are processed with a low gas flow rate and by keeping the atomizing gas hot, the metal particles are prevented from rapidly freezing into irregular shapes (Capus, 2017). The virgin powder is compared to one recycled over than one hundred cycles collected from the Arcam A2X machine (complete specifications are accessible on the web) after the cycles have been run using the standard process settings recommended by Arcam based on their expertise. The process was carried out in a vacuum chamber, with the pressure decreasing from $5x10^{-3}$ mbar at the start to $2x10^{-5}$ mbar at the end of the cycle, while before starting the manufacturing cycle, the powder bed was heated to 750 °C to obtain partial sintering of the powder. The sintering is frangible but robust enough to hold powder and sustain the impact of the electron beam during the EBM process, decreasing the number of supports needed. When the EBM process is completed, the powder aggregation may be broken apart by external force, releasing the enclosed samples (He et al., 2011).
Because the powder circulates continuously in the EBM machine, a definite number of recycles cannot be specified. Moreover, the powder tested in this study cannot be linked to specific areas of the building chamber, therefore it may be considered as a mix of particles found both in the pre-sintered and in the surrounding regions. Before reintroducing the reused powder for the next cycle after completing one, the powder was sieved to remove large agglomerates.
Some early information about the powders concerns the chemical composition.

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

	% Al	% V	% C	% N	Fe %	% O	% Ti
Virgin	6.50	4.03	0.01	0.02	0.205	**0.11**	Remaining
Recycled	6.46	4.03	0.01	0.03	0.202	**0.30**	Remaining

As indicated in Table 1, which shows the chemical composition of both batches of powder, the oxygen percentage in reused powders has grown to 0.3%, exceeding the standard of 0.2%, which is specified as the limit value in the aerospace fields according to ASTM F2924 (Ghods et al., 2020). Therefore, the recycled powders analyzed in this work cannot meet the requirements for the production of aerospace components.
The morphology of powder particles was analysed using the FEI Quanta 650 Scanning Electron Microscope (SEM) to make a preliminary comparison between the two batches of powder and to study changes in shape, roughness, and surface imperfections after recycling. Following this first observation, which did not need any powder preparation, the powder particles were mounted in phenol-formaldehyde resin, producing 30 mm cylindrical molds that facilitated the powder handling. The resin/powder samples were then exclusively polished on porous woven wool felt with a Struers polishing machine using 1 µm and 0.3 µm Alumina solutions. Grinding procedures are not considered appropriate because they are too aggressive, increasing the number of particles that escape from the resin during the grinding process. The polished samples were initially examined with a Nikon Epithot inverted Metallurgical Microscope to look for any internal porosities caused by entrapped gas during the atomization process. Following this first examination, the samples were etched with a 0.1 molar HF solution and examined again under the same inverted Metallurgical Microscope to look for microstructural and phase changes within the powders.
Three samples for each batch of powders were manufactured through the Electron Beam Melting process, Figure 1 – (a). The samples were cut into 9 sections and some sections were mounted for defects and microstructural investigation. Using a Struers LaboPress-1 hot mounting press, the samples were mounted in phenol-formaldehyde resin. As seen in Figure 1 – (b), the exposed surfaces of the mounts included a top and bottom cross sectioned surface as well as a top and bottom longitudinal surface; consequently, for each sample there were four mounted surfaces to

be analysed. The top and the bottom areas were chosen to investigate possible influences of the building plate. Following mounting, each sample was sequentially grinded using SiC paper with increasing mesh counts of # 240, 400, 600, 800, 1000, and #1200. Mirror finish polishing was performed on porous woven wool felt using a 1 µm and 0.3 µm Alumina suspension employing the standard polishing method. Internal porosity and other internal imperfections were examined with the Nikon Epithot inverted Metallurgical Microscope on the mirror-finished surfaces. Following this first examination, the samples were etched by immersing in a 0.1 molar HF solution for 15 seconds and then washed with ethanol. A second observation was carried out using the same optical microscope, this time to study the microstructure and any phase changes.

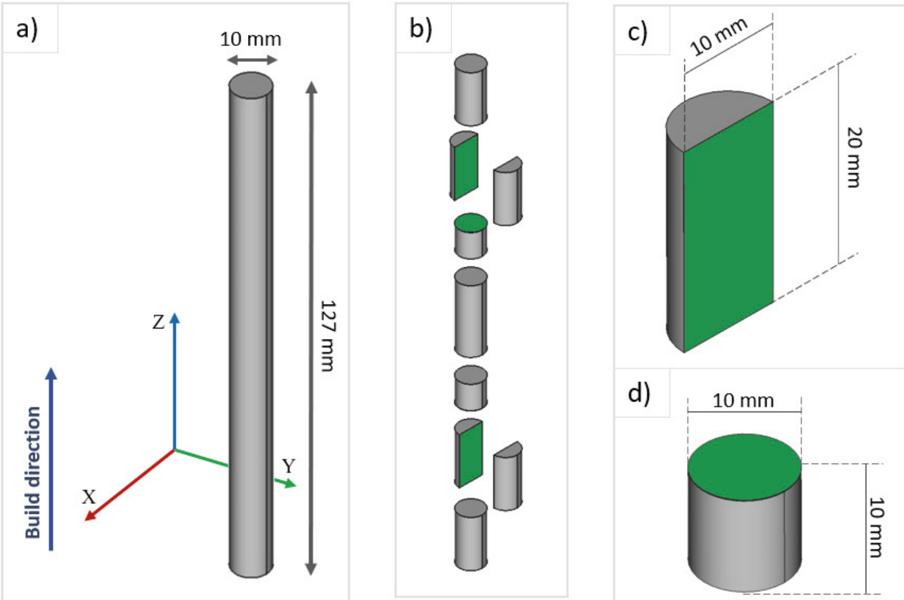

Figure 1. *(a) CAD model of the manufactured samples; (b) surface representation to be analysed (highlighted in green); (c) detailed view of the longitudinal surface; (d) detailed view of the cross-sectional surface.*

3. Results and Discussions

3.1 Defects

Figure 2 shows the morphology of virgin powder. This powder, which has been plasma atomized, has a few imperfections indicated by tiny particles that agglomerate or adhere to the surface of larger ones, known as satellites, and which are known to reduce the flowability of the powder (Seyda et al., 2012). The shape of particles is almost perfectly spherical with very few imperfections, and with smooth surfaces. Some irregular particles may also be found; however, their presence is low.

The first major change seen in the recycled powder, as shown in Figure 3, is the almost complete disappearance of satellites, which is consistent with the results obtained from other research (Tang et al., 2015). This significant change can be ascribed to the temperature conditions, i.e., the pre-heated environment in the Arcam 2X machine, allowing the satellites to melt on the surface of the bigger particles. As a direct result of this phenomena, there is an increase in surface deformation and a slight decrease in sphericity. Other imperfections include elongated, fractured, or severely damaged particles; these variances are caused by both temperature conditions, which are the pre-heating of the environment and the prolonged thermal holding of the powder bed (Tang et al., 2015), and mechanical factors such as the preliminary sieve operation employed (an indispensable process in recycling procedures) (Slotwinski et al., 2014). The investigation on particle size distribution was not conducted in this work, however other authors have reported a smaller particle size dispersion in recycled powder (Sun et al., 2018b).

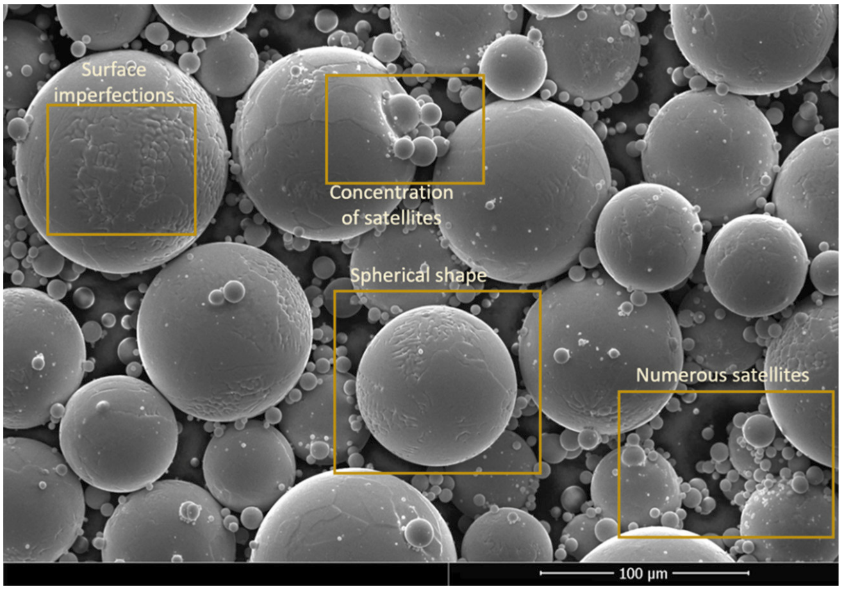

Figure 2. *SEM examination of virgin powder. The high sphericity of the particles, numerous tiny particles (satellites) around the larger ones, and a few imperfections are highlighted.*

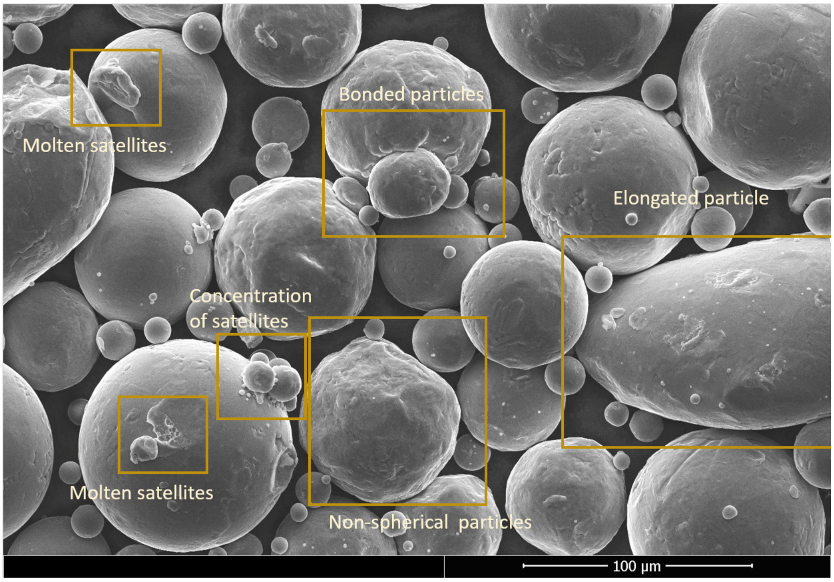

Figure 3. *SEM observation of recycled powder. Highlighted are uneven and rough particle surfaces, elongated particles, and almost all satellites that have disappeared.*

In Figure 4 the cross-sectional views of both powder batches are shown. As can be seen, both batches have an almost spherical shape, with few imperfect particles. The voids inside the particles are also spherical in shape, suggesting that these pores are due to trapped gases during the atomization process (Susan et al., 2006). As noticeable, the microporosities are almost completely eliminated in the recycled powders. This behavior can be explained by the preheating in the Arcam machine environment, which has contributed to increasing the density.
Powder porosity generally has a spherical morphology, possibly due to the high pressure inside the liquid droplet acting on the gas bubble (Gowtham, 2022).

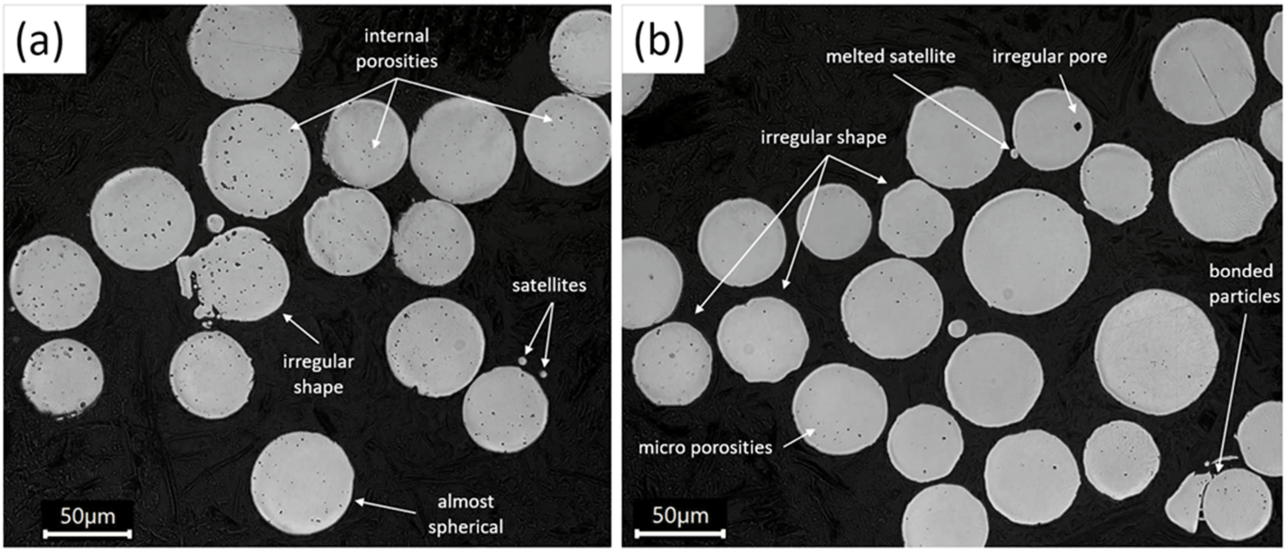

Figure 4. *Optical observations of (a) virgin powder; (b) recycled powder.*

From this point of view, it is almost impossible to comment on the size of the particles since, being enclosed in resin, the maximum observable diameter is unknown. The same applies to porosities: since they are mainly spherical-shaped voids, commenting on their size would be an underestimate. However, with the same underestimate, a quantitative analysis of the percentage of defects present may be performed. This was already carried out in our previous work (Bellini et al., 2022), using the commercial software ImageJ, finding that the virgin powder had 3.03% defects, while the recycled ones had 0.68% defects.

In this study, an additional 25 particles were considered for each powder batch to perform a new quantitative analysis. The results showed an average value of 3.723% for the virgin batch and 0.974% for the recycled batch. It is noticeable that although the percentage values are different, the trend remains almost the same, indicating that pre-heating within the EBM production chamber causes a reduction in internal micro porosity.

Regarding the components manufactured with the Electron Beam Melting process starting with the two batches of powder particles, it was found that both kind of samples exhibit the typical defects found in the EBM components, which are the metallurgical pores (or hydrogen porosity) and macro pores that have regular shape, and they are due to the pores existing inside the gas atomized powder particles, or they may be related to the entrapped gas during solidification when the scan speed is low (Xiao & Zhang, 2014), (Tammas-Williams et al., 2015). Keyhole porosities, have an irregular shape and they mostly result from keyhole instability, when powder particles receiving insufficient energy (Bellini, Berto, et al., 2021), (Svenungsson et al., 2015). Lack of fusion defects are also called "incomplete fusion zones," and they mainly appear when the energy density is not sufficient to melt the entire intended region (Darvish et al., 2016). Surface roughness results from the approximation of sloped surfaces by tiny vertical steps, and therefore thinner layer thicknesses result in smoother, higher-quality surfaces because the staircase effect is diminished (Brooks et al., 2012).

The results of the ImageJ software performed on the cross-sectional images of the components, in the upper part and in the lower part, and in both longitudinal and transversal sections, exhibits an average value of 1.919% of defects within components manufactured with virgin powders, and an average value of 1.247% of internal defects within components manufactured with recycled powders.

The lower difference between the two kinds of samples, compared to the values found in powders itself, is mainly due to the process parameters influence, and therefore the influence of the recycling steps is minimized by the influence of the parameters themselves. The only defects that can be found and attributed to recycling are microporosities resulting from gas trapped in the powders and transferred to the components during manufacturing.

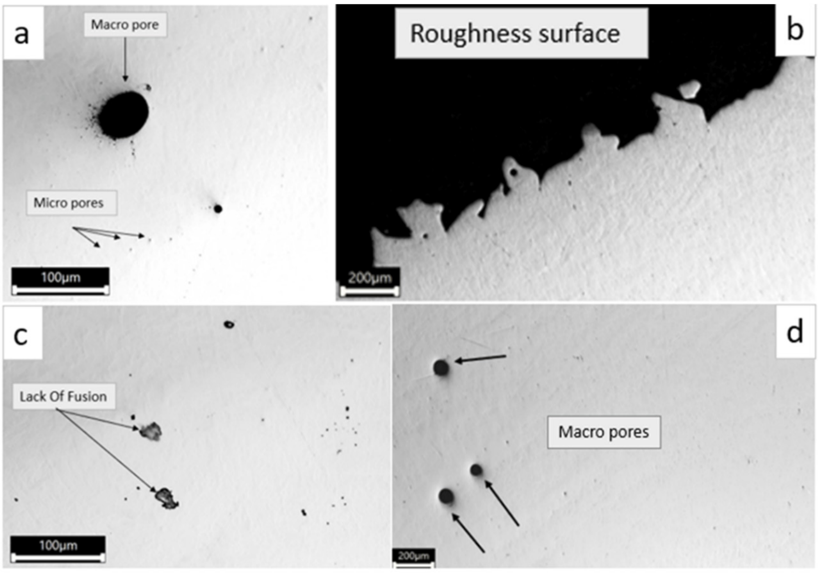

Figure 5. *Optical images of the EBMed components showing the typical defects found up.*

3.2 Microstructure

The virgin particles exhibited a microstructure that was predominantly martensitic, consisting of α' phase characterized by finely dispersed acicular grains of the α phase. This microstructure, which is a result of the rapid cooling of the β phase, is notable for its unique and intricate pattern. The nature of this microstructure underscores the significance of the atomization process in determining the final properties of the powder particles.

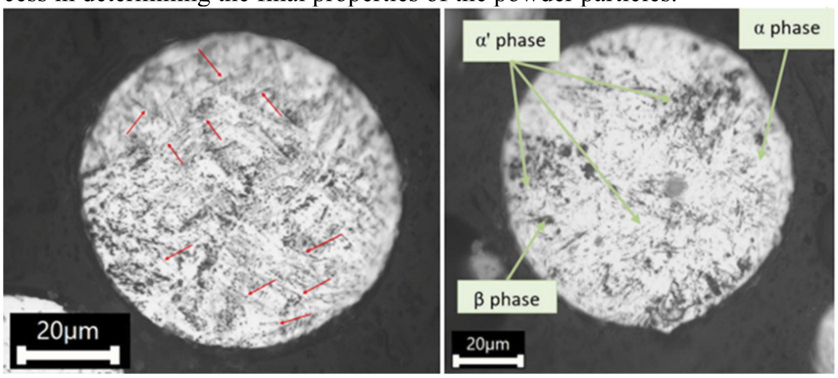

Figure 6. *Optical micrographs of virgin powders showing the mostly predominantly martensitic structure (indicated with red arrows).*

Regarding the recycled powders, as is known from the literature, the coarsening of the microstructure increases with increasing reused cycles. In the batch investigated in this work, it was found that the particles consisted almost entirely of heat-affected particles, with relatively few particles still showing the virgin microstructure, Figure 7 – (c), while most exhibit the coarsened microstructure. This suggests that individual powder particles experienced significantly different thermal histories.

Additionally, the presence of alpha-case was observed on the recycled particles, Figure 7 – (d), but not in the virgin ones. This is due to the dissolved oxygen content inside the analysed titanium alloy. Further studies in this regard plan to perform a nano-hardness test at the alpha-case layer to possibly confirm the higher hardness in those zones.

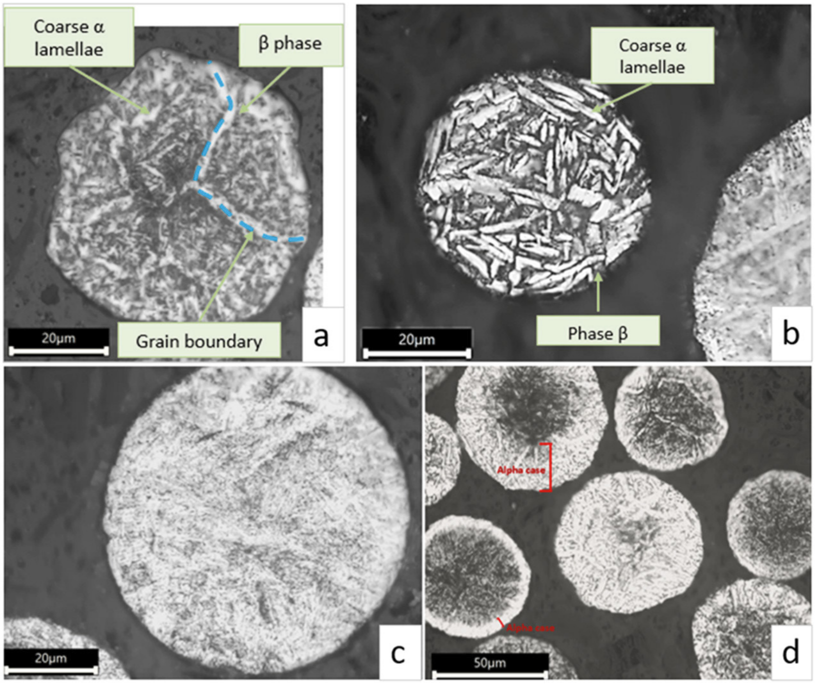

Figure 7. *Optical micrographs of recycled powders showing the mostly predominantly coarsened structure and (d) the presence of Alpha-case.*

Regarding the microstructure of the samples, there were no differences between the longitudinal and cross-sectional sections. The two sections had the same basketweave microstructure. This was observed for samples produced with virgin powder and those produced with recycled powder, and both for the upper and lower parts.

Something interesting was that for the longitudinal view, the β grain priors tended to follow the direction of the building, Figure 9. This was observed only in the centre of the sample, while close to the edges this was not found. Here, the prior beta grains did not follow any preferred direction. This was observed for both virgin and recycled. And for both parts upper and lower.

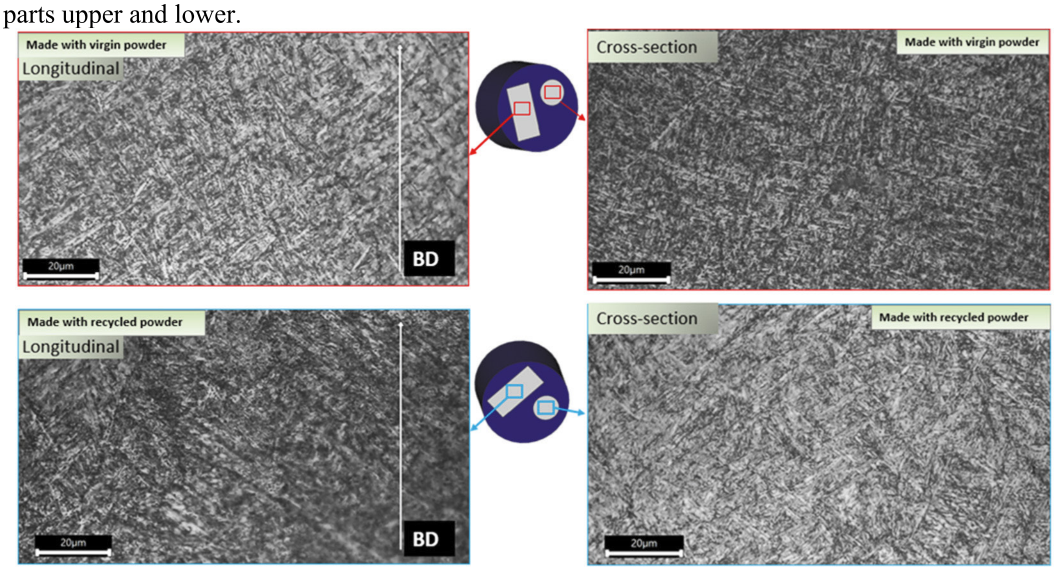

Figure 8. *Optical micrographs of the virgin and recycled powders showing no differences regarding the microstructure.*

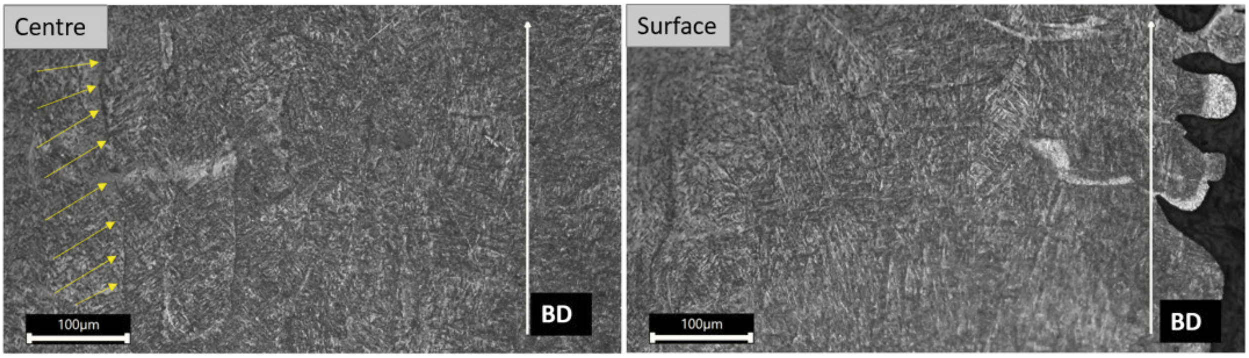

Figure 9. *Optical micrographs of the EBMed components showing the alignment of the prior β grains with the building direction (BD).*

4. Conclusions

The influence of recycling on powder microstructure, samples microstructure and samples defects were investigated by means of the Optical Microscope and the commercial software ImageJ.
The following has been found:

- Components produced by Powder Bed Fusion (PBF) processes and particularly the Electron Beam Melting (EBM) subprocess suffer from inherent defects in the process itself. These defects can be minimized by optimizing the process parameters.
- A higher defect rate was found in components produced with virgin powder than those printed with recycled powder. This is not entirely attributable to the recycling of powders, but also, more importantly, to the choice of process parameters.
- The microstructure of samples does not vary when considering longitudinal or cross-sections. The microstructure is basketweave, for both types of components.
- In the central zone of the samples, it was seen that the prior beta grains follow the direction of construction, while in the more superficial zone, this was not found. In the more superficial zone, the prior beta grains follow a not specified direction.
- Regarding powders, the virgin batch is characterized by the alpha prime martensitic phase, characterized by a kind of scratching. Although recycled powders also exhibit this phase, it is in smaller quantities. This is consistent with what has been seen in the literature, in that recycled powders are as if they undergo heat treatment, as a result, the martensitic phase decomposes into the alpha phase.
- Recycled powders are characterized by a layer called alpha case, which is characterized by a high concentration of oxygen. This zone is much more frequent in recycled powders, as it is known from the literature and our preliminary results that the amount of oxygen increases as the number of recycling increases. However, a nano-hardness test is necessary to be able to characterize the hardness of this outer layer.

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