

Fragmentation and Branch Elimination of Primary α' Martensite in Additively Manufactured Ti-6Al-4V Alloy

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Abstract: Fragmentation and branch elimination are generally noticed in the conventionally processed Ti-6Al-4V. Such a key morphological change produces a positive change in certain mechanical properties. We, for the first time, observe fragmentation and branch elimination of the primary α' martensite in additively manufactured as-built Ti-6Al-4V and the effect of scan speed on these is studied. Nanovoids inside and on the surface of the primary α' martensite are assumed to be the starting points of the fragmentation and branch elimination, respectively. At a lower scan speed (250 mm/s) a relatively shorter branch length than that of a higher scan speed (500 mm/s) is observed. Such change in the morphology of primary α' martensite has positively impacted hardness. This has been discussed in terms of additive manufacturing parameters. Such a fundamental morphological change will further help the understanding of laser powder bed fusion metal additive manufacturing. The hardness of the samples is measured and correlated with the fragmentation of the primary α' martensite.

Keywords: additive manufacturing; Ti-6Al-4V; primary α' martensite; fragmentation; branch elimination; laser scan speed



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1. Introduction

Laser powder bed fusion (LPBF) is an additive manufacturing (AM) technique, also called 3D printing technology, which has been used for the manufacture of metallic components. It offers several unique advantages over other conventional manufacturing processes, such as flexibility in geometric design, rapid production of components with complex geometry and high spatial resolution, customisation of products at an acceptable cost (due to the lack of tooling), and little material waste through the recycling of unprocessed powder [1–3]. Components fabricated via LPBF under optimised manufacturing parameters can be almost fully dense and have equivalent mechanical properties compared with their wrought counterparts.

Numerous studies have been performed on the LPBF Ti-6Al-4V (Ti-64) alloy because of its wide range of applications in the biomedical and aerospace sectors. It has been found that LPBF processing parameters, e.g., laser power, scan speed, spot size, hatch space, and heat treatment, significantly affect the microstructure features and porosity of as-built Ti-64. Special attention has been paid to the defects, porosity, and powder features of LPBF Ti-64 [1–8]. During LPBF processing, phase transformation of phases in Ti-64 also occurs. Microstructure evolution during processing of Ti-64 alloy via AM has been studied [7,9–14]. The influence of laser scan speed on the microstructure features of LPBF Ti-64 is reported in [14,15]. Han et al. [16] reported that the size of prior β columnar grains increased with decreased laser scan speed, but they did not provide an explanation for

this. Microstructure studies show α' martensite as an inevitable part of AM Ti-64. Their types, morphology, dependence on the processing and role in mechanical properties have been elaborately discussed [17–20]. Morphologically, the primary α' phase resembles the lamellar microstructure of α phase in the conventionally processed Ti-64 alloy. Semiatin et al. and others [21–24] have established that the lamellar microstructure of the α phase breaks up or globularises both dynamically during deformation and statically during post-deformation annealing in the two-phase field. The AM process has all components including force by the spreading blade [25] and heat dissipation to the previous layers during cooling that can induce the atmosphere for breaking or globularisation of the primary α' phase. Surprisingly, there is no report of fragmentation and branch elimination of the primary α' martensite phase in the as-built Ti-64 alloy. However, spheroidisation of the α' phase is reported after post-processing [26,27]. We have for the first time observed the fragmentation of primary α' martensite in as-built AM Ti-64. Poor mechanical performance, especially tensile ductility of the AM Ti-64 alloy, is attributed to the primary martensite [28]. Fragmentation of the primary α' phase into smaller sized α' phases can be an important phenomenon for enhancing mechanical properties. It is mentioned that fragmentation of the primary α' phase facilitates easy movement of dislocation [29] and thus higher ductility and fatigue properties can be anticipated. The fragmentation mechanism of the primary α' phase is also illustrated. Finally, the influence of laser scan speed on the phenomenon is established. Hence, the aim of this work is to show the evidence of fragmentation of primary α' martensite and the effect of laser scan speed on the fragmentation of primary α' martensite of as-built LPBF Ti-64 alloys. Then, the impact of the changes in hardness resulting from the fragmentation of primary α' martensite is also observed.

2. Materials and Methods

Ti-64 ELI plasma atomised powders (LPW, Bristol, UK) with a size range of 20–60 μm and a 3D printing system (SLM 50, ReaLizer, London, UK) were employed to fabricate the Ti-6Al-4V samples with a dimension of $10 \times 10 \times 10 \text{ mm}^3$. SLM 50 has a 1070 nm yttrium scanning laser with a power range of 20–120 W, and a laser spot diameter of 50–100 μm . The experiment can be performed in an inert gas (argon, nitrogen) medium, with oxygen content below 0.4% during the process. The samples were vertically built on a $\varnothing 300 \text{ mm}$ Ti substrate. Laser processing parameters used in this study include a laser power of 85 W, layer thickness of 45 μm , hatch distance of 80 μm , and a scan speed of 250 and 500 mm/s. The scanning strategy follows a zigzag pattern with a rotation angle of 67° between adjacent layers. A relative density of over 98% was measured for both conditions studied here.

Following the manufacture of the samples, metallographic specimens were prepared by cutting the samples longitudinally, followed by polishing to observe the microstructure along the building direction. SiC grit papers and a mixture of colloidal silica and hydrogen peroxide were used in the polishing process. Samples were chemically etched using Kroll's reagent (2-4HF:2HNO₃:H₂O) to reveal the microstructures. A Field Emission Scanning Electron Microscope (FE-SEM, Carl Zeiss ULTRA, Oberkochen, Germany) equipped with an EDS detector at an accelerating voltage of 5 kV and a working distance of 5–6 mm was used to observe the fragmentation of the primary α' martensite.

In order to measure the Vickers hardness, the samples were initially polished to obtain a smooth and flat parallel surface before indentation testing was performed. Then, the polished samples were placed in a Zwick Roell hardness tester to measure the Vickers' hardness. The applied load was 0.5 kg for 30 s and at least 30 readings of different indentations were taken at room temperature to obtain the mean value.

3. Results and Discussion

3.1. Microstructure

Figure 1a shows several primary α' martensite in the sample processed at 250 mm/s. Both fragmentation (F) and branch elimination (B.E.) can be observed.

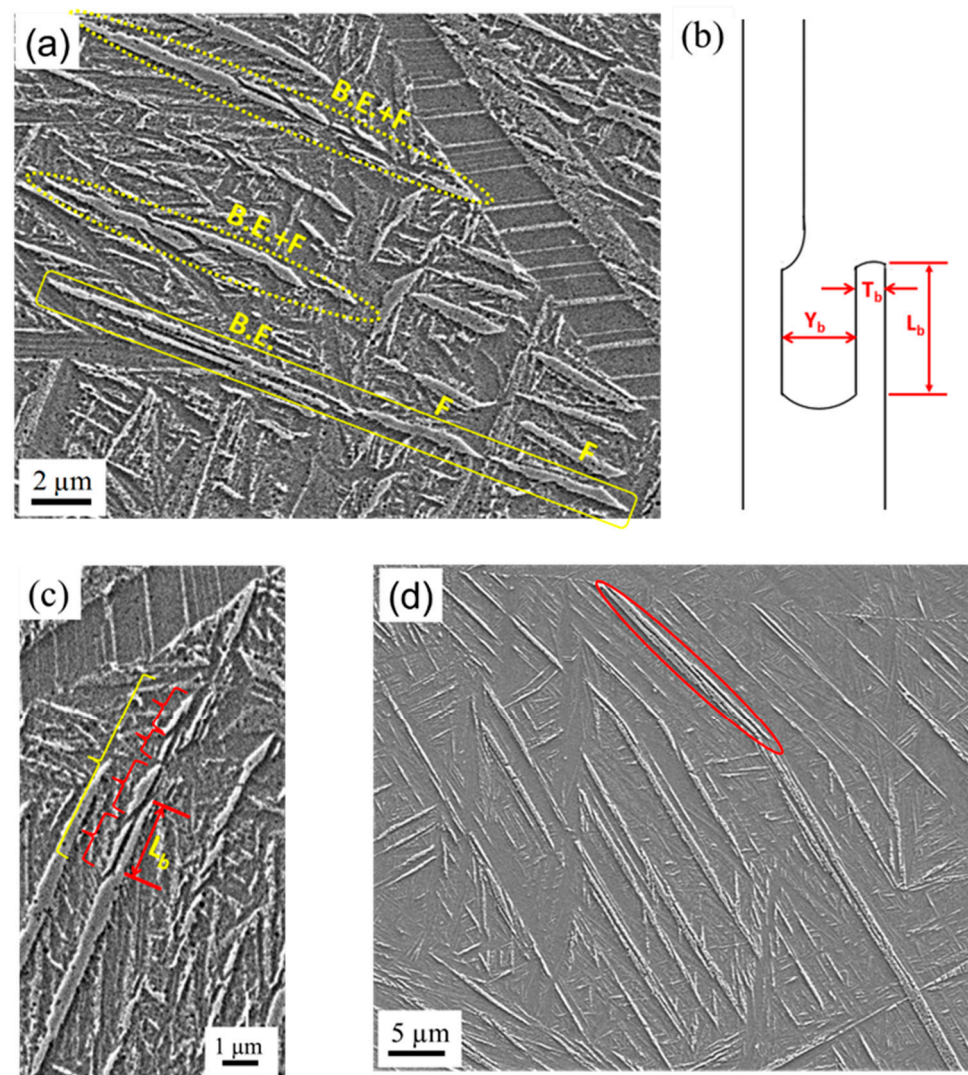


Figure 1. Fragmentation and branch elimination of primary α' martensite. Yellow coloured boxes in (a) showing the fragmentation (F) and branch elimination (B.E.) of primary α' martensite. (b) A schematic illustration of branch elimination. (c) A higher magnification image of one of the primary α' martensites showing that branch elimination and fragmentation occurred together. Red brackets indicate the resultant smaller-sized martensite. (d) Primary α' martensite at 500 mm/s, fragmentation and/or branch elimination are marked by red circles.

The fragmentation of a martensite into three smaller parts indicated by a rectangular box can be seen. Branch elimination, a phenomenon first realised by the Semiatin group [21,30] in Ti-64, is schematically shown in Figure 1b where L_b , T_b , and Y_b represent branch length, branch thickness, and spacing between the branch and the martensite lamellae to which it is detached, respectively. It is evident that the marked martensite in Figure 1a are the result of the combination of fragmentation and branch elimination. To clearly illustrate this, a closer view of one of the martensite lamellae in Figure 1a is shown in Figure 1c. Smaller-sized α' indicated by red parentheses resulting from branch elimination and fragmentation of larger α' can be seen here. In the sample fabricated at a higher scan speed (500 mm/s), several primary α' martensite lamellae can be seen in Figure 1d. Branch elimination is marked by the red ellipse and few fragmentations can be seen. It can be said, more pronounced fragmentation and branch elimination occurred in the sample processed at 250 mm/s than that in the sample at 500 mm/s. In addition, fewer primary α' martensite display fragmentation and branching, in the sample processed at 500 mm/s.

To understand the process of fragmentation and branch elimination, a higher magnification image was captured (Figure 2). Figure 2 shows two primary α' martensite lamellae with nanovoids highlighted by yellow colours. Nanovoids are arranged in such a way that they can merge and subsequently split the martensite lamellae into smaller sized α' . Rectangular yellow boxes show the near merge and near split conditions of these α' martensite. The red V-shaped free-form may indicate the commencement of the fragmentation.

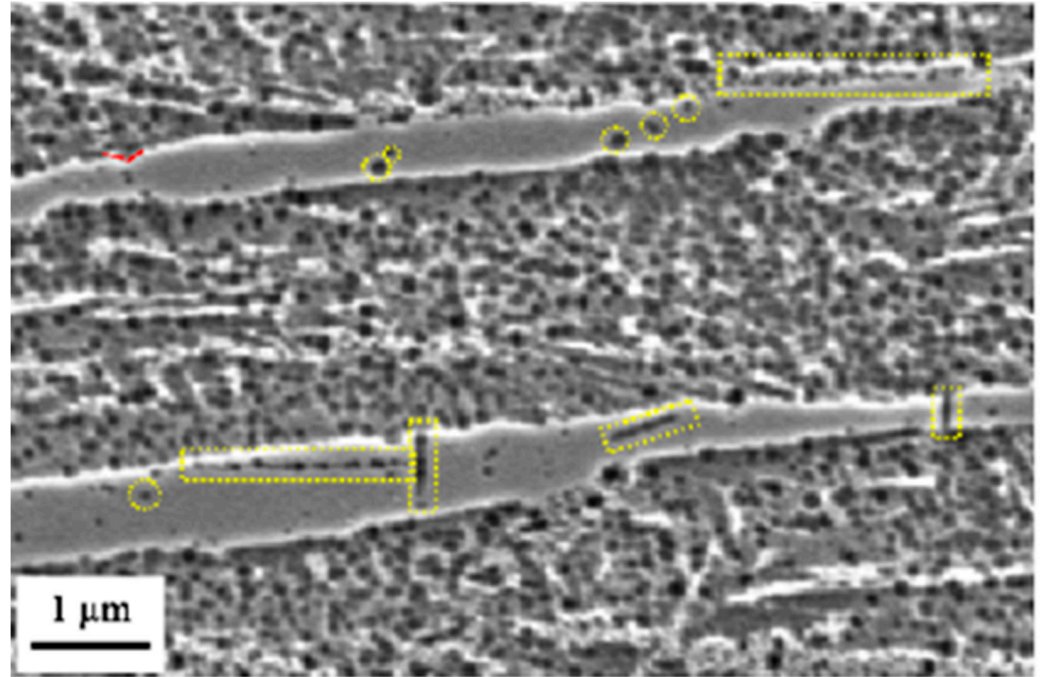


Figure 2. Process of fragmentation and branch elimination shown in the 250 mm/s sample. Nanovoids highlighted by circles and the merge of nanovoids and split of martensite are marked by rectangle boxes. Red V-shape showing fragmentation starting from the edge.

In this study, the morphological change of primary α' martensite resulting from a combination of fragmentation and branch elimination is predominantly observed in samples processed at the lower scan speed (250 mm/s). Therefore, it is discussed with respect to the laser scan speed, i.e., volumetric energy density (VED). The volumetric energy density (VED) is calculated using the following equation.

$$VED = P/v \cdot t \cdot h \quad (1)$$

Here, laser power is represented by (P), scan speed by (v), layer thickness by (t), and hatch spacing by (h). As mentioned in [6], higher VED means higher melt pool temperature. During cooling, heat transfers through conduction to the previous layers. The heat gained through conduction in the previous layers can be regarded as an annealing treatment. As such, the branch elimination rate can be correlated with different laser scan speeds used here. The branch elimination rate, $\frac{dL_b}{dt}$, can be calculated using the equation below [30].

$$\frac{dL_b}{dt} = \frac{\pi D C_F V_m \gamma}{R T T_b (Y_b + T_b/2)} \quad (2)$$

Here, D represents the diffusivity of the solute, C_F represents the composition factor, V_m represents the molar volume, γ represents the interface energy between the phases, R represents the gas constant, and T represents the absolute temperature. Assuming D ($= 0.052 \mu\text{m}^2/\text{s}$), C_F ($= 61.3$), V_m ($= 1.044 \times 10^{-5} \text{ m}^3/\text{mol}$), γ ($= 0.4 \text{ J/m}^2$), and R parameters are constant, Semiatin and Poteet [21] showed a higher branch elimination rate at 955 °C

(10 $\mu\text{m/h}$) than at 900 $^{\circ}\text{C}$ (4 $\mu\text{m/h}$) for Ti-64. Furthermore, the changes in the values of Y_b and T_b between the conditions are reported to be insignificant. Therefore, it can be claimed that the temperature is the only variable that determines the branch elimination rate, provided that all other parameters remain constant. Hence, a shorter branch length (L_b) is anticipated at a lower scan speed. In fact, our data support this argument, i.e., L_b of $2.8 \pm 0.4 \mu\text{m}$ (250 mm/s) vs. $6.2 \pm 0.3 \mu\text{m}$ (500 mm/s). Fragmentation, on the other hand, occurs on the same lamellae either prior to branch elimination or subsequently following the branch elimination. The fragmentation mechanism includes two steps (i) boundary splitting during the initial stage, and (ii) termination migration during the later stage [22,31]. Following the boundary splitting (V-shape free form in Figure 2), the termination migration stage commences through the merging of nanovoids. This stage is controlled by the diffusivity of the alloying elements [22,31,32]. In the context of laser powder bed fusion, the diffusivity is controlled by the melt pool temperature, cooling rate, and cooling time, indicating that a lower scan speed might have higher diffusivity as VED is higher at 250 mm/s and more time allows for diffusion of the alloying elements. Therefore, higher fragmentation is anticipated in samples fabricated at the lower scan speed, 250 mm/s. The discussion on fragmentation is validated with respect to the laser scan speed as more fragmentation is seen in the 250 mm/s sample. Finally, based on the experimental observation, a schematic illustration of the fragmentation and branch elimination during the process is shown in Figure 3. Based on the observation, two lamellae are drawn where one starts branch elimination followed by fragmentation and the other one is oppositely drawn. In Figure 3a, yellow dotted nanovoids in the martensite form in the initial stage; this can be seen in Figure 2. In the intermediate stage (Figure 3b), nanovoids in one α' martensite reached the other side of the martensite and fragmented α' into three, while nanovoids in other α' created a hole inside. Later, in the intermediate stage (Figure 3c), the large fragmented α' underwent branching, and the other α' suffered fragmentation and branching resembling that shown in Figure 1a. Figure 3d depicts the more advanced stage where both fragmentation and branching can be seen at a higher degree, and thus more fragmented particles are seen.

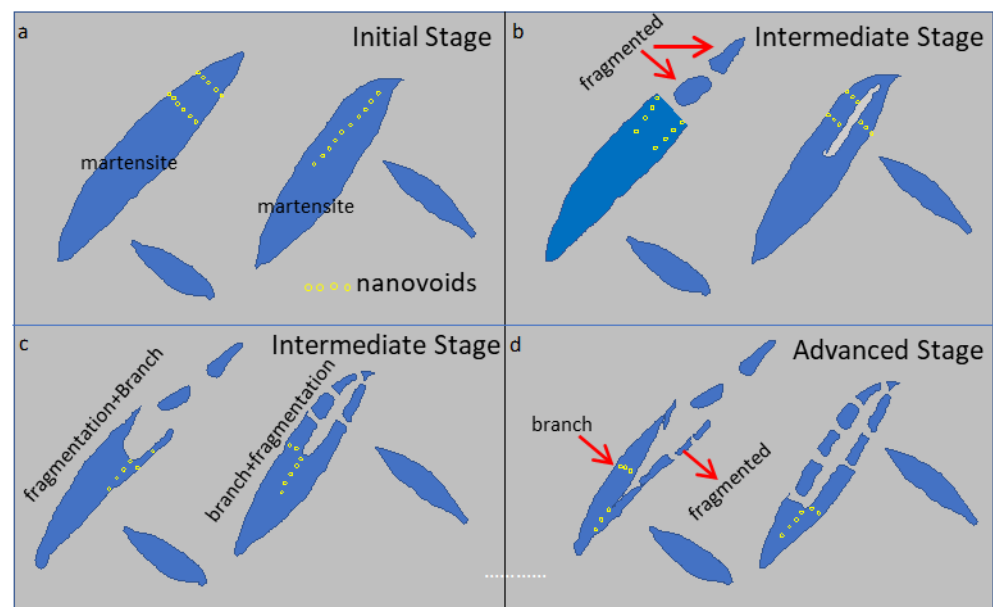


Figure 3. Schematic illustration of morphological changes of primary α' martensite during LPBF. Two α' lamellae showing the fragmentation and branch elimination process during LPBF. (a) shows the initial stage, (b,c) intermediate stages, and (d) advanced stage.

3.2. Hardness

Vickers hardness is measured to determine the influence of fragmentation of martensite on properties. Figure 4 shows the Vickers hardness number for the studied conditions. It is evident that the hardness value decreased with decreased scan speed. The lower hardness number for samples processed at 250 mm/s can be attributed to the morphological change caused by the fragmentation of the primary α' martensite. That means the measured higher degree of split of martensite into smaller sizes and solute redistribution at 250 mm/s affects the mechanical properties of Ti-64. These make the samples softer and thus the lower hardness number is obtained. On the other hand, samples fabricated at 500 mm/s showed fewer morphological changes that caused a higher hardness value. However, other potential factors, residual stresses, phase ratio/composition, presence of other microstructural constituents that can occur due to processing could also contribute to the hardness. In this study, residual stress can be neglected because of the small sample dimensions. The evolution of a new phase through phase transformation such as α' to α is anticipated; however, it is not possible to quantitatively measure the phase ratio of the phases due to the crystallographic conformity of the phases. In addition, there could be such phase transformation locally with a very small extent, and their effect on hardness is difficult to quantify and would be small. Therefore, we can assume that the change in hardness is predominantly due to fragmentation with minor contributions from the aforementioned factors.

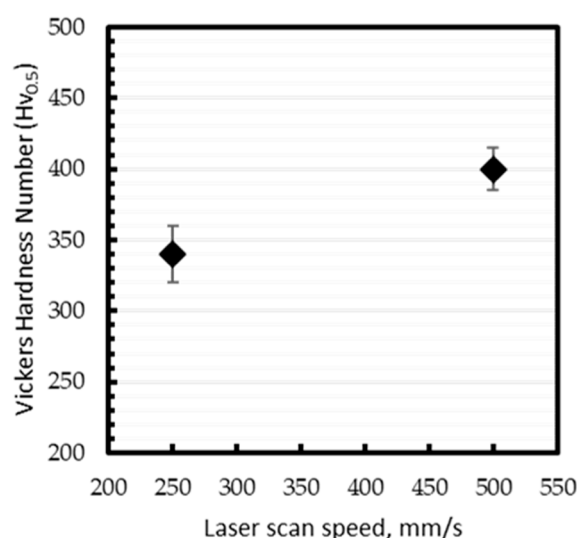


Figure 4. Vickers hardness number of samples plotted against the laser scan speed studied.

4. Conclusions

In this study, fragmentation and branch elimination were seen in additively manufactured Ti-6Al-4V. We found that nanovoids formed inside a primary α' martensite facilitate branch elimination, whereas nanovoids at the surface may act as the start of the fragmentation process. Both branch elimination and fragmentation were found to be more pronounced at 250 mm/s. As a result, a lower hardness value was recorded for samples processed at 250 mm/s.

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Data Availability Statement: The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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Conflicts of Interest: Authors Fionnan McNamara and Charles Tomonto were employed by the company Johnson & Johnson Services Inc.; Author Greg Duggan was employed by the company DePuy Synthes. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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