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Effects of heat treatment on microstructure and mechanical behaviour of additive manufactured porous Ti6Al4V

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Abstract. Titanium and its alloys such as Ti6Al4V play a major role in the medical industry as bone implants. Nowadays, by the aid of additive manufacturing (AM), it is possible to manufacture porous complex structures which mimic human bone. However, AM parts are near net shape and post processing may be needed to improve their mechanical properties. For instance, AM Ti6Al4V samples may be brittle and incapable of withstanding dynamic mechanical loads due to their martensitic microstructure. The aim of this study was to apply two different heat treatment regimes (below and above β -transus) to investigate their effects on the microstructure and mechanical properties of porous Ti6Al4V specimens. After heat treatment, fine acicular α' martensitic microstructure was transformed to a mixture of α and β phases. The ductility of the heat-treated specimens, as well as some mechanical properties such as hardness, plateau stress, and first maximum stress changed while the density and elastic gradient of the porous structure remained unchanged.

1. Introduction

Numerous products have been influenced by advent of additive manufacturing (AM). Complex designs and structures with a wide range of materials and sizes, which would have previously been just a fantasy, are nowadays available in the market, all thanks to AM [1]. AM itself has been evolved over time, changing from a simple method of prototyping to manufacturing high precision products in high-tech industries [2]. A wide range of materials are developed for AM from polymers to metals among which titanium has gained a remarkable position.

Given their outstanding mechanical properties, titanium and its alloys are widely used in various industries including the aerospace, automotive, and medical industries [3, 4]. In particular, titanium is a preferred material for fabrication of orthopaedic implants due to its three main advantages: lightness, biocompatibility, and strength [5]. Solid titanium implants remained unrivalled for decades, until the introduction of the new generation of porous titanium made by AM. AM porous implants have two substantial benefits: space for bone ingrowth and mimicking the mechanical properties of bone [6, 7]. Tailored AM porous titanium meets almost all the biomedical design requirements. However, it requires post-processing, since AM delivers near net shape and not the final product. So far, only a few studies have focused on the effects of different heat treatments on such porous structures. The aim of the current study is to investigate the effects of heat treatment on the microstructure and mechanical properties of AM Ti6Al4V porous structures. Two different heat treatment regimens (below and above β -transus) were selected and applied to the specimens. The resulting static mechanical properties such as the elastic gradient and plateau stress as well as the microstructure and hardness profile of the heat treated samples were compared with the as-processed material.



2. Materials and methods

Porous Ti6Al4V specimens with dimension of $\Phi 15$ mm x 20 mm were manufactured using a commercial selective laser melting (SLM) machine (SLM125, Realizer GmbH, Borchten, Germany) with the highest laser power of 400 W (IPG Photonics Corporation, Oxford, USA) and wavelength range of 1070 ± 10 nm. The specimens were designed using the regular diamond unit cell [8] with a unit cell size of 1 mm and an average relative density of 30.25 % (± 1.5), which was measured by the dry weighting technique.

Three different groups were defined:

1. *As-processed* samples which are considered as the reference material.
2. Samples heat treated at 800 °C, further denoted as *T800*. For this group, the *as-processed* samples were first stress relieved at 600 °C, followed by tempering at 800 °C for 4 hours.
3. Samples heat treated at 1050 °C, further denoted as *T1050*. For this group, the *as-processed* samples were first stress relieved at 600 °C and then heat treated at 1050 °C for two hours.

The heat treatments were performed under an inert atmosphere (Argon) with a heating rate of 10 °C/min, after which the furnace was allowed to cool down to the room temperature.

The mechanical properties of Ti6Al4V samples were determined using quasi-static compression tests with a static test machine (INSTRON 5985, 100 kN load cell). A constant deformation rate of 1.8 mm/min was applied to three specimens per group. The mechanical properties such as the elastic gradient, plateau stress, and first maximum stress were calculated in accordance with NEN ISO 13314:2011 [9]. Structure density, which is the ratio of the density of each strut to density of the same strut if it was 100 % solid, was calculated by measuring the internal pore area of the strut and deduct the number from the total surface area.

The micro-hardness measurements were carried out using an automated Vickers hardness machine, Dura Scan (Steurs) with a load of 0.3 Kgf. The hardness measurements were carried out along the cross section of the sample (XY plane) to analyze the influence subsequent layer deposition during SLM along the building direction.

The specimens were first ground (SiC 320 and MD Largo 9 μ m) and polished (MD Chem with OPS solution 0.04 μ m). Following which the samples were etched by immersion for 12-15 seconds in a mixture of 50 ml distilled water, 25 ml HNO₃ and 5 ml HF solution. The samples were observed using an optical microscope Keyence VHX5000.

X-Ray diffraction (XRD) patterns were obtained using Bruker D8 Advance diffractometer Bragg-Brentano geometry with graphite monochromator and Vantec position sensitive detector and CoK α radiation with the following measurement method: Diffraction patterns were scanned with 2θ range of 10 ° to 110 °, step size 0.034 ° 2θ and counting time per step 8 s.

3. Results and discussion

The compressive stress strain curves of *as-processed* and heat treated samples are illustrated in figure 1. From these graphs, static mechanical properties were extracted and presented in table 1. These results indicate that *as-processed* and below β -transus (*T800*) samples show similar mechanical behaviour. It should however be noted, that there is a slight difference in trend of the stress-strain curve after 0.5 compressive strain (figure 1). The reason behind this trend could be the effect of non-optimised processing parameters, which is out of the scope of this paper and will be presented in our follow up publication. Structure density of three groups were in the same range, indicating that another post treatment methods such as hot isostatic pressing (HIP) should be applied in order to reduce process induced porosity and defects [10].

In contrast with the first two groups, the specimens heat treated above the β -transus (*T1050*) showed different mechanical behaviour. Although the elastic gradient is in the same range as for the two other groups, however, the first maximum stress shows significant decrease while the plateau stress substantial increase.

The other main difference is the energy absorption (the energy required for deforming a specimen up to 50 % strain, i.e. area under the strain-stress curve) which is higher for *T1050* specimens. The

obtained results indicate a ductile behaviour of *T1050* samples, which can be further explained by looking at their microstructure.

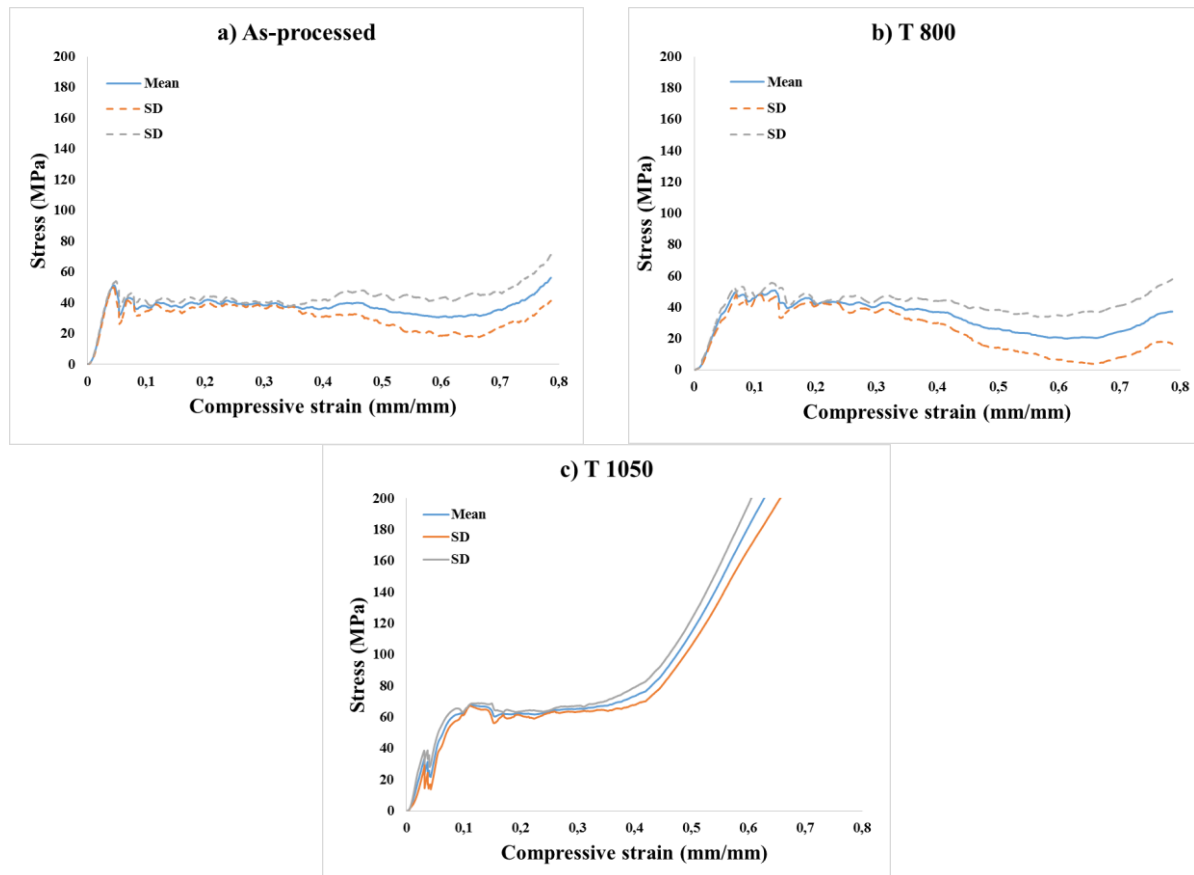


Figure 1. Compression stress-strain curve of a) *as-processed*, b) *T800* and, c) *T1050*

A fully fine acicular α' martensitic microstructure was observed in *as-processed* samples (figure 2 a) and XRD data (figure 3) did not reveal any β phase. After stress relieving at 600 °C followed by heat treatment at 800 °C (*T800*) the martensitic fine α' structure transformed to a combination of α and β phases. As shown in table 1, the β -fraction for *T800* samples is about 10 %. Comparing figure 2 a and figure 2 b, it is observed that α plates are coarser changing from less than 1 μm width to about 4 μm . Heat treatment at 1050 °C (*T1050*) results in a larger amount of β -fraction (table 1), which is in a good agreement with the bulk AM samples [11]. According to literature and phase diagrams [12], only β phase is expected when heating up the specimens above 995 °C (β transus). However, once specimens are furnace cooled, a mixture of lamellar $\alpha+\beta$ appears (Figure 2.c). It should also be noted that a further coarsening of α plates is observed for this heat treatment regime.

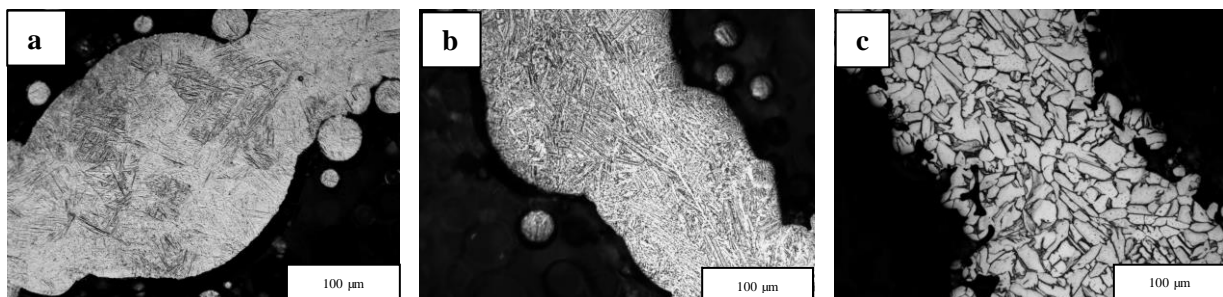


Figure 2. Microstructure of a) *as-processed*, b) *T800* and c) *T1050*

XRD data show significant peak shifting of *as-processed* and heat treated samples (figure 3). Peak shifting in the XRD data for the heat treated samples can be attributed to the formation of the equilibrium phases consisting of a two phase lamellar ($\alpha+\beta$) structure. The transformation occurs from the non-equilibrium α' phase in the *as-processed* condition (due to the high cooling rate involved in the SLM process) to the equilibrium α phase for the heat treated samples [11]. This transformation can explain the decrease in the width and increase in the intensity of the XRD peaks. It should also be noted, that peak shifting might also be effected by residual stress, which is rather unlikely since *as-processed* and stress relieved (T800) samples do not show any significant differences (see an enlarged insert in Figure 3). For the two heat treated T800 and T1050 samples, a peak shift was observed on the specimens surface (Figure 3). However, there were no significant peak shifting observed when measurements were performed in cross section (in core) of the heat treated sample (Figure 3). This could be due to the high temperature involved in the heat treatment resulting in a possible solute redistribution from the surface which causes a change in the lattice parameters for the two phases and hence the peak shift for the 1050 °C heat treated sample at the surface to lower 2θ angles.

Another important observation from Figure 3 is the reduction in the peaks width of the heat treated samples when compared to the *as processed* material, which is most likely attributed to the increased crystallinity size observed in Figure 2 [13].

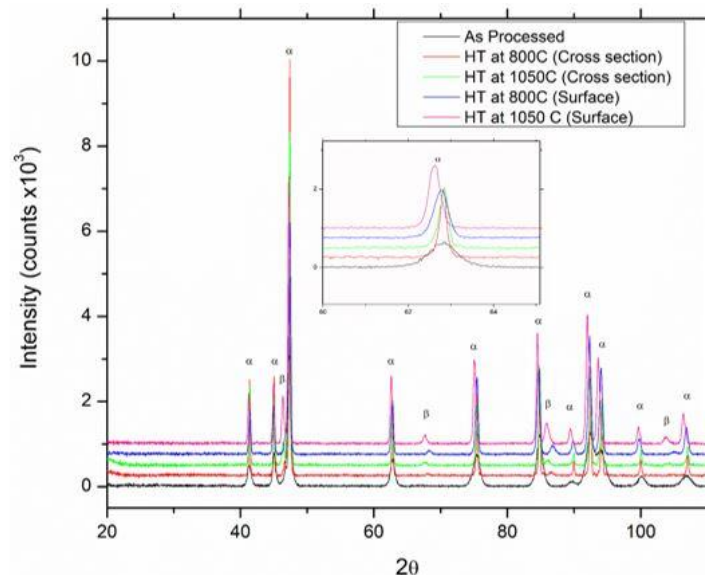


Figure 3. XRD measurement of *as-processed*, T800 and T1050.

Finally, the results of hardness measurement showed reduction from 412 (± 23) of *as-processed* samples to 353 (± 34) for T1050. Hardness reduction can be explained by microstructural evolution from needle-shape α' martensitic structure to a mixture of $\alpha+\beta$ and by increment of β phase, shown in table 1).

Table 1. Mechanical properties

	Elastic gradient (SD) GPa	Hardness (SD) HV _{0.3}	Plateau stress (SD) MPa	Max stress (SD) MPa	β fraction (SD) %	Structure density (SD) %
<i>As-processed</i>	1.65 (0.046)	412.24 (23)	38.53 (0.63)	52.35 (1.91)	0	98.96 (0.74)
<i>T800</i>	1.60 (0.058)	388.15 (45)	42.44 (2.24)	52.50 (1.17)	9.6 (0.6)	98.15 (0.47)
<i>T1050</i>	1.49 (0.22)	353.25 (34)	66.34 (2.63)	38.41 (1.85)	20.4 (0.5)	98.61 (0.58)

4. Conclusion

The effect of different heat treatments (below and above β -transus) on microstructure and mechanical properties of porous additive manufactured Ti6Al4V structures was studied. It was observed that heat treatment substantially changes the microstructure of *as-processed* Ti6Al4V samples. XRD data showed peak shifting for both heat treatment regimes. However, the only considerable mechanical properties alteration was for plateau stress of *T1050* (heat treated above β -transus). Hardness profile showed reduction by incensement of temperature. The selected post treatment regimes produced a mixture of $\alpha+\beta$ phases and as previously observed [14] $\alpha+\beta$ interface is an effective barrier of crack propagation, which makes these structures attractive for load bearing applications. It is suggested to carry out further fatigue tests in order to investigate the impact of microstructure modification on dynamic mechanical properties of porous Ti6Al4V structures. Also, it is concluded that structure density of AM structures does not improve by heat treatment and in order to solve this problem other post treatments method such as hot isostatic pressing (HIP) must be considered.

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