INVESTIGATION OF MICROSTRUCTURAL CHARACTERISTICS OF HEAT TREATED HIGH SPEED SELECTIVE LASER MELTING FABRICATED TI6AL4V COMPONENTS

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ABSTRACT

This study presents an investigation of the effect of different heat treatments on the microstructure of Ti6Al4V fabricated components produced by high speed selective laser melting. 10 mm x 10 mm x 10 mm cubes were heat treated in an inert oven at temperatures of 700 °C and 950 °C. The samples were heat treated at their respective temperatures for 2 hours before air cooling to room temperature. Subsequent to heat treatment, the microstructures of the heat treated samples and the as-built were studied. The as-built sample showed a martensitic alpha phase which was also observed on the 700 °C sample. Elongated columnar grains were observed on the as-built and the 700 °C heat treated sample. Furthermore, a fully transformed lamella α+ß microstructure was observed for the 950 °C heat treated sample.

1. INTRODUCTION

Additive manufacturing (AM) is currently a topic of interest in the aerospace industry due to its various advantages over conventional methods. These advantages include possibility of manufacturing parts of any geometric complexity, great design freedom and reduced production time, **Galti et.al** [1]. There are various types of AM techniques/platforms which are currently being used. These platforms include powder beds, direct energy deposition, material extraction and sheet laminating, **Gockel et.al** [2]. This study focus on selective laser melting (SLM), a powder bed AM technique which build components by selectively melting powder layer-by-layer with focused laser beam, **Vrancken et.al** [3]. The advantages of SLM include high level of flexibility (able to change easily according to situation), material efficiency, near net shape production and the build-up of complex geometries in a very short period of time, **Vrancken et.al & Eylon et.al** [3-4]. Various materials can be used in AM processes including Titanium alloys [2].

Titanium alloy grade 5 (Ti6Al4V) is an attractive material for aerospace industry because of its light weight, high strength properties and excellent corrosion resistance at high temperatures, **Eylon et.al & Wanying et.al** [4-5]. Ti6Al4V was developed in early 1950s for aerospace application and it is now qualified and widely used. Ti6Al4V is widely used in the aerospace industry because it possesses high specific strength and high corrosion resistance at temperatures up to 500°C, **Yaidroitsev et.al & Vilaro et.al** [6-7]. It has a crystal structure of α+ß, which allows full transformation to beta phase during heat treatment and transform back to α+ß when cooling [5].

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Another interesting feature of Ti6Al4V powder is that it has an excellent combination of ductility and low weight ratio which makes it a suitable alloy for aerospace industry, **He et.al** [8]. Ti6Al4V is used for high performance engineering solutions in aerospace motor cases, aircraft turbines, pressure vessels and marine components, **Imam et.al** [9-10].

Ti6Al4V component produced from SLM, in the as-built state, are unable to achieve high material performance to wrought counterparts (**Ter-Haar et.al** [11]) for aerospace application. This is because Ti6Al4V component produced from SLM exhibit α' phase which is associated with residual stresses that are generated during processing, **Zaeh et.al** [12]. These stresses are as a result of high temperature gradients which form during fabrication. **Kong et.al** [13] investigated the microstructure of Ti6Al4V samples produced by SLM and found that it exhibited fine martensitic α' phase with tensile strength of about 1.45 GPa and elongation of 4,4%. **Thijs et.al** [14] found that Ti6Al4V materials processed by SLM shows a very fine, non-equilibrium microstructure due to high temperature gradients. Post production heat treatment is one of the methods that can be used to relieve these residual stresses that result in a part post production.

Several studies [3, 5, 7, 11, 15] showed that heat treatment can improve the microstructure and mechanical properties of SLM Ti6Al4V components. The Ti6Al4V microstructures of the produced components were complex and differed within a built. The types of microstructures depend largely on the heat treatment temperature and the cooling rate. It is possible to achieve a highly desirable microstructural features such as a highly refined precipitate, **Collins et.al** [16]. The aim of this paper was to investigate the effect of heat treatment on the microstructure of Ti6Al4V produced by a high speed SLM system.

2. METHODOLOGY AND RESULTS

2.1 Methodology

Gas atomized Ti6Al4V powder with a particle distribution of 20-60 µm was used in this study. The powder was supplied by TLS and used as received. Ti6Al4V samples were produced with energy densities of 42.41 J/mm^3 and 78.46 J/mm^3 using the high speed SLM system available at CSIR (NLC). The energy densities were calculated using this formula $E=\frac{P}{hvt}$ where P is the laser power, h is the hatch spacing, v is the speed and t is the layer thickness. A schematic diagram of the SLM process is shown in Figure 1. During processing, a CAD model of the part is loaded onto the machine working station where a pre-processing software slices the model into layer of finite thickness. A powder is deposited onto a plate above the built platform then a focused laser beam scans over the powder bed based on the sliced CAD data. The scanning results in localised melting and solidification of the powder form layer of the part. Subsequent layers are built one over the other by lowering the platform equivalent to the layer thickness until a part is completed.

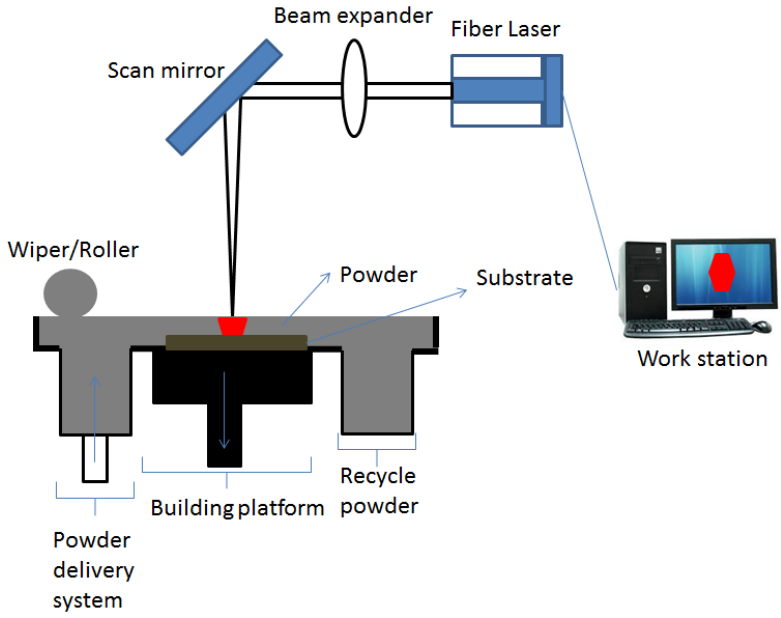

Figure 1: A schematic diagram of SLM process.

During the printing process 10 mm x 10 mm x 10 mm cubes of Ti6Al4V samples were produced by scanning very fast the focused high power fibre laser beam with wavelength of 1073 nm over the pre-placed powder bed. Post

manufacturing, the produced samples were then heat treated in an air rich oven at temperatures of 700 °C and 950 °C, respectively. The temperatures were chosen on the context that 700 °C is a stress relieving temperature and 950 °C is the ß-transus temperature, **Vracken et.al** & **Dong et.al** [3, 17]. During heat treatment the condition was such that the set temperature was held for 2 hours. The samples were allowed to air cool to room temperature.

The as-built and heat treated samples were mechanically prepared and polished for microstructural analyses. The samples were cross sectioned in direction perpendicular to the built direction. The samples were then etched with Kroll's reagent before metallographic examination. Metallurgical samples (as-built and heat treated) were analysed for microstructural evolution using Joel JSM-6010PLUS/LA Scanning Electron Microscope (SEM) with an accelerating voltage of 20 kV.

2.2 Results

2.2.1 Microstructures

Figure 2 below shows microstructures of the as-built samples.

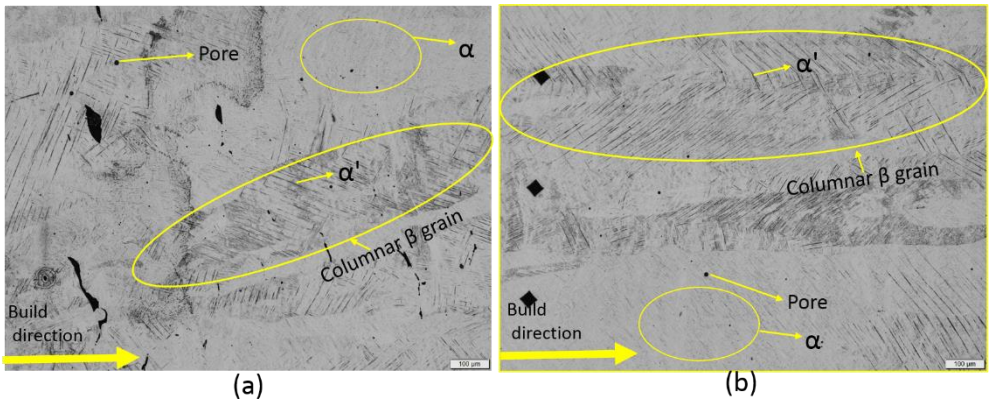

Figure 2: Microstructures of the as-built samples: (a) 42.41 J/mm^3 and (b) 78.46 J/mm^3.

Both samples show large columnar grains as highlighted on the optical images. These grains are orientated along the build direction. Additionally, both samples show an acicular martensitic α' phase which forms as a result of high cooling rates. Figure 2a shows regions where there are sharp crack-like pores which are attributed to the low energy density that was used. The crack-like pores are not observed on the microstructure sample in Figure 2b which was manufactured a high energy density. Both of the samples show the presence of three phases (α, ß and α'), but ß phase is present in small quantity. These observations are detailed in the high magnification SEM microstructures that are presented in Figure 3.

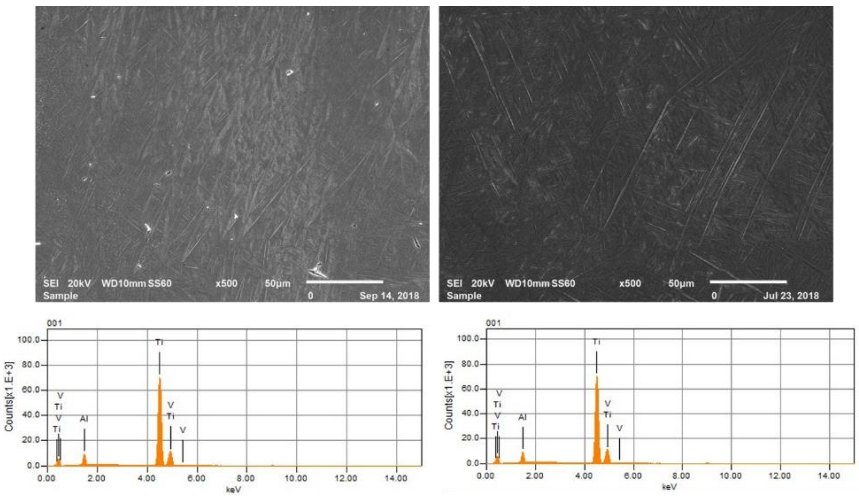

Figure 3: EDS graphs (a) 42.41 J/mm^3 and 78.46 J/mm^3.

From Figure 3, α' martensitic structure are rods or needle like structures that are seen on the image. This phase is somewhat precipitated with higher energy density (Figure 3b). The bright-white particles on the image are ß-

phase while α is the overall darker matrix of the sample. The microstructures of the heat treated samples are given in Figures 4 and 5 for 700 °C and 950 °C, respectively.

The microstructures of the samples that were heat treated at 700 °C.

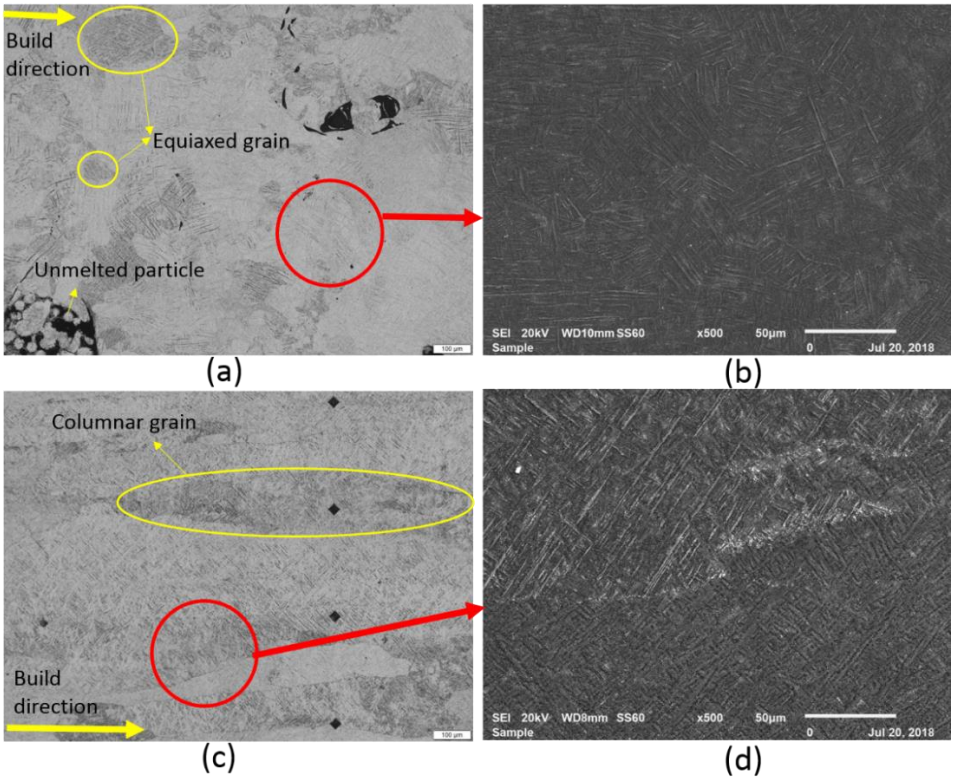

Figure 4: Micrographs of the samples after heat treated at 700 °C: (a and b) 42.41 J/mm^3 and (c and d) 78.46 J/mm^3.

Heat treatment at 700 °C, for 2 hours followed by air cooling, resulted in no phase transformation, however the grain sizes of the sample that was manufactured with energy density of 42.41 J/mm^3 (Figure 4a) were refined to smaller size and became more equiaxed as compared to the as-built (Figure 2a). This is due to nucleation when ß columnar grain changed to ß equiaxed grains. The sample that was built at 78.46 J/mm^3 and heat treated to 700 °C seem to remain the same since no grain growth or nucleation can be reported, however, the SEM microstructure clearly depict that at this heat treatment temperature ß phase was highly precipitated and the α'martensitic phase grew longer in the rich ß-precipitated columnar grain (top of the b-image) and shorter on the α grain (bottom part of image b). In both samples all three phases can still be identified. The microstructures of the sample after heat treatment at 950 °C are shown in Figure 5.

Figure 5 below shows the microstructures samples after heat treatment at 950 °C.

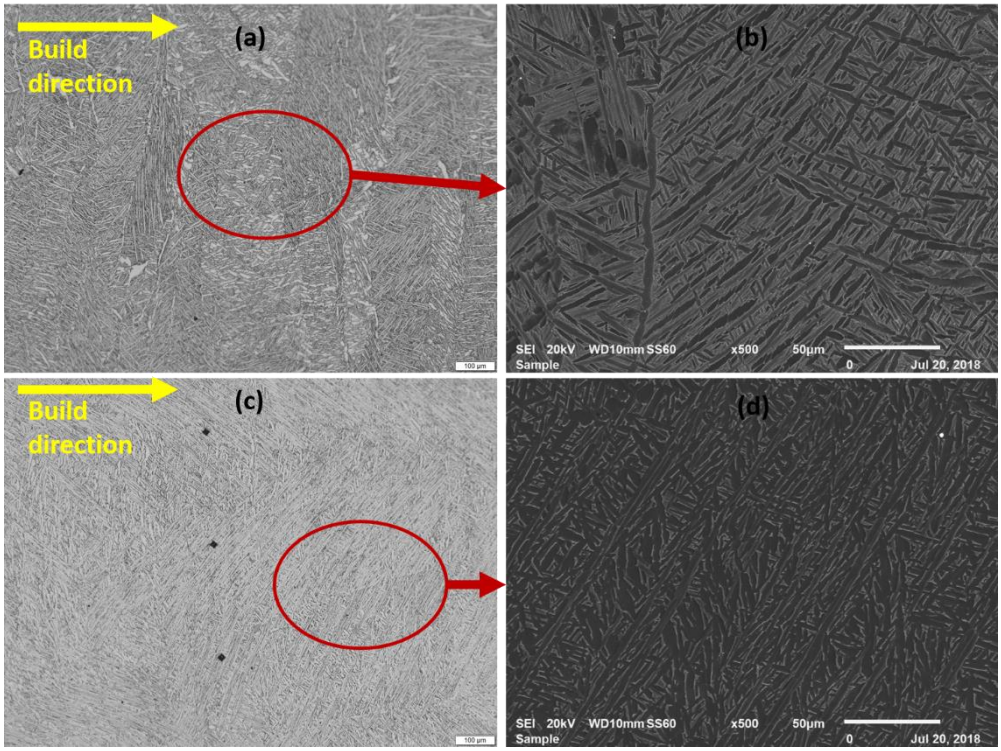

Figure 5: Micrographs of the samples after heat treated at 950 °C: (a and b) 42.41 J/mm^3 and (c and d) 78.46 J/mm^3.

Heat treatment at 950°C altered the grain sizes and phases for both samples. Full phase transformation was observed on the microstructures of the two samples. α' martensitic phase present in both the as-built samples (Figure 2a & 2b) and samples that were heat treated at 700 °C (Figure 4a & 4c) can no longer be observed after heat treatment at 950 °C as shown on Figures 5a & 5c. It has been cited that when Ti64 is heat treated at ß-transus temp or above α' martensitic phase will fully transform to ß phase. This transformation leads to a lamellar α+ß structure being formed (**Vracken et.al & Vilaro et.al** [3, 7]) as seen on Figures 5 (a and b). Study by **Vrancken et. al** [3] has shown that when Ti6Al4V is heat treated above the ß-transus temperature, it would homogenise, and all the beta phase present will transform to lamella α+ß during furnace cooling. They also observed grain growth with increase in heat treat temperature. Similar results [Figure 4d (700 °C) and Figure 5d (950 °C)] were observed in this study when temperature was increased.

2.2.2 Hardness

Hardness is the measure of strength of material and is influence main by material consolidation and homogeneity. Meanwhile a sample that underwent heat treatment will have its hardness influence by formed grains, precipitates and the resulting phases. The Hall-Petch relationship is the accepted relationship that explains the influence of mechanical] properties to the grains of the alloy (**Malheiros et.al & Ruan** [18-19]). This background is necessary for the interpretation of the hardness results presented in this study. Figures 6 and 7 show the hardness profiles of the samples in both as-built and heat treated conditions.

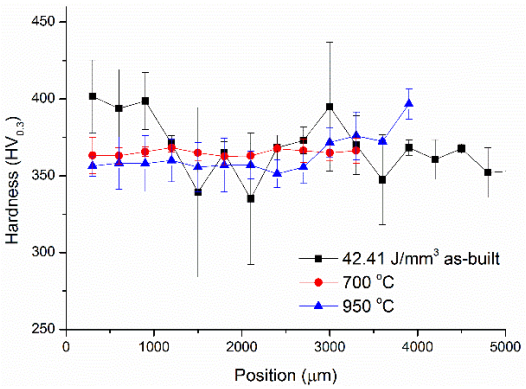

Figure 6: Average hardness profile of 42.41 J/mm³ sample.

Figure 6 presents the micro-hardness profile of the as-built sample and its corresponding heat treated samples. The sample was built using 42.41 J/mm³ energy density, and heat treated at 700 °C and 950 °C followed by air cooling, respectively. Hardness was measured across the sample perpendicular to the build direction. The hardness profile of the as-built sample is wavy. This observation can be attributed to the microstructural inhomogeneity, defects, and also might be due to the observed α' martensitic phase. The hardness profile of the heat treated samples remained constant. This might be due to a homogenised microstructure, and the refinement α' martensitic and its partial transformation to ß-precipitate. It should be noted that the hardness profile of the sample that was heat treated at 950 °C is a bit lower than the sample that was heat treated at 700 °C. This can be attributed to the over refinement in the microstructure and full transformation of α' to ß leading to a lamellar α+ ß microstructure.

The micro-hardness profiles of the sample that was built at 78.46 J/mm³ energy density and its corresponding heat treated samples are reported in Figure 7.

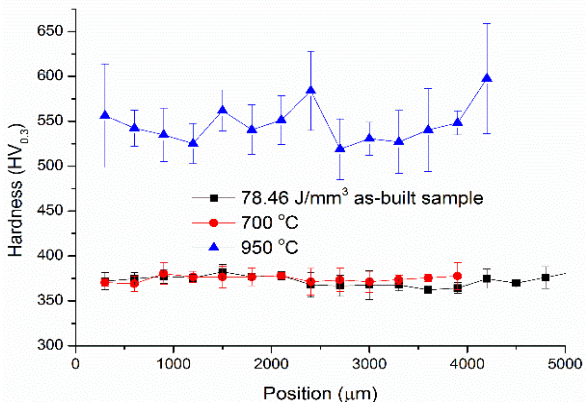

Figure 7: Average hardness profile of 78.46 J/mm³ sample.

It can be said from Figure 7 that the hardness profile of the as-built sample and its heat treated sample (700 °C) are the same. Both samples had similar microstructure. The hardness profile of the sample that was heat treated at 950 °C is wavy and significantly increased when compared to the hardness of the as-built sample and the sample that was heat treated at 700 °C. The increase in hardness cannot be attributed to the presence of α' phase as it was fully transformed. **Dong et. al** [17] reported a similar jump in hardness and attributed it to the thermal oxidation. This study would investigate the effects of thermal oxidation and its impact on the hardness of the Ti64 alloy as a future study. The overall hardness values of the samples are summarised in Table 1 with the error.

Table 1: Overall hardness ($HV_{0.3}$) values.

Sample ID	As-built	700 °C	950 °C
42.41 J/mm^3	367±30	365 ±30	363±30
78.21 J/mm^3	377±30	375 ±30	547±30

Table 1 summarises that there was no significant difference in the as-built and heat treated sample for the sample that was built at 42.41 J/mm^3, but a significant jump was observed in the sample that was heat treated at 950 °C for the sample that was built at 78.21 J/mm^3.

3. CONCLUSION

High Speed SLM process was used to produce 10 mm x 10 mm x 10 mm cubes Ti6Al4V samples. The effect of heat treatment on Ti6Al4V microstructure was investigated. It was observed that the resulting Ti6Al4V microstructure after annealing depend mainly on the heat treatment temperature. It was also observed that grain size increases with heat treatment temperature for the same cooling method. It was also observed that heat treatment at 700 °C does not allow full α' transformation but 950 °C allow full phase transformation to lamella α+ß.

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REFERENCES

[1] **Galti, M, Luliano, L**, 2018, A literature review of powder-based electron beam melting focusing on numerical simulations, *Additive Manufacturing*, 19, pp 1-20.
[2] **Gockel, J, Beuth, J**, 2012, Understanding Ti6Al4V Microstructure Control in additive Manufacturing via Process Maps, *Department of Mechanical Engineering*, Carnegie Mellon University, PA15213, pp 666-674.
[3] **Vrancken, B, Thijs, L, Kruth, J, Humbeeck, J**, 2012, Heat treatment of Ti6Al4V produced by Selective Laser Melting: Microstructure and mechanical properties, *Journal of Alloys and Compounds*, 541, pp 177-185.
[4] **Eylon, D, Newman, JR, Thorne, JK**, 1990, Metal Handbook, 10th ed. Material Park, OH: ASM Int, vol 2.
[5] **Wanying, L, Yuanhau, L, Yuhai, C, Taihe, S, Singh A,** 2017. Effect of different heat treatment on microstructure and mechanical properties of Ti6Al4V titanium alloy, *Rare metal Materials and engineering*, 46(3), pp 0034-0039.
[6] **Yaidroitsev, J, Krakhmalev, P, Yaidroitsava, I**, 2014, Selective Laser Melting of Ti6Al4V alloy for biomedical application: Temperature monitoring and microstructural evolution, *Journal of Alloys and Compounds*, 583, pp 404-409.
[7] **Vilaro, T, Collin, C, Bartout, JD**, 2011, As-Fabricated and Heat- Treated Microstructures of the Ti6Al4V Alloy Processed by Selective Laser Melting, *Metallurgical and Materials Transactions*, 24A, PP 3190-3199.
[8] **He, B, Wu, W, Liang, Z, Lu, L, Yang, Q, Long, Q, Chang, K**, 2018, Microstructure characteristic and mechanical property of Ti6Al4V alloy fabricated by selective laser melting, *Vacuum*, 150, pp 79-83.
[9] International Titanium Association, <www.titanium.org.>
[10] **Imam, MA, Franker, AC, Brown, SA, Lemons, JE**, 1996, Medical applications of Titanium and its Alloy, *ASTM Special Technical Publication*, pp 3-16.
[11] **Ter-Haar, GM, Becker, TH**, Selective Laser Melting Produced Ti6Al4V: Post-Process Heat Treatment to Achieve Superior Tensile Properties, *Materials*, 2018, 146, pp 1-15.
[12] **Zaeh, MF, Btanner, G**, 2009, Investigation on residual stresses and deformations in selective laser melting, *German Academic Society for Production Engineering*, 4, pp35-45.
[13] **Kong, CJ, Tuck, CJ, Ashcroft, A, Wildman, RD, Hauge, R**, 2011, High density Ti6Al4V via SLM processing: microstructure and mechanical properties, *proc. Of Solid Freeform Fabricated Symposium*, pp 337-483.
[14] **Thijs, L, Verhaeghe, F, Craeghs, T, Van Humbeeck, J, Kruth, J**, 2010, A study of the microstructural evolution during selective laser melting of Ti6Al4V, *Acta Materialia*, 58, pp-3303-3312.
[15] **Semiatin, S, Knisley, SL, Fagin, PN, Zhang, F, Barker, DR**, 2003. Microstructural Evolution during Alpha-Beta Heat Treatment of Ti6Al4V, *Metallurgical and Materials transections*, 34A, pp 2377-2386.
[16] **Collins, PC, Brice, DA, Samini, P, Ghamarian, I, Fraser HL**, 2016, Microstructural control of Additive Manufactured Metallic Materials, *Annual Review of Materials Research*, 46, pp 63-91.

[17] **Dong, E, Yu, W, Cai, Q**, 2017, Alpha-case kinetics and high temperature plasticity of Ti6Al4V alloy oxidized in different phase regions, *Procedia Engineering*, 207, pp 2149-2154.
[18] **Malheiros, LR, Figueredo, RB, Langdon, TG**, 2015, Grain size and microhardness evolution during annealing of a magnesium alloy processed by high-pressure torsion, *Journal of Material Research and Technology*, 4(1), pp 14-17.
[19] **Ruan, Y, Mohajerani, A, Dao, M,** 2016, Microstructural and Mechanical property Manipulation through Rapid Dendrite Growth and Undercooling in an Fe-based Multinary alloy, *Scientific report*, 6:1038, pp 1-11.