

DEPENDENCE of MICROSTRUCTURE and MECHANICAL PROPERTIES on HEAT TREAT CYCLES of ELECTRON BEAM MELTED Ti-6Al-4V

S. Raghavan, Mui Ling Sharon Nai*, Pan Wang, Wai Jack Sin, Tao Li, Jun Wei**

Singapore Institute of Manufacturing Technology (SIMTech), 73 Nanyang Drive, Singapore
637662

Corresponding author:* Email: mlnai@simtech.a-star.edu.sg, Phone: +65 6793 8976

** Email: jwei@simtech.a-star.edu.sg, Phone: +65 6590 3186

Abstract

The EBM Ti-6Al-4V alloy has generally superior mechanical properties, owing to finely spaced α - β laths which give a good combination of strength and ductility. The grain structures in the as-printed structures are long columnar which can give rise to anisotropic mechanical properties. Moreover the non-uniformity in microstructure can also arise from part geometry where the thin features have propensity to form martensite phase. Heat treatment provides a viable solution to modify the microstructure and to tailor to the properties as desired. A wide range of heat treatment experiments were performed, followed by microstructure and tensile property analyses. It was observed that the microstructure and the tensile properties significantly changed depending on the heat treat cycle performed. Tensile properties of solution treated air-cool plus aged samples yielded globular equiaxed grains with fine α - β lath structure, which were found to be the best among the different heat treated samples and better than ASTM F1472 specifications.

Introduction

Electron Beam Melting (EBM) machine developed by Arcam AB, is capable of producing near net shape complex parts and fully dense Ti-6Al-4V with very fine α - β lath microstructure. The superior microstructural features in the EBM printed parts are mainly due to preheat scan carried out to maintain the powder bed at 650°C-700°C, which transforms the as-printed martensitic α' structure to an annealed fine α - β lath structure [1, 2]. However, non-uniformity in the microstructure and mechanical properties are observed with regard to the part's height [3], part's dimensions [4] and part's geometries [1] due to the different thermal conditions experienced during the build process. Moreover, the high thermal gradients developed during solidification produce long columnar grains of a few mm length along the build direction [1, 5] which can also contribute to anisotropic properties in the printed part [5].

Heat treatment provides a viable method to modify and tailor microstructures to alter the mechanical properties for the applications desired. There has been very limited study on post heat treatment of EBM printed Ti-6Al-4V to improve the properties other than the HIP treatments, possibly because of the superior mechanical properties of the EBM printed parts and any standard

heat treatment does not improve the properties significantly as recently published by Charlotte *et al.* [6]. On the other hand, selective laser melting printed Ti-6Al-4V parts are completely martensitic and require a compulsory post heat treatment operation to improve the properties [7]. In the current research, a comprehensive set of heat treatment cycles were designed to study the influence of the process on the microstructure and eventually the tensile properties. The best heat treatment practice of EBM Ti-6Al-4V was also identified which gives refined microstructures for superior tensile properties in the alloy.

Materials and Methods

The samples for post heat treatment study were fabricated in Arcam A2X EBM machine using the Ti-6Al-4V Grade 5 powders with powder size of range 45-105 μm and build layer thickness of 50 μm . Rectangular bars (10 x 30 x 100 mm) parallel to build direction (Z) and perpendicular to build direction (XY) were fabricated as schematically shown in Figure 1, with the build direction marked by an arrow. The bars were later machined to ASTM E8 dimensions for tensile testing under the as-printed and post heat treatment conditions. The heat treatments were carried out in a vacuum furnace (Thermal Technology LLC) which can attain high vacuum levels of $1\text{e-}5$ torr. For samples that required air cooling, the samples were vacuum sealed in a quartz tube and heat treated in a Nabertherm muffle furnace. For microstructural observation, the samples were ground, fine polished and etched using Kroll's reagent (2% HF, 4% HNO_3 in water). Images were taken using the Olympus GX51 optical microscope for heat treated samples and Zeiss Field Emission Scanning Electron Microscope (FESEM) for as-printed sample, since the α lath morphology is very fine. The tensile tests were also carried out using the Instron 5982 machine with the strain rate of $3.3\text{e-}4 \text{ s}^{-1}$.

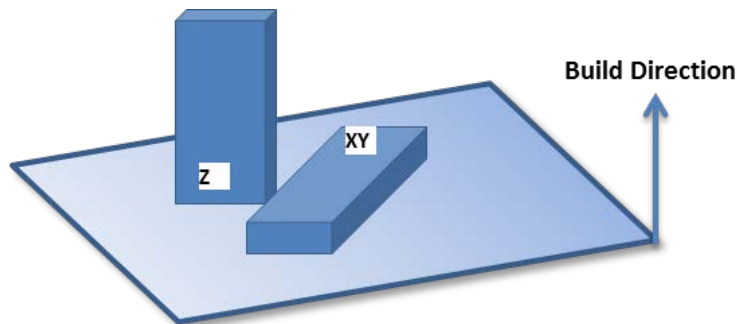


Figure 1. The orientation of rectangular blocks printed by EBM for tensile tests.

The heat treat experiments conducted in this study were classified to three categories: a) sub β -transus, b) super β -transus and c) solution heat treated, air cooled followed by ageing heat treatment. The classification is done to mark the difference in the process conditions and also to emphasize the difference in the observed microstructure and mechanical properties. Figure 2 schematically explains the regions for different heat treatments in the phase diagram and Table 1 shows the different heat treatments performed.

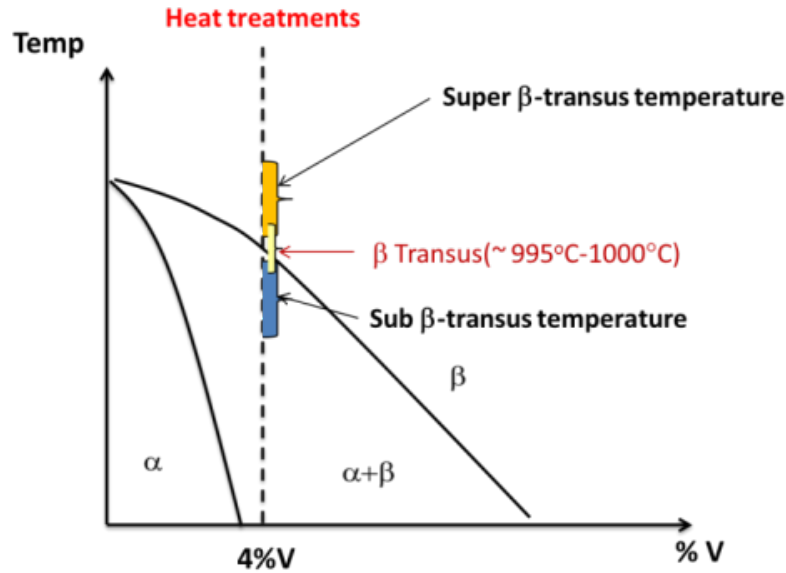


Figure 2. Schematic showing different heat treatment regions.

Table 1. Heat treatment conditions used.

Heat Treatment	Temperature/time	Orientation & no. samples
Sub β -transus	900°C/2h	XY-3, Z-3
Sub β -transus	950°C/2h	XY-3, Z-3
Super β -transus	1050°C/2h	XY-3, Z-3
Solution Air cooled and Aged	1030°C/2h (Air cooled) 500°C/8h (Aged)	XY-3, Z-3

Results and Discussion

The microstructure of as-printed EBM sample was long columnar prior β grains with very fine α - β lath structure as shown in Figure 3, which was in good agreement to that observed by other researchers [1, 8-10]. The SEM image of the as-printed microstructure (Figure 4) revealed both lamellar and Widmanstätten morphologies with dark α phase and bright β phase. The optical images of microstructures for the different heat treatments taken along the build directions are shown in Figures 5-8 for 900°C/2h, 950°C/2h, 1050°C/2h and 1030°C/2h air cooled and aged 500°C/8h, respectively. A distinct difference was observed in the microstructures for the different heat treatments. The sub β -transus heat treatments still displayed columnar grain structures that are similar to the as-printed microstructure. The β -transus temperature is at 995-1000°C [11] where the α -phase completely dissolves into β -phase. However, in the sub β -transus temperatures, the presence of α -phase restricted the growth of prior β grains. Coarsening of α laths occurred in the sub β -transus heat treatments due to diffusion of elements and also the precipitation of α phase in the grain boundaries with increasing temperature was observed. In contrast, the grain structures were completely modified from columnar grains to equiaxed grains after the super β -transus heat treatment. This was caused due to the absence of α -phase in the

matrix and the long columnar β grains were free to rearrange into a more equiaxed morphology, which has a lower energy and stable morphology. The α lath width for the super β -transus heat treatment principally depended on the cooling rate from the solutionizing temperature (above β -transus temperature), hence the furnace cooled samples exhibited coarser α laths (Figure 7) as compared to air cooled samples (Figure 8). The solution air cooled samples were aged at 500°C for 8h to remove any residual stresses and any martensite that may have been formed. Of all the heat treatments, the solution air cooled and aged samples exhibited equiaxed grains with very fine α - β lath structure which are good for mechanical properties. The α lath width is very critical in determining the strength of the alloy because with fine α - β lath structure, the dislocation slip length is reduced which contributes to increase in strength in the alloy.

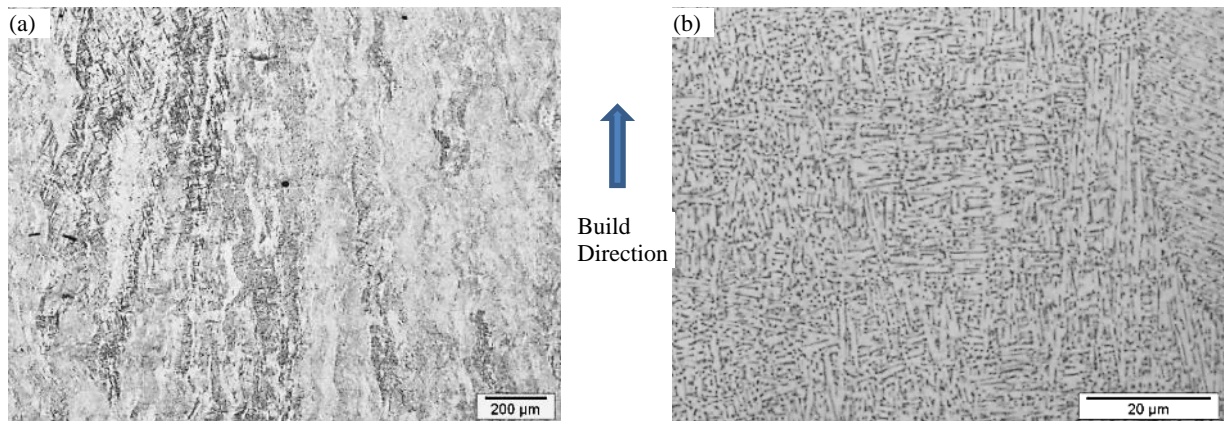


Figure 3. The microstructure of as-printed Ti-6Al-4V showing (a) columnar grains and (b) fine α - β lath structure.

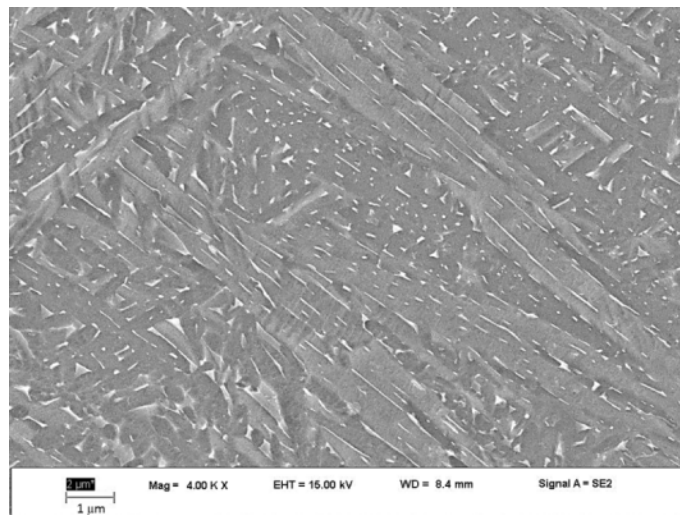


Figure 4. FE-SEM image showing white streaks of β and dark α phases.

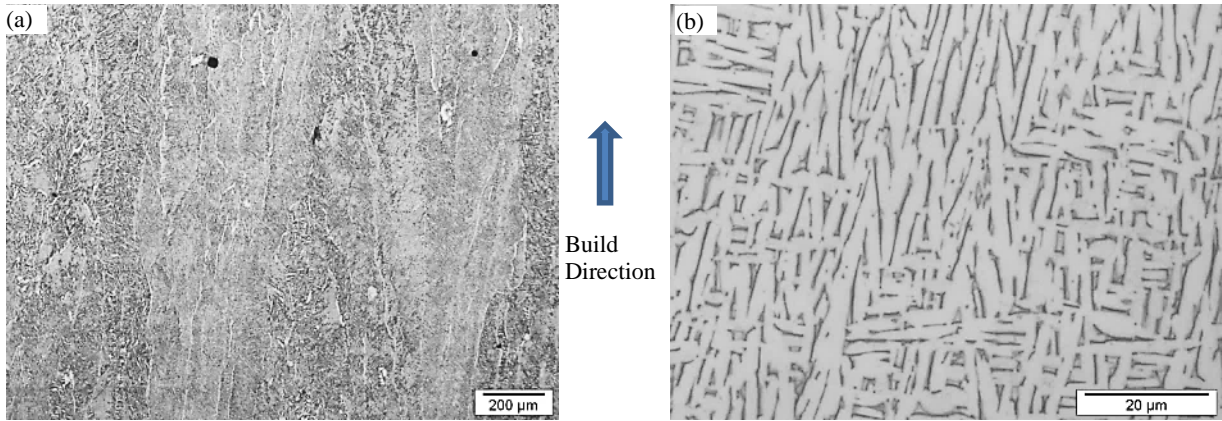


Figure 5. Sub β -transus heat treatment at 900°C/2h, showing (a) columnar grains and (b) coarsened α - β laths.

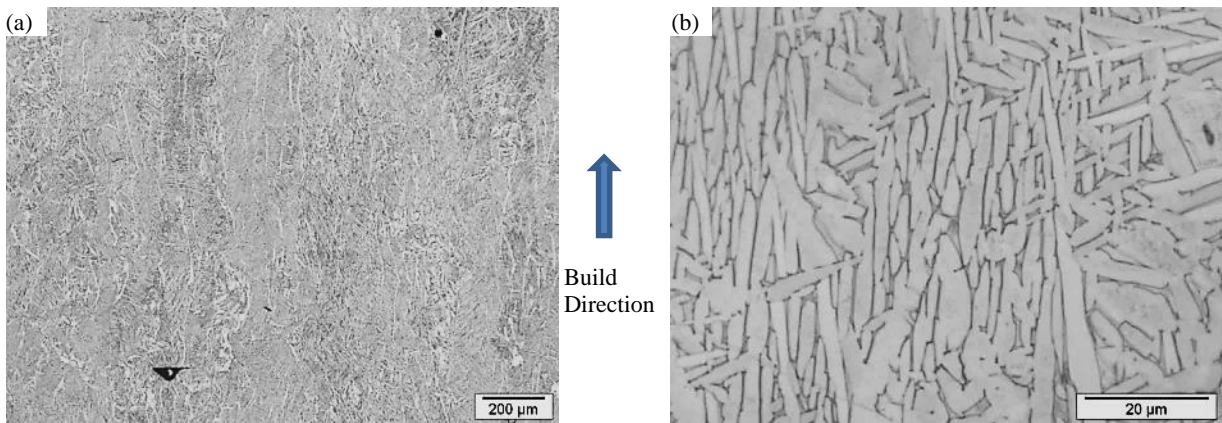


Figure 6. Sub β -transus heat treatment at 950°C/2h, showing (a) columnar grains with α precipitation in grain boundaries and (b) coarsened α - β laths.

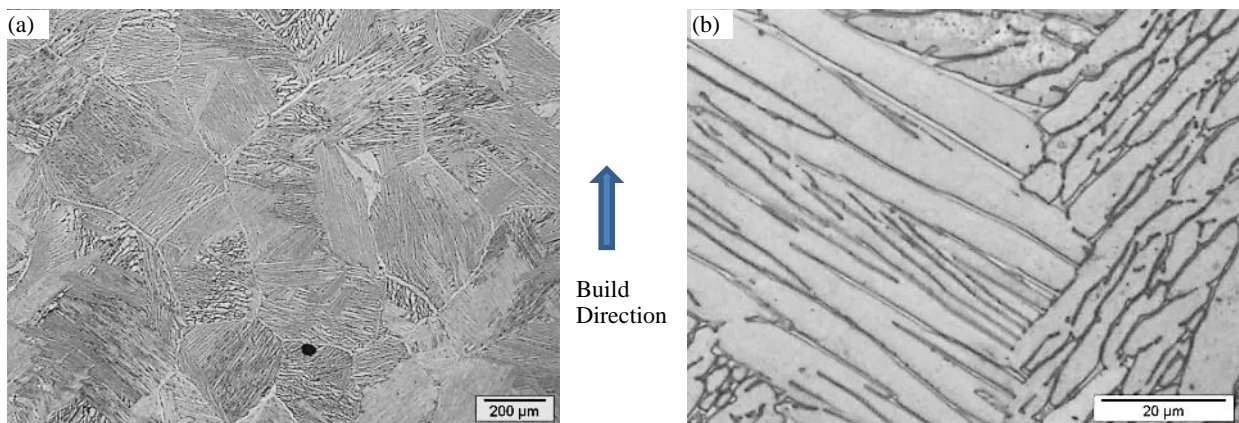


Figure 7. Super β -transus heat treatment at 1050°C/2h, showing (a) fully equiaxed and (b) very coarse α - β lath structure.

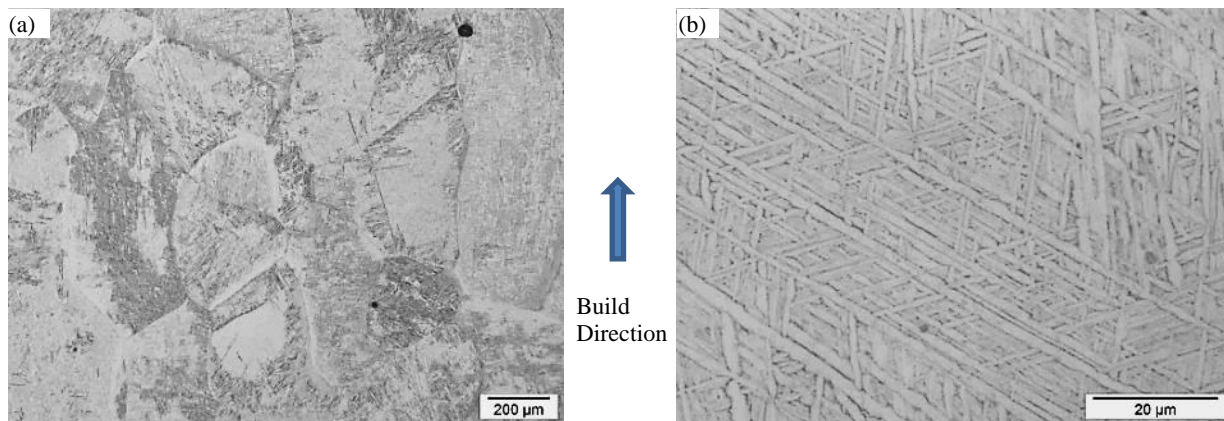


Figure 8. Solution air cooled and aged heat treatment showing (a) fully equiaxed and (b) very fine α - β lath structure.

Tensile tests were carried out for the samples of all the different heat treatments and for the samples' orientations XY and Z to the build plane. The tensile results for ultimate tensile strength (UTS), yield strength (YS) and elongation to failure (El %) are presented in Figure 9. The as-printed EBM samples exhibited the best combination of strength and ductility. The solution air cooled and aged samples also displayed good UTS and YS, which is close to that of as-printed sample. It is interesting to note that only the as-printed samples and the solution air cooled, aged samples exhibited strength higher than the ASTM1472 (wrought and annealed) values in both build directions, marked by the line in Figure 9. The reason for the superior tensile strengths of the as-printed samples followed with solution air cooled samples is attributed to the fine α - β structure. It is also noteworthy that 900°C/2h heat treated samples exhibited the best ductility (~19%) among all the samples. With increase in temperature for sub β -transus heat treatments, the α laths coarsened and the precipitation of α phase on grain boundary also increased, which contributed to the drop in strength for 950°C/2h heat treat samples, as seen in Figure 9. For the super β -transus heat treatment 1050°C/2h, the samples exhibited the worst properties as the microstructures displayed coarse α - β structure and coarse β -grains due to the high heat treat temperature and slow furnace cooling.

In Figures 10-12, the fracture surface of the three heat treated samples (900°C/2h, 1050°C/2h and 1030°C/2h air cooled and aged) are presented. The 900°C/2h fracture surface exhibited a complete ductile failure with the presence of microvoids that led to cavitation and dimpled texture throughout the fracture surface, as seen in Figure 10. On the other hand, the super β -transus heat treatment 1050°C/2h exhibited a mixed failure with fracture surface showing dimples and cleavage planes that indicated brittle failure, as seen in Figure 11. A similar mixed fracture surface with dimples and cleavage planes was also observed for the solution air cooled (1030°C/2h) and aged samples, as shown in Figure 12. The 1050°C/2h heat treated and the solution air cooled samples showed an elongation at failure of 12.1-13.9% which was significantly less than 17.8-18.3% elongation exhibited by 900°C/2h heat treated sample. This difference in ductility was substantiated by the observed difference in the fracture surface of the samples.

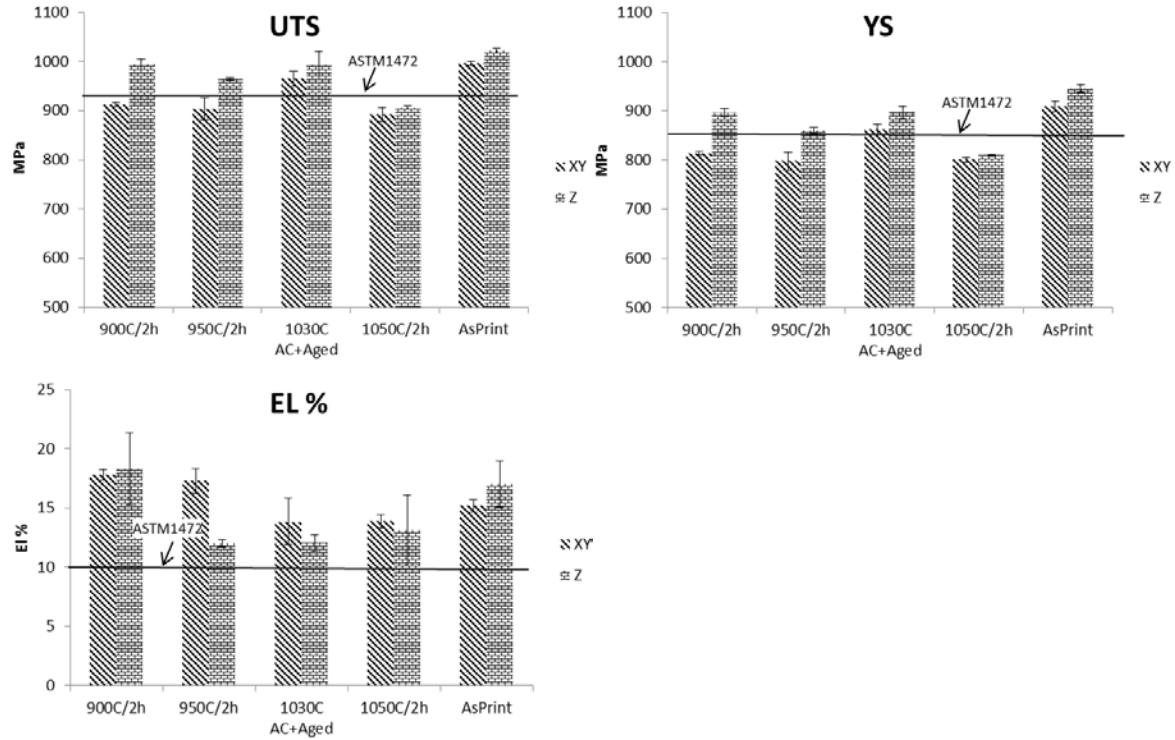


Figure 9. The (a) ultimate tensile strength (UTS) (b) yield strength (YS) and (c) elongation % of the different heat treated samples in both XY and Z orientations.

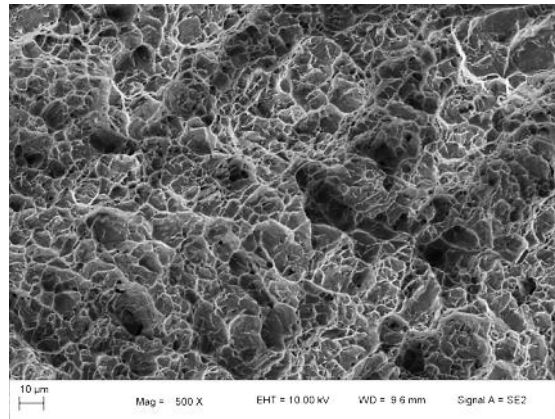


Figure 10. Fracture surface of sub β -transus, 900°C/2h sample showing ductile failure with dimples and microvoids.

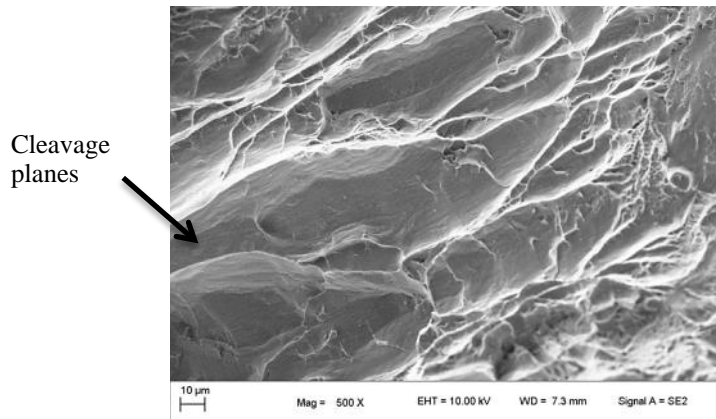


Figure 11. Fracture surface of solution super β -transus , 1050°C/2h showing predominantly cleavage planes with some dimples, indicating mixed failure.

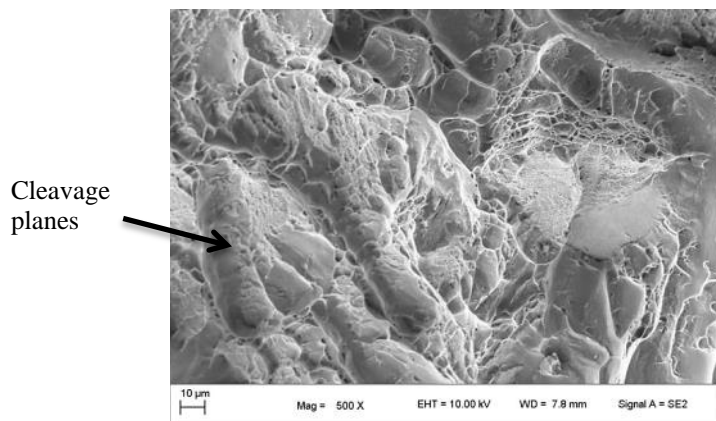


Figure 12. Fracture surface of solution air cooled and aged sample showing mixed failure with both dimples and cleavage planes.

Conclusions

The as-printed EBM Ti-6Al-4V exhibited long columnar grains with very fine α - β microstructure that contributed to its superior tensile properties. For the sub β -transus heat treatments, coarsening of α laths and precipitation of α on grain boundaries occurred with increase in temperature which contributed to loss in strength with increasing temperature. In the super β -transus heat treatment, the absence of α phase allowed the grain morphology to completely change from columnar to equiaxed. The α lath size is mainly controlled by the cooling rates and a clear difference was observed between the furnace cooled and the air cooled samples. The best refinement in microstructures was observed for the solution air cooled and aged samples which also exhibited the best combination of tensile strength among all the heat treated samples. One of the potential post-processing operation in future could be to combine the solutionizing, quench process with HIPping process to obtain a good microstructure with reduced porosity.

Acknowledgments

The authors are grateful for the financial support provided by A*STAR Industrial Additive Manufacturing Program: Work Package 3 (Electron Beam Melting), grant no. 132 550 4103.

References

- [1] P. Wang, X. Tan, M.L.S. Nai, S.B. Tor, J. Wei, Spatial and geometrical-based characterization of microstructure and microhardness for an electron beam melted Ti-6Al-4V component, *Mater. Des.* 95 (2016) 287-295.
- [2] X. Tan, Y. Kok, Y.J. Tan, M. Descoins, D. Mangelinck, S.B. Tor, et al., Graded microstructure and mechanical properties of additive manufactured Ti-6Al-4V via electron beam melting, *Acta Mater.* 97 (2015) 1-16.
- [3] P. Wang, M.L.S. Nai, W.J. Sin, J. Wei, Effect of Building Height on Microstructure and Mechanical Properties of Big-Sized Ti-6Al-4V Plate Fabricated by Electron Beam Melting, *MATEC Web Conf.* 30 (2015) 02001.
- [4] X. Tan, Y. Kok, Y.J. Tan, G. Vastola, Q.X. Pei, G. Zhang, et al., An experimental and simulation study on build thickness dependent microstructure for electron beam melted Ti-6Al-4V, *J. Alloys Compd.* 646 (2015) 303-309.
- [5] P. Wang, M.L.S. Nai, X. Tan, W.J. Sin, S.B. Tor, J. Wei, ANISOTROPIC MECHANICAL PROPERTIES IN A BIG-SIZED Ti-6Al-4V PLATE FABRICATED BY ELECTRON BEAM MELTING, *TMS 2016 Supplemental Proceedings* (2016) 5-12.
- [6] C. de Formanoir, S. Michotte, O. Rigo, L. Germain, S. Godet, Electron beam melted Ti-6Al-4V: Microstructure, texture and mechanical behavior of the as-built and heat-treated material, *Mater. Sci. Eng., A* 652 (2016) 105-119.
- [7] B. Vrancken, L. Thijs, J.-P. Kruth, J. Van Humbeeck, Heat treatment of Ti6Al4V produced by selective laser melting: microstructure and mechanical properties, *J. Alloys Compd.* 541 (2012) 177-185.
- [8] S. Al-Bermani, M. Blackmore, W. Zhang, I. Todd, The origin of microstructural diversity, texture, and mechanical properties in electron beam melted Ti-6Al-4V, *Metall. Mater. Trans. A* 41 (2010) 3422-3434.
- [9] A. Safdar, L.-Y. Wei, A. Snis, Z. Lai, Evaluation of microstructural development in electron beam melted Ti-6Al-4V, *Mater. Charact.* 65 (2012) 8-15.
- [10] L. Facchini, E. Magalini, P. Robotti, A. Molinari, Microstructure and mechanical properties of Ti-6Al-4V produced by electron beam melting of pre-alloyed powders, *Rapid Prototyping J.* 15 (2009) 171-178.
- [11] S. Semiatin, S. Knisley, P. Fagin, D. Barker, F. Zhang, Microstructure evolution during alpha-beta heat treatment of Ti-6Al-4V, *Metall. Mater. Trans. A* 34 (2003) 2377-2386.