CHARACTERISATION OF DMLS MANUFACTURED TI6AL4V POROUS STRUCTURES FOR BIOMEDICAL APPLICATIONS

K.M. Raselabe[1*], P. Mendonidis[2] & N.M. Baloyi[3]

[1] Department of Metallurgical Engineering
Vaal University of Technology, Vanderbijlpark Gauteng South Africa,
211086932@edu.vut.ac.za

[2]Department of Metallurgical Engineering
Vaal University of Technology, Vanderbijlpark Gauteng South Africa,
peter@vut.ac.za

[3]Department of Metallurgical Engineering
Vaal University of Technology, Vanderbijlpark Gauteng South Africa,
nkeleb@vut.ac.za

ABSTRACT

The aim of this paper to study how heat treatment and build orientation affect the hardness and microstructure DMLS porous Ti6Al4V. The porous structures were manufactured by using the EOSINT M280 DMLS system. The stress-relieved microstructure revealed prior beta phase grains along the build direction. At the same time, the vacuum heat-treated samples showed alpha + beta lamellae microstructure with increments of epitaxial prior-ß grains. Vertically built samples for both stress relieved and vacuum heat-treated show higher hardness than the horizontally built samples.

[1] The author was enrolled for an M Tech (Metallurgy) degree in the Department of Metallurgical Engineering, Vaal University of Technology
[2] The author was enrolled for a D Tech (Metallurgy) degree in the Department of Metallurgical Engineering, Vaal University of Technology
*Corresponding author

1. INTRODUCTION

Ti6Al4V is one of the most utilised biomaterials globally; this is mainly due to its favourable characteristics as compared to other biomaterials like cobalt chromium CoCr alloys and stainless steel. These characteristics include high corrosion resistance, biocompatibility and high strength-to-weight ratio [1]. Recently, research has shifted its direction from dense products towards porous structures of Ti6Al4V because porous structures provide better oesintergration and reduced stress shielding effect of implants than fully dense Ti6Al4V [1-6].

Research has shown that the manufacturing of porous structures using conventional methods is limited because of their geometry. However, additive manufacturing (AM) processes have been found to produce these structures with ease. The superiority of AM processes over conventional methods include freedom of design, short lead time and maintaining dimensional control of the components produced [2]. AM processes make use of computer-aided design (CAD) models to develop three-dimensional (3D) parts by building layer by layer and offer the beneficial capacity to construct parts with geometric complexities [3].

Researchers have succeeded in manufacturing porous Ti6Al4V structures using various AM processes. By using electron beam melting type of AM, Cheng et al., (2012) [4] investigated two different types of porous Ti6Al4V, namely stochastic and reticulated mesh in as-built conditions. The microstructures of both the porous structures revealed α' martensite, which research has shown to be expected in all as-built Ti6Al4V manufactured by AM, this was also observed in work done by Sallica-Leva et al., (2013) [5], that showed similar microstructures of α'martensite, with a little β phase present. The as-built Ti6Al4V manufactured by additive manufacturing processes contains α' martensite, which is hard and brittle, due to the non-equilibrium solidification of the processes during manufacturing [5]. Research has shown that heat treatment can be employed to change the hardness and brittleness associated with as-built Ti6Al4V [6-7]. The primary purpose of stress relieving heat treatment is to remove the thermal stresses induced by rapid solidification of AM processes. In contrast, other heat treatments methods at various conditions will result in a change of the microstructure and mechanical properties of Ti6Al4V [7].

The decomposition of α' martensite accomplishes the alteration in microstructure to α+β phase through heat treatments [8]. Becker et al., (2015) [9] evaluated the microstructure and mechanical properties of DMLS Ti6Al4V heat-treated under various conditions, HIPed and compared to as-built and wrought Ti6Al4V. They reported that mechanical behaviour of Ti6Al4V was directly dependant on the microstructure and the crack growth, tensile behaviour and fracture toughness were comparable to the wrought sample. In contrast, as-built Ti6Al4V performance was low due to the induced thermal stresses. Wauthle et al., (2015) [10] indicated that stress relieved T6Al4V porous structures result in no variation of microstructure and mechanical properties, while the HIPed Ti6Al4V showed α+β lamellae structure, with a reduction of tensile strength and increased ductility. Furthermore, Wauthle et al., (2015) [10] stated that this porous Ti6Al4V is favourable in load-bearing applications.

In a study by Ahmadi et al., (2017) [11], which investigated the effect of heat treatments, both below and above the β-transus, of Ti6Al4V porous structures, α+β phases replaced the acicular martensitic structure after heat treatment, while mechanical properties such as ductility and hardness of the heat-treated porous samples remained the same. Fousová et al., (2017) [12] compared selective laser melted (SLM) fully dense and porous Ti6Al4V with hot-rolled Ti6Al4V. The microstructure of SLM Ti6Al4V showed very thin lamellae α phase with small amounts of inter-lamellae β phase. Thus, it concluded that heat treatment below the β-transus of SLM Ti6Al4V showed microstructure like that of hot-rolled Ti6Al4V. Nevertheless, Fousová et al., (2017) [12] stated that while fully dense Ti6Al4V may provide better mechanical properties than porous structures, the latter provides better biological fixation and tissue ingrowth. Chauke et al., (2013) [13] compared the tensile properties, hardness, and microstructure of vacuum heat-treated (VHT) above β-transus temperature (1200°C) to those of as-built Ti6Al4V. This work showed that the VHT Ti6Al4V gave lower ultimate tensile strength and percentage elongation than the as-built Ti6Al4V, even though the resultant microstructure showed α+ß microstructure.

Fully dense Ti6Al4V induces stress shielding by the metal implants when used in high loading areas and their substitution with porous structures is advised. According to Monkova *et al.* (2017) [14], porous structures give rise to advantageous properties like low weight and good energy absorption. Due to insufficient information in literature, there is a need to study porous structures [15]. Consequently, the present work was aimed at studying how heat treatment and build orientation affect the microstructure and hardness of Ti6Al4V porous structures manufactured by direct metal laser sintering

2. METHODOLOGY

2.1 Materials and methods

The nominal chemical composition of the argon-atomised Ti6Al4V ELI (-45μm) from TLS Technik is shown in Table 1 where it is compared to ASTM F-136 standard, with Titanium being the balance of the total composition. The percentiles of the equivalent diameter of the powder particles are as follows D_{10}= 8.13μm, D_{50}=23.56 μm and D_{90}=38.73 μm.

Table 1: Chemical composition of Ti6Al4V

	Al	*V*	*O*	*N*	*H*	*Fe*	*C*	*Y*
ASTM F-136	5.50-6.50	3.50-4.50	Max-0.13	Max-0.05	Max-0.012	Max-0.25	Max-0.08	Max-0.005
Actual value	6.15	4.10	0.12	0.09	0.002	0.19	0.016	<0.001

The Ti6Al4V porous samples with a dimension 65x10x12 mm^3 were manufactured using the EOSINT M280 DMLS system. The samples were designed with a square type unit cell of 5 mm and strut size (thickness) of 1.5mm (Fig.1).

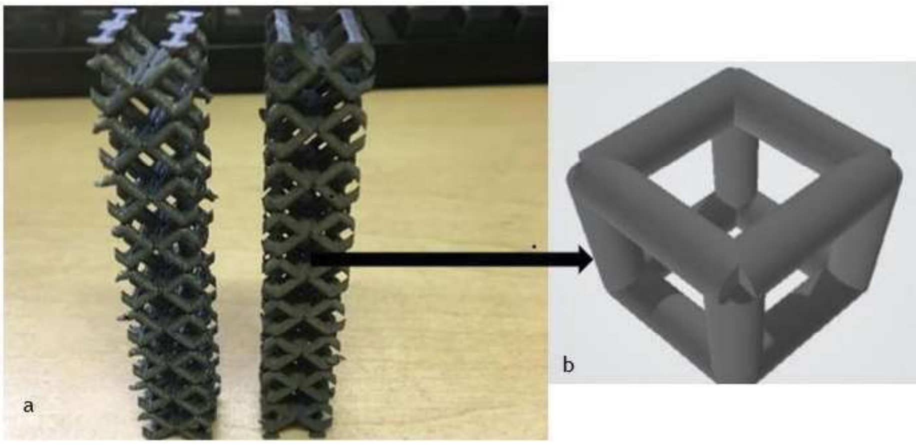

Figure 1. Front view of the Ti6Al4V porous structure a) and the CAD model of the porous structure b).

During the manufacturing of the porous structures, argon was used as a protective atmosphere, and the oxygen level in the chamber was between 0.07-0.12%. The samples were manufactured in two different orientations, i.e. vertical and horizontal. After AM, the samples were stress relieved in an argon atmosphere at 650 °C for 3 hours. After stress relieving, the samples heat-treated under vacuum (annealed) for 2 hours at 950 °C with a heating rate of 10 °C/min, soaking for 90 minutes, and furnace cooled at a rate of 10-15 °C till room temperature.

2.2 Sample preparation and characterisation

After heat-treatment, the porous structure samples were sectioned by mechanical cutting under flowing coolant solution to prepare for characterisation. Standard metallographic sample preparation was done, and the samples were etched using Keller's reagents (Distilled water 190 ml, Nitric Acid 5 ml, Hydrochloric acid 3ml and Hydrofluoric acid 2 ml). Microstructural analysis was accomplished by using an optical microscope Olympus BX41M-LED and Jeol JSM IT500 SEM to reveal the microstructure of the stress relieved (SR) and vacuum heat-treated (VHT) samples. An Innova test falcon 500 Vickers hardness tester was used to measure the hardness of the SR and VHT samples using a load of 1kgf with a dwell time of 10 seconds per indentation.

3. RESULTS AND DISCUSSIONS

3.1 Microstructural analysis

Figures 2 and 3 show the optical and SEM micrographs of the Ti6Al4V porous structures. Figure 2 and 3 shows the optical and SEM micrographs of the Ti6Al4V porous structures. Figure 2 (a) and (b) reveal horizontally build microstructures of SR and VHT samples, respectively. Similarly, in figure 2 (c) and (d) shows microstructures of vertically build samples SR and VHT.

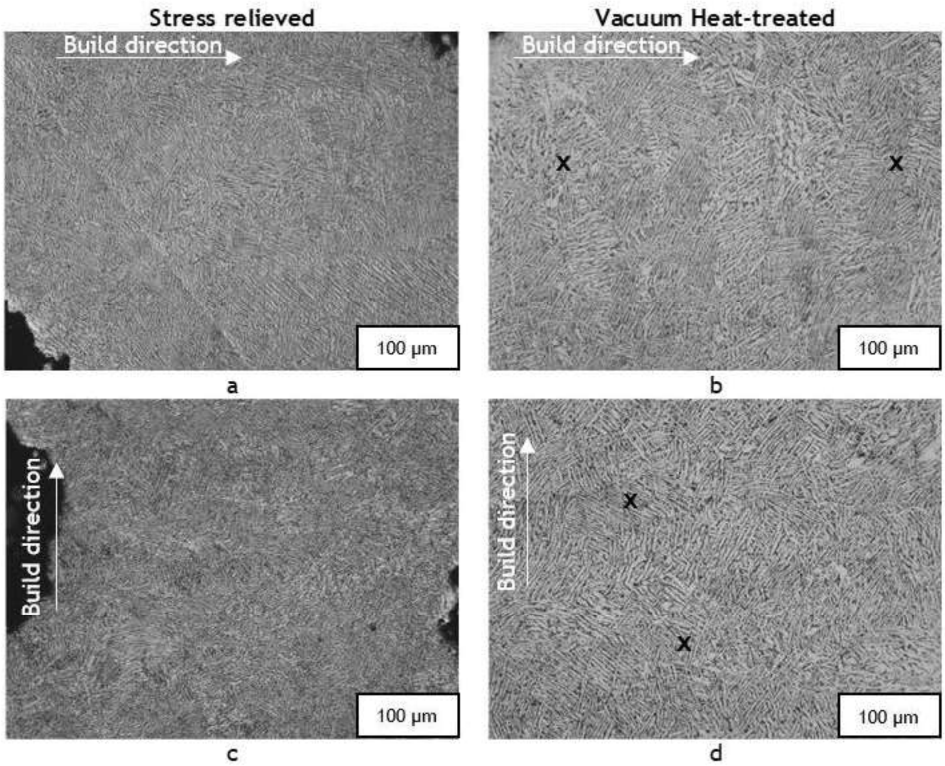

Figure 2. Optical micrographs of Horizontally built-in stress relieved condition (a), vacuum heat-treated condition (b), Vertically built-in stress relieved condition (c) and vacuum heat-treated condition

Stress relieved microstructure in figure 2 (a) and (c) reveals prior β along the build direction, with small amounts of α' martensite still visible. This type of microstructure has been shown in the literature [1-10], and it is due to fast cooling rates associated with AM processes [6]. Figure 2 (b) and (d) shows α+β type lamellae with small amounts of β, while area marked x on the micrographs shows a small amount of epitaxial prior β grains. Heating just below the β-transus temperature has shown the decomposition of α’ martensite to α+β phase if slow cooling and furnace cooling is applied [11]. The furnace cooling rate coarsens the microstructure, hence the formation of α+β lamellae structure shown in Figures 2 (c and d) and 3 (c and d).

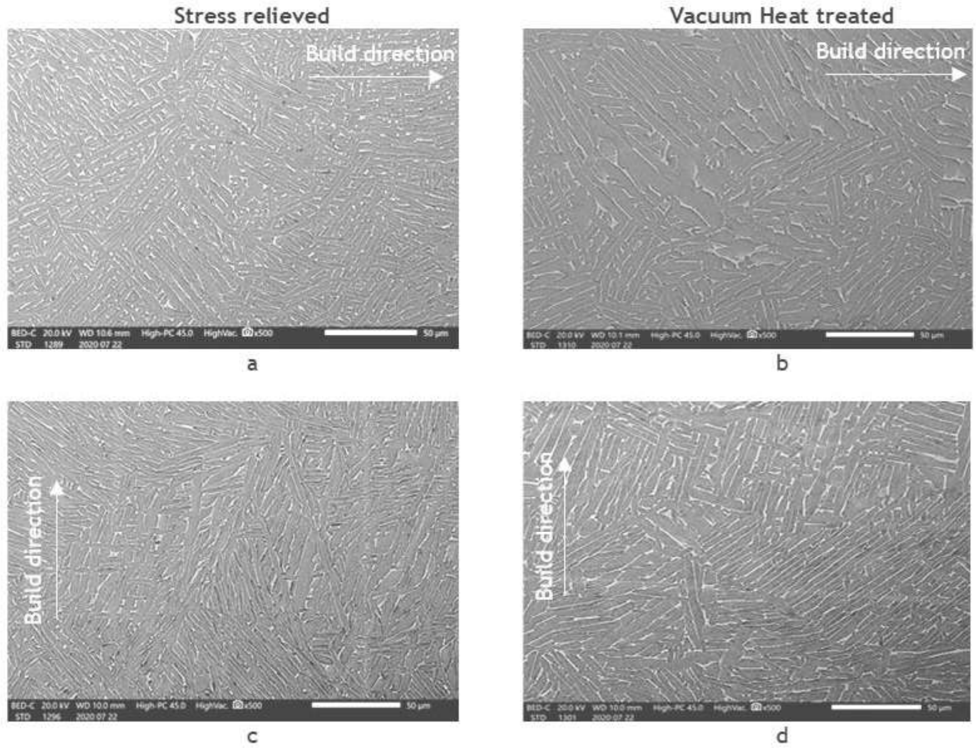

Figure 3. SEM micrographs of Horizontally built-in stress relieved condition (a), vacuum heat-treated condition (b), Vertically built-in stress relieved condition (c) and vacuum heat-treated condition.

The SEM micrographs Figure 3 (c and d) show that the VHT Ti6Al4V laths are thicker than the SR Ti6Al4V in figure 3 (a) and (b), which is in agreement with previous studies of additively manufactured Ti6Al4V [11-13]. The variation in thickness of SR and VHT samples is attributed to the heat treatment in the region of (α-β), which lead to the coarsening of the laths of VHT samples.

3.2 Hardness evaluation

Figure 4 shows the hardness profile conducted on the porous Ti6Al4V. The VHT porous samples show high hardness than the SR samples (Table 2). Moreover, vertically built samples show higher hardness than the horizontally built ones (Table 2). In terms of heat treatment of Ti6Al4V below β-transus temperature, the literature suggests that the hardness of the heat-treated Ti6Al4V is lower than the stress relieved and as-built Ti6Al4V.

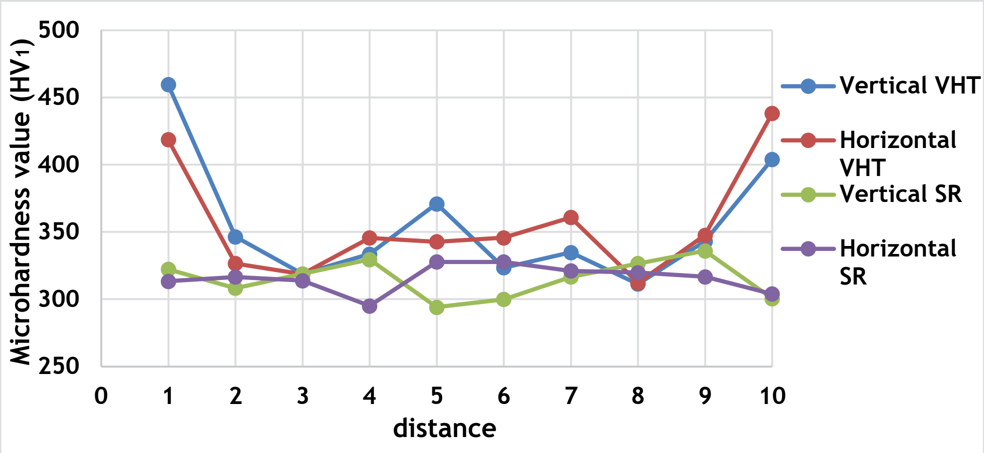

Figure 4. Hardness profile of VHT (horizontal-vertical) and SR (horizontal and vertical) porous Ti6Al4V structures.

Table 2: Average Hardness Values comparison of stress relieved with vacuum heat-treated of vertically and horizontally build Ti6Al4V porous structures

Sample Condition	Build orientation	Average Hardness HV/1 (SD)
Stress Relieved	Vertical	315.83 (14.22)
	Horizontal	313.23 (10.56)
Vacuum Heat-treated (Annealed)	Vertical	385.24 (42.62)
	Horizontal	370.36 (50.55)

Literature [11;13] further suggests that this decrease in hardness resulted from decomposing of α' martensite to α+β lamellae structure. However, the results in Figure 4 and Table 2 reveal the opposite of what literature has presented [11,13], but is corroborated by the work done by Chauke et al., (2013) [13], where VHT Ti6Al4V showed higher hardness than the as-built Ti6Al4V. Furthermore, such behaviour of high hardness of VHT Ti6Al4V was not explained.

4. CONCLUSION

The aim of this work was to evaluate the effect of heat treatment and build orientation on the microstructure and hardness of DMLS manufactured Ti6Al4V porous structures. From the characterisations and analyses of the results, the following conclusions were made:

- The microstructure of the stress relieved samples revealed prior β-grains along the build direction and the vacuum annealed heat-treated samples showed an α+β lamellae with small amounts of epitaxial prior β grain structure upon furnace cooling.
- The vacuum heat-treated samples showed higher hardness values (horizontally built: 370.36 HV and vertically built: 385.24 HV) than the stress relieved samples (horizontally built: 313.23 HV and vertically built: 315.83 HV), which is at variance with most previous studies.
- The effect of build orientation on the hardness revealed that vertically built porous Ti6Al4V were more resistant to indentation than the horizontally built porous samples.

REFERENCES

[1] Parthasarathy, J., Starly, B., Raman, S. and Christensen, A., 2010. Mechanical evaluation of porous titanium (Ti6Al4V) structures with electron beam melting (EBM). *Journal of the mechanical behavior of biomedical materials*, *3*(3), pp.249-259.

[2] Guo, N. & Leu, M. C. 2013. Additive manufacturing: technology, applications and research needs. Frontiers of Mechanical Engineering, 8, 215-243.

[3] Grünberger, T. & Domröse, R. 2015. Direct metal laser sintering. *Laser Technik Journal,* 12, 45-48.

[4] Cheng, X., LI, S., Murr, L., Zhang, Z., Hao, Y., Yang, R., Medina, F. & Wicker, R. 2012. Compression deformation behavior of Ti–6Al–4V alloy with cellular structures fabricated by electron beam melting. journal of the mechanical behavior of biomedical materials, 16, 153-162.

[5] Sallica-Leva, E., Jardini, A.L. and Fogagnolo, J.B., 2013. Microstructure and mechanical behavior of porous Ti–6Al–4V parts obtained by selective laser melting. *Journal of the mechanical behavior of biomedical materials*, *26*, pp.98-108.

[6] Du Preez, W., Yadroitsev, I., Yadroitsava, I., Van Zyl, I., ELS, J., Monaheng, L. & Dzogbewu, T. C. Evaluation of the compressive mechanical properties of cellular dmls structures for biomedical applications. 2016. SUNConferences, 17th Annual Conference of the Rapid Product Development Association of South Africa.

[7] Wang, X., Xu, S., Zhou, S., Xu, W., Leary, M., Choong, P., Qian, M., Brandt, M. and Xie, Y.M., 2016. Topological design and additive manufacturing of porous metals for bone scaffolds and orthopaedic implants: A review. *Biomaterials*, *83*, pp.127-141.

[8] Xu, W., Brandt, M., Sun, S., Elambasseril, J., Liu, Q., Latham, K., Xia, K. and Qian, M., 2015. Additive manufacturing of strong and ductile Ti–6Al–4V by selective laser melting via in situ martensite decomposition. *Acta Materialia*, *85*, pp.74-84.

[9] Becker, T.H., Beck, M. and Scheffer, C., 2015. Microstructure and mechanical properties of direct metal laser sintered Ti-6Al-4V. *South African Journal of Industrial Engineering*, *26*(1), pp.1-10.

[10] Wauthle, R., Vrancken, B., Beynaerts, B., Jorissen, K., Schrooten, J., Kruth, J.-P. & Van Humbeeck, J. 2015. Effects of build orientation and heat treatment on the microstructure and mechanical properties of selective laser melted Ti6Al4V lattice structures. *Additive Manufacturing,* 5, 77-84.

[11] Ahamdi, S., Jain, R. A. K., Zadpoor, A., Ayas, C. & Popovich, V. 2017. Effects of Heat treatment on microstructure and mechanical behaviour of additive manufactured porous Ti6Al4V. *IOP Conference series: Materials Science and Engineering,* IOP Publishing, 293(1), p 012009.

[12] Fousová, M., Vojtěch, D., Kubásek, J., Jablonská, E. & Fojt, J. 2017. Promising characteristics of gradient porosity Ti-6Al-4V alloy prepared by SLM process. *Journal of the mechanical behavior of biomedical materials*, 69, pp 368-376.

[13] Chauke, L.; Mutombo, K.; Kgomo, C. Characterization of the direct metal laser sintered Ti6Al4V Components.In Proceedings of the RAPDASA 2013 Conference, Clarens, South Africa, 29 October–1 November 2013.

[14] Monkova, K., Monka, P., Zetkova, I., Hanzl, P. & Mandulak, D. 2017. Three Approaches to the Gyroid Structure Modelling as a Base of Lightweight Component Produced by Additive Technology. DEStech Transactions on Computer Science and Engineering. DEStech Publications, Lancaster pp. 124–129

[15] Du Plessis, A., Broeckhoven, C., Yadroitsava, I., Yadroitsev, I., Hands, C.H., Kunju, R. and Bhate, D., 2019. Beautiful and functional: a review of biomimetic design in additive manufacturing. *Additive Manufacturing*, *27*, pp.408-427.