# IMPROVING THE MICROSTRUCTURE OF HIGH SPEED SELECTIVE LASER MELTED Ti6AI4V COMPONENTS BY VARYING RESIDENCE TIME DURING HEAT TREATMENT

P. Lekoadi<sup>1\*</sup>, M. Tlotleng<sup>2, 3</sup>, N. Maledi<sup>1</sup> & B.N. Masina<sup>2</sup>

#### **ABSTRACT**

Selective laser melting (SLM) is a powder bed additive manufacturing technique that produces components layer-by-layer from computer aided designs as opposed to conventional manufacturing methods. Although this manufacturing technique offers various advantages, the microstructure of the produced components exhibits an acicular martensitic  $\alpha$ ' phase which exhibits low ductility and high hardness on the produced components. Heat treatment is known to improve the microstructure and ductility and hardness of SLM produced Ti6Al4V components. This study reports on the effect of residence time during heat treatment of Ti6Al4V samples that were built with Aeroswift 3D printing SLM machine. Samples were heat treated to a temperature of  $1000^{\circ}$ C and held for 2hrs, 4hrs, 8hrs, 10hrs and 12hrs before furnace cooling. It was found that the microstructure transformed from martensitic to lamella  $\alpha$ + $\beta$ , then to globular  $\alpha$ + $\beta$  as residence time was increased. Furthermore, grain growth was observed with an increase in residence time.

<sup>&</sup>lt;sup>1</sup> University of the Witwatersrand, School of Chemical & Metallurgical Engineering, Private Bag 3, Wits 2050, Johannesburg, South Africa, Plekoadi@csir.co.za, nthabiseng.maledi@wits.ac.za

<sup>&</sup>lt;sup>2</sup> CSIR National Laser Centre, Laser Enabled Manufacturing Group, PO BOX 395, Pretoria, 0001, South Africa, Bmasina@csir.co.za, Mtlotleng@csir.co.za

<sup>&</sup>lt;sup>3</sup> University of Johannesburg, Department of Mechanical Engineering Science, Auckland Park Campus, Johannesburg, 2012, South Africa, daopase@gmail.com

<sup>&</sup>lt;sup>1\*</sup> University of the Witwatersrand, School of Chemical & Metallurgical Engineering, Private Bag 3, Wits 2050, Johannesburg, South Africa

<sup>&</sup>lt;sup>2</sup> CSIR National Laser Centre, Laser Enabled Manufacturing Group, PO BOX 395, Pretoria, 0001, South Africa <sup>3</sup>University of Johannesburg, Department of Mechanical Engineering Science, Auckland Park Campus, Johannesburg, 2012, South Africa

## 1. INTRODUCTION

Selective Laser Melting (SLM) is a type of powder bed fusion additive manufacturing (AM) technique whereby parts are produced from a metal powder, layer-by-layer, using the laser beam as an energy source to selectively melt powder and promote fusion according to a previously defined computer aided design (CAD) model [1, 2, 3]. SLM products, such as aircraft turbines, pressure vessels, and surgical implants, are used in various industries including aerospace, automotive, electronic, chemical, biomedical and other high technology areas [4, 5]. The success of SLM is linked with its ability to produce complex geometrical structures, reduction of material waste, reduced production time, production of near net shapes and high level of flexibility [4].

Ti6Al4V also known as Ti64 is an  $\alpha+\beta$  titanium alloy with 6 wt% aluminum stabilizing the  $\alpha$  phase and 4 wt% vanadium stabilizing the  $\beta$  phase. Ti6Al4V is the most commonly used titanium alloy, accounting for more than 50% of all titanium usage worldwide [6, 7, 8]. The advantages of Ti6Al4V, over other alloys, include good stability at high operating temperatures, high specific strength, and good corrosion resistance properties [15]. Ti6Al4V is widely used in aerospace applications such as components in aero-engine and space shuttles because of their superior strength to weight ratio [13]. The microstructure of Ti6Al4V consists of hexagonally closed packed (hcp)  $\alpha$  phase and some retained body centered cubic (bcc)  $\beta$  phase at room temperature [9]. The microstructure of Ti6Al4V can be varied significantly in the process of heat treatment [10].

SLM produced Ti6Al4V parts, in the as-built state, are unable to achieve high material performance compared to their wrought counterparts [11, 12, 13]. This is because the starting microstructure of SLM produced Ti6Al4V is significantly different from that of its wrought alloys of the same composition. A study by **Kasperovich et al.** [14] compared the microstructures of SLM produced Ti6Al4V to that of wrought counterparts. The microstructures are shown in Figure 1.

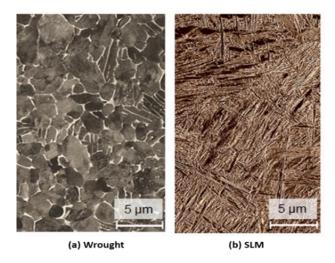


Figure 1: Microstructures of Ti6Al4V (a) Wrought, (b) SLM [14].

The microstructure of the wrought components consists of  $\alpha$  globular phase in a matrix of  $\alpha$ +B (Figure 1a) [14]. This type of microstructure is characterized by a good balance of strength and ductility [14]. SLM produced samples consist of acicular martensitic  $\alpha$ ' phase which is characterized by low ductility and high residual stresses [15]. In order for the microstructure of SLM produced components to match that of wrought components, heat treatment must be performed on the SLM produced Ti6Al4V components [15, 18]. It is well known from literature that a bimodal microstructure achieves good balance of ductility and strength [19, 20].

Heat treatment is used to transform the martensitic  $\alpha'$  phase, improve the microstructure and reduce residual stresses of SLM produced Ti6Al4V components [3, 16, 17]. Heat treatment temperature, residence time and cooling method are the three parameters that play an important role during heat treatment. In this study, residence time was varied during heat treatment above ß transus temperature (at  $1000^{\circ}$ C) to allow grain growth. This was done in order to improve the microstructure in an attempt to produce globular  $\alpha+\beta$  phases.

## 2. METHODOLOGY AND RESULTS

# 2.1 Methodology

## 2.1.1 Materials

Spherical Grade 5, titanium alloy (Ti6Al4V) powder with a particle size distribution in the range of  $20-60\mu m$  was used as deposition material during the production of the samples. The powder was supplied by TLS Technik GmbH & Co and was used as received. Ti6Al4V base plate of dimension  $320 \times 340 \times 30 \text{mm}^3$  was used as a substrate.

#### 2.1.2 Powder Characterization

Powder characterization of the Ti6Al4V was conducted at North West University (Potchefstroom Campus). A Malvern PANalytical G3 advanced particle characterization machine (RAMANXRN System) was used for the measurement of powder shape from 0,5µm to several millimetres. Joel JSM-6010PLUS/LA Scanning Electron Microscope (SEM) was used to study particle morphology and to get images of the powder.

Figure 2 shows the morphology and shape distribution curve of the Ti6Al4V powder. The powder particles were spherical in shape (Figure 2a). The same observation was complemented by the high sensitive (HS) circularity curve in Figure 2b which shows a high percentage peak at approximately 1.0 (a perfect circle has a HS circularity of 1).

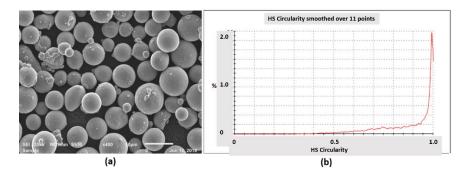


Figure 2: Powder morphology of the Ti6Al4V powder.

# 2.1.3 Methods

A high speed SLM 3D printing machine (Aeroswift), with a single mode fibre laser of wavelength of 1073 nm available at CSIR NLC, was used to produce the samples. The samples were received and heat treated at a temperature of 1000°C. The samples were heat treated in a columnar tube furnace under an argon protected environment at a gas flow rate of 2l/min and heating rate of 10°C/min. The residence times during heating were varied from 2hrs, 4hrs, 8hrs, 10hrs, and 12hrs before furnace cooling. It should be noted that most literature uses 2hrs as their residence time and not much work has been done on residence times above 2hrs [16, 17]. This study aimed at evaluating other residence times above 2hrs.

After heat treatment, the samples were mounted and mechanically ground using SiC papers up to a grit 4000 and then polished for surface finish. Kroll's reagent was used as an etchant to reveal the microstructure of the samples. Samples were analyzed for microstructural evolution using Joel JSM-6010PLUS/LA Scanning Electron Microscope (SEM) and hardness using Matsuzawa Seiko Vickers model MHT-1 micro-hardness machine.

### 2.2 Results

# 2.2.1 Microstructures

All the microstructures were taken on the top left corner of the samples and it should be noted that the microstructural features across the samples were homogeneous. Figure 3 shows the microstructure of the as-built and a corresponding 2hrs heat treated sample.

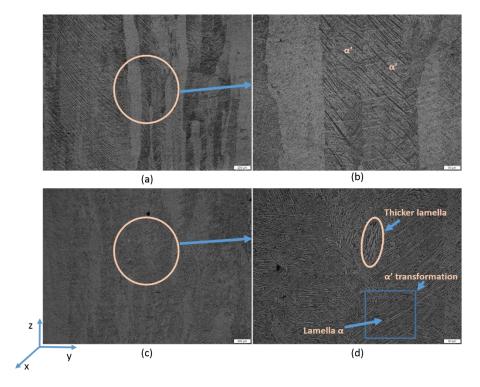


Figure 3: Microstructures of the As-built sample (a) and 2hrs heat treated sample (c) and their specific zoom in (b and d)

As expected, large columnar prior  $\beta$  grains microstructure was observed along the build direction for the as-built sample (Figures 3a, 3b). According to **Vilaro et al.** [11] these prior  $\beta$  grains grow epitaxially along the direction of the heat conduction in a columnar way and are due to partial remelting of the previous layer during SLM processing. An acicular sharp martensitic  $\alpha$  phase (Figure 3b) was observed inside the formed columnar prior  $\beta$  grains of the as-built sample. SLM process is associated with high heating and cooling rates which are the cause of these martensite phase [2, 4, 10, 13]. **Kasperovich et al.** [14] discussed that these martensite  $\alpha$  phase contributes to the low ductility of SLM produced Ti6Al4V components and suggested that heat treatment can be used to transform the martensite  $\alpha$  phase and improve the ductility of the components.

The columnar prior  $\beta$  grains were still observed after heat treatment for 2hrs (Figure 3c) and evidence of phase transformation was also observed. The martensitic  $\alpha$ ' phase transformed into lamella  $\alpha+\beta$  phases of different sizes and thickness (Figure 3d) and this might be due to the temperature used. It was shown that the lamella structure is preferred in applications where fracture toughness and fatigue crack propagation are a priority [14].

The microstructure of the 4hrs and 8hrs are shown in Figure 4.

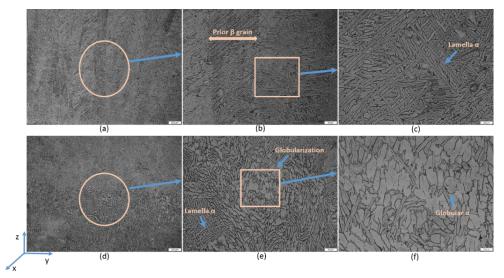


Figure 4: Microstructures of the heat treated samples (a, b & c) 4hrs, (d, e & f) 8hrs.

The large columnar prior ß grains were still observed after heat treatments at 4hrs (Figure 4a) but were no longer observed after heat treatment for 8hrs (Figure 4d). It should be noted that as the residence time increases, the large columnar grains tend to be fading until the disappeared at 8hrs heat treatment.

Lamella  $\alpha+\beta$  growth was evidenced after 8hrs heat treatment. The lamella  $\alpha+\beta$  changed shape and size to globular  $\alpha+\beta$ , a process known as globularization (Figure 4e). **Kasperovich et al.** [14] showed that the globular structure is preferred because of better strength, ductility and crack initiation properties. Heat treatment for 8hrs was sufficient to allow the grains to change from lamella to globular.

More evidence of grain lamella  $\alpha+B$  transformation into globular  $\alpha+B$  was observed as the residence time was increased. Figure 5 shows the microstructures of the samples that were heat treated for 10hrs and 12hrs, respectively.

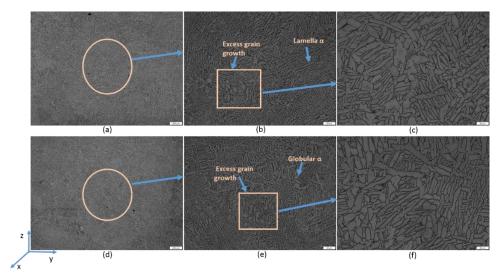


Figure 5: Microstructures of the heat treated samples (a. b & c) 10hrs, (d, e & f) 12 hrs.

The large columnar prior  $\beta$  grains were no longer visible on the microstructure of both samples (Figure 5a, 5d). More of thicker globular  $\alpha+\beta$  grains were observed on the microstructure of the sample that was heat treated for 10hrs (Figure 5b) but with some of the phases starting to show an irregular shape. Figure 5c demonstrates well the irregular grains. As the residence time was increased to 12hrs, excess grain growth was observed (Figure 5e). Some of the globular  $\alpha+\beta$  phases were growing thicker and others were showing more of the irregular shape (Figure 5f). This implies that an increase in the residence allows the lamella  $\alpha+\beta$  phases enough time to grow to different shapes. Furthermore, lamella growth was also observed on the sample that was heat treated for 12hrs.

### 2.2.2 Hardness

The hardness profiles of the as-built and its heat treated samples is shown in Figure 6.

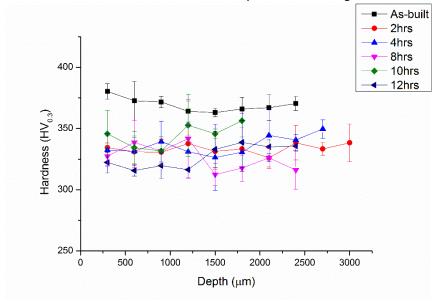


Figure 6: Average hardness profiles of the as-built and heat treated samples.

The as-built sample together with the samples that were heat treated for 2hrs and 4hrs displayed a linear pattern. The sample that was heat treated for 8hrs, 10hrs and 12hrs showed wavy patterns. The as-built sample showed the highest hardness profile with a decrease in hardness observed after heat treatment at different residence times. This decrease in hardness profiles was associated with the transformation of the martensitic  $\alpha$ ' phase and grain growth. From this observation, it can be discussed that the martensite  $\alpha$ ' phase is harder and brittle than the lamella  $\alpha+\beta$  and globular  $\alpha+\beta$  phases. This means that heat treatment at  $1000^{\circ}$ C softened the components.

The hardness profiles in Figure 6 are summarized in Table 1.

Table 1: Summarized hardness of the as-built and heat treated samples.

Sample	As-built	2hrs	4hrs	8hrs	10hrs	12hrs
Hardness (HV)	369±8	333±9	336±15	327±14	342±12	327±9

The as-built sample showed the highest hardness value as compared to the heat treated samples. This high hardness was associated with the martensitic  $\alpha$ ' phase that was observed on the microstructure of the as-built sample. There was no much variation between the hardness values of the heat treated samples.

#### 3. CONCLUSION

High Speed SLM process was used to produce Ti6Al4V samples. The effect of residence time during heat treatment on Ti6Al4V microstructure and hardness were investigated. It was observed that the martensite  $\alpha$ ' phase transformed into lamella  $\alpha$ +B after heat treatment for 2hrs and 4hrs and further transformed into globular  $\alpha$ +B after heat treatment for 8hrs, 10hrs and 12 hrs with lamella phases still observed. The large columnar prior B grains were observed on the as-built, 2hrs and 4hrs heat treated samples were no longer observed after heat treatment at 8hrs, 10hrs and 12 hrs. It was also observed that residence time influence grain growth. Furthermore, it was found that hardness remained the same as residence time was increased.

## **REFERENCES**

- [1] Sallica-Leva, E. Caram, R. Jardini, A.L. Fogagnolo, J.B. 2016, Ductility improvement due to martensitic a'decomposition in porous Ti6Al4V parts produced by selective laser melting for orthopaedic implants, *Journal of the Mechanical Behavior of Biomedical Materials*, 54, pp 149-158.
- [2] Wysocki, B. Maj, P. Krawczynska, A. Rozniatowski, K. Zdunek, J. Kurzydlowski, K.J. Swieszkowiski, W. 2017, Microstructure and mechanical properties investigation of CP titanium processed by selective laser melting (SLM), *Journal of Material Processing Technology*, 241, pp 13-23.
- [3] Xu, W. Brandt, M. Sun, S. Elambasseril, J. Liu, Q. Latham, K. Xia, K. Qian, M. 2015, Additive manufacturing of strong and ductile Ti6Al4V by selective laser melting via in situ martensite decomposition, *Acta Material*, 85, pp 74-84.
- [4] Yadroitsev, I. Krakhmalev, P. Yadroitsava, I. 2014, Selective laser melting of Ti6Al4V alloy for biomedical application: Temperature monitoring and microstructural evolution, *Journal of Alloys and Compounds*, 53, pp 404-409.
- [5] Tong, J. Bowen, C.R. Persson, J. Plummer, A. 2017, Mechanical properties of titanium based Ti6Al4V alloy manufactured by powder bed additive manufacture, *Material Science and Technology*, 33, pp 138-148.
- [6] Semiatin, S.L. Seetharaman, V. Weiss, I. 1997, The Thermomechanical Processing of Alpha/Beta Titanium alloys, *Metals*, 49, pp 33.
- [7] Alcisto, J. Enriquez, H. Garcia, S. Hinkson, T. Steelman, E. Silverman, P. Valdovino, H. Gigerenzer, J. Foyos, J. Ogren, J. Dorey, K. Karg, T. McDonald, O.S. 2011, Es-said, Tensile properties and microstructure of laser-formed Ti6Al4V, *Journal of Material Engineering Performance*, 20, pp 203-212.
- [8] Lutjering, G. 1998, Influence of processing on the Microstructure and Mechanical Properties of  $\alpha+B$  Titanium Alloys, *Materials Science Engineering*, A243, pp 32-45.
- [9] Pederson, R. Babushkin, O. Skystedt, F. Warren, R. 2003, Use of high temperature X-ray diffractometer to study phase transitions and thermal expansion properties in Ti6Al4V, *Material Science and Technology*, 19, pp 1533-1538.
- [10] ] Markovskya, P.E. Semiatin, S.L. 2011, Tailoring of microstructure and mechanical properties of Ti6Al4V with local rapid heat treatment, *Material Science and Engineering*, A528, pp 3079-3089.

- [11] Khorasani, A. Gibson, I. Golberg, M. Littlefait, G. 2017, On the role of different annealing heat treatments on mechanical properties and microstructure of selective laser melted and conventional wrought Ti6Al4V, *Rapid Prototyping Journal*, 23, pp 295-304.
- [12] Joshi, VA. 2006, An Atlas of Structures and Fracture Features, *Titanium Alloys*, ISBN-9781420006063, 1st edition, CRC Press: Boca Raton, FL, USA.
- [13] Ter Haar, G.M. Becker, T.H. 2018, Selective Laser Melting Produced Ti6Al4V: Post-Process Heat Treatments to achieve Superior Tensile Properties, *Materials*, 11, pp 1-15.
- [14] Kasperovich, G. Hausmann, J. 2015, Improvement of fatigue resistance and ductility of Ti6Al4V processed by selective laser melting, *Journal of Materials Processing Technology*, 220, pp 202-214.
- [15] Shipley, H. McDonnell, D. Culleton, R. Coull, R. Lupoi, G. Odonell, D. Trimble, D. 2018, Optimization of process parameters to address fundamental challenges during selective laser melting of Ti6Al4V: A review, *International Journal of Machine Tool and Manufacture*, 128, pp 1-20.
- [16] Villaro, T. Colin, C. Bartout, J.D. 2011, As-Fabricated and Heat-Treated Microstructures of the Ti6Al4V Alloy Processed by Selective Laser Melting, *Metallurgical and Materials transactions*, 42A, pp 3190-3199.
- [17] Vrancken, B. Thijs, L. Kruth, P.J. Van 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.
- [18] Yan, M. Yu, P. 2015, An overview of densification, microstructure and mechanical properties of additively manufactured Ti6Al4V-comparison among selective laser melting, electron beam melting, laser metal deposition and selective laser sintering, and with conventional powder, *Sintered Technology Material*, 23, pp 374-386.
- [19] Ter Haar, M. Becker, H. 2018, Selective laser melting produced Ti6Al4V: Post process heat treatment to achieve superior tensile properties, *Materials*, 11, pp 5-15.
- [20] Nalla, R.K. Boyce, B.L. Cambell, J.P. Peters, J.O. Ritchie, R.O. 2002, Influence of Microstructure on high cycle fatigue Ti6Al4V: Bimidal vs lamella structure, Metallurgical and Materials Transection, 33A, pp 202-218.