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Microstructure and fatigue performance of SLM-fabricated Ti6Al4V alloy after different stress-relief heat treatments

Martin Frkan[a]*, Radomila Konecna[a], Gianni Nicoletto[b], Ludvik Kunz[c]

[a]*University of Zilina, Univerzitna 1,01026 Zilina, Slovakia*
[b]*University of Parma, Parco Area delle Science 181/A, Italy*
[c]*Institute of Physics of Materials, Ziskova 22, 61662 Czech Republic*

Abstract

The main interest in Additive Manufacturing (AM) technology relates to its ability to produce complex components with relatively reduced weight that are difficult to produce or cannot be produced by other conventional technologies. Selective laser melting (SLM) is extensively used, as one of the AM technologies to fabricate metallic parts. This advanced method allows to produce various parts with complex geometries with high three-dimensional (3D) accuracy from fusion powders in a layer-by-layer style.
Ti6Al4V alloy is a widely used material for structural applications in aerospace and biomedical due to high specific fatigue strength. SLM processing makes this alloy attractive when weight reduction is a design objective. The SLM Ti6Al4V microstructure is influenced by process parameters and build orientation. The localized high energy input during very short interaction times leads to the formation of very fine structures and to the generation of internal stresses. Therefore, the SLM parts are heat treated to decrease or completely remove residual stresses.
The present study aims at evaluating the effect of stress-relief heat treatments on the microstructure, the mechanical properties and the fatigue performance of SLM Ti6Al4V alloy. Ti6Al4V alloy specimens were manufactured according to the SLM process with an EOS M290 system. Post fabrications heat treatments at different temperatures (i.e. 740°C vs. 900°C) resulted in different structure and mechanical properties that were identified and measured. Fatigue testing of specimens with as-built surfaces was performed at room temperature on modified Schenk-type fatigue testing machine applying a pulsating plane bending (load cycle ratio R = 0) to the specimens at a frequency f = 15 Hz.

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Keywords: Additive manufacturing; Selective laser melting; Ti6Al4V; Microstructure, Heat treatment; Fatigue

* Corresponding author. Tel.: +421 41 513 2632
E-mail address: martin.frkan@fstroj.uniza.sk

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1. Introduction

Additive Manufacturing (AM) has experienced more than 20 years of development since the first AM technology became available in the late 1980s to fabricate models and prototypes. AM is presently a rapidly developing manufacturing technique that has become competitive with traditional manufacturing techniques in terms of cost, speed, reliability, and accuracy (Gu 2015).

Selective laser melting (SLM), one of the AM technologies, offers a wide range of advantages. Short time-to-market, wide variety of metallic materials (aluminum, copper, iron, stainless steel, chromium, nickel alloys, titanium), direct production from three-dimensional CAD model, high level of flexibility (different geometries can be produced in the same batch), high production rates, versatility, precision, geometric freedom, and the ability to create unique designs with internal structures (Yadroitsev 2009; Li 2014; Malca 2018). For these reasons, in recent years, the SLM has proven to be the most efficient additive process for the production of metal prototypes and parts with complex geometries and has been used in different industries, e.g., motorsport, biomedical, aerospace.

SLM enables the direct fabrication of parts from three dimensional (3D) model data by layer-by-layer manufacturing process. A laser beam interacts with feedstock powders and produces a solid material (Shin 2018). Large temperature gradients and high cooling rates develop in the material due to the highly localized heat input and very short application time. The SLM process affects the microstructure and develops high residual stresses in the parts (Parry 2016). Therefore, parts are usually stress relieved before separation from the substrate to minimize residual stresses.

Titanium alloys are among the most important advanced materials and are key to improved performance in both aerospace and terrestrial systems, due to an excellent combination of specific mechanical properties and excellent corrosion behavior (Froes 2014). Ti6Al4V alloy is ($\alpha + \beta$) titanium alloy with high strength, low density, high fracture toughness and excellent corrosion resistance. Ti6Al4V is the most popular alloy accounting of half of the titanium used in the world today. This lightweight and strong alloy is ideal for the production of jet engines, gas turbines and many structural components (Donachie 2000; Liu 2019).

The most important properties of SLM Ti6Al4V for load-bearing applications are static mechanical properties and fatigue and fracture performance. The purpose of this work is therefore the study of the influence of two specific stress-relieving heat treatments on a microstructure and fatigue properties of SLM Ti6Al4V.

2. Material and Methods

The Ti6Al4V (Ti64 ELI) alloy powder with reduced content of interstitial elements supplied by EOS GmbH was used as an experimental material. The powder was made by melt spraying in argon gas resulted in the formation of a spherical powder.

Powder fraction contains 28 % of particles smaller than 25 μm and 12 % of particles with a particle size above 45 μm. The largest percentage ratio of 60 % represented particles with sizes between 25 and 45 μm and therefore a particle size of 45 μm was determined.

Analysis of the chemical composition of the powder was made by TLS Technik GmbH & Co.

The chemical composition of the powder (Tab. 1) meets the ASTM F136 requirements, which defines the maximum concentration of impurities in the titanium alloy.

Table 1. Chemical composition of the Ti6Al4V powder.

	Al	V	O	N	H	Fe	C	Ti
[wt. %]	6.27	4.2	0.09	0.006	0.002	0.18	0.013	Bal.

The specimens were manufactured by SLM on EOS M290 machine. The EOS M290 system operates an Yb fiber laser unit with a wavelength of 1075 nm, supplying a power of 200 W or 400 W. The EOS M290 machine is equipped with a building chamber filled by argon gas to avoid oxidation of the titanium powder. A 400 W laser power, a process chamber temperature of 80 °C and 60 µm layer thickness, were used in the manufacturing process. The scan strategy was based on a shell and core concept, where the internal part of the layer is first melted by raster laser motion then the contour of the layer is melted. The melting of a successive layer is performed after rotation of the scanning pattern of 30-deg angle.

Heat treatment. During the SLM process considerable thermal-related internal stresses develop in the parts (Shiomi 2004). For this reason, the stress relieving heat treatment is an important step in production of parts by the SLM technology. The specimens were heat-treated before separation from the substrate to avoid undesirable deformations. Two different heat treatment temperatures were selected and the influence on the microstructure and the properties of the alloy were investigated. To prevent oxidation of the surface, specimens were heat treated in a vacuum furnace (TAV Caravaggio, Italy). The heat treatment consists of heating to a maximum temperature (740 ºC or 900 ºC), with soaking period of about 2 hours, followed by a control cooling to a temperature 520 ºC. The last stage consists of fast cooling from a temperature 520 ºC to a room temperature by argon gas injected to a hot temperature chamber under pressure. Both heat treatments reduce residual stresses due to fabrication to negligible levels.

Microstructure of specimens was observed using a light optical microscope Zeiss Axio Observer Z1M on polished and etched (10% HF for 10 s) specimens that were cut from broken fatigue specimens. The metallographic specimens were prepared according to standard practice for Ti alloys. Observation of the microstructure characterized the material anisotropy arising from the specific process conditions, (i.e. layer by layer generative principle, short interactions, high temperature gradients and the high localization of this manufacturing process).

Tensile tests were carried out on tensile specimens fabricated in vertical orientations (C orientation) on servo-hydraulic MTS 810 machine according to the ASTM 8/E 8M-08 standard.

Vickers hardness tests were performed on metallographically prepared specimens in polished state using HPO 250/AQ machine with a load of 10 kP and 10 s of loading time. The hardness was the average of 10 measurements.

Fatigue specimens. Special miniature specimens (5 x 5 mm in cross-section and 22 mm in length) designed by Nicoletto (2016) were used for fatigue testing. The aim of the specimen design was the minimization of material required for manufacturing. Three sets of specimens were produced according to three orientations with respect to build direction (i.e. horizontal and vertical, see Fig. 1a) to evaluate the influence of build direction on the fatigue behavior.

a)

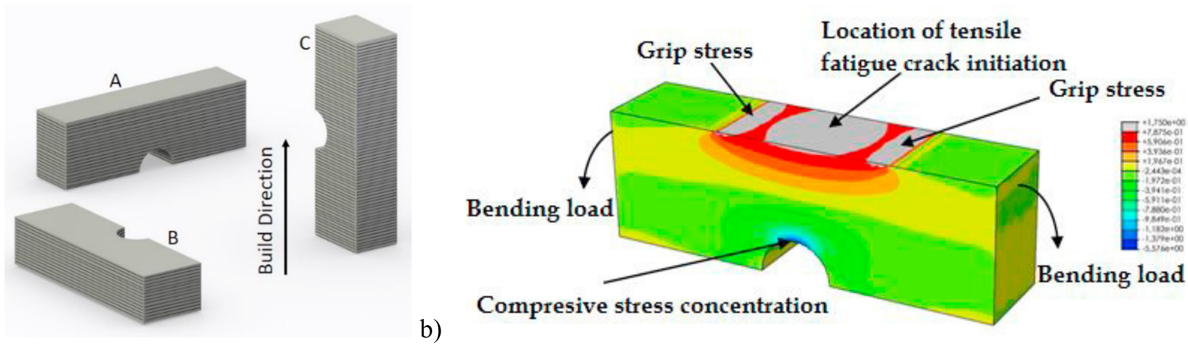


Fig. 1. (a) Build orientation; (b) FEM analysis of stress distribution in miniature specimen.

Fatigue testing. It was performed at room temperature on a modified Schenk-type fatigue testing machine applying a pulsating plane bending (load cycle ratio R = 0, test frequency f = 15 Hz) to the specimen so that the surface under the cyclic tensile loading was opposite to the notch. The notch was inserted in the specimen to control crack initiation in the central region of the specimen. Fig. 1b shows the elastic FE stress distribution in the notched mini specimen due to an applied bending moment. The surfaces of the specimens were in the as-build state.

3. Results and discussion

3.1. Structure

The macrostructure of heat treated specimens at 740 °C and 900 °C show characteristic texture, which is similar for the lateral planes and differs from orthogonal planes. During the material processing, the primary β-phase grains grow parallel to the build direction as a consequence of the thermal history experienced by the layer-wise fabrication. They are much longer (primary β columnar grain grows through several building layers) than the layer thickness of about 60 µm. The microstructure of orthogonal planes shows a cross-section of the primary β columnar grains, which are observed as polyhedral grains. The size of these grains corresponds to the width of the primary β columnar grains.

The microstructural analysis after heat treatment showed a two-phase structure formed by α and β phases. The specimens heat treated at 740 °C show a microstructure characterized by fine needles of α´/α-phase in β matrix (Fig. 2). Dark spots, generated by the local intense laser energy input, were locally observed. These areas are characterized by a finer microstructure. The average hardness for this microstructure is 392 HV10. Tensile properties were determined as yield strength = 1104 MPa, ultimate tensile strength = 1176 MPa, A = 13 %.

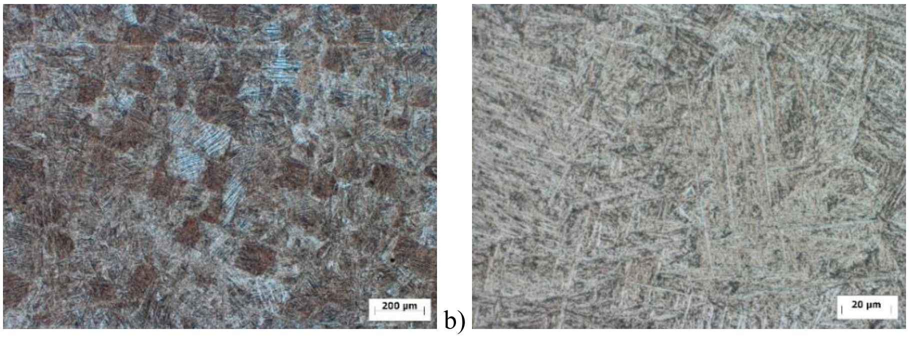
a)  b)

Fig. 2 Microstructure of Ti6Al4V at 740 ˚C, (a) orthogonal plane to the build direction; (b) detail of microstructure.

Microstructure of heat treated specimens at 900 °C (Fig. 3) is characterized by lamellas of the (α+β) phases, arranged in the Widmanstätten form. The thickness of the (α+β) is different and depends on the orientation of the lamellas and the metallographic cross-section. The higher stress relieving temperature results in a homogeneous structure without black spots compared to the heat treated specimens at 740 °C. The average hardness is corresponding to a coarser structure and is 347 HV10. Tensile properties are yield strength = 990 MPa, ultimate tensile strength = 1078 MPa, A = 18 %.

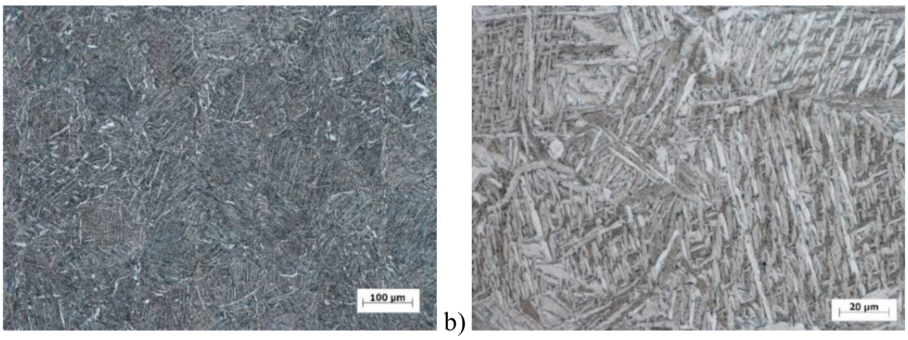
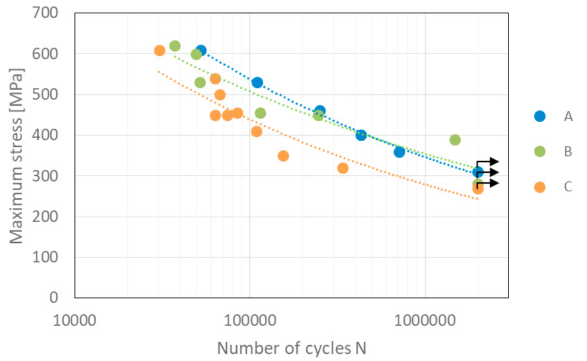
a)  b)

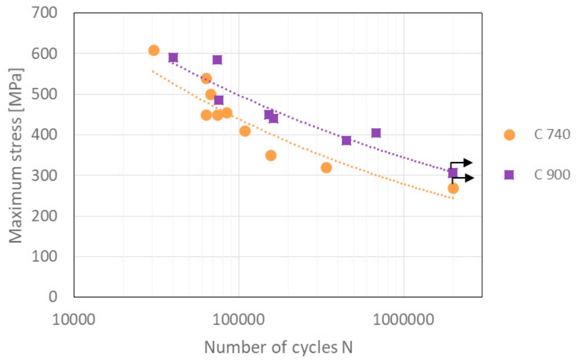
Fig. 3. Microstructure of Ti6Al4V at 900 ˚C, (a) orthogonal plane to the build direction; (b) detail of microstructure.

3.2. Fatigue

The results of the high-cycle fatigue tests of SLM Ti6Al4V alloy heat treated to 740 °C/2 h are shown in the form of the Wöhler (S-N) curves in Figure 4a. For this series of specimens, fatigue tests were performed for all three orientations (Fig. 1a). The measured results show that the orientation of the specimens influences the fatigue life. Specimens with orientation A and B of Fig. 1a have similar fatigue behavior. A fatigue strength of $\sigma_{c,R=0} \approx 300$ MPa can be determined at 2.10^6 cycles for these two orientations. The fatigue strength of specimens oriented in a direction C is lower than the previous ones with orientations A, B and is estimated $\sigma_{c,R=0} \approx 270$ MPa at 2.10^6 cycles.



a)



b)

Fig. 4. Directional fatigue behavior of SLM Ti6Al4V (R=0), (a) 740 ˚C; (b) 740 ˚C vs 900 ˚C

A set of specimens with C orientation and heat treated at 900 °C/2 h was tested in the same way as the previous series of specimens. The results of fatigue tests for C orientation specimens heat treated at 900 °C are shown in Fig. 4b and compared the similarly orientated specimens heat treated at 740 °C. The comparison shows that the fatigue behavior of both series of specimens are very similar. The fatigue strength of the specimens heat treated at 900 °C is higher compared to specimens heat treated at 740 °C and is estimated in $\sigma_{c,\,R=0} \approx 300$ MPa at 2.10^6 cycles.

Since all specimens were manufactured in the same AM system and according to the same process parameters, the observed fatigue behavior has to be discussed in dependence of maximum heat treatment temperature and its influence on microstructure and mechanical properties. Available literature has shown that the very fine martensitic structure of Ti6Al4V titanium alloy leads to the higher mechanical properties (Vrancken et al. 2012; Krakhmalev et al. 2016), but fatigue strength (Fan et al. 2016) and resistance to fatigue crack growth (Masete et al. 2016; Agius et al. 2018) is lower compared to the lamellar or bimodal structure. Fan et al. (2016) which examined the relationship between microstructure, mechanical properties and fatigue behavior in the Ti6Al4V alloy, reported that lower elongation deteriorates resistance to fatigue crack growth at the prevailing tensile load, but on the other hand, higher strength can slightly increase resistance to crack initiation.

The heat treated specimens at 740 °C exhibit higher yield strength, higher tensile strength and a finer microstructure compared to the material heat treated at 900 °C. These characteristics are in favor of high fatigue strength (ASM, 1996). However, the material heat treated at 900 °C has a greater elongation to rupture (i.e. higher ductility). The similar fatigue strength data determined in this study suggest that the two heat treatment temperature are equivalent in terms of their influence on fatigue strength.

4. Conclusion

The aim of this study was the determination of influence of the two specific post-fabrication heat treatment on the microstructure and fatigue behavior of Ti6Al4V titanium alloy fabricated by SLM technology. This study leads to the following conclusions:

- Both series of specimens after heat treatment exhibit a texture of the structure formed by primary β columnar grains oriented parallel to the building direction, typical for SLM materials.
- The microstructure of Ti6Al4V specimens prepared by SLM and then heat treated is characterized by primary β columnar grains that are filled with fine needles of α′/α-phase in case of heat treatment at 740 °C or lamellas of (α+β) matrix in case of heat treatment at 900 °C.
- Fatigue behavior of the specimens Ti6Al4V at 740 °C shows certain anisotropic behavior. Specimens with orientation C have a lower fatigue strength compared to specimens with orientation A and B.
- The fatigue strength of Ti6Al4V at 900 °C is slightly higher than that of Ti6Al4V at 740 °C and may be due to a greater ductility (i.e. A = 18 % instead of A = 13 %).
- Taking into account all factor, the fatigue strength of both series of specimens can be estimated to $\sigma_{c, R=0} \approx 300$ MPa at 2.10^6 cycles.

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