Microstructure and mechanical properties of selective laser melted Ti6Al4V alloy

M Losertová and V Kubeš
VŠB – Technical University of Ostrava, Faculty of Metallurgy and Materials Engineering, 17. listopadu 15, 708 33 Ostrava-Poruba, Czech Republic

mlosertova@vsb.cz

Abstract. The present work was focused on the properties of porous Ti6Al4V specimens processed by selective laser melting (SLM) and tested in tension and compression before and after heat treatment. The SLM samples were annealed at 955 °C, water quenched and aged at 600 °C with following air cooling. The values of the mechanical tests showed that the samples exhibited high mechanical properties. The anisotropy of tensile and compressive strength was observed, which was related to the occurrence of voids. The plastic properties of specimens were improved by means of the heat treatment that led to the transformation of martensitic to lamellar structure composed of $\alpha + \beta$ phases. The microstructure of SLM samples were evaluated before and after the heat treatment. The brittle nature of failures of non-heat treated samples can be explained by synergy of martensite presence, microcracks and residual stresses produced by SLM.

Keywords: Selective laser melting, Ti6Al4V, Microstructure, Porosity, Mechanical properties.

1. Introduction
The process of the Selective Laser Melting (SLM) represents advantageous production techniques that allows reduction of production steps, a high material use efficiency and a near net shape production [1, 2]. The SLM assures the layer-wise building of parts with a high geometrical complexity and thus, offers broad applications in different industries. However, the final products can displayed some problems related with large thermal gradients occurring in the SLM process due to short interaction times and highly localized input of heat. The influence of the different process parameters as well as of the heat treatments were studied for Ti6Al4V [1-3] that is still the material of particular interest for biocompatible implants in orthopedics and traumatology for its higher biological and biomechanical compatibility in comparison with 316L steel or CoCr alloys [4]. Unlike the parts produced of the compact Ti6Al4V alloy having the Young’s modulus equal to 110 GPa [5], the material containing certain level of porosity decreases its modulus of elasticity toward that one of human bones and in surgical implants allows to avoid the stress shielding effect due to the mismatch between the mechanical properties of titanium alloy and bone.

The study of this work was focused on the properties of Ti6Al4V specimens processed by SLM technology and containing certain porosity. The material was tested in tension and compression in the condition with and without heat treatment.
2. Experimental
Two sets of samples were built vertically (in a direction $z$), it means in the direction of intended tensile and compressive loading (Figure 1), by SLM method in a layer-by-layer manner. The square patterns produced by building strategy of the SLM method are evident in Figure 1a. First set included ten cylindrical tensile test specimens with a gauge length of 30 mm and gauge diameter of 5 mm; second set represented ten cylinders of 10 mm in diameter and 12 mm in height for the compressive tests.

![Figure 1. Ti6Al4V specimens built in the direction of height: a) general view with square patterns resulting from building strategy; b) tensile specimens; c) compressive specimens.](image)

Half of the specimens of both sets were processed by heat treatment (HT) under argon atmosphere that consisted of solution annealing at 955 °C followed by furnace cooling to 855 °C and water quenching and ageing at 600 °C followed by air cooling. The surface of heat treated specimens changed the colour to blue-black (Figure 1b and 1c) due to oxidation reactions in argon atmosphere even though the gas purity reached 5N. The thermal regime was chosen in accordance with findings in [3, 6] and is presented in Figure 2.

![Figure 2. Thermal treatment consisting of two steps: 1) solution annealing followed by furnace cooling (FC) and water quenching (WQ) and 2) ageing followed by air cooling (AC).](image)

The tensile and compression tests at room temperature were performed with strain rate 0.0025 s$^{-1}$ on the specimens with and without heat treatment using Inova Fu-0-350-1700-V1 machine. The porosity was evaluated using image analysis on an optical microscope Olympus IX70. The
Microstructure features of both sets in different conditions of heat treatment were performed by means of optical microscopy on both cross and longitudinal electrolytically polished sections before and after etching in Kroll's solution (6% HF, 8% HNO₃, 86% H₂O).

3. Results and discussion

The porosity that was determined from ten measurements on five specimens is shown in Figure 3 and reached average values of 5%. The etched microstructure of non-heat treated specimen is formed of a fine acicular martensite (α' phase) resulting of the imposed high cooling rate in the SLM process, as seen in Figure 4. The evident building patterns (so called tracks) were processed due to the molten pool boundaries (MPBs) with a special shape in the internal regions and on the surfaces of SLM specimens [7].

![Figure 3](image1.png)

**Figure 3.** Distribution of the voids in the specimens prepared by SLM and before heat treatment: a) longitudinal and b) cross sections (non-etched).

![Figure 4](image2.png)

**Figure 4.** Microstructure of as-built specimen: a) low magnification morphology of building tracks and pores; b) acicular α' martensite in prior β grains (etched).

After the heat treatment the acicular α' martensite transformed into equilibrium (α + β) microstructure, at the same time with reducing thermal stresses that have been built up during the SLM process. It is evident that annealing in the vicinity of the β transus (the temperature of the α ↔ β transition is called β transus and for Ti6Al4V reaches 980 °C) led to growing β grains from columnar
to large and semi-equiaxed shape that transformed to lamellar ($\alpha + \beta$) after the cooling, as seen in Figure 5.

As summarized in Table 1, the mechanical properties in tensile test reached a high yield stress (with average value of 1 056 MPa), a high ultimate tensile strength (1 351 MPa) but a relatively low ductility (up to 6 %). The thermal treatment resulted in slightly increasing yield strength (YS) (by 4 %) but in markedly decreasing ultimate tensile stress (UTS) and elongation (deformation) by 13 and 9 %, respectively. Concerning Young’s modulus, the measured values dropped to the 42 and 49 GPa for tensile tested specimens in non-heat treated and heat treated stages, respectively.

![Figure 5. Microstructure of heat treated specimen: a) low magnification morphology of lamellar ($\alpha + \beta$) grains; b) detail of lamellar ($\alpha + \beta$), $\alpha$ is white, $\beta$ is dark (etched).](image)

### Table 1. Mechanical properties of both sets of Ti6Al4V specimens in tensile and compressive testing.

| Specimen tested in | Porosity (\%) | YS (MPa) | UTS (MPa) | Deformation (\%) | E (GPa) |
|--------------------|---------------|----------|-----------|------------------|---------|
| **Tensile**        |               |          |           |                  |         |
| 1                  | 5             | 1060     | 1312      | 6                | 43      |
| 2                  | 1083          | 1373     | 5.9       | 44               |
| 3                  | 1025          | 1369     | 4.5       | 40               |
| Average value      | 1056±29       | 1351±34  | 5.5±0.8   | 42±2             |
| 6 + HT             | 5             | 1055     | 1184      | 4.9              | 49      |
| 7 + HT             | 1102          | 1171     | 5.1       | 50               |
| 8 + HT             | 1137          | 1189     | 5         | 49               |
| Average value      | 1098±41       | 1181±9   | 5±0.1     | 49±0.6           |
| **Compression**    |               |          |           |                  |         |
| 1                  | 5             | 1099     | 1763      | 40               |
| 2                  | 1101          | 1620     | 42        |
| 3                  | 1300          | 1661     | 23        |
| Average value      | 1167±115      | 1681±74  | 35±10     |
| 6 + HT             | 5             | 1140     | 1622      | 25               |
| 7 + HT             | 1100          | 1657     | 27        |
| 8 + HT             | 1100          | 1602     | 27        |
| Average value      | 1113±23       | 1627±28  | 26±1      |
The compression properties achieved of higher values, as seen in Table 1. The heat treatment resulted in decreasing all values of the mechanical properties, by 5, 3 and 16 % for YS, UTS and deformation in compression, respectively. Comparing the results in tensile and compression tests, it is clear that strongly textured structures led to significant anisotropic mechanical behavior. As it can be seen from the Figure 6, the compression properties are higher by 10, 24 and 536 % in values of YS, UTS and deformation, respectively, for as-built specimens, and by 1, 37 and 420 % in YS, UTS and deformation, respectively, for heat treated specimens. The failure surfaces of non-heat treated samples were characterized by brittle nature that can be explained by synergy of martensitic microstructure, microcracks and residual stresses produced by SLM. The lamellar feature of microstructure after heat treatment led to brittle fracture but at lower strength values.

Figure 6. Behavior of the heat treated SLM specimens under loading in a) tension and b) compression.

4. Conclusion
The Ti6Al4V specimens produced by SLM process were mechanical tested in heat treated and non-heat treated conditions. Based on the results of tensile and compression loading the following conclusions can be drawn:

1. Ti6Al4V specimens in the as-built stage consisted of prior-β grains filled with acicular α′ martensite, and usually display high yield strength, but limited ductility.
2. The combined effects of acicular martensite, microcracks and residual stresses were responsible for lower ductility comparing tensile and compressive tests.
3. The martensitic microstructure was the main reason for the higher strength of the as-built specimens.

The high mechanical properties together with lowered Young’s modulus are suitable improvement of material properties; however material evaluation of the biocompatible porous alloy is necessary to accomplish by fatigue and corrosion tests in near future.

Acknowledgement
This article has been elaborated in the framework of the project No. LO1203 "Regional Materials Science and Technology Centre - Feasibility Program" funded by Ministry of Education, Youth and Sports of the Czech Republic and co-financed by the European Social Fund and of the projects SGS SP2017/77 and SGS SP2017/58 supported by Ministry of Education, Youth and Sports of the Czech Republic.

References
[1] Sun J, Yang Y and Wang D 2013 Parametric optimization of selective laser melting for forming Ti6Al4V samples by Taguchi method Optics & Laser Technology 49 118-124
[2] 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 74-84

[3] Wauthle R, Vrancken B, Beynaerts B, Jorissen K, Schrooten J, Kruth J-P and 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

[4] Niinomi M 2008 Mechanical biocompatibilities of titanium alloys for biomedical applications J Mech Behav Biomed Mater 1 30-42

[5] Banerjee R, Nag S and Fraser H L 2005 A novel combinatorial approach to the development of beta titanium alloys for orthopaedic implants Mater Sci Eng C 25 282-289

[6] Vrancken B, Thijs L, Kruth J-P and Van Humbeeck J 2012 Heat treatment of Ti6Al4V produced by Selective Laser Melting: Microstructure and mechanical properties J Alloy Compd 541 177-185

[7] Shifeng W, Shuai L, Qingsong W, Yan Ch, Sheng Z and Yusheng S 2014 Effect of molten pool boundaries on the mechanical properties of selective laser melting parts J Mater Process Tech 214 2660-2667