Influence of microstructure on fracture feature of Ti6Al4V alloy prepared by 3D printing

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Abstract. In this work, the Ti6Al4V specimens produced by selective laser melting were mechanically tested in non-heat treated condition and fracture surface features were compared with this one of the material prepared using conventional casting and forging. The results of the fractography observation for both types of the samples were explained on the base of the microstructure analysis. The fracture surface of selective laser melted specimens showed more brittle feature that was in relation with the microstructure composed almost fully of martensite as opposed to conventionally prepared alloy with bimodal (α + β) microstructure and more ductile character of fracture surfaces.

1. Introduction

New materials can be prepared by alternating both advanced technologies and alloying elements. Three biocompatible materials mostly used in traumatology or orthopedics are steels (ASTM F55 or F138), CoCr alloys (ASTM F75 or F90) and Ti6Al4V alloy (ASTM F136) [1-3]. Considering the increasing sensitivity of human body to nickel presence in materials, most of implants are produced now from Ti6Al4V alloy that possesses higher biocompatibility, higher corrosion resistance and favorable mechanical properties comparing with 316L stainless steel. Mechanical properties and strain distribution in relation to microstructure of Ti6Al4V resulting from mechanical and thermal treatment are important features influencing the reliability and safety of implants [3]. The Young modulus that is the critical parameter to avoid stress shielding in healing process, is in the case of compact Ti6Al4V implants produced by conventional method still high comparing modulus of human bone [1].

Biomechanical compatibility of Ti6Al4V can be improved using advanced producing methods, as for example selective laser melting (SLM). However, the material prepared by this method can display some defects, as pores, phase inhomogeneity and residual stresses [4-5] that can imply different mechanical and fracture behavior unlike conventional wrought Ti6Al4V products. Due to the vertical layer-by-layer build process during selective laser melting technology a highly directional microstructure in as-built condition comprises of fine acicular α’ martensite that transforms to bimodal or lamellar (α + β) equilibrium structure after heat treatment. Relieving residual stresses and
modifying rapidly solidified microstructure provide a more desirable combination of strength and toughness.

The present work is focused on the evaluation of the fracture surfaces in relation with microstructure characteristics and mechanical properties that were determined for the as-forged and SLM as-built samples after tensile testing.

2. Experimental
Two sets of Ti6Al4V specimens were studied. First group of five tensile test samples (furthermore denoted as forged) was machined from traditionally forged rods (supplied by BIBUS METALS CZ). Second group represented five tensile test samples (furthermore denoted as SLM) that were prepared by selective laser melting (supplied by ProSpon spol. s r.o.) and machined before testing. The SLM samples were built in the vertical orientation (in the stress axis of tensile specimens) with specified parameters: laser power of 200 W, slice thickness of 30 µm, 5x5 mm islands rotated by 45 ° one from another, scanning speed of 800-900 mm/s.

Tensile testing of both sets was realized at the room temperature applying the force $F = 17.3 \text{ kN}$ and at the strain rate $\dot{\varepsilon} = 0.0025 \text{ s}^{-1}$ by GALDABINI, Sun 10 device. For the metallographic study, specimens were ground, polished and etched by means of the Kroll’s solution (6 % HF, 8 % HNO₃, 86 % H₂O) for 10 - 60 seconds. The microstructure was studied using the optical microscope OLYMPUS GX51. The porosity was measured using an Image-Pro Plus software of Media Cybernetics. Fracture surfaces were observed by means of the PHILIPS SEM 505 microscope.

3. Results and discussion
The microstructure of Ti6Al4V forged samples in the as-received state is represented in figure 1. Transversal and longitudinal sections in figures 1a and 1b, respectively, show very fine grains of α phase with size under 5 µm (white phase) and (α + β) lamellar grains (dark grey phase) formed in prior β phase. Unlike cross section in the longitudinal direction the grains are elongated due to hot forging.

![Microstructure of Ti6Al4V forged sample](image)

**Figure 1.** Microstructure of Ti6Al4V forged sample: a) bimodal morphology with fine (α + β) grains in the cross section and b) details of elongated α and β transformed grains in the longitudinal section.

The macrostructure of vertical cut of the SLM samples was characterized by patterns related with melted pools while in cross section the texture corresponding to scanning strategy (figure 2a) was observed. The microstructure of as-built specimens is formed of martensite needles in the prior β phase due to rapid cooling of melted pools, as seen in figure 2b. When heat treated the martensitic needles are transformed to α + β lamellar structure, as it was confirmed in [6]. The average value of
the porosity measured in the as-built material (figure 2a) reached about 3% whereas size pores varied from 2 to 159 µm.

**Figure 2.** Microstructure of Ti6Al4V sample after SLM: a) cross section with pores, scanning strategy morphology and martensite needles; b) detail of acicular martensite structure.

**Table 1.** Mechanical properties of both sets of Ti6Al4V specimens in forged and SLM state.

| Forged specimens | YS (MPa) | UTS (MPa) | ε_f (%) | E (GPa) |
|------------------|----------|-----------|---------|---------|
| 1                | 881      | 974       | 13.69   | 118     |
| 2                | 897      | 978       | 14.08   | 101     |
| 3                | 903      | 989       | 12.79   | 126     |
| 4                | 806      | 979       | 14.19   | 111     |
| 5                | 828      | 969       | 13.39   | 117     |
| **Average value** | **863±43** | **978±7** | **13.6±0.6** | **115±9** |

| SLM specimens | YS (MPa) | UTS (MPa) | ε_f (%) | E (GPa) |
|---------------|----------|-----------|---------|---------|
| 1             | 1091     | 1271      | 7.23    | 93      |
| 2             | 1005     | 1260      | 7.82    | 95      |
| 3             | 924      | 1263      | 7.61    | 102     |
| 4             | 1052     | 1260      | 8.16    | 92      |
| 5             | 1033     | 1261      | 5.34    | 91      |
| **Average value** | **1021±63** | **1263±5** | **7.2±1.1** | **95±4** |

**Note:** YS – yield strength, UTS – ultimate tensile strength, ε_f – strain to fracture; E – Young’s modulus.

**Figure 3.** Macrographs: a) view of break after tensile testing of Ti6Al4V specimens (left – forged and right – SLM); b) and c) necking feature for forged (ε_f = 13.6%) and SLM (ε_f = 7.2%) samples, respectively.
Figure 4. Tensile test diagrams of Ti6Al4V specimens: a) forged and b) prepared by SLM.

Figure 5. SEM fractographs of tensile tested Ti6Al4V specimens produced by forging: a) ductile failure in central part of fracture surface; b) detail of dimples and voids related with more brittle $\alpha$ phase; c) transition of ductile and shear fracture at the specimen edge; d) detailed deep dimples and shear facets at the transition area.
Character of the rupture differed for both sets, as seen in figure 3. The forged samples formed slight necking of the tensile samples (figure 3b). Due to the martensitic microstructure the SLM samples showed macroscopically more brittle feature of the damage than the forged samples.

The results of tensile mechanical properties of both specimen sets are summarized in table 1. Comparing the average mechanical values it is evident that martensite microstructure showed both higher yield strength (YS = 1021 MPa) and ultimate tensile stress (UTS = 1263 MPa). Unlike the bimodal (α + β) forged samples with YS = 863 MPa and UTS = 978 MPa, the martensitic microstructure of SLM material increased the average values of YS by 18 % and UTS by 29 %.

Figure 4 shows the typical tensile engineering stress - strain curves of both Ti6Al4V specimens sets tested to the fracture. As it can be seen, the deformation behavior of both materials with high strength characteristics differs under loading to the break. The bimodal (α + β) forged sample showed higher strain (εf = 13.6 %) comparing to the SLM sample with martensitic structure (εf = 7.2 %).

The SLM technology producing the porosity in the volume of the samples provided decreasing of the Young’s modulus by 17 % (from 115 to 95 GPa - see table 1). The higher elasticity of the SLM material studied can be beneficial for medical applications but there is no evidence of the effect of the microstructure morphology and porosity volume on the fatigue and corrosion behavior.

![Figure 6. SEM fractographs of tensile tested Ti6Al4V specimens prepared by SLM method: a) shear fracture with pores at specimen edges; b) detail of very shallow dimples and pores on shear surface; c) detail of powder particle not remelted; d) detail of martensite laths and pore.](image-url)
The evident differences in ragged fracture surfaces of tensile tested samples consisted in feature of dimples and shear facets (figures 5 and 6). Ductile failure with deep dimples is characteristic for the central part of the fracture surface in forged samples where existing holes are corresponding to more brittle \( \alpha \) phase and transition area between ductile and shear fracture is observed at the specimen edge (figure 5a and 5b).

In some areas of SLM fractures, cavities with unmelted powder particles could be observed (figure 6b and 6c). Shallow dimples with cracked martensite needles rounded by ductile \( \beta \) phase in the fracture surfaces of the SLM samples (figure 6d) are corresponding to more brittle damage feature.

From the fractography study it is clear that SLM process-induced imperfections were the driving factor at the failure initiation on the microscopic scale. Moreover, the pores occurring in the specimen surfaces (figure 2a and 6a) served as notches localizing stresses at the loading. Generally, the Ti6Al4V alloy shows lower ductility comparing austenitic steels but the SLM process can contribute to further decreasing of the stress accommodation by plastic behavior due to providing the residual stresses, important porosity and metastable phases in rapidly solidified microstructure.

Conclusion
Based on the results of the comparative study of the tensile behavior, microstructure and fractography analysis of the Ti6Al4V specimens prepared of forged rods and by selective laser melting, it is possible to draw the following conclusions:

1) The as-received state of forged specimens was characterized by very fine bimodal microstructure composed of \( \alpha \) and \( (\alpha + \beta) \) grains due to previous mechanical treatment. Owing to this the mechanical properties reached 863 MPa and 978 MPa for YS and UTS, respectively. Due to the higher plasticity more ductile fracture feature with deep dimples were determined.

2) The SLM technology prepared specimens showed the microstructure consisted of fine martensite needles in prior \( \beta \) grains that led to the higher mechanical properties in YS and UTS (1021 and 1263 MPa, respectively) but the strain decreased by 47 %. According to the martensitic microstructure feature and porosity presence the damage at tensile tests was characterized by more brittle fracture feature.

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