Influence of processing technology on fracture feature of Ti6Al4V

V Kubeš, M Losertová, J Škoda, K Konečná and F M Ijaz

VŠB - Technical university of Ostrava, Faculty of Metallurgy and Materials Engineering, Ostrava - Poruba, Czech Republic

E-mail: mlosertova@vsb.cz

Abstract. Selective laser melting is an advanced manufacturing technology providing an alternative method of producing complex components directly from 3D computer models. The paper is focused on problems related with selective laser melting technology (SLM) used for preparing titanium based implants. Two series of ten Ti6Al4V samples were mechanically tested in tension and compression respectively before and after heat treatment. Annealing under argon atmosphere consisted of solution treatment at 955 °C followed by furnace cooling to 855 °C with water quenching and ageing at 600 °C with air cooling. Fractography study of fracture surfaces was performed using scanning electron microscopy and the observation was correlated with structural and mechanical properties of the SLM specimens in the as-built or heat treated conditions. The failure features of the specimens corresponded to the microstructure with relatively high porosity, martensite needles, microcracks and residual stresses. The heat treatment resulted in the transformation of martensite to (alpha + beta) lamellar structure modifying plastic properties that corresponded to the morphology of fracture surfaces. In spite of the porosity occurrence both specimen series exhibited high mechanical properties in tension as well as in compression.

1 Introduction

Selective laser melting (SLM) is an additive manufacturing process in which successive layers of powder are selectively melted by the interaction of a high energy density laser beam. Molten and re-solidified material forms parts, while non-melted powder remains in place to support the structure. This layer-wise production technique offers some advantages over conventional manufacturing techniques such as high geometrical freedom, short design and manufacturing cycle time and made-to-order components. Layer-wise production techniques have evolved rapidly in the last 10 years and SLM has changed from a rapid prototyping to an additive manufacturing technique [1].

Consequently, the static and dynamic material properties must be sufficient to meet in service loading and operational requirements. The SLM process is characterized by rapidly solidified and non-equilibrium microstructures due to high temperature gradients. Residual stresses developed at high localized thermal gradients and very short interaction times lead to rapid volume changes. Furthermore, the change of process parameters can have a strong influence on the microstructure, density and surface quality. As a result, the mechanical properties of SLM parts can differ substantially from one another and from those produced by conventional techniques. In this respect, it is recognized that the advantages of SLM are only performed when the mechanical
behavior of the final products is at least similar to conventionally produced components of the same material [2].

In recent years, much research [2 – 5] has focused on optimizing the SLM process. The SLM parts and material properties specifications were studied in order to characterize how the mechanical properties obtained with SLM may differ from the ones of conventional material and improve the quality of the resulting products. At present, Ti – based alloys can be processed by SLM technology with high reproducibility and hence low variation in material density and mechanical properties whereas the material density can be significantly affected by varying scanning parameters and scanning strategies. The effect of applying specific heat treatments of Ti6Al4V produced using SLM has been studied in order to produce a preferred final microstructure.

Although the quasi-static material properties such as tensile strength, hardness and impact toughness have been well characterized and reported to match those of conventional wrought materials, the dynamic mechanical behavior during crack initiation and propagation is more complex to explain because of critical sensitivity to the relations between fracture path, orientation, microstructure and loading conditions. Previous studies [3, 6] have shown that the residual stresses have detrimental effects on the mechanical behavior and fatigue life of SLM parts though the knowledge of failure mechanisms remains limited and consequently insufficient confidence to predict product life-time. Nevertheless, the SLM parts have great potential to meet the fatigue life requirements.

More recently, the influence of post-built heat treatment was investigated [7 – 9]. The two-step annealing of as-built products was performed at 955 °C for one hour with subsequent furnace cooling (FC) to 850 °C and water quenching (WQ) followed by ageing at 600 °C for six hours and air cooling (AC). The heat treatment of as-built material not only relieve residual stress, but also modify rapidly solidified microstructure which comprises of fine acicular α' martensite. The as-built microstructure is highly directional as a result of the imposed solidification mode during the vertical layer-by-layer build process. Hence, the purpose of the post-built heat treatment is to generate the preferred lamellar α + β equilibrium structure which provides a more desirable combination of strength and toughness. However, the maximum heat treatment temperature does not exceed the β transus temperature and the time at temperature does not result in excessive grain growth.

The present work is focused on the evaluation of the fracture surfaces in relation with as-built defects, heat treatment and mechanical properties that were determined for the as-built and annealed conditions in the previous study [9].

2 Experimental

The specimens of Ti6Al4V ELI for both tensile and compressive test were prepared by selective laser melting with vertical building orientation (in the stress axis) and specified parameters: laser power 200 W, slice thickness 30 µm, 5x5 mm islands rotated 45° one from another.

As-built products were heat treated (HT) in accordance with the schema presented in figure 1. For the metallographic study, the specimens were ground on grinding papers with grain size ranged from 600 to 2500, electro-chemically polished using Struers LectroPol-5 with A3 electrolyte and etched by Kroll’s solution (6 % HF, 8 % HNO₃, 86 % H₂O) for 10 - 60 seconds. The microstructure was observed by means of Olympus IX70 microscope equipped with digital camera DP12. The porosity was measured using Image-Pro Plus software of Media Cybernetics. The mechanical tests were performed at strain rate of 0.0025 s⁻¹ and room temperature by devices Walter + Bai UTM 100 and Inova Fu-0-350-170-V1 for tensile and compression tests, respectively, and the results were evaluated elsewhere [9]. The fractographic study was realized using an SEM JOEL JSM-649OLV microscope.
Results and discussion
The mechanical properties of SLM specimens in tension and compression that have been evaluated and discussed elsewhere [9], are summarized in table 1. The as-built porosity occurring in the as-built material ranged locally from 1.7 to 10.6 %. The macrostructure of vertical cut was characterized by patterns related with melted pools, while in cross section the texture corresponding to scanning strategy was observed.

Table 1. Tensile and compressive mechanical properties of Ti6Al4V (average values from 3 tests) [9].

| Specimen         | YS (MPa) | UTS (MPa) | $\varepsilon_{\text{fracture}}$ (%) |
|------------------|----------|-----------|-------------------------------------|
| **Tensile test** |          |           |                                     |
| As-built         | 1056±29  | 1351±34   | 5.5±0.8                             |
| Heat treated     | 1098±41  | 1181±9    | 5.0±0.1                             |
| **Compression test** |         |           |                                      |
| As-built         | 1167±115 | 1681±74   | 35±10                               |
| Heat treated     | 1113±23  | 1627±28   | 26±1                                |

Note: YS – yield strength, UTS – ultimate tensile strength, $\varepsilon_{\text{fracture}}$ – strain to fracture

The microstructure of as-built specimens before the HT was formed of martensite needles in the prior $\beta$ phase due to rapid cooling of melted pools, as seen in figure 2a. After the two-step HT the $\alpha'$ martensite was transformed to $\alpha + \beta$ lamellar structure (figure 2b). The lath thickness measured ranges from 3 to 6 µm and it is depending on maximum temperature of treatment below $T_\beta$ transus and cooling rate whereas their influence is increasing with increasing vicinity to $T_\beta$.

After tensile test, failures of both as-built and annealed conditions were ragged and consisted of shear and transcrystalline quasi-cleavage surfaces (figures 3 and 4). In some areas of fractures, cavities with unmelted alloy particles could be observed (figure 4b). It is clear that SLM process-induced imperfections were the driving factor at the failure initiation for both microstructural modifications on the microscopic scale. Moreover, the pores present on the specimen surfaces acted as notches localizing stresses at the loading. Generally, the Ti6Al4V alloy shows low ductility and the SLM-process providing the residual stresses and metastable microstructure can
contribute to that the possibility of plasticity-induced accommodation of these elevated stresses is limited. Comparing the average mechanical values (table 1) it is evident that martensite microstructure showed rather lower yield strength (YS) but higher ultimate tensile stress (UTS) which is corresponding to more ductile feature of the fracture surfaces with cracked martensite needles rounded by ductile β phase. Unlike martensitic microstructure the lamellar α + β structure increased the YS value by 4 % but decreased the UTS by 13 %.

Macroscopically, the failure feature of the fine lamellar microstructure seemed to be very similar to martensitic one, the evident difference consisted in thickness of martensitic needles and α laths that determined the size of brittle cleavage facets. As both phases are inserted in the ductile β phase the impact of the martensite to α + β transformation on the fracture feature was less apparent.

Figure 2. Microstructure of Ti6Al4V specimens produced by SLM process: a) martensite needles in prior β phase and voids (as-built before HT) and b) α + β lamellae (after HT).

Figure 3. Tensile fracture of Ti6Al4V specimen tested in as-built condition: a) view of rugged fracture surface, b) detail of mixed failure feature with shear areas, transgranular quasi-cleavage surfaces and voids.
Figure 4. Tensile fracture of Ti6Al4V specimen tested in heat treated condition: a) view of rugged failure surface, b) detail of mixed fracture feature with shear areas, transgranular quasi-cleavage surfaces, voids and unmelted powder particles.

Figure 5. Compression fracture of Ti6Al4V specimen tested in as-built condition: a) view of smooth failure surface, b) detail of mixed feature of fracture with shear areas and ductile dimples.

Figure 6. Compression fracture of Ti6Al4V specimen tested in heat treated condition: a) view of mixed failure feature, b) detail of steps, secondary cracks, shear areas and transgranular quasi-cleavage surfaces.
After compressive tests both as-built and HT conditions showed the failures with smooth surfaces (figures 5 and 6). The HT changed the predominant shear feature of the fracture with elongated very shallow dimples (figure 5a) to more rugged one with occurrence of secondary cracks, sharp steps, transgranular quasi-cleavage surfaces and elongated ductile dimples (figure 6b) that corresponded to microstructure changes as it was stated for tensile tests. In this case, martensitic structure in compression showed higher mechanical values by 3 % and 5 % for the YS and UTS, respectively (table 1), together with higher ductility by 35 %.

4 Conclusion

The fractographic study was performed on the specimens prepared by the selective laser melting process and tested in tensile and compressive loading. The SLM material originally consisted of fine α' martensite needles and after two-step heat treatment the microstructure transformed to α + β lamellae. The failure surfaces for both type of loading corresponded to microstructure states in accordance with very small changes in mechanical values.

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