3D PRINTING OF BIOMEDICAL TITANIUM ALLOY

FOUSOVÁ Michaela, VOJTĚCH Dalibor, KUBÁSEK Jiří

University of Chemistry and Technology Prague, Czech Republic, EU

Abstract

Selective Laser Melting (SLM), an additive manufacturing technology, shows a high potential for preparation of biomedical implants, especially from reason of its capability to achieve geometrically complex shapes. Therefore, a preliminary research was done in order to characterize microstructure and mechanical properties of titanium alloy Ti-6Al-4V, one of the most frequently used materials for orthopaedic implants. As results show, SLM yields comparable strength properties as the wrought material, thus makes it an appropriate manufacturing alternative. Only drawback is represented by reduced elongation associated with defect porosity, which can be, however, optimized by an ideal combination of process parameters. In conclusion, SLM has a good outlook for the preparation of customized medical devices exactly reproducing the patient's defect being treated and porous implants with properties corresponding to human bone enhancing biological and mechanical fixation.

Keywords: 3d printing, slm, titanium, ti-6al-4v alloy

1. INTRODUCTION

Owing to its capability to reach complex geometry details, additive manufacturing (AM) that was originally confined to the production of prototypes has recently attracted the attention of modern domains in industry. These domains include manufacturing of aerospace components, biomedical implants, tooling inserts or artwork. Among all the AM methods that have been developed until now, Selective Laser Melting (SLM) dominates [1].

Selective Laser Melting (SLM) is a powder-based method, in which solid products are formed in a layer-by-layer fashion by melting powder particles and fusing them together. Part fabrication takes place in a chamber filled with an inert atmosphere (argon or nitrogen gas) to avoid oxidation. The input material is a single-composition metallic powder prepared by atomization process. Many different metal powders can be processed, such as stainless steel, maraging steel, cobalt-chromium and titanium alloys or others. A metallic powder is deposited by a feeder onto the base plate and spread into a thin layer (tens of micrometers). A laser beam carried by an optic fiber is focused onto the powder bed, selectively melts the powder particles according to the data derived from a 3D CAD model and fuses them to the previous layer. As laser scanning of one layer is finished, the base plate is lowered and new powder is deposited. This process is repeated until the whole part is formed [2-4]. For illustration, a simplified SLM schema is shown in **Figure 1**. The image of the input material represents a gas atomized powder of Ti-6Al-4V alloy used in the present experimental work and the output is directly a tensile test specimen.

In comparison with conventional fabrication routes, SLM offers a whole range of benefits. The layer-by-layer manufacturing fashion provides freedom in product geometry and allows obtaining complex shapes that would be impossible to achieve by another way. Since the product is provided in the near-net form, no component-specific tooling is needed, which saves not only production time, but also costs. Furthermore, the costs are also reduced thanks to the high material utilization. As the input material is in the form of powder, which is selectively melted by laser beam only in areas destined to solidify to form the final product, the remaining unmelted powder can be easily recycled and reused in other production cycles. Thus, there are almost no material losses [1, 5].

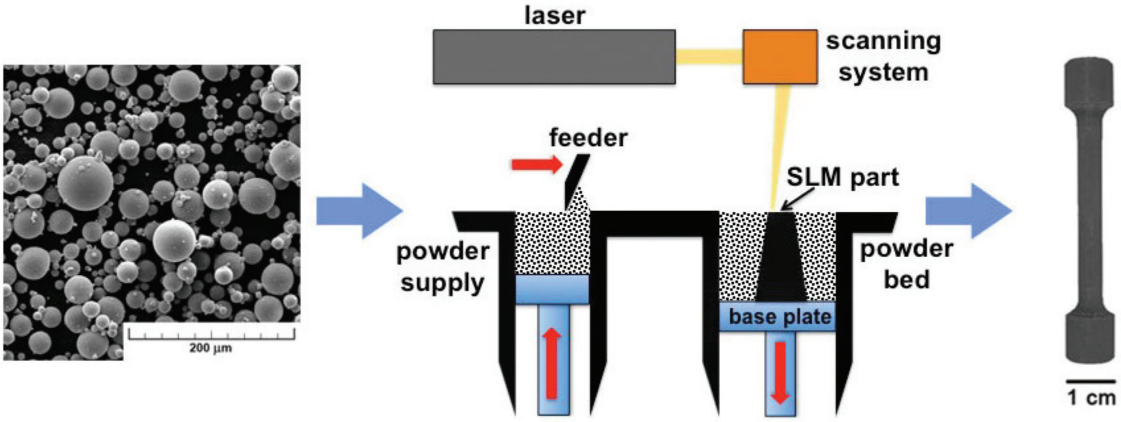

Figure 1 SLM process schema with an input powder material and an output tensile specimen

For the fabrication of customized orthopaedic implants, titanium alloy Ti-6Al-4V is an appropriate material. Its predominance in biomedical field is given by high strength-to-density ratio, superior corrosion resistance and excellent biocompatibility determined by biological inertness. As a duplex structured alloy, it contains a hexagonal close-packed (hcp) α phase and a body-centered cubic (bcc) β phase. However, when prepared by laser melting, typically acicular martensite α' is formed due to the rapid cooling. In order to eliminate high residual stresses associated with martensitic phase, sub-β heat treatment is suggested to ensure its decomposition into a fine α+β structure [6, 7].

Present paper deals with novel approach in preparation of biomedical alloys - additive technology Selective Laser Melting (SLM). In particular it concerns titanium alloy Ti-6Al-4V (Grade 5) widely used for orthopedic or dental implants. It focuses on characterization of microstructure and mechanical properties of model specimens of this alloy prepared under the process parameters set-up according to the manufacturer recommendation. It represents the first step to a future optimization.

2. METHODOLOGY

2.1. Selective Laser Melting

The Selective Laser Melting process was carried out on a M2 Cusing machine (by ConceptLaser) with one 200 W fiber Yb:YAG laser. The process parameters were set as given in **Table 1**. Laser scanning was accomplished in a continuous regime under a protective atmosphere of high purity argon gas (max. 0.5 vol.% O_2).

Table 1 SLM process parameters

Laser power (W)	Beam spot (µm)	Scanning speed (mm/s)	Layer thickness (µm)	Hatching distance (µm)
200	200	1250	30	80

Four specimens intended for tensile tests were prepared directly from Ti-6Al-4V alloy gas atomized powder (rematitan® CL, Dentaurum, 15-45 µm). In order to eliminate internal stresses built up during the melting and subsequent solidification process, the specimens were subjected to a heat treatment at 1093 K (1.5 h, argon atmosphere).

2.2. Metallography

Microstructure of laser melted Ti-6Al-4V alloy was studied on prepared metallographic sections. Several cross sections through the whole length of the sample were moulded into an acrylic resin VariDur 200, grinded on SiC grinding papers and polished on a diamond paste D2 and final polished on colloid SiO_2 mixed with 30% H_2O_2 (20 vol.%). At first, porosity was evaluated on as-polished samples with the use of light microscopy (OLYMPUS PME3 light metallographic microscope) and image analysis (software ImageJ). Subsequently, samples were etched in Kroll's reagent (100 ml H_2O, 3 ml HF, 6 ml HNO_3) and microstructure was studied by a TESCAN VEGA-3 LMU scanning electron microscope (SEM).

2.3. Chemical analysis

Chemical composition was determined by Oxford instruments INCA 350 EDX analyzer (SEM-EDX). Samples consisted of titanium with 6.4 wt.% of Al and 4.0 wt.% of V, which is in accordance with ASTM standard, where allowed range is 5.5-6.75 wt.% for Al and 3.5-4.5 wt.% for V. X-ray diffraction was carried out to reveal phase composition (PANalytical X'Pert PRO diffractometer equipped with Cu anode). However, this method did not provide accurate quantitative results. Therefore, it was complemented by an image analysis of BSE (back scattered electrons) images.

2.4. Mechanical properties

Mechanical properties were tested in uniaxial tension. Tensile tests were carried out on three specimens using a universal testing machine LabTest 5.250SP1-VM. Strain rate was set up to 0.001 s^{-1}. Morphology of fracture surfaces was studied by the scanning electron microscope. Also, the Vickers hardness with 1 kg load (HV1) was measured on a Future-Tech FM-700 hardness tester (according to ČSN EN ISO 6507).

3. RESULTS AND DISCUSSION

3.1. Microstructure

Microstructure of SLM Ti-6Al-4V alloy is shown in **Figure 2**. In the overview image (**Figure 2a**) some porosity is visible. The overall porosity was determined to be 1.6 %. The pores are of irregular shapes rather then spherical, more or less flat. Existence of such pores can be attributed to insufficient melting, which is related to the process parameters set-up. Energy density (*E*) determining the melt pool temperature is influenced by laser power (*P*), scanning speed (*v*), layer thickness (*t*) and hatching distance (*h*) as shown in equation (1).

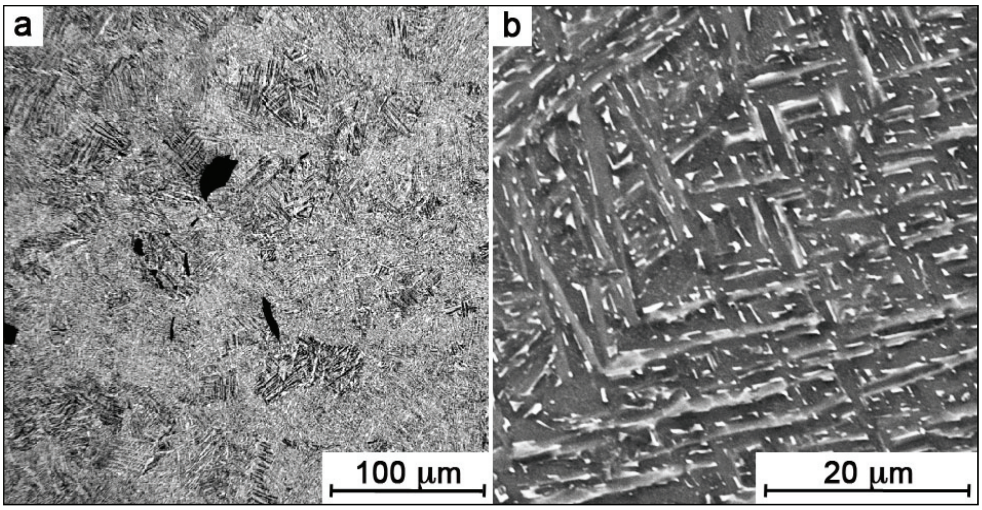

Figure 2 Microstructures of SLM Ti-6Al-4V

$$E = \frac{P}{v + h + t} \quad (1)$$

When the energy input is insufficient, successive scan tracks do not fuse together properly and defects appear along the scan lines [2]. The quality of final product is also dependent on properly selected scanning strategy.

At higher magnification **(Figure 2b)**, two-phase lamellar microstructure can be observed. The two-phase composition was confirmed by X-ray diffraction. Diffraction pattern in **Figure 3** shows peaks of majority α phase with hexagonal packing and much lower intensity peak for β phase (BCC packing). Therefore, it can be concluded the matrix is formed by fine grains of α with β phase lamellae along the boundaries. In backscattered electrons contrast in **Figure 2** β phase richer in vanadium, as it was proved by point EDX analysis, represents bright lamellae (thinner than 0.5 µm) in between the plates of α. The volume fraction of β phase was evaluated as 12.5 %.

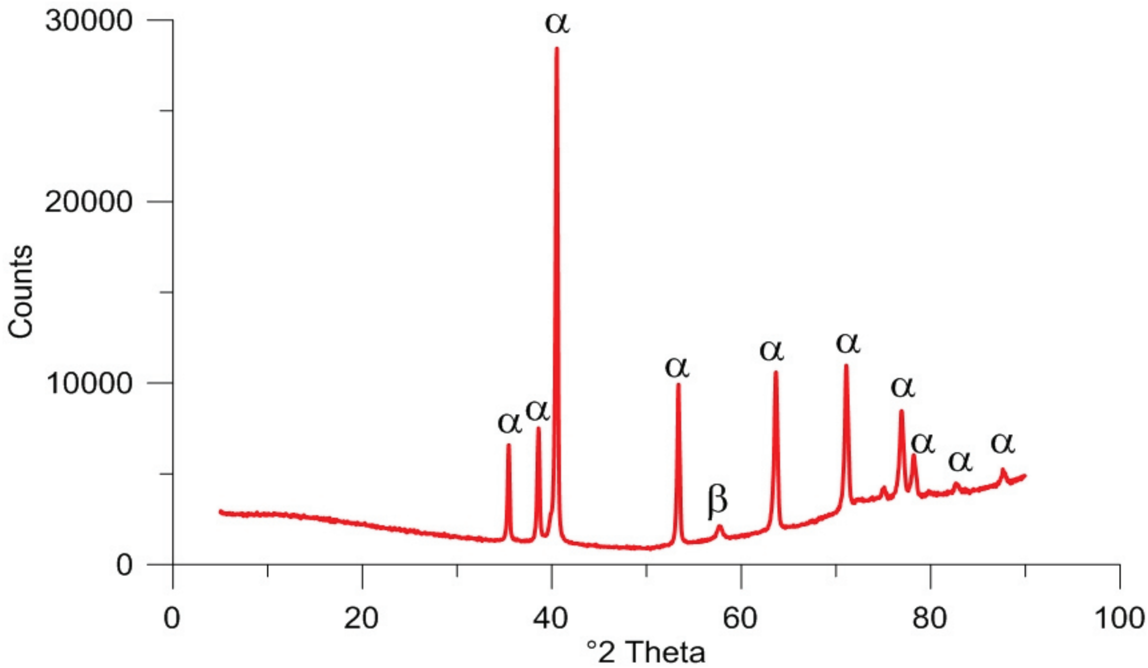

Figure 3 XRD pattern of Ti-6Al-4V alloy prepared by SLM and subsequent heat treatment

The reason for current phase composition corresponding to conventional wrought alloy lies in the post-production heat treatment. Due to rapid cooling of selectively laser melted regions during SLM process, thermodynamically unstable martensitic phase α′ was formed. However, by heating the as-fabricated sample up to the temperature of 1093 K lying in the α+β region, the originally formed acicular martensite transformed into α+β structure.

3.2. Mechanical properties

The mechanical properties determined by tensile test and hardness measurement are summarized in **Table 2**. The average yield strength and ultimate tensile strength reached 940±10 MPa and 989±10 MPa, respectively. These values are comparable to those given for wrought Ti-6Al-4V alloy (according to ASM International [8]). The same goes for Young's modulus (119±10 GPa) and hardness (347 HV1). Only measured elongation was significantly lower. That may be explained by a certain porosity observed within the SLM samples (**Figure 2a**), since the defects facilitate the crack initiation and propagation.

Table 2 Mechanical properties of SLM Ti-6Al-4V (*YS* = yield strength, *UTS* = ultimate tensile strength, *A* = elongation, *E* = Young's modulus, *HV*1 = Vickers hardness)

sample	*YS* (MPa)	*UTS* (MPa)	*A* (%)	*E* (GPa)	*HV*1
wrought [8]	830-924	900-993	14	105-116	349
SLM 1	950	1000	3	115	347
SLM 2	939	988	1.3	132	347
SLM 3	931	980	2.1	110	347

Concerning mechanical properties of SLM materials, there are two contradictory effects. On one side, lower grain size, obtained thanks to the rapid melting and subsequent cooling during which grains do not have enough time to coarsen, is associated with an increase in strength. On the other hand, this increase is hindered by porosity, which has an adverse effect. As a result, strength properties remain almost equivalent to the wrought material.

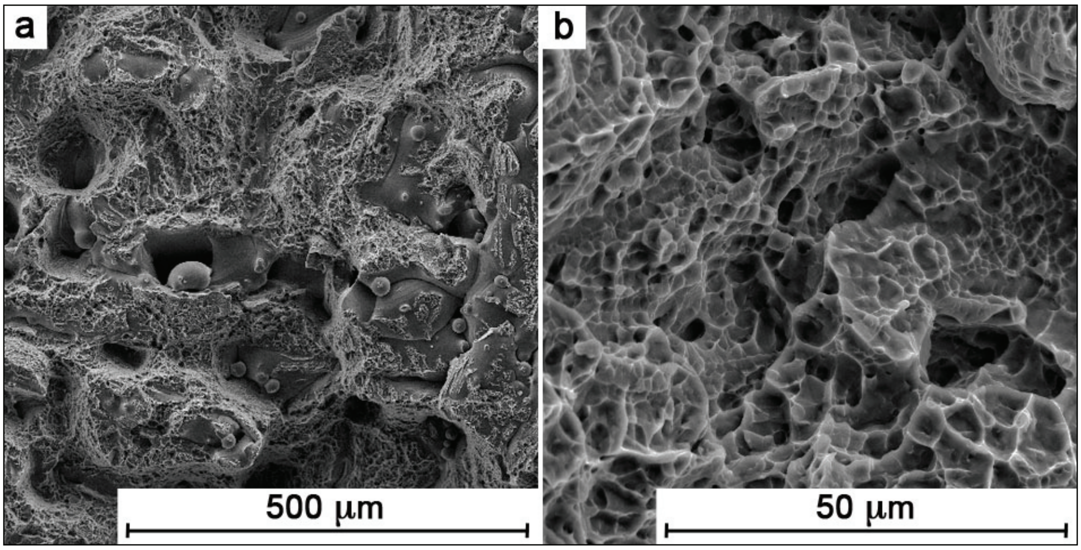

Figure 4 Fracture surface

In **Figure 4** fracture surface after tensile test is displayed. At the first sight, fracture surface shows ductile morphology with shallow dimples. The fracture surface is relatively rugged as the crack propagates not perpendicularly to the loading direction, but through the pores representing the weakest parts in the structure. In **Figure 4a** defects present inside the specimen can be clearly seen. The surface inside the defects is smooth with no sign of fracture, as this part was not interconnected with the subsequently melted layer in the layer-by-layer laser melting process. Also some unmelted spherical particles of the input powder material can be noticed.

4. CONCLUSION

Our paper has shown that Selective Laser Melting (SLM) is an appropriate alternative to the conventional preparation of titanium alloy Ti-6Al-4V in term of mechanical properties. Microstructure of the model specimen with two-phase composition was very fine, with plate like α grains separated by submicrometer thin β lamellae. Yield strength (*YS*) and ultimate tensile strength (*UTS*) even slightly overpassed the average values given for the wrought material. That is probably related to a lower grain size brought about by rapid cooling during SLM process. On the other hand, elongation suffered from significant reduction compared to the wrought material resulting from present porosity. However, with future optimization of process parameters the reduction in porosity is presumed. Obvious advantage of SLM over conventional way of casting, forging and machining is one-step manufacturing. In terms of implant production, it is especially the geometric freedom allowing preparation of customized devices. Therefore, our future efforts will be focused on deeper characterization of selective laser melted titanium alloy and preparation of a suitable orthopaedic implant.

ACKNOWLEDGEMENTS

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