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Phase Composition and Microstructure of Ti-Nb Alloy Produced by Selective Laser Melting

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Abstract. The phase composition and microstructure of Ti-Nb alloy produced from composite titanium and niobium powder by selective laser melting (SLM) was studied. Produced monolayered Ti-Nb alloy enhanced the formation of fine-grained and medium-grained zones with homogeneous element composition of 36-38% Nb mass interval. Alloy phase composition responded to β-alloy substrate phase (grain size was 5-7 μm) and non-equilibrium martensite α''- phase (grain size was 0.1-0.7 μm). α''-phase grains were found along β-phase grain boundaries and inside grains, including decreased niobium content. Alloy microhardness varied within 4200-5500 MPa.

1. Introduction

Bioinert low-modulus Ti-Nb alloys [1] are more preferable as medical alloys. Due to their physic-mechanical characteristics—mainly low elastic modulus and bioinert properties—these alloys are excellent materials for medical implants [1, 2]. It should be mentioned, that Nb-doped alloy including 40 wt% of Nb (Ti40Nb) exhibits the lowest elastic modulus for such a system (55-70 GPa*)* [1]. The elastic modulus for most alloys is within the range of 100-120 GPa [4, 6], which, in its turn, is significantly higher than in the case of bones (15-50 GPa). Based on the above-mentioned facts, an upcoming trend in medical materials science is the production of biocompatible titanium alloys with low elastic modulus, for example, alloys of Ti-Nb or Ti-Nb-Zr systems [6]. Traditional methods used in producing the titanium-niobium alloys are extraction metallurgical methods. Nevertheless, the application of traditional methods for producing titanium-niobium alloys with homogeneous microstructures is not always effective. There are also so-called methods of powder metallurgy, which could include additive manufacturing technology. Applying additive technology, for example 3D-printing mode selective laser melting (SLM) could be, in most cases, the only alternative method in manufacturing composite mold castings and/or parts using NC (numerical control) machine [3-5]. In this respect, the SLM method could be rather challenging in the production of implants and endoprostheses. Another important aspect is that this method produces items with specified porosity, which, in its turn, improves the implant material to bones osseointegration. This is today's current issue involving both the development of a SLM method and the study of phase composition and structure formation. This paper describes the investigation of specific features of alloy structure formation and phase composition of Ti40Nb produced by SLM.

2. Experimental Procedure

Ti-Nb alloy samples produced by SLM using VARISKAF 100mV were investigated [6-8]. As the melting temperature of titanium (1725ºC) and niobium (2468ºC) are significantly different, composite titanium and niobium powder (60 wt% Ti and 40 wt% Nb) with average particle size of 20 μm was used as the reference material. Powdered material was sifted on VT1-0 substrate as a thin layer with homogeneous thickness and density. The following SLM modes were selected: laser radiation power, 105W; laser beam scan speed, 2000-3000 mm/min; laser beam diameter, 0.4 mm; scan step, 0.1 mm; substrate preheating, 200 °C. The scanning direction of each succeeding layer was altered by 90°. To produce a monolayer, the composite powdered layer thickness was 0.5 μm, determining the melt layer thickness and deleting the monolayer from the substrate.

Optical microscopy X-ray phase analysis, SEM, TEM and microhardness testing were performed using DRON-7, LEO EVO 50, AXIOVERT-200MAT, JEM-2100 and Duramin-5 (SKC ”NANOTECH”, ISMPS SB RAS, Russia).

3. Results and Discussion

Molding of bulk sample by SLM method was based on scanning along a predetermined trajectory by melting thin powder layer and crystallizing the material on interlaying substrate tracks. Multi-layered sample surface topography is identical to a monolayer, as the molding of each succeeding layer in the sample replicates the previous monolayer. In this case, the molding of a monolayer in the first cycle is the most important phase and determines the entire homogeneous structure-phase molding of the bulk produced sample. The total thickness of melted monolayer amounts up to 300 μm.

SEM investigation of sample cross-sections showed that the bulk sample is molded as a result of the superposition of the layers, which could be described as monolayer (Figure 1). Such a monolayer embraces completely melted and crystallized material. There are two zones within the monolayer. The first one involves fine-grained structure with grain size of 2 to 8 μm, further merging into medium-grained structure (zone 2) within an interval of 7 to 24 μm. Pores can be observed across the monolayer section.

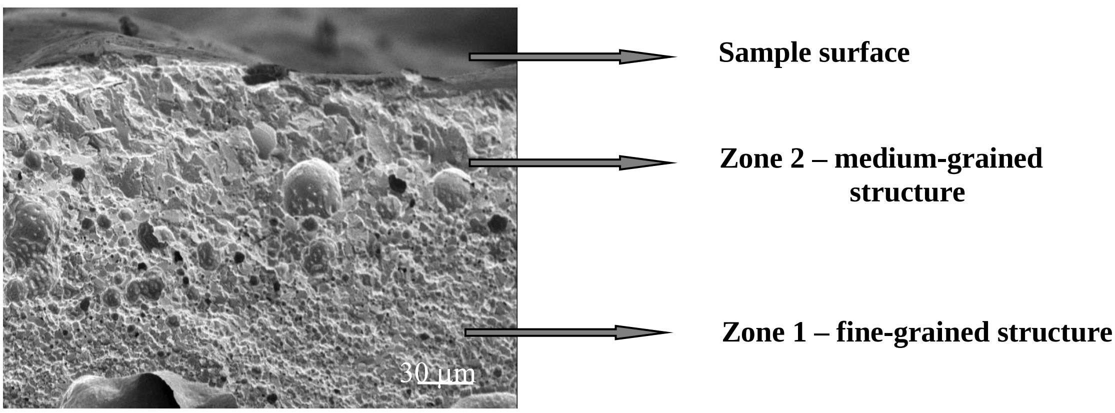

Figure 1. SEM image of fracture surface

In terms of zone structure aspects, the monolayer molding could be described as follows. Under exposure of a laser beam, the powdered material in the vicinity of the heating source, i.e. upper powdered layer, is melted. This results in the formation of lower spongy monolayer zone. During cooling, heat dissipation is evident in the zones of sintered powder and substrate in the upper melt material. Inversely from the boundaries, the sintered powdered material crystallizes forming clowholes. In the lower crystallizing bath, the increase of cooling rate furthers the formation of fine-grained structure with inclusions of small gas pockets along the grain boundaries. In the upper crystallizing bath, where heat dissipation is impeded in the substrate, cooling provides rather balanced material crystallization conditions. In this case, a medium-grained structure zone with average porosity and large grains is formed in the upper monolayer. Between the upper and lower crystallization zones large rounded pores emerge. As a result of layering, the translation of the monolayer structure itself occurs within the monolayer sample.

Thus, it can be stated that the formation of distinct fine-grained and medium-grained structured zones in the sample section is related to different temperature-time conditions of powder melting, crystallization and cooling in selective laser melting. It should be noted that monolayer microhardness in axial section ranges from 4200 to 5500 MPa.

Regardless of the non-uniform structure of the monolayer, titanium and niobium are distributed evenly throughout all the zones, as indicated by the mapping results of fractures by SEM-EDST (Figure 2). Quantitatively, the basic melt components are insignificant, 36-38 wt% of Nb.

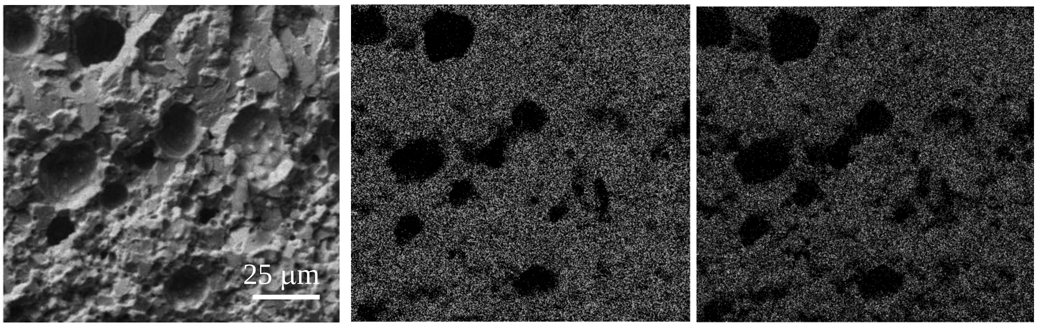

Figure 2. SEM images of fracture (a); Ti distribution (b); Nb distribution (c)

Optical image of zone 1 with fine-grained structure is depicted in Figures 3a and 3b. Basically, the microstructure embraces equiaxial grains of 5 µm. Grain boundaries are decorated with less than 1 µm grains of different phase (Figure 3a). These tabular grains are characteristic for the martensite phase (Figure 3b). These samples can be identified as reflections from the diffracting plane of β-BBC (body-centered cubic) of Ti and Nb solid solution and non-equilibrium α"- phase with orthorhombic lattice on X-ray diffractograms (Figure 3c). The existing α"-phase is associated with the unbalanced cooling and crystallization conditions of composite powder [9, 10].

TEM method was used to investigate the microstructure of produced melt samples. Light field images of Ti40Nb microstructure sample with corresponding microdiffractions are depicted in Figures 4a and 4b.

Rather small grains of 0.1-0.7 µm are located on the β-alloy grain boundaries, which in turn corresponds to ultra-fine grained structure (Figure 4b). These tabular grains indicate the fact that they were formed in unbalanced conditions during cooling of melted powder. Microdiffractions show reflections of metastable titanium α''-phase (orthorhombic crystalline lattice), which could be characteristic for possible martensite phase formation [10]. It should be noted that α''-phase grains include low content of Nb of 13-19 wt%. Reflexes of α''-phase for zone axis $[\bar{1}01]_{\alpha''}$ were identified on the microdiffraction.

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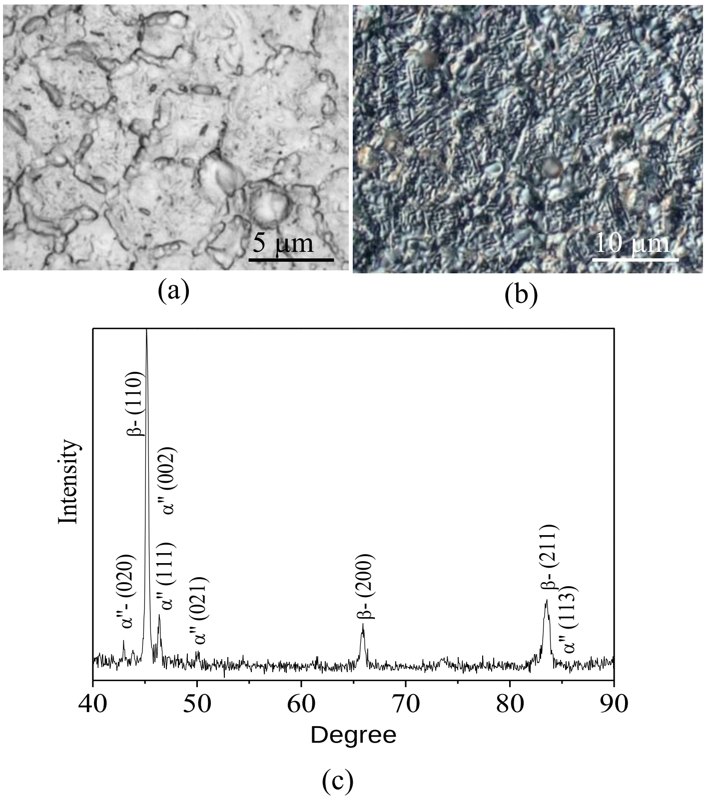

Figure 3. Optical images of the microstructure (a, b) and X-ray diffractogram fragment of the sample (c)

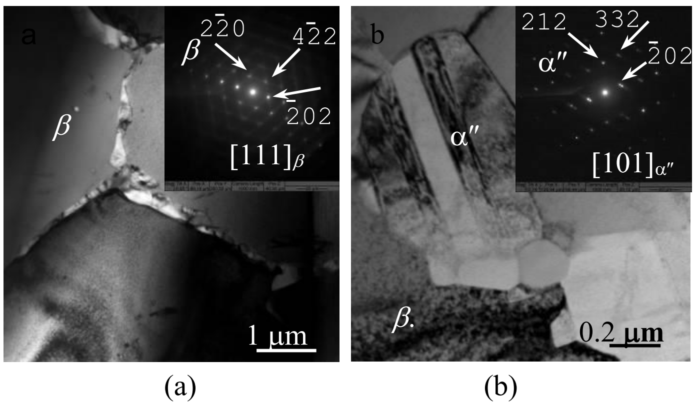

Figure 4. Light-field electron-microscopic images with corresponding microdiffractions of β-phase (a) and α''- phase (b)

Consequently, SLM method leads to molding of Ti-Nb alloy monolayer from composite powder of homogeneous element composition, which in its turn includes medium-fine grained structure zones. Forming highly homogeneous microstructure of bulk samples with specific geometrical dimensions requires specific conditions for powder cooling and crystallization. In this case, the monolayer and the item itself will have identical microstructures embracing rather fine grains and insignificant porosity.

4. Conclusions

Ti-Nb alloy monolayer with homogeneously distributed titanium and niobium was produced from composite titanium and niobium powder by SLM method. This monolayer embraced upper zone of fine-grained structure and lower zone of medium-grained structure. Average Nb content in the alloy within the monolayer along and across the sample changes insignificantly 36-38 wt%. Ti-Nb alloy includes two-phase state, i.e. base β-phase (grain size, 5-7 μm) and non-equilibrium martensite α″-phase (grain size is 0.1-0.7 μm), located on grain boundaries of β-phase. Grain of β-phase involves high Nb content (up to 40 wt%). Decreased Nb content (up to 20 wt%) is observed in grains of α''-phase. Formation of fine-grained and medium-grained structure zones are related to different temperature time conditions of crystallization and cooling of a layer melted during SLM.

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