The Investigation of the Influence of Formation Conditions on the Structure of Ti-40Nb Alloy

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Abstract. It was determined that there is components segregation in Ti-Nb alloy under electro arc melting and selective laser melting. It leads to two phase formation. The β-phase forms in the areas enriched in Nb. α"-phase forms in the Nb-depleted areas. It is recommended to increase the Nb concentration in the alloy up to 45 wt. % to eliminate heterogeneity of phase and elemental composition.

INTRODUCTION

Important requirements for metallic biomaterials are the absence of toxicity of alloying elements in the alloy composition and low elasticity modulus comparable to the human bone module [1]. Because of its bioactive and biocompatible properties calcium phosphate (CaP) coatings are applied to prevent post-operative complications [2]. CaP coating is formed on the surface of metallic implant. Thus, the problem of biocompatible properties of implant material is solved [3].

The Ti-Nb alloys are used for the production of medical materials and bioimplants [4]. Previously it was founded that the alloys containing 40-45 wt. % Nb are the most suitable for bioimplants production. These alloys have the lowest Young's modulus which is retained after external influences. It is very important for mechanical compatibility of implant and human bone [5].

Primary dendritic structure is formed during the process of Ti-Nb ingots crystallization. Parameters of this structure depend on crystallization conditions and size of obtained ingot [6]. The formation of dendritic structure is accompanied by the formation of chemical heterogeneity in the bulk of grain called as dendritic segregation. The dendritic segregation retaining in subsequent processing steps causes heterogeneity of physical and mechanical properties of the hardware. It is unacceptable for the materials used in medicine.

The advanced treatment methods as severe plastic deformation, electron arc and laser treatment can eliminate the structural heterogeneity in the alloy. They can increase strength characteristics of the alloy and insignificantly increase modulus of elasticity of the hardware at the same time [7-9].

One of the modern methods of implants production is the method of selective laser melting (SLM). It is possible to obtain products with prescribed porosity by SLM method. Thereby, structure of implant can be corresponded to the human bone structure with the precise form repeating shape of any bone fragment [9].

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The aim of this investigation was to study conditions of the formation of the Ti-Nb alloy in different crystallization conditions. Two methods of electron arc melting and selective laser melting were used. It was necessary to estimate changes of conditions of the alloy structure formation at significant increase of cooling rate in the process of SLM.

MATERIALS AND METHODS

Ingots produced by the electron arc melting method using a nonconsumable electrode in the water cooled copper crucible are investigated in this paper [10]. The mass of each ingot was 300 g. The shape of the ingot was a "button" form with diameter of 80 mm and high of 20 mm. The structure of the ingot after melting and subsequent quenching with preheating up to 1000°C during 3 hours and cooling in water was investigated.

SLM was carried out on "VARISKAF 100MV" installation [11]. The composite powder material obtained by mechanical alloying Ti and Nb was overlaid on VT1-0 titanium substrate [12]. SLM conditions were as follows. Power of laser beam was 105 W. Scanning speed of laser beam was 2000 mm/min. The spot diameter was 0.7 mm, scanning step was 0.05 mm and the substrate temperature at the beginning of melting was 200°C. Laser beam scanning direction was changed by 90° for each subsequent layer. The specimens consisting of seven layers were investigated.

The investigations were carried out in the shared use centers "NANOTECH", ISPMS SB RAS Tomsk and NSTU, Novosibirsk, in the laboratory MIMAM, NR TPU Tomsk, on DRON-7 diffractometer (Burevestnik, Russia), scan electron microscope with energy dispersive microanalysis (EDMA) LEO EVO 50 (Zeiss, Germany) and metallographic microscope Carl Zeiss Axio Observer (Zeiss, Germany).

RESULTS AND DISCUSSION

Primary dendritic-cell structure in the contrast with secondary grain structure is observed on metallographic images of cast alloy (fig. 1a). The mean distance between secondary dendrite branches is about 100 μm and the size of secondary grains was varied between 80 and 1000 μm [10]. The size of structural elements indicates that cooling rate of the alloy during ingot crystallization obtained by electron arc melting is about 100 deg/s.

The dendritic structure in the ingot indicates presence of dendritic segregation in the alloy. Dendritic segregation leads to heterogeneity of alloy components content in different zones of dendrites. EDMA showed that quantitative ratio of the main components of the alloy in the ingot is approximately 58 wt. % Ti and 42 wt. % Nb. The Nb concentration in dendrites is greater than that in interdendritic space (fig. 1b). Presence of dendritic segregation is typical for the alloys of Ti with β-stabilizers. Analyzing constitution diagram of Ti-Nb system [13] it can be concluded that during crystallization of the alloy in equilibrium conditions the difference between Nb concentrations in different zones could be equaled up to 16 wt. % [10]. The range of possible concentrations decreases with increasing of cooling rate as can be seen by the results of EDMA. Despite of a small scatter of Nb concentrations the presence of segregation is a negative factor. It leads to the formation of different phases in dendrites areas with different Nb concentration. Different structures can be formed in the alloy.

This conclusion is confirmed by the results of x-ray diffraction analysis (XRD). The main phase in the alloy is β-phase (bcc solid solution of Ti and Nb). The main β-phase peaks can be seen on X-ray diffraction patterns (fig. 1c). Furthermore, the faint α''-phase peaks can be identified on x-ray diffraction patterns (solid solution of Nb in Ti with orthorhombic cell). α''-phase is nonequilibrium and forms in conditions of quenching from liquid state. Consequently, the process of the ingot formation occurs in nonequilibrium conditions. This conclusion is confirmed by the results of investigation of Ti-Nb alloy ingots with other chemical composition but the same mass [10].

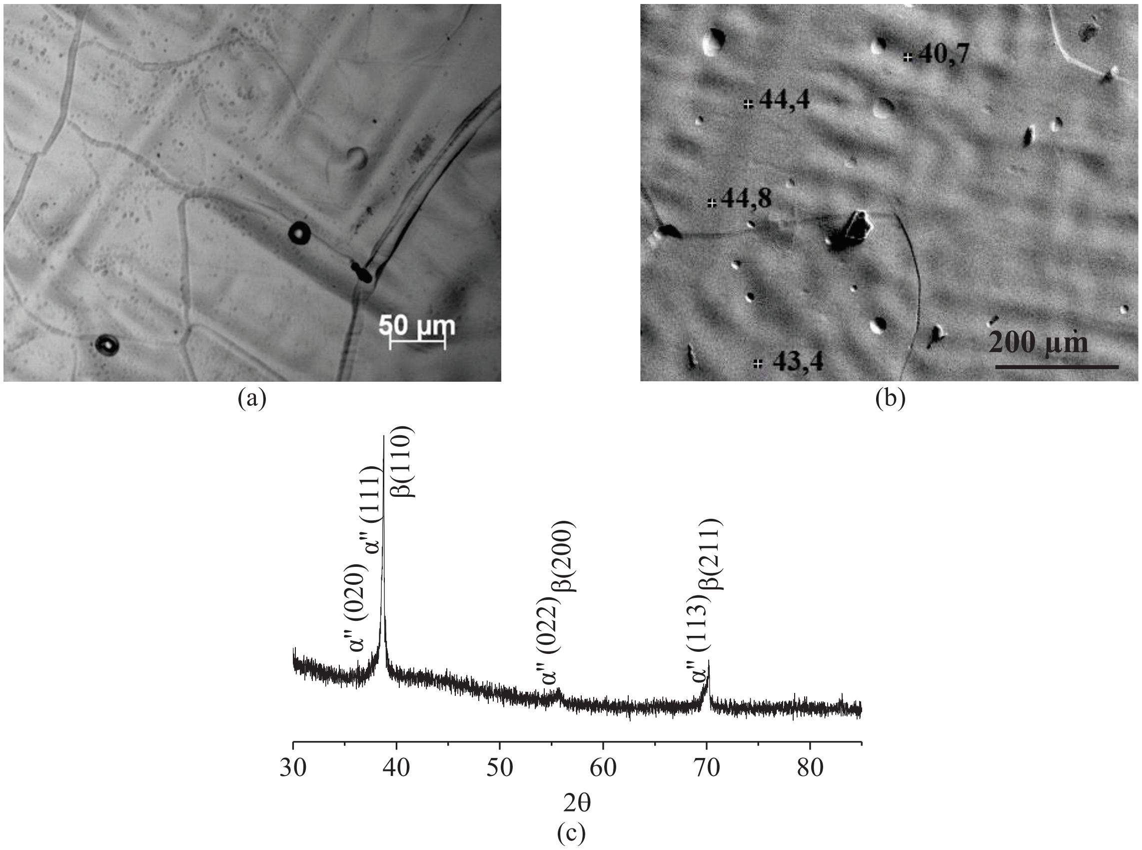


FIGURE 1. The image of microstructure (a), concentration of Nb (wt. %) in structural elements (b) and fragment of X-ray diffraction pattern (c) of the cast specimen

Only β-phase grains are observed on metallographic image of the ingot (fig. 1a). They have distinct clear visible boundaries. Martensitic structure which is typical for the phases obtained in nonequilibrium cooling conditions is not observed. Probably, its quantity so small that the phase with nonequilibrium structure is not visualized.

Quenching of obtained ingot was carried out to identify areas with nonequilibrium structure. The structure typical for the alloy after nonequilibrium cooling is formed in the alloy after quenching. The structure of two types is observed on metallographic images of quenched specimen (fig. 2a). These types are areas with needle structure and needles-free areas.

The grains of nonequilibrium α''-phase (martensite) has needle-like structure. Needle-free areas likely belong to β-phase. As can be seen at low magnifications primary dendritic structure with some blurring of the contrast is retained. The needle-free areas are formed in dendrites branches. The needle structure is observed in interdendritic space. Consequently, the two-phase structure of the alloy after quenching is caused by partially retaining of dendritic segregation after heating for quenching. EDMA results have shown that Nb concentration in needle-free areas is about 46 wt. %. The Nb concentration decreases to 39 wt. % in martensite needles. The difference of Nb concentrations in different dendrites zones after quenching is 6-7%. Decrease of Nb concentrations scatter did not occur despite diffusional processes which accompany aging of the specimens at high temperature during quenching. Three hours aging did not eliminate dendritic segregation. Nonequilibrium cooling conditions of the alloy have led to the formation of martensitic structure in Nb-depleted areas.

α''-phase peaks are identified on X-ray diffraction patterns (fig. 2c). Some peaks typical for α''-phase are absent. There is a redistribution of peaks intensities. These features of pattern indicate the formation of directed structures in the specimen during quenching. Nonequilibrium α''-phase can be formed in alloys with Nb concentration up to 40.5 wt. % as shown in previous researches [7, 10]. β-phase is not identified by XRD. It can be explained by its small quantity in the bulk of material.

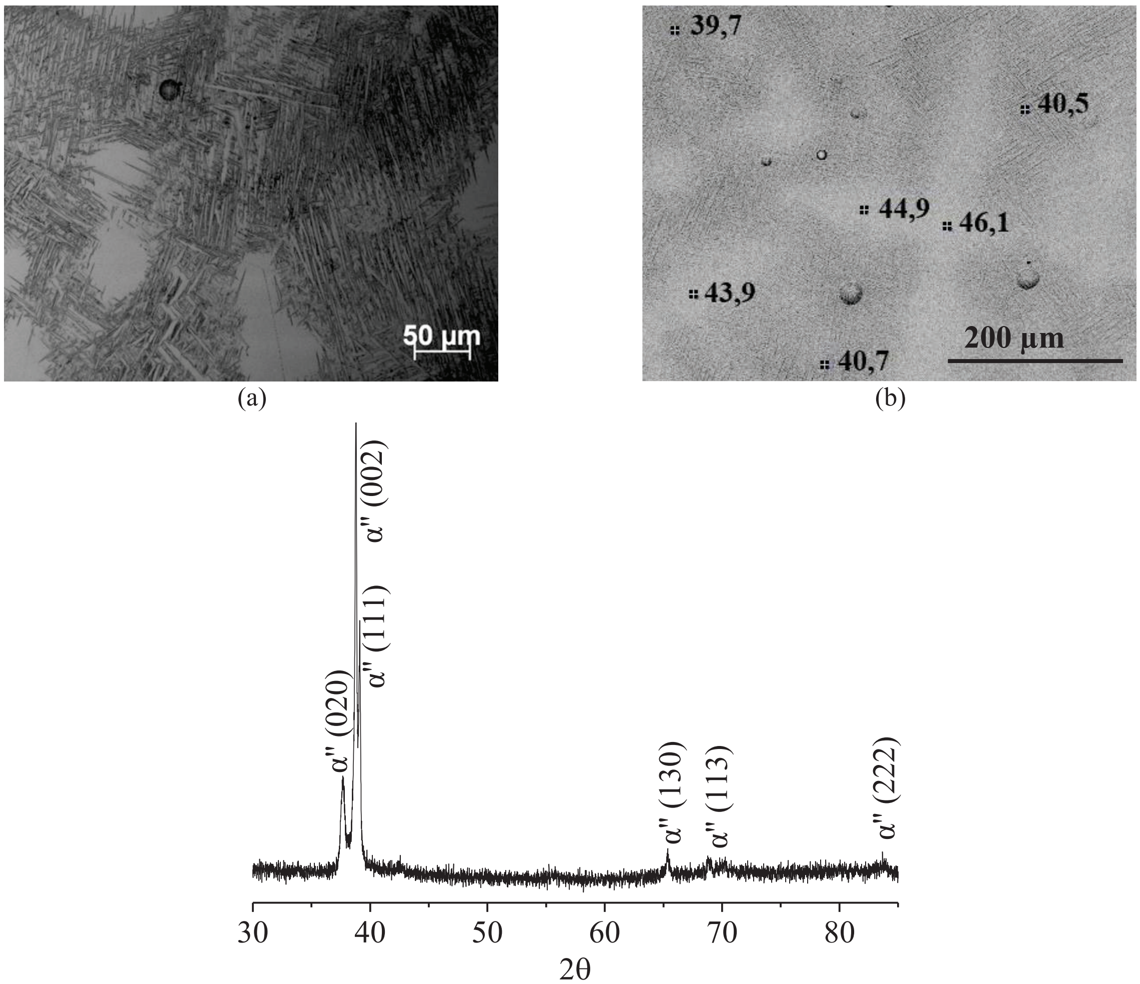
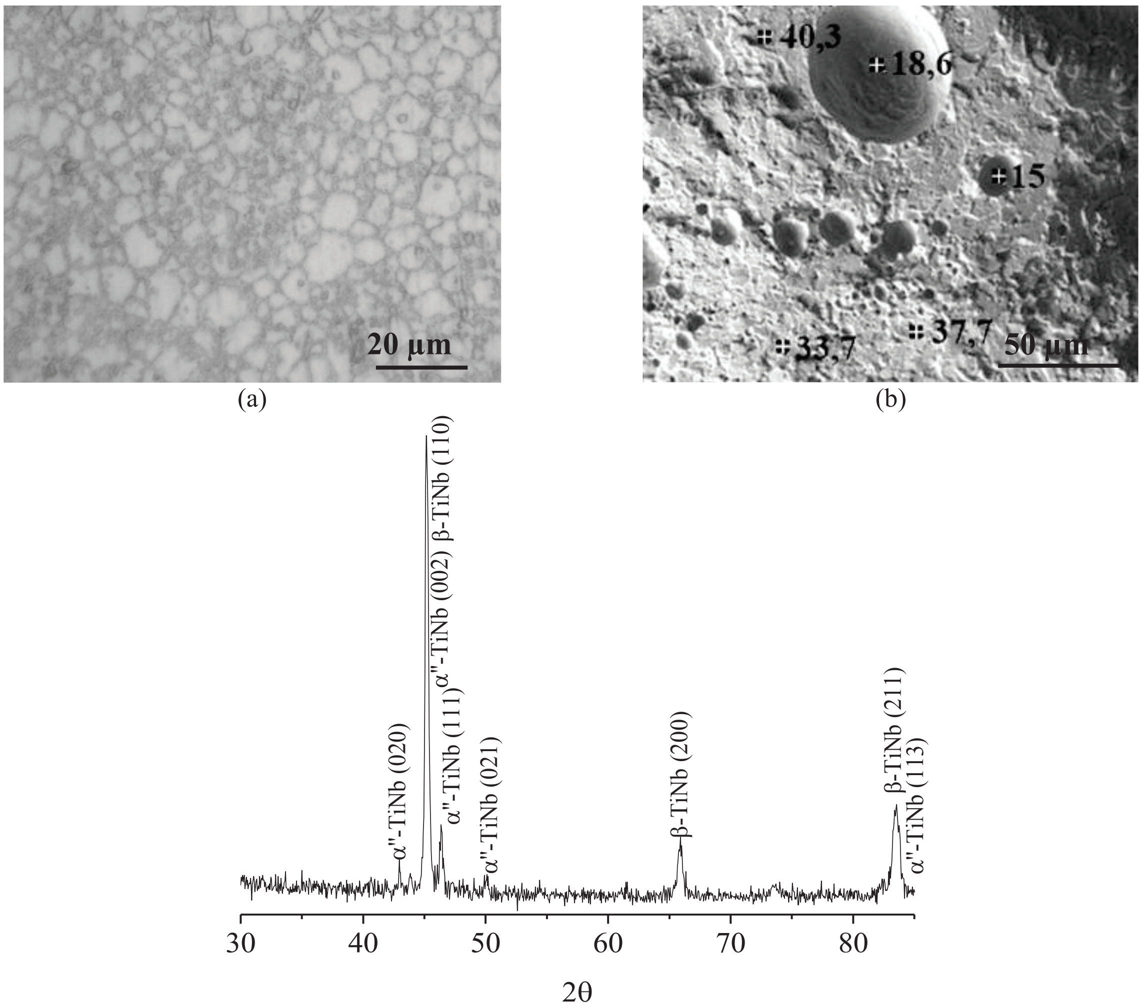


FIGURE 2. The image of microstructure (a), concentration of Nb (wt. %) in structural elements (b) and fragment of X-ray diffraction pattern (c) of the cast specimen with followed quenching

The structure of the alloy during SLM is formed in completely different temperature and rate conditions in comparison with that of the ingot. The alloy is formed from powder material overlaid on movable substrate. The laser beam scans a predetermined path, sinters portions of the powder in the form of tracks with overlap and forms single layer of hardware. The substrate is lowered by a distance equal to the value of single layer for the formation of the next layer. Then the procedure is repeated. The high cooling rates about 105 deg/s must ensure the formation of fine grain structure and nonequilibrium phases [14].

Analysis of fractures of multilayered SLM specimens has shown that the process of the formation of the structure is caused by the layers stacking on the top of each other [14]. The process of layers stacking is accompanied by the formation of macrodefects such as interlayer boundaries, large shrink holes, etc. The structure of single layer is transmitted in each layer throughout the section of multilayered specimen. Each layer has nondendritic grain structure with average grain size about 8 μm (fig. 3a).



(c)

FIGURE 3. The image of microstructure (a), concentration of Nb (wt. %) in the structural elements (b) and fragment of X-ray diffraction pattern (c) of the specimen obtained by SLM

Nondendrtitc grain grows in the alloy during the process of crystallization in the case when intensity of nucleation predominates over the growth of dendrites branches. High cooling rates facilitate this process. From the size of formed grains it can be concluded that cooling rate of the alloy during SLM is 104 deg/s [14]. Whether the segregation formed herewith? The distribution of the alloy components throughout the section of the layer and the whole specimen is equilibrium as can be seen on EDMA mapping. The quantitative ratio of min components of the alloy ranges from 36 to 38 wt. % Nb. EDMA in separate points have shown wide scatter of components concentrations (fig. 3b). The Nb concentration can be reduced to 15 wt. % on the pores surface where the grain boundary is situated. It also can be raised up to 42 wt. % in the center of the grain. Consequently, heterogeneity of components distribution is retained in Ti-Nb alloy with predetermined Nb concentration up to 38 wt. %.

This conclusion is confirmed by XRD. The reflections from the β- and α"-phase planes are identified on X-ray diffraction patterns of investigated specimens (fig. 3c). Exclusion of presence of nonequilibrium α"-phase is possible by long annealing but it may lead the growth of grain.

CONCLUSION

1. Primary dendritic structure is formed throughout the whole bulk of the ingot during crystallization in Ti-Nb alloy with 40 wt. % of Nb. There is dendritic segregation in the alloy. The secondary structure is large polyhedral β-phase grains.
2. β → α" phase transformation occurs in Ti-40 wt. % Nb alloy as the result of quenching. The dendritic segregation retaining causes structural heterogeneity of quenched alloy.
3. The structure consisting of fine grains is formed after SLM. The two-phase composition indicates that the alloy heterogeneity retains at high cooling rates.
4. It is recommended to increase Nb concentration in the alloy up to 45 wt. %. It will allow the formation of β-monophase structure in the alloy.

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