# Investigation of high-energy external influences on structural heredity of the Ti-Nb alloy

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**Abstract.** The effects of high-energy external influences on structural heredity of Ti-Nb alloy is investigated in this paper. By the methods of XRD, SEM, EDX and optical microscopy it was founded that thermal treatment and severe plastic deformation lead to the phase transformations in the alloy, the dendritic segregation occurs and retains in the alloy under external influences.

## 1. Introduction

The primary dendritic structure forms in the process of the ingots crystallization during production of Ti-Nb alloys. Parameters of this structure depend on conditions of crystallization and bulk of produced ingot [1]. The formation of dendritic structure goes with the formation of chemical heterogeneity of grains which is termed as dendritic segregation. Retaining of dendritic segregation on the next stages of material treatment (structural heredity) can provide heterogeneity of physical and mechanical properties in finished product. It is unacceptable for materials which are applied in the field of medicine [2].

It was explored earlier that the alloy containing 40 mas % niobium is most suitable of all Ti-Nb system alloys for producing of medical materials including bioimplants [3].

This alloy has the lowest elasticity modulus retaining after external influences. This is very important for biocompatibility of implant and bone tissue [2]. Progressive methods of external high-energy influence on material such as electron beam and laser treatment, severe plastic deformation, electron beam and laser treatment can remove structural heredity, to increase strength properties of the alloy and to increase elasticity modulus of finished product insignificantly [4]. The question of structural heredity in the alloy of titanium with 40 mas % niobium after thermal treatment and severe plastic deformation is studied in this paper.

## 2. Materials and methods

Ti-40 mas % Nb alloy ingots were used as research material. They were produced by the method of electro-arc melting with nonconsumable electrode into water-cooled copper crucible [3]. Ingots after remelting were quenched in two different regimes: heating in argon atmosphere to 1100 ºC during one

hour and heating to 1000 ºC during three hours. Cooling was carried out into water. The specimens were cut from the ingots after quenching. Specimens had parallelepiped form. They were exposed to the SPD different schemes different schemes of SPD. These schemes included forging in cramped conditions and free pressing and rolling [5]. The temperature was decreased during each operation of forging and pressing in the range 500-400 ºC. The multistage rolling in grooved rolls at a room temperature was the third stage of SPD. The last one was annealing at 350 ºC to remove the residual stresses.

X-ray diffraction analysis was carried out to determine the phase composition. The Bragg-Brentano scheme on Panalytical Empyrean diffractometer (Almelo, Netherlands) in monochromatic $CuK_{\alpha}$- and $CoK_{\alpha}$-radiations was used. Optical microscope Carl Zeiss Axio Observer (Jena, Germany) and scanning electron microscope Zeiss EVO 50 XVP (Jena, Germany) were used to study the microstructure. Energy dispersive analysis was also carried out on Zeiss EVO 50 XVP.

## 3. Results and discussion

The primary morphology of a cellular-dendritic structure can be seen on metallographic images of the alloy in the cast state. This morphology is observed in contrast mutually to the secondary grain structure (fig. 1 a).

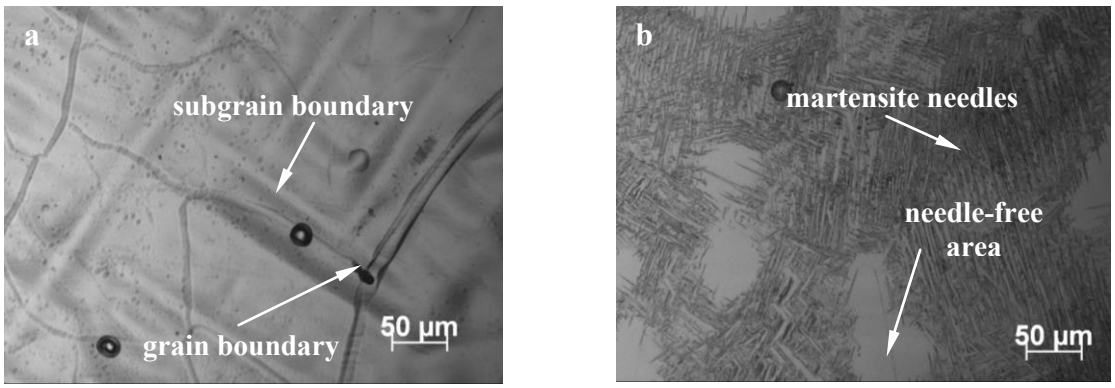


**Figure 1**. Microstructural images of the specimens in cast state (a) and after quenching (b).

The dendritic structure in the ingot means presence of dendritic segregation in the alloy. The dendritic segregation leads to the heterogeneity of the alloy component's content in different dendrites zones. The results of EDX analysis have shown that niobium concentration in dendrites is more than in interdendritic space. Niobium concentration in dendrites is about 45 mas %. Niobium concentration in interdendritic space is about 39 mas %. Thus, the difference of the niobium concentration in different dendrites zones is 6 %. Presence of dendritic segregation is the feature of the alloys of Ti with β-stabilizing elements [3]. This fact is negative. Following treatment can lead to the formation of different phases in the areas of dendrites with different niobium concentration. Different structures can be formed in the alloy. Usually this problem is solved by many hours alloy annealing [6].

Peaks of α" and β can be seen on X-ray diffraction pattern of the specimen in cast state. Both phases are solid solution of niobium in titanium with the difference at the type of lattice: α" is orthorhombic lattice and β is bcc one. The content of β-phase in the ingot is significantly more than that of α"-phase (fig. 2 a). α"-phase peaks are mild. β-phase is equilibrium and forms as a result of crystallization at equilibrium conditions. α"-phase is non-equilibrium and forms at conditions of quenching from liquid stage. Therefore, process of the ingot's formation occurs at non-equilibrium conditions. The results of investigation of the Ti-Nb alloy ingots with other chemical composition confirm this conclusion [3, 7].

β-phase grains can be seen on metallographic image of the ingot. They have clear and strongly pronounced boundaries. The martensitic structure which is characteristic of non-equilibrium cooling

conditions is not observed. Probably, its quantity is so small that the structural non-equilibrium phase is not visualized.

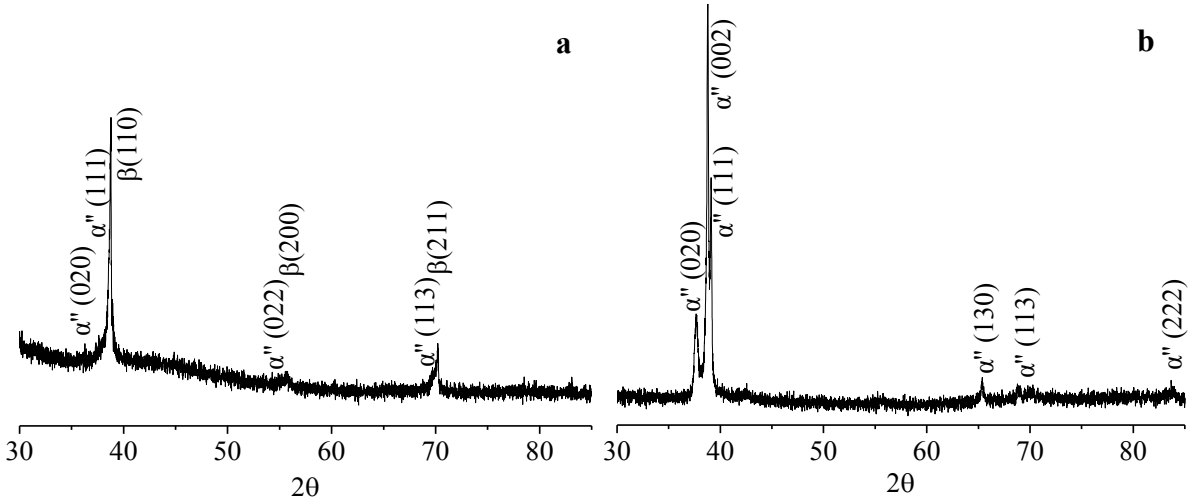


**Figure 2**. X-ray diffraction patterns of the specimens in cast state (a) and after quenching at 1000 ºC (b).

The structure which is characterized of the alloy after non-equilibrium cooling forms in the alloy after quenching. Two types of structure are observed on metallographic images of quenched specimen (fig. 1 a). These two types are areas with needle structure and needle-free areas. Grains of nonequilibrium α"-phase (martensite) have the needle microscope one can see that the primary dendritic structure with some blur contrast retains in the structure. The needle structure forms in the branches of dendrites. Needle-free areas are formed in interdendritic space. Hence, double-phase structure of the alloy after quenching is due to partially retaining of dendritic segregation after heat. The results of EDX analysis shown that niobium concentration in the martensite-free areas is about 44 mas %. The niobium concentration in martensite is equaled to 40 mas %. Difference of Nb concentration after quenching in different dendrites zones is 4%. The variability of the niobium concentration reduce is due to the diffusion processes accompanying keeping of the specimens at a high temperature in the process of quenching. However, complete disappearance of dendritic segregation on standing during three hours was failed. Nonequilibrium conditions of cooling of the alloy led to the formation of martensitic structure in the niobium depleted fields.

Peaks of α"-phase can be seen on X-ray diffraction patterns of quenched specimens. Some peaks which are characterized of α"-phase are absent. The redistribution of peaks intensities is observed. These features of X-ray diffraction pattern indicate the formation of directed textures in the specimen after quenching. These features of structural-phase state is typical as for quenching at 1100 ºC during one hour as for quenching at 1000 ºC during three hours (fig. 2 b).

However, the values of the alloy's all peaks intensities on X-ray diffraction patterns are higher at three hours that at one hour. X-ray peaks-reflections from the planes of the specimen quenched at 1000 ºC are much wider than that of the specimen quenched at 1100 ºC. The simultaneous broadening and increase of the values of X-ray peaks intensities could indicate increasing of phases quantity in the specimen. Consequently, increasing the time of quenching leads to the increasing of non-equilibrium α"-phase quantity. Peaks of other phases are not identified at X-ray diffraction pattern of the specimen quenched at 1000 ºC (fig. 2 b).

Severe plastic deformation leads to the phase transformation. The complete α" → β phase transformation occurs at SPD as shown by XRD (fig. 3 a). The β-phase peaks are identified on X-ray diffraction pattern of transverse section of the specimen subjected to the three-stage SPD. Broadening of X-ray peaks means reducing of grains size. A small shift of X-ray peaks toward small angles

indicates a change of the lattice parameter. The peaks of other phases are not identified on X-ray diffraction pattern. The redistribution of all peaks intensities means the formation of directed textures.

Band contrast created by the directed slip bands can be seen on metallographic image of longitudinal section (fig. 3 b). It seems that presence of dark and light areas caused by partial retaining of dendritic segregation after thermal treatment [3]. However, the results of EDX analysis have shown the following. Concentration of niobium in different areas of the section is not same. The distribution of the areas of enriched and depleted Nb does not coincide with the metallographic contrast. Areas of depleted Nb can have as light as dark contrast. Areas of enriched titanium also are not connected with metallographic contrast. Hence, presence of metallographic contrast is due to the structural transformations which characterize plastic deformation. And it is also due to the formation of crystallographic grain's orientation and formation of dislocation substructure, fragments and subgrains [8].

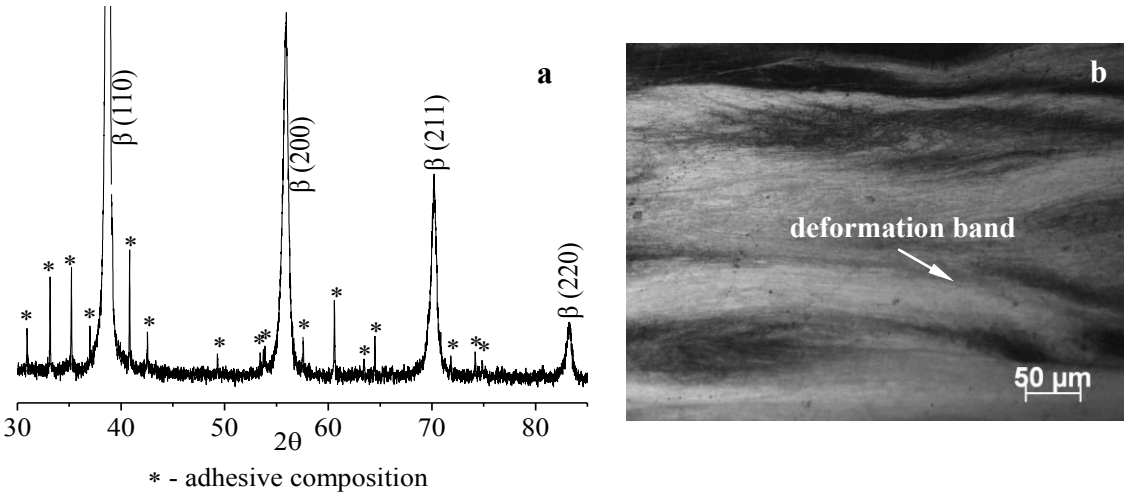


**Figure 3.** X-ray diffraction pattern of transverse section (a) and microstructural image of longitudinal section (b) of the specimens after SPD

## 4. Conclusions

1. The primary dendritic structure forms in the alloy with 40 mas % Nb during crystallization in the entire volume of the ingot. Polyhedral grains of β-phase are the secondary structure. The dendritic segregation has place in the alloy.

2. The β → α" phase transformation occurs in the Ti-40 mas % Nb alloy as a result of quenching. Retaining of dendritic segregation causes structural heterogeneity of the quenched alloy.

3. The structure consisting of oriented refined grains forms after SPD. The reverse α" → β phase transformation occurs in the process of plastic deformation. The dendritic segregation is not inherited from previous operations. Heterogeneity of elemental composition forms during plastic deformation and is due to the structural transformations.

**Acknowledgement**

This work is performed under financial support of RSF, project № 15-19-00191.

The authors wish to acknowledge the Leonhard Euler DAAD program for support and personally thank Prof. M. Epple, Prof. C. Mayer, Dr. O. Prymak (University of Duisburg-Essen, Essen), Prof. Q. Zhu and Prof. Z. Sun (General Research Institute for Nonferrous Metals, Beijing) for their assistance and help in research.

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