Dislocation structure and phase composition of Ti13Nb13Zr after high-pressure torsion

E A Rudenya¹, I V Ivanov¹, A Yu Ognev¹ and A M George Jr²

¹Department of Material Science in Mechanical Engineering
Novosibirsk State Technical University, 20, Prospekt K. Marksa, Novosibirsk, Russia
²Materials Engineering Department, Federal University of San Carlos, Rod.
Washington Luiz, s/n, São Carlos - SP, 13565-905, Brasil

E-mail: i.ivanov@corp.nstu.ru

Abstract. The approach based on synchrotron X-ray diffraction was applied in this research to investigate the dislocation and phase structures of severely deformed disk of Ti13Nb13Zr alloy. It was found, that the final structure of the material is inhomogeneous along the disk diameter. The local area of highest dislocation density of α'-phase matches with the zone of passed deformation-induced phase transformations.

1. Introduction
High-pressure torsion (HPT) is one of the severe plastic deformation (SPD) methods. It is widely used to study the behavior of metallic alloys under high pressure and strains. A characteristic feature of this technological process is different conditions of external loading of peripheral and central parts of the deformed material. For this reason, the structural transformations occurring along the diameter of the sample are different. This study describes the results of structural and phase analysis of martensitic titanium alloy Ti13Nb13Zr subjected to SPD by the HPT method. Several great properties of this alloy (e.g. the great mechanical resistance, low elasticity modulus and high corrosion resistance) make it a promising alternative to a classical biomedical titanium alloys (such as cp-Ti and Ti6Al4V) [1]. This research is based on a complex approach. It combines several deconvolution profile analysis methods to fully describe the transformations of micro- and dislocation structures of deformed by HPT material.

2. Experimental procedure
The material used in this study was the biomedical Ti13Nb13Zr titanium alloy. A disk with a thickness of 1 mm and a diameter of 10 mm was used for high-pressure torsion. Before performing deformation, it was annealed in a vacuum furnace for 1 hour at a temperature of 750⁰C and cooled in water. HPT was performed by using an equipment of Federal University of San Carlos, Brazil. The pressure during the experiment was 4.5 GPa. The upper anvil rotated 3 turns. The rotation speed was 3 rpm.

Scanning electron microscopy was performed by using a Phillips XL 30 FEG scanning microscope. Conventional transmission microscopy and orientation-phase mapping were accomplished by transmission electron microscopy (TEM) using a FEI TECNAI G2 operated at 200 KV. This facility is equipped with an orientation and phase mapping precession unit NanoMEGAS (model ASTAR) and Digistar P1000 unit.
The microhardness was evaluated using a WOLPERT Group 402 MVD Vickers hardness tester under a load of 100 g and a dwell time of 10 s.

Diffraction experiments were performed in transmission mode using synchrotron radiation on the P07 beamline (The High Energy Materials Science) at Deutsches Elektronen-Synchrotron (DESY) (Hamburg, Germany). The used wavelength was 0.014 nm. A 16-inch 2D PerkinElmer XRD 1621 scintillation detector with resolution of 2048 × 2048 pixels was used to record the diffraction patterns. The distance from the test material to the detector was 1.8 m. The images were taken along the diameter of the disk, the beam size was 1×1 mm.

Two-dimensional diffraction patterns were obtained in diffraction experiments. These patterns were azimuthally integrated using pyFAI open-source software package [2]. X-ray diffraction peaks were fitted by Pseudo-Voigt function. The instrumental contribution to broadening of the peaks was calculated using diffraction pattern of LaB$_6$ standard and fitted by Caglioti function. Subsequent analysis of dislocation structure was performed by using deconvolution profile analysis approach described elsewhere [3].

3. Results and Discussion

Before performing plastic deformation, the sample was annealed and water-cooled. The scanning electron microscopy results shows the formation of a martensitic structure in alloy after heat treatment (Figure 1). Before plastic deformation the structure is represented by large grains and needle-shaped crystals.

![Figure 1. The structure of Ti13Nb13Zr alloy before HPT. SEM.](image)

According to the classical concepts presented by Il’in [4], the boundaries of the observed large grains correspond to the boundaries of the former grains of the β-phase of titanium. Besides, the plate-type (needle-shaped) crystals located in the grains are the martensitic α'-phase of the titanium. Thus, it can be considered that the structure of the material before plastic deformation is represented only by the titanium martensitic α'-phase.

The transmission electron microscopy results (Figure 2) show that during the plastic deformation some changes in phase composition occurs. The presence of α' and β-phases was experimentally confirmed by using ACOM-TEM method (Figure 2 b). At the same time, results of other research groups [1] indicate the presence of a metastable hexagonal ω-phase (P6/mmm) in the Ti13Nb13Zr alloy after HPT deformation with 4.5 GPa loading.

The diffraction map of the deformed alloy is shown in the Figure 3. The obtained results confirm that in case of α'-titanium alloys the severe plastic deformation can lead to phase transitions. Firstly, the
β-phase of titanium appears in the central area of the disk, which is consistent with the results of transmission electron microscopy. Secondly, obtained results indicate the presence of the ω-phase of titanium. However, this phase is present in small amounts (the ratio of intensities of the diffraction maxima (01.1)-ω to (10.2)-α’ and (200)-β is 0.05 and 0.2, respectively).

![Figure 2](image1.png)

**Figure 2.** a – pseudo-light-field image; b – automatic phase distribution map constructed by the ACOM-TEM method of Ti13Nb13Zr alloy after HPT. The yellow color indicates the α'-phase, the blue color indicates the β-phase.

![Figure 3](image2.png)

**Figure 3.** Diffraction map and phase composition of the Ti13Nb13Zr alloy after HPT processing along the sample diameter. Zero on the X axis corresponds to the center of the deformed disk.

Figure 4 shows the distribution of several structural parameters obtained by analyzing results of synchrotron X-ray diffraction data: size of coherent scattering regions (<x<sub>area</sub>>; dislocation cut-off radius (R<sub>e</sub>); Wilkens constant (M); total dislocation density (ρ) as well as density dislocations with of <a>, <c> and <c+a> Burgers vectors types; relative distribution of dislocations (h) with same Burgers
vectors types. The results of the analysis of the dislocation structure allow to conclude that the dislocations distribution in the α'-phase is inhomogeneous. It should be noted that most common (from 40 to 60 %) type of dislocations is the medium-energy dislocations (<c> type of Burgers vectors). Simultaneously, the relative amount of low-energy dislocations <a> does not exceed 25 %.

The dislocation structure of the material is also heterogeneous along the deformed disk diameter. Low values of the Wilkens parameter indicate that the dislocations form ordered structures. In this case, the regions with the lowest dislocation density correspond to the maximum values of dislocations outer cut-off radius (R_e), which indicates a relatively low stress shielding of dislocations from each other.

Figure 4. Distribution of structural parameters of the α'-phase of the Ti13Nb13Zr alloy along the diameter of the deformed disk relative to the diffraction maxima (01.1)-ω, (10.2)-α’ and (200)-β.
Figure 5 shows the result of the microhardness of the deformed disk surface. It should be noted that the uneven distribution of phases and structural parameters leads to a corresponding distribution of the microhardness of the material. The regions of the disk that are characterized by the presence of the $\beta$-phase, as well as the highest density of dislocations of the $\alpha'$-phase, correspond to the highest values of microhardness.

![Microhardness distribution](image)

**Figure 5.** Microhardness distribution over the disk surface.

4. **Conclusion**

The approach used in the work based on scanning the diameter of the $\alpha'$-titanium alloy disc showed that the high-pressure torsion leads to significant structure changes. It was found that the central zone of deformed material is characterized by the presence of $\beta$- and $\omega$-phases. Furthermore, that local area possesses an increased dislocation density in the $\alpha'$-phase compared with the peripheral zones of the disk. Finally, the distribution of phases and dislocation density in a severely deformed material has a significant effect on its microhardness.

**Acknowledgments**

This study was funded according to Russian Science Foundation research project № 20-73-10215 « In-situ study of the evolution of the dislocation structure of plastically deformed high-entropy alloys under high-pressures and temperatures using synchrotron radiation».

**References**

[1] Pérez D A G, Jorge Jr A M, Kiminami C S, Bolfarini C and Botta W J 2017 *Materials Research* **20** 404

[2] Ashiotis G, Deschildre A, Nawaz Z, Wright J P, Karkoulis D, Picca F E and Kieffer J 2015 *J. Appl. Crystallogr.* **48** 510

[3] Ivanov I V, Lazurenko D V, Stark A, Pyczak F, Thömmes A and Bataev I A 2020 *Metals and Materials Int.* **26** 83

[4] Il’in A A, Kolachev B A and Pol’kin I S 2009 *Titanium alloys. The composition, structure and properties* (Moscow: VILS-MATI) p 520