Refinement of Ti18Zr15Nb alloy structure exposed to accumulative high-pressure torsion deformation

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Abstract. The strain $\gamma$ processing at high-pressure torsion (HPT) deformation for many hard materials is really much smaller than expected. This is a result of slippage of sample surface and surface of anvils. In this context, the authors develop a new method - “accumulative high-pressure torsion” (ACC HPT). This method enables producing much higher true strains in metallic and alloys than the traditional HPT method. HPT and ACC HPT were applied to $\beta$-Ti-alloys Ti-18Zr-15Nb. As TEM studies showed, the size of grains after HPT n=10 revolutions is about 40 nm while the size of grains after ACC HPT with n=10 revolutions (in total) is about 20 nm.

1. Introduction

Titanium alloys are widely used in medicine as materials for medical implants [1]. $\beta$-Ti- alloys (alloyed with Nb, Zr, Mo) are most perspective for medical implants [2-4]. The Ti18Zr15Nb (at% hereinafter) [2-5] alloy is developed by the team of the Moscow Institute of Steel and Alloys. Ti18Zr15Nb contains non-toxic and bio-compatible components. Young’s modulus of this alloy is much lower than Young’s modulus of pure titanium and $\alpha$ titanium alloys [3-5]. Moreover, the Ti18Zr15Nb alloy has superelasticity effects and shape memory (SM) effects. These effects are ensured by $\beta\leftrightarrow\alpha''$ martensitic transformation taking place in these alloys [5]. Superalasticity effects and SM effects increase the potential of Ti18Zr15Nb alloy for medical application.

The effect of severe plastic deformation (SPD) in pure titanium and titanium alloys results in the formation of nano-structure state and increased strength and performance of these materials [7-13]. Increased strength is advantageous for use of Ti alloys in medicine. High-pressure torsion (HPT) is a well-known method of crushing the metal grain to nanosizes [6]. HPT processing of Ti-Ni SM alloys leads to the nanocrystalline state and amorphization to be formed in them [11-13]. The grain size of $\beta$-Ti alloy Ti-15Mo as a result of the HPT is refined to 80 nm. The $\omega$-phase appears at some stages of HPT. In $\beta$-Ti alloys Ti-Nb-Zr system grain refinement to 50 nm was revealed during HPT processing. The $\beta$-phase after HPT Ti-Nb-Zr alloy remains the primary phase, but some part of the $\omega$ phase may appear in the HPT Ti-Nb-Zr alloy [9, 10].

The degree of shear strain $\gamma$ at High-pressure torsion is predicted by the formula (1) [6]:

$$\gamma = \frac{2\pi n R}{h},$$

(1)
where \( R \) is the radius from the center of HPT disc to the point of measurement \( \gamma \), \( n \) is the number of revolutions, \( h \) is the specimen thickness.

But it has been found in [14-19] that the real degree of shear strain at HPT of some hard or hardened metallic materials is much lower than \( \gamma \) calculated under formula (1). This is a result of slippage [17, 18]. In [19] we proposed an original “accumulative high-pressure torsion” (ACC HPT) method. The ACC HPT method enables producing a higher degree of shear strain in hard metallic materials than the traditional high-pressure torsion. In the present work, the Ti18Zr15Nb alloy was treated by traditional high-pressure torsion and accumulative high-pressure torsion, and the actions of two strain methods upon this material were compared.

2. Experimental part

The initial Ti18Zr15Nb (at\%) alloy by vacuum arc remelting in NITU MISIS was produced. The HPT die-set had anvils 20 mm in diameter and with a groove 0.4 mm deep. The pressure \( P \) at HPT was 6 GPa, the process temperature was room temperature. For traditional high-pressure torsion \( n \) - number of revolutions - was 10. In this study, the ACC HPT method was as follows: the stage I – a disc (diameter 20 mm) was subjected to HPT with the \( n = 2 \). As a result, the HPT disk sample \( \varnothing 20 \text{ mm} \), thickness \( h \approx 0.6 \text{ mm} \) was obtained. Stage II – this HPT disk was cut into 4 segments. We placed these four segments on the anvils on top of each other, and HPT \( n = 2 \) was performed again. As a result, the disk of the same size \( \varnothing 20 \text{ mm} \ h \approx 0.6 \text{ mm} \) was obtained. At the II stage, the sample receives large strain by shrinkage and torsion strain simultaneously. In this study, the cycles of HPT - cutting disk sample into segments - and the following HPT \( n = 2 \) were repeated 3 times. At stage IV the HPT disk was cut into 4 segments, and then these segments were subjected to HPT at \( n = 4 \). As a result of stage IV, a solid disk was obtained. As a result, the total number \( n \) (revolutions) at “ACC HPT” was \( n = 10 \). It can be claimed that the total \( \gamma \) achieved at “ACC HPT” due to the summation of torsional strains and compressive strains were larger than \( \gamma \) achieved at traditional HPT \( n = 10 \).

The measurements of microhardness Hv were performed with Durascan-50 at a load of 100 g and retention time of 10 s. JEOL JEM-2100 transmission electron microscope was used to study the structure.

3. Results and discussion

Since the structure obtained at traditional HPT is irregular [6], micro-hardness was determined in areas “Center” and areas “Edge” of HPT disc. Microhardness of sample after traditional high-pressure torsion \( n = 10 \) is increased approximately 1.5X as compared to the initial state (Table 1). The Hv in the center of the disc samples after traditional HPT \( n = 10 \) revolution, is a little lower than at the edge of the disc. After ACC HPT \( n \Sigma = 10 \) microhardness is significantly increased as compared to traditional HPT \( n = 10 \). At the center of the ACC HPT disc-sample, Hv appears to be even higher than at the edge of disc. This result is unusual and requires additional studies.

Table 1. Microhardness of Ti-Zr-Nb alloy at Initial state, after traditional HPT and ACC HPT.

| Strain          | Microhardness, HV (± 8) |
|-----------------|------------------------|
|                 | Center of disc | Edgeof disc |
| Initial        |                |             |
| HPT, \( n = 10 \) | 285           | 310         |
| ACC HPT, \( n = 10 \) | 370           | 335         |

Initial Ti-Zr-Nb, as the TEM SAED patterns show, has a structure of the \( \beta \)-phase (Figure 1). A grain size of initial Ti-Zr-Nb is of about 10 \( \mu \text{m} \) (Figure 1).
Figure 1. Structure of the Ti18Zr15Nb alloy in initial state/ TEM, Bright field (BF), SAED patterns.

Figure 2. TEM of the Ti18Zr15Nb alloy after traditional HPT n=10: a) bright-field images, b) dark-field images, SAED pattern.

Figure 3. Structure of the Ti18Zr15Nb alloy after accumulative HPT: a) bright-field images, b) dark-field images, SAED pattern.

The Ti18Zr15Nb alloy after traditional HPT has a nanocrystalline structure (Figure 2) with an average grain size of about 40 nm. Grains have high-angle misorientations. The SAED pattern
corresponding of β-phase, reflexes on SAED pattern form diffraction rings, reflexes from individual grains overlapped.

After ACC HPT the structure is finer than after traditional HPT (Figure 3). The grain size after ACC HPT is about 20 nm. The SAED pattern also corresponds to β-phase. The SAED pattern indicates the formation of fine nanocrystalline grains with high-angle misorientations. Thus, the accumulative high-pressure torsion leads to a greater grain refinement than the traditional HPT. Also, the efficiency of ACC HPT for the grinding of the structure of other alloys and steels was shown in [18, 19].

4. Conclusions
The main phase in the Ti18Zr15Nb alloy is a β-phase both in an initial state and after high-pressure torsion under various conditions. The β-phase in this alloy is stable under HPT. According to TEM, the traditional HPT with n=10 leads to grinding of grains down to 40 nm. As a result of traditional HPT, the microhardness has increased by about 1.5 times. Increased microhardness indicates an increase in strength of alloy. As the TEM studies showed, the size of grains after accumulative high-pressure torsion with n=10 revolutions (in total) is about 20 nm. Hence, accumulative high-pressure torsion leads to the greatest grain refinement and strain hardening of the Ti18Zr15Nb alloy. This is a result of achieving a higher degree of strain at ACC HPT than at traditional HPT.

5. References
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