Accumulative high-pressure torsion of steel 316 and $\beta$-Ti alloy

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Abstract. As revealed by studies, in the process of high-pressure torsion of hard or work-hardenable metals and alloys, including bulk metallic glasses, the actual produced strain is much smaller than the expected one. The authors proposed a new technique called “accumulative high-pressure torsion” for producing high strains in hard materials, including bulk metallic glasses.

1. Introduction

As revealed by the studies [1–4], in the process of high-pressure torsion (HPT) of hard or work-hardenable metals and alloys including bulk metallic glasses (BMGs), the actually produced strain, is much smaller than the one expected according to the formula $\gamma = 2\pi n R / h$ (1) [1]. This discrepancy can be attributed to the effect of “slippage” of anvils on the sample surface during the HPT processing of hard and work-hardenable materials.

The authors earlier have proposed a new technique, accumulative high-pressure torsion (ACC HPT), for producing high strains in hard materials including BMGs [5]. In the process of accumulative HPT, a disk-shaped sample is subjected to several cycles of deformation according to the following route (figure 1): “HPT processing up to $n = 1$ or $n = 2$ anvil revolutions $\rightarrow$ cutting of the sample into parts $\rightarrow$ compression of the stacked parts on anvils and subsequent HPT processing up to $n = 1$ (or another number of anvil revolutions)” [5]. Such cycles can be repeated several times. At the final stage, the stacked segments are subjected to HPT with a large number of revolutions ($n \geq 3$), which consolidates the sample into a monolithic disk (similarly as observed in the HPT-consolidation of amorphous ribbons and powders [1,6]). As a result of ACC HPT, the material experiences a significant total compressional or torsional strain, since in the process of upsetting the sample is known to experience a considerable strain.

In [5] it was shown, that the structure of bulk metallic glasses (BMG) Zr$_{52.5}$Cu$_{17.9}$Ni$_{14.6}$Al$_{10}$Ti$_5$ (at. %) (Vit105) underwent a more significant transformation during accumulative high-pressure torsion processing than in the case of regular HPT with a similar number of revolutions ($n = 5$), the X-ray diffraction pattern revealed a larger shear and increment in the broadening of the amorphous halo. It should be noted that the microhardness of initial BMG is very high (HV>5000 MPa), which causes
slippage of samples during regular HPT processing. In contrast, in the process of accumulative HPT at the upsetting stage the material is known to experience strain.

This paper reports the first results on the effect of ACC HPT processing on crystalline materials such as β-Ti alloy Ti-18Zr-15Nb and austenitic steel 316. Each of these alloys contains several alloying elements but have a single-phase composition in the initial as-quenched state. This makes it easier to monitor possible phase transformations during the SPD processing of these materials. It should be noted that there have been many previous studies on the effect of regular HPT processing on steel 316 [7,8], which enables making a comparison between the results on the effect of accumulative HPT processing reported in this study and the previously obtained results.

2. Materials and methods

The starting materials were austenitic steel 316 and β-Ti alloy Ti-18Zr-15Nb. Initial samples of conventional stainless steel 316 (Fe-0.03C-17Cr-0.41Si-1.72Mn-0.01P-0.03S-12.9Ni-2.36Mo, wt%) were received from Prof. T. D. Shen, Yanshan University, Qinhuangdao, China. Experimental Ti-18Zr-15Nb (at.%) alloy is developed in the National University of Science and Technology “MISIS”, Moscow, Russia by prof S.D. Prokoshkin’s group. The Ti-18Zr-15Nb titanium alloy is the most preferred material for medical implants [9,10]. This shape-memory alloy (SMA) composed of non-toxic components exhibits a high biomechanical compatibility and corrosion resistance. The reversible thermoelastic $\beta\leftrightarrow\alpha''$ martensitic transformation provides the shape memory effect in this alloy [9]. The ingot was initially produced by vacuum arc remelting, then transformed into a rod by multi-axis forging at a temperature 1050 °C.

Figure 1. Principle of accumulative HPT and a photo of a sample produced according to this procedure [4].

High-pressure torsion die-set with 20 mm diameter anvils having a 0.4 mm deep groove was used for HPT processing, which was conducted at a pressure of 6 GPa, room temperature, and various numbers of revolutions. The rotation speed of the anvils was 1 rpm. In the “accumulative HPT” procedure, at the first stage, a sample was subjected to HPT up to n = 2 rotations, as shown on figure 1. Obtained disks at the second stage were cut into 4 segments, then these segments were stacked on the HPT anvils on top of each other and HPT up to n = 2 was performed again. The cycles of “accumulative HPT” n=2 were repeated 3 times. At the fourth stage, the procedure was repeated again, but rotation number was n = 4,for both steel 316 and Ti-18Zr-15Nb alloy. As a result, solid HPT disks from all materials were obtained. The total number of revolutions during “accumulative HPT” processing was n=10. It can be asserted that the total strain produced during “accumulative HPT” processing due to the summation of compressive and torsional strains was much greater than that resulting from conventional HPT processing.
The structure and phase composition were studied using Rigaku Ultima IV X-ray diffractometer under Cu-Kα radiation. The microhardness measurements were performed using Durascan-50 at a load of 100 g for 10 s.

3. Results and discussion

To find the actual strain that can be produced by high-pressure torsion in steel 316, two halves of a disk (figure 2, a) were subjected to simultaneous HPT up to the rotation angle of 90° (n = 1/4) according to the principle first presented in [4] (figure 2, b). The samples obtained by this way are shown in figure 1, c. One can see that there was no shear of the lower surface of the half with respect to the upper surface, i.e. the HPT deformation did not occur due to the effect of slippage. Apparently, this is due to the fact that the steel sample was hardened already at the initial stages of deformation. i.e., at the beginning of HPT (up to H, = 400 after n=1/4). Thus, the experiment on the HPT with n=1/4 for steel 316 halves demonstrates that torsional deformation may be not implemented already at the initial stages of HPT processing by selected modes (figure 1, c). On the other hand, a huge amount of studies indicates a strong refinement of the structure of various materials during HPT [1,2] despite the possible slippage during processing. For example, in steel 316, after HPT n = 10, the grain is refined down to 40 nm [7,8]. Hence, the reasons leading to the structure refinement during HPT, in spite of slippage, require additional studies and analyses.

For comparison, a soft metal, copper, was subjected to HPT for n= 1/4 under a similar scheme. The observed rotation angle of the sample halves corresponds to the rotation angle of the anvils (figure 1, d), i.e. the strain introduced into the sample is consistent with the known formula.

![Figure 2](image)

**Figure 2.** Scheme and view of the samples, subjected to joint HPT: a – two disk halves, b – the principle of joint HPT [4], c – two halves of steel 316 after joint HPT for n=¼, d – two halves of Cu after joint HPT for n=¼.

Therefore, the results presented above demonstrate that the strain actually produced during regular HPT processing for many materials is smaller than the expected one, and the use of accumulative HPT is required to produce really large strains.
An analysis of the XRD data shows that the BCC β-phase is the main phase component in the initial Ti-18Zr-15Nb alloy, both after HPT n=10 and ACC HPT n=10 processing. The X-ray line width of the β-phase increases after HPT n=10. But the largest increase in the X-ray line width is observed after the ACC HPT which produces the largest strain. The increase in the X-ray line width indicates an increase in the dislocation density or/and grain refinement. Thus, β-phase in the Ti-18Zr-15Nb alloy turned out to be stable even to very large real deformations.

Table 1. X-ray line width B_{110}β and microhardness of the Ti-18Zr-15Nb alloy after the specified HPT processing.

|                      | Initial | HPT, n = 10 | ACC HPT, n = 10 |
|----------------------|---------|-------------|-----------------|
| X-ray line width B_{110}β*, deg. | 0.31    | 0.98        | 1.12            |
| Microhardness, HV (± 8), area ½ R | 265     | 345         | 330             |

As a result of HPT processing, the microhardness of Ti-18Zr-15Nb alloy grows noticeably (table 1). After HPT processing for n = 5, HV increases both in the center and at the edge of the disc samples. The increment in the microhardness after HPT processing indicates an increase in strength and yield stress. As the number of revolutions increases above n > 5, HV does not increase further after HPT n = 10 and even after accumulative HPT processing n = 10. This result is unusual and calls for additional investigations.

Analysis of the XRD data shows that the γ phase is the main phase component in the initial steel 316 (figure 3, a). The steel also contained a small amount of the α-phase, although it should be noted that in most other works original steel 316 does not contain the α-phase after correct heat treatment. HPT led to an increase in the α-phase or α'-martensite peaks (figure 3, b). Earlier, the appearance of about 20% α'-martensite in steel 316 after HPT n = 10 was noted in [7]. As a result of ACC HPT processing, phase transformations have become much stronger. The α'-martensite peak after ACC HPT processing became more intense than the γ-phase peak. This indicates that most of the γ-phase has turned into α'-martensite as a result of the very high strain achieved in the ACC HPT.

In addition, after HPT up to n = 10, a very weak line appeared at the left foot of the γ(111) line at 41-41.5°. After ACC HPT with n = 10, this line increased noticeably, and on the right foot of the α'(110) line at 46.5-47° another weak line grew. These peaks are related to the ε-martensite with a hexagonal close packed lattice. The epsilon phase is the “high pressure phase” in the iron, often appearing during severe plastic deformation of the material [11,12].

As a result of HPT, the Hc of steel 316 also grows significantly (table 2). Hc as a result of ACC HPT increased slightly more than after regular HPT, and an increase in Hc is observed both in the
center and at the periphery of the HPT sample-disk. A greater increase in H$_v$ indicates a more efficient refining of the structure of 316 steel after ACC HPT than after regular HPT.

**Table 2.** Microhardness of 316 steel in different parts of the sample in the initial state and after treatment with the HPT.

| Condition      | centre | $\frac{1}{2}$ R | edge |
|----------------|--------|-----------------|------|
| Initial        | 170    | -               | 150  |
| HPT n=10       | 430    | 450             | 465  |
| ACC HPT n=10   | 475    | 490             | 525  |

4. Conclusions
The studies demonstrate that during HPT of samples of some materials (in particular, austenitic steel) with increasing number of revolutions the strain is not imparted into the sample due to the effect of slippage. The new technique of accumulative high-pressure torsion (ACC HPT) is efficient for producing high strains in metallic materials. The studies show that HPT and accumulative HPT do not lead to a change in the phase composition of Ti-18Zr-15Nb alloy, β-phase remains the main phase even after deformation. In steel, however, HPT n = 10 led to an increase in the content of the α-phase and α' martensite, but apparently by no more than 20%. At the same time, as a result of ACC HPT, a significant part of the γ-phase in steel 316 has turned into α'-martensite and high-pressure ε-martensite. Hence, the ACC HPT can more efficiently transform the structure of alloys than regular HPT.

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