



Effect of Quasi-Hydrostatic Pressure on Deformation Mechanism in Ti-10Mo Alloy

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Abstract: The deformation mechanisms of Ti-10Mo (wt.%) alloy subjected to different quasi-hydrostatic pressure values were investigated under constrained compression using stage of high-pressure torsion apparatus. Deformation products contain {332}<113> mechanical twinning, stress-induced α'' martensitic phase and stress-induced ω phase. A volume expansion accompanied stress-induced α'' martensitic phase transformation is 2.06%. By increasing the applied pressure from 2.5 GPa to 5 GPa, the dominant deformation mechanism underwent a transition from stress-induced α'' martensitic phase transformation to {332}<113> mechanical twinning.

Keywords: titanium alloys; quasi-hydrostatic pressure; deformation structure; martensitic phase transformation; twinning

1. Introduction

Metastable β titanium alloys are potentially attractive for applications in several industrial fields, such as aerospace applications and biomedical devices, thanks to their unique combination of high specific strength-to-density ratio, low elastic modulus, excellent corrosion resistance, good biocompatibility and good formability [1–3]. One characteristic of metastable β titanium alloys is that the mechanical properties can be greatly influenced by the deformation mechanisms during loading. There are several possible deformation mechanisms in metastable β titanium alloys, including dislocation slip, mechanical twinning, stress-induced martensitic (SIM) transformation or a combination of these, as a function of β phase stability [4–11]. In general, deformation by SIM α " or mechanical twinning results in a lower yield stress, improved work hardening responses and a higher elongation to failure when compared with the deformation by slip [6–8,12].

The β phase stability, which is a function of its composition, is commonly gauged by the Mo_{eq}, an equivalent binary Ti-Mo alloy concentration. It is reported that, in order to obtain a full β phase structure in a binary Ti-Mo alloy, a minimum of 10wt.% Mo is required to prevent the martensitic transformation upon quenching to ambient temperature [13]. In recent years, the phase-stability diagram based on the mean bond order (Bo) and the mean d-orbital energy (Md) has been applied to predict the β phase stability and the plastic deformation behavior [14,15]. The decrease in the β phase stability is reflected by the decrease in the Mo_{eq} value and the decrease in the Bo value or increase in the Md value. The dominant deformation mechanisms will change from dislocation slip to mechanical twinning and then to SIM transformation with decreasing levels of β phase stability. However, there is some overlap in the activity of deformation mechanisms because of some microstructural features, such as grain size or the presence of metastable phases, which can promote or suppress a particular mechanism. In addition, some studies about the temperature sensitivity and strain rate sensitivity

of deformation mechanisms activated in metastable β titanium alloys have been published [16–18]. Zhan et al. [16] reported that the dynamic deformation mechanism in Ti-25Nb-3Zr-3Mo-2Sn (wt.%) alloy changes from a combination of mechanical twinning, stress-induced phase transformation and slip to only slip by increasing the temperature from 293 K to 873 K. Ahmed et al. [17] reported that the compressive deformation mechanism of Ti-10V-3Fe-3Al (wt.%) alloy changes from SIM α'' and slip to mechanical twinning and slip by increasing the strain rate from 10^{-3} s⁻¹ to 10^2 s⁻¹. However, relatively little work has been carried out to date to investigate the hydrostatic pressure sensitivity of deformation mechanisms activated in metastable β titanium alloys. In the present study, the deformation modes in Ti-10Mo (wt.%) alloy at different quasi-hydrostatic pressures was investigated.

2. Materials and Methods

An ingot with a weight of around 1 kg was prepared by cold crucible levitation melting (CCLM) under Ar gas atmosphere. After solidification, the ingot was homogenized at 1273 K for 3.6 ks, hot forged at 1273 K into a block of 90 mm (l) × 40 mm (w) × 40 mm (t), and then hot rolled into a plate of 290 mm (l) × 50 mm (w) × 10 mm (t) at 1173 K followed by air cooling. The plate was cut into 40 mm (l) \times 50 mm (w) \times 10 mm (t) pieces and solution treated at 1173 K for 3.6 ks followed by water quenching. All such treatments were carried out in air. Disc samples with a diameter of 10 mm and a thickness of 0.85 mm were cut by electro discharge machine and processed by high-pressure torsion (HPT) equipment (RIKEN ENTERPRISE Co. Ltd., Fukuoka, Japan). HPT refers to processing which consists of half-constrained compression stage and compression-torsion stage under a high quasi-hydrostatic pressure [19]. In the present study, only the compression stage was carried out. Two nominal pressure values of 2.5 GPa and 5 GPa were imposed on the disc samples at room temperature. Compression loading time was set as 5 min. Edalati et al. reported that the real hydrostatic pressure in HPT sample is somewhat lower than the applied pressure, which is due to the outflow of the sample between the two anvils [20]. However, disc samples of Ti-10Mo alloy in the present study exhibited very limited outflow and almost retained their original dimensions, because there is no a rotation stage. The real hydrostatic pressure can be considered as similar level to the nominal hydrostatic pressure. Planar samples were mechanically polished to mirror surface for X-ray diffraction (XRD) characterization which was performed on a RIGAKU RINT-TTR3 diffractometer (Rigaku, Tokyo, Japan) with Cu-Kα radiation (40 kV, 150 mA). Electron backscatter diffraction (EBSD) scans were performed on the cross-section using a Zeiss Sigma field emission gun scanning electron microscope (Carl Zeiss AG, Jena, Germany) equipped with a TSL Orientation Imaging Microscopy (OIM) EBSD system and operated at 20 kV. The observed region was located at around 2 mm away from the edge of the disc sample. Transmission electron microscopy (TEM) was performed on a JEOL-2100/2800 microscope (JEOL Ltd., Tokyo, Japan) operated at 200 kV. Disc samples with a diameter of 3 mm were cut for TEM observation. TEM foils were prepared by twin-jet polishing using an electrolyte containing 5% perchloric acid (Fujifilm Wako Pure Chemical Co., Tokyo, Japan), 35% methanol (Kanto Chemical Co. Inc., Tokyo, Japan) and 60% butanol (Junsei Chemical Co. Ltd., Tokyo, Japan) at -35 °C.

3. Results and Discussion

The EBSD inverse pole figure (IPF) map of Ti-10Mo alloy after solution treatment is shown in Figure 1a. It exhibits a typical equiaxed microstructure with a single β phase. The β grains exhibit different colors based on their crystallographic orientation. Here it should be noted that, in the present research, the crystallographic orientation is presented based on a cylindrical coordinate (Thickness, Radial, and Hoop direction) instead of the ordinary Cartesian coordinate (i.e., ND, TD, RD), as shown in Figure 1a. The EBSD observations were carried out on the cross-section of the disk sample, which can be described as the observed plane is parallel to the Hoop direction. The TEM dark field (DF) image shown in Figure 1b and inserted selected area electron diffraction (SAED) pattern taken from [011] $_{\beta}$ zone axis suggest the formation of a certain amount of nano-sized athermal ω particles in the β matrix.



Figure 1. (a) EBSD inverse pole figure map of Ti-10Mo alloy after solution treatment, (b) dark field image of athermal ω phase and the corresponding SAED pattern along [011]_β zone axis.

Figure 2 presents the XRD patterns. Diffraction peaks are indexed to be β phase and athermal ω phase in the solution treated specimen. Diffraction peaks from orthorhombic α'' phase can be identified in the specimens after compression, suggesting that SIM α'' phase transformation occurs during deformation. The intensities of the diffraction peaks related to ω phase become significantly stronger after compression, indicating the occurrence of stress-induced ω phase. By increasing the compressive pressure from 2.5 GPa to 5 GPa, the diffraction peaks for SIM α'' become weaker, but the diffraction peaks for ω phase become stronger. The lattice parameters of the β phase and SIM α'' were determined by the XRD experiments. The lattice parameter of the β phase is $a_{\beta} = 3.266 \pm 0.004$ Å, while that of the SIM α'' is $a_{o} = 3.040 \pm 0.001$ Å, $b_{o} = 4.979 \pm 0.002$ Å and $c_{o} = 4.697 \pm 0.006$ Å.



Figure 2. XRD spectrums for specimens compressed at different hydrostatic pressures.

A general view of the deformation microstructure after compression at 2.5 GPa is displayed by EBSD orientation maps which are shown in Figure 3a,b. Deformation bands can be indexed as twins or SIM α'' . Figure 3b shows that most of the deformation bands can be indexed as SIM α'' . The twins have been identified as {332}<113> twins with characteristic 50.5° misorientation angle [16,21], as shown in Figure 3c. The area fractions of SIM α'' and {332}<113> twins are calculated to be 14.2% and 5.4%, respectively. The TEM dark-field (DF) image of SIM α'' and its corresponding selected area electron diffraction (SAED) pattern are presented in Figure 3d,e, respectively. The thickness of the shown SIM α'' is around 0.45 µm. Plate-like structures can be seen inside the SIM α'' and they are considered to be β phase.



Figure 3. EBSD and TEM analysis of the specimen compressed at a hydrostatic pressure of 2.5 GPa: (a) EBSD inverse pole figure map of β phase, (b) EBSD inverse pole figure map of SIM α'' phase, (c) line traces across the region indicated by white arrows in (a) showing the misorientation angle, (d) dark field image of SIM α'' phase and (e) the corresponding SAED pattern.

Figure 4 shows EBSD and TEM analysis of the deformation microstructure after compression at 5 GPa. EBSD analysis shown in Figure 4a–c suggests that most of the deformation bands can be indexed as (332)<113> twins. The area fractions of SIM α'' and (332)<113> twins are calculated to be 1.2% and 13.0%, respectively. For the EBSD analysis, it should be noted that, some deformation bands, as indicated by red arrows in Figures 3a and 4a, cannot be indexed. This is considered to be due to the extensive distortions of the lattice by the deformation. The finer features of a (332)<113> twin in the specimen compressed at 5 GPa were characterized by TEM analysis. The DF image in Figure 4d shows that the deformation band is around 1 µm in thickness and contains some parallel plate-like structures. The (332)<113> twinning relationship between the matrix and the deformation band can be confirmed by the SAED pattern along the $[011]_{\beta}$ zone axis, as shown in Figure 4e. The SAED pattern shown in Figure 4e also confirms the presence of ω phase in the matrix and in the twin. The DF image of the plate-like structures (332)<113> twin is shown in Figure 4f. The SAED pattern (Figure 4f) taken from the plate-like structures (332)<113> twin boundary suggests that these plate-like structures are secondary (332)<113> twins. The DF image in Figure 4h shows that a large amount of nano-sized

ω phase uniformly distributed in the β matrix. The DF image in Figure 4i shows the formation of plate-like ω phase in the primary {332}<113> twin. It is evident from the DF images that the plate-like ω phase formed in the twin are much coarser than the ω phase formed in the β matrix, although it is quite difficult to calculate the accurate size of ω phase. The TEM and XRD results suggest that a high pressure can promote the nucleation and growth of ω phase. The mechanism of phase transformation from β to ω has under high-pressure has been well documented [22].



Figure 4. EBSD and TEM analysis of the specimen compressed at a hydrostatic pressure of 5 GPa: (a) EBSD inverse pole figure map of β phase, (b) EBSD inverse pole figure map of SIM α'' phase, (c) line traces across the region indicated by white arrows in (a) showing the misorientation angle, (d) dark field image of primary {332}<113> twin, (e) the corresponding SAED pattern illustrates twinning plane and twinning axis of primary {332}<113> twin, (f) dark field image of secondary {332}<113> twins, (g) the corresponding SAED pattern illustrates twinning (i) dark field image of ω phase in the matrix and the corresponding SAED pattern and (i) dark field image of ω phase in the primary {332}<113> twin and the corresponding SAED pattern. The subscripts "M", "Pt" and "St" denote β matrix, primary twin and secondary twin, respectively.

For Ti-10Mo alloy, the average values of Bo and Md are calculated to be 2.805 and 2.421 based on the equation proposed in [15]. It locates at the region where SIM α'' phase transformation is the

dominant deformation mechanism in the Bo-Md diagram. Min et al. [8] reported that the tensile deformation modes of Ti-10Mo alloy underwent a transition from SIM α'' phase transformation to (332)<113> mechanical twinning and further to dislocation slip by increasing the oxygen addition. In the present study, the deformation-induced products in Ti-10Mo alloy contain (332)<113> mechanical twinning, SIM α'' phase and stress-induced ω phase. The dominant deformation mechanism is SIM α'' phase transformation at the hydrostatic pressure of 2.5 GPa. This is consistent with the previous studies mentioned above. However, the SIM α'' phase transformation is suppressed at the hydrostatic pressure of 5 GPa and (332)<113> mechanical twinning becomes the dominant deformation mechanism.

Martensitic transformation is generally accompanied by some volume change, expansion or contraction. Because the shear strain associated with martensitic transformation has not only a component parallel to, but also one perpendicular to the shear plane, it is not pure shear but quasi shear. The effects of hydrostatic pressure on martensitic transformation were discussed by some previous studies based on the volume change caused by martensitic transformation [23–26]. Hydrostatic pressure may oppose the volume expansion accompanied martensitic transformation, suppressing the martensitic transformation and lowering the martensite start temperature (Ms), such as in an Invar Fe-29.9Ni (at.%) alloy and a non-Invar Fe-24.6Ni-1.8C (at.%) alloy [23]. On the contrary, hydrostatic pressure may assist the volume contraction accompanied martensitic transformation, promoting the martensitic transformation and raising the Ms, such as in a Cu-28.8Al-3.8Ni (at.%) alloy [24]. During the SIM α'' phase transformation in metastable β titanium alloys, the crystal lattice of α'' can be derived from the crystal lattice of β by {011}<011> shuffles with a certain shuffle amplitude [27–29]. According to the lattice correspondence between bcc β phase and orthorhombic α'' martensite, the lattice parameters of the α'' martensite, a_0 , b_0 and c_0 , correspond to a_β , $\sqrt{2}a_\beta$ and $\sqrt{2}a_\beta$, respectively. The shuffle processing leads to three principal transformation strains, the lattice strains due to α'' martensite transformation, which are defined as $\varepsilon_1 = (a_0 - a_\beta) / a_\beta$, $\varepsilon_2 = (b_0 - \sqrt{2}a_\beta) / \sqrt{2}a_\beta$ and ε_3 $= (c_0 - \sqrt{2a_\beta}) / \sqrt{2a_\beta}$. The substitution of the lattice parameters measured by XRD into the above equations gives $\varepsilon_1 = -6.91\%$, $\varepsilon_2 = 7.80\%$ and $\varepsilon_3 = 1.70\%$ for the current Ti-10Mo alloy. These strain values are quite high and correspond to a change in the shape and volume. The volume of SIM α'' martensite is expanded by 2.06% compared with the β phase. Therefore, it is considered that the high hydrostatic pressure of 5 GPa can oppose the volume expansion more effectively during compression, suppressing the SIM α'' phase transformation.

4. Conclusions

In the present study, the deformation modes in Ti-10Mo (wt.%) alloy at different quasi-hydrostatic pressures had been investigated. Two quasi-hydrostatic pressure values of 2.5 GPa and 5 GPa were imposed on the samples by the compression stage of HPT processing. The deformed microstructures were analyzed by XRD, EBSD and TEM. The following conclusions were drawn from the results:

- 1. The deformation-induced products in Ti-10Mo alloy contain $\{332\}<113>$ mechanical twins, SIM α'' phase and stress-induced ω phase.
- 2. The dominant deformation mechanism changes from SIM α'' phase transformation to {332}<113> mechanical twinning by increasing the quasi-hydrostatic pressure from 2.5 GPa to 5 GPa.
- 3. The volume of SIM α'' martensite is expanded by 2.06% compared with that of β phase. A higher quasi-hydrostatic pressure of 5 GPa can oppose this kind of volume expansion during compression, suppressing the SIM α'' phase transformation.

For future works, quantitative evaluation of amount of ω phase and SIM α'' phase should be helpful to assess the thermodynamic stability of these phases and to gain deeper insight on transformation mechanism under hydrostatic pressure.

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