Influence of strain amount on stabilization of $\omega$-phase in pure Ti by severe plastic deformation under high-pressure torsion

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Abstract. In pure Ti, the influence of shear deformation on the $\alpha$ to $\omega$ transformation and the development of texture in the $\omega$-phase under high-pressure torsion (HPT) straining were investigated by means of X-ray and neutron diffractions. The fraction of $\omega$-phase increased with strain in the $\omega$-phase state. Bulk submicrocrystalline $\omega$-Ti was fabricated by HPT-straining under the compressive pressure $P = 5$ GPa with the equivalent strain $\varepsilon_{eq} > 110$ at the rotation speed of $3.3 \times 10^{-3}$ rev. per sec. (0.2 rev. per min.) at room temperature. The texture of $\omega$-phase evolved by HPT-straining with the prismatic planes parallel to the shear direction of HPT-straining and the basal planes perpendicular to it.

1. Introduction

Severe plastic deformation (SPD) techniques have been applied to the design of bulk ultrafine-grained materials with improved properties, because intense strain can be introduced and high-density lattice defects created in the SPD-processed samples [1]. Among the SPD techniques, high-pressure torsion (HPT) process has a unique characteristic: deformation under high pressure. The principle of the HPT process is based on the procedure developed by Bridgman [2]. A thin disk is placed between two anvils and subjected to torsional straining under high pressure. Thus, the two parameters, i.e. the number of turns applied to the disk, $N$, (an amount of shear strain, $\gamma$) and the magnitude of the imposed pressure, $P$, are important in the HPT process.

The pressure-induced transformations of Ti, Zr, Hf were first observed by Jamieson [3]. It is well-known that pure Ti stabilizes in the hexagonal close-packed $\alpha$-phase at ambient condition, and under high pressure ($\geq 2$ GPa) the $\alpha$-phase transforms into the simple hexagonal $\omega$-phase [4]. Recently, in pure Ti [5, 6] and pure Zr [7] after HPT-straining, the high-pressure $\omega$-phases of these materials were observed at ambient condition. Todaka et al. reported that the stabilization of $\omega$-phase at ambient condition occurs via the application of shear deformation in the $\omega$-phase state [6].

In this work, to investigate the influence of shear deformation on the $\alpha$ to $\omega$ transformation in pure Ti, HPT-straining was applied. The texture evolution of $\omega$-phase during HPT-straining was also examined by means of X-ray and neutron diffractions.
2. Experimental Procedures

The experiments were conducted using pure Ti (Ti-0.045O-0.025Fe-0.01N-0.01C-0.004H in mass%). This material was annealed at 1073 K for 3.6 ks in a pure Ar atmosphere. The annealed Ti was cut into disks of 10 and 20 mm in diameter × 0.85 mm thick for HPT-straining. The HPT process was carried out using anvils with a depression of 0.25 mm in depth and 10 or 20 mm in diameter. The disk was held between the two anvils and was torsion-strained under \( P = 5 \) GPa up to \( N = 10 \) turns at a rotation speed of \( 3.3 \times 10^3 \) rev. per sec. (0.2 rev. per min.) at room temperature. The shear strain, \( \gamma \), for HPT-straining is given by the following equations: \( \gamma = 2\pi N r / t \), where \( r \) is distance from the disk center and \( t \) is the disk thickness; and also the von Mises equivalent strain, \( \varepsilon_{eq} \), given by \( \varepsilon_{eq} = \gamma / \sqrt{3} \).

The disk flowed to the radial direction during HPT-straining due to the lack of side constraint, resulting in the reduction of thickness to 0.6 mm (10 mm diameter disk) and 0.65 mm (20 mm diameter disk). The increment of temperature at the region of anvil around 1 mm (10 mm diameter anvil) away from the surface of disk center was around 5 K during deformation. The HPT process has been described in detail elsewhere [8,9].

The HPT-processed disks were characterized at ambient condition by X-ray diffraction (XRD; Rigaku RINT-2500, Cu K\( \alpha \), 40 kV, 250 mA, step size 0.02°, collection time 1 s), neutron diffraction (ND: RESA in the Japan Research Reactor No.3 (JRR-3) at the Japan Atomic Energy Agency, wavelength 0.165 nm, slit size 2 (width) × 15 (height) mm\(^2\)) and transmission electron microscopy (TEM; Hitachi H-800, 200 kV). The 10 mm diameter disks for XRD with the center position of \( r = 5 \) mm in the HPT-processed 20 mm diameter disks were prepared. The specimens for ND were prepared by stacking the HPT-processed 20 mm diameter disks to 5 mm thick (Figure 1 (a)). The ND was carried out from two different directions: radial and hoop directions (Figure 1 (b), (c)). Diffraction planes in the radial- and hoop- direction measurements were parallel and perpendicular to the shear direction in the HPT-straining, respectively. The TEM observation was performed from the direction parallel to the torsion axis.

![Figure 1](image)

3. Results and discussion

Figure 2 (a) shows the XRD patterns of the samples after HPT-straining for varying turns. In the sample compressed at \( P = 5 \) GPa for 3 ks in the HPT anvils without torsional deformation, no peaks of \( \alpha \)-phase are seen. However, the peak intensity ratio of \( \alpha \)-phase changed by compression in the comparison with that of the initial annealed (non-deformed) sample. This change might be caused by reverse-transformation from \( \omega \) to \( \alpha \)-phase after pressure-release and / or by compressive deformation. The increase in the peak intensity of \( \omega \)-phase and the simultaneous decrease in that of \( \alpha \)-phase are observed with increasing numbers of turns, indicating that the \( \alpha \) to \( \omega \) transformation occurred by shear deformation. The volume fractions of \( \omega \)-phase estimated from the XRD profiles of Figure 2 (a) by using the Rietveld program Quanto [10] were plotted in Figure 2 (b) as a function of \( \varepsilon_{eq} \) calculated using the following values: \( t = 0.65 \) mm and \( r = 5 \) mm (the center of XRD-measured disks). The fraction of \( \omega \)-phase increased rapidly with \( \varepsilon_{eq} \), and level off at around 90 vol % with \( \varepsilon_{eq} > 100 \). This result is consistent with that of ND measurement. The ND profiles at the various radial positions of the sample after HPT-straining for \( N = 5 \) are shown in Figure 3. The both peaks of \( \alpha \)- and \( \omega \)- phases were observed near the disk center. However, the peak intensity ratio of \( \alpha \)- and \( \omega \)- phases, \( I_{\alpha}/I_{\omega} \), decreased
with increase in \( \varepsilon_{eq} \) and the (101) \( \alpha \) peak disappeared with \( \varepsilon_{eq} > 110 \) (\( r \geq 4 \text{ mm} \)). Figure 4 shows the ND profiles of several diffraction planes at the region of \( r = 8 \text{ mm} \) (\( \varepsilon_{eq} = 223 \)) away from the disk center after HPT-straining for \( N = 5 \). In the radial direction measurements, the (002) \( \alpha \) peak of basal plane was not observed, meanwhile the peaks diffracted by the non-basal planes of (211) \( \omega \), (300) \( \omega \) and (220) \( \omega \) could be seen. As well, in the hoop ones, the (002) \( \omega \) peak appeared, and also the peak of the (112) \( \alpha \) and/or (301) \( \alpha \) pyramidal planes was seen. These ND experiments indicate that the prismatic planes are aligned parallel to shear direction and the basal planes are done perpendicular to it during HPT-straining.

Figure 2. (a) XRD patterns of the samples after HPT-straining for varying turns. (b) Volume fractions of \( \omega \)-phase estimated from the XRD profiles of (a) as a function of \( \varepsilon_{eq} \).

Figure 3. ND profiles at the various radial regions in the HPT-processed sample for \( N = 5 \). (These profiles are not correct by the length of beam path.)

Figure 4. ND profiles at the region of \( r = 8 \text{ mm} \) (\( \varepsilon_{eq} = 223 \)) away from the disk center in the HPT-processed sample for \( N = 5 \).
Figure 5 shows the microstructure at the region of \( r = 3.2 \) mm (\( \varepsilon_{eq} = 97 \)) away from the disk center after HPT-straining (\( N = 5, 10 \) mm diameter disk). In the dark-field (DF) image, the strongly reflecting regions correspond to the ring of \((110)_{\alpha}\) and/or \((100)_{\alpha}\) marked in the selected-area electron diffraction (SAD) pattern taken from an area 1.2 \( \mu \)m in diameter in the bright-field (BF) image. Equiaxed grains of a few hundred nanometers in diameter with high dislocation density can be seen. The SAD spots almost corresponded to those of \( \omega\)-phase, which is consistent with the results of X-ray and neutron diffractions. The arrangement of these spots in semicontinuous circles demonstrates that this submicrocrystalline structure contains large misorientations. This is consistent with the DF image where bright regions in grains are non-uniform due to the significant distortions.

Figure 5. Microstructure at the region of \( r = 3.2 \) mm (\( \varepsilon_{eq} = 97 \)) away from the disk center in the HPT-processed 10 mm diameter disk for \( N = 5 \).

4. Conclusions
Bulk \( \omega\)-Ti with grain size of a few hundred nanometers was fabricated by HPT-straining under \( P = 5 \) GPa with \( \varepsilon_{eq} > 110 \) at \( 3.3 \times 10^{-3} \) rev. per sec. (0.2 rev. per min.) at room temperature. The fraction of \( \omega\)-phase increased with strain in the \( \omega\)-phase state. The prismatic planes of \( \omega\)-phase were aligned parallel to shear direction during HPT-straining, and also the basal planes were done perpendicular to it.

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