Evolution of deformation texture of high-pressure ω-phases in pure Ti and Zr during high-pressure torsion straining

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Abstract.
High-pressure torsion process is the only way to obtain bulk-shaped samples of nearly 100% of high-pressure ω-phase of pure Ti and Zr at ambient condition. The neutron diffraction experiment of HPT-processed pure Ti and Zr revealed that the deformation of high-pressure ω-phase mainly occurs at the prismatic plane, which is completely different as α-phase. This paper also reports the correlation between deformation textures and mechanical property of ω-phase obtained by HPT process.

1. Introduction
Group IV transition metals such as Ti and Zr display three phases, depending on the temperature and pressure. At ambient temperature and pressure, these metals have a hexagonal close-packed (hcp) structure (α-phase), which transforms into a body-centered cubic (bcc) structure (β-phase) with increasing temperature. The α-phase structure also transforms into a simple hexagonal structure (ω-phase) under high pressure (3.4 GPa [1]); this pressure-induced α-to-ω transformation was first observed by Jamieson [2]. The high-pressure ω-phase essentially exhibits a reverse phase transformation upon pressure reduction or cooling. Therefore, the application of these phases as structural material could not be achieved so far. However, recent studies have revealed that, by using the high-pressure torsion (HPT) process, the high-pressure ω-phases in both pure Ti and Zr can be stabilized under ambient conditions [3–5]. The HPT process can realize a severe plastic deformation and involves the application of an intense shear strain under high pseudohydrostatic pressure on the gigapascal scale. This process is based on a procedure developed by Bridgman [6], in which a disk-shaped specimen is placed between two anvils, and a torsional shear strain is applied to the specimen by rotating the lower anvil under high pressure. The number of revolutions, N (accompanied by an amount of shear strain, γ) and the compression pressure, P, that the specimen is subjected to are the critical parameters. The fraction of the stabilized ω-phase after the HPT process is dependent on both N and P [3,7]. The HPT process is the only method that can result in nearly 100 vol% of the ω-phase in pure group IV metals. This fact enables the determination of the mechanical properties of single high-pressure ω-phase, such as elastic constants [8] and slip systems.

In this study, we investigated the deformation behavior of high-pressure ω-phase of pure Ti and Zr during the HPT process by using the neutron diffraction technique. The correlation between the texture of ω-phase and the mechanical properties was also investigated.

2. Experimental procedures
The materials used in this study are commercially pure Zr (99.2 %) and Ti (99.9 %). First, the Ti and Zr disks—with 20 and 10 mm diameters, respectively, and 0.85 mm thickness—were annealed at 1073 K for 3.6 ks in vacuum to homogenize their microstructures. The disks were individually placed between two anvils with a depression of 10 mm in diameter and 0.25 mm in depth, and a torsional strain was applied under high pressure. A compression pressure, P, of 5 GPa was used for each sample, and N was up to 20 revolutions. The rotating speed was 0.2 rpm, and the HPT process was performed at room temperature. The γ introduced by the HPT process was calculated using the expression $\gamma = \frac{2\pi N}{t}$, where r is the distance from the disk’s center and t is the specimen thickness.
The neutron diffraction (ND) experiment was performed at the Japan Research Reactor No. 3 (JRR-3) of the Japan Atomic Energy Agency. The wavelength of the neutron beam was 0.164 nm and the slit size was 2 (width) \times 15 (height) mm. The experiment was performed under ambient conditions, using RESA-1 (diffractometer for the residual stress analysis). The ND samples consisted of stacks of the HPT-processed disks with a total thickness of 5 mm and neutron beam is irradiated from the lateral side of the disks. The diffraction patterns were measured along two directions of the incident beam—hoop and radial—at each diffraction angle. The diffraction plane of the sample in the hoop (or radial) direction was perpendicular (parallel) to the direction of shear applied during the HPT process (Fig. 1).

3. Results and discussion

The XRD profiles of pure Ti and Zr after the HPT process measured at ambient condition were shown in Figs. 2(a) and (b). Note that measured diffraction planes are parallel to disk surface (i.e., perpendicular to hoop and radial). The stabilization of high-pressure \(\omega\)-phase was confirmed. The peak intensities of stabilized \(\omega\)-phase increases dependent on the introduced strain (i.e., \(N\)) and the \(\alpha\)-phase peaks almost disappeared after the HPT process of \(N = 5\), these results consistent with the previous results [3]. The \(P\) required to obtain \(\omega\)-phase has threshold value; when the applied \(P\) is 3 GPa or lower, the \(\omega\)-phase could not be observed. Zhilyaev et al. reported that the threshold value in pure Zr (99.8%) is 1 GPa, which is much lower value compared with the present study. This difference is probably caused by the purity of material. The effect of impurities are still unclear, but it can be expected that the oxygen content affects threshold value, as oxygen is \(\alpha\)-phase stabilization element in Zr and Ti and increases the energy barrier for \(\alpha\)-to-\(\omega\) transformation [9].

Using scanning transmission electron microscopy, we could confirm that both the \(\beta\)-phase and the \(\omega\)-phase existed in high-purity Zr after the HPT process; this result will be published elsewhere. During the ND experiment, however, the \(\beta\)-phase and the \(\omega\)-phase cannot be distinguished, even when a long-wavelength beam is used. This is because the \(\omega\)-phase’s superlattice structure includes a bcc structure in its interior, and all the diffraction peaks of the \(\beta\)-phase overlap with those of the \(\omega\)-phase [1,10]. Therefore, the existence of the \(\beta\)-phase was not considered in this study.

![Fig. 1 Schematic illustrations of the beam incident direction in the neutron diffraction experiment.](image)

![Fig. 2 XRD profiles of the HPT-processed (\(P = 1.5, 5\) GPa, \(N = 1/2 \sim 20\)) (a) pure Ti and (b) pure Zr samples measured at ambient condition.](image)
We previously reported the results of the ND experiment in pure Ti after the HPT process [11]. The obtained results in pure Zr have almost same tendency. By employing pure Zr instead of pure Ti for ND experiment, however, more detailed information of deformation texture of \(\omega\)-phase was obtained since Zr have larger lattice constant compared with Ti and thus diffraction peaks which can not be separated in Ti can be separated in Zr. The observed diffraction planes in both the hoop and radial directions were summarized in Fig. 3. The \(\omega\)-phase after the HPT process has quite strong texture. For instance, in the radial direction (parallel to shear direction applied in the HPT process), the \{11-20\}_\omega\) deformation texture were gradually formed with increasing \(\gamma\) (and \(r\)), and basal plane could not observed. In contrast, basal plane was mainly observed in the hoop direction (perpendicular to shear direction). These results suggest that the active slip plane of \(\omega\)-phase during the HPT process is prismatic plane. It is well known that, in the case of \(\alpha\)-phase, the main slip system is close-packed plane and direction: \{0001\}_<11\overline{2}0>\). After \(\alpha\) to \(\omega\)-phase transformation, \{11-20\}_\omega\) prismatic plane becomes close-packed plane and close packed direction is \(<0001>_\omega\). Therefore, the main slip system changed to \{11-20\}_<0001>_\omega\) and aligned parallel to shear direction applied in the HPT process.

In the hoop direction, pyramidal planes were also observed in addition to basal planes, indicate that the c-axis of some part of \(\omega\)-phase tilted to the shear direction. The observed lattice plane of \(\omega\)-phase in the ND experiment and the interplanar angles between observed lattice planes and reference planes (basal plane, prismatic planes) were listed in Table 1. As seen in table, diffraction peaks were observed when the interplanar angle is below 36°; if an interplanar angle between basal (prismatic) plane is below 36°, a diffraction peak is observed in hoop (radial) direction. In the case of \{11-21\}_\omega\), every interplanar angles are over 36°, thus this plane could not observed in both the hoop and radial directions. The pyramidal plane \{30-31\}_\omega\) could not observed in spite of low interplanar angle. This is probably owing to the quite small structure factor of this plane.

In order to investigate the effect of texture on the mechanical properties of \(\omega\)-phase, the samples for the tensile tests were cut out from the HPT processed pure Ti as shown in Fig.4. Two types of tensile sample (parallel area: 1.0 mm width, 0.5 mm thickness, and 3.0 mm length) were prepared as shown in Fig.5(a). The tensile direction of one sample is parallel to shear direction applied in the HPT process (sample-A) and thaxt of the other sample is perpendicular to shear direction (sample-B). The obtained tensile stress – strain curves are shown in Fig.5(b). The sample-A has the high strength of about 1250 MPa, but fractured with brittle manner after yielding. In contrast, the sample-B showed the tensile elongation of about 1 %. Such difference in the deformation behavior is probably caused by the

![Fig.3 Observed diffraction planes in the neutron diffraction experiment of the HPT-processed pure Ti and Zr.](image)

**Table 1** List of the observed diffraction planes in the neutron diffraction experiment, and interplanar angle between the observed and basal or prismatic planes.

| Lattice plane | Observed direction | Interplanar angle (°) |
|---------------|--------------------|----------------------|
|               |                    | (0001)    | (10-10) | (11-20) |
| (10-11)       | Hoop               | 35.7      | 54.3    | 59.6    |
| (11-21)       | ×                  | 51.2      | 47.5    | 38.8    |
| (11-22)       | Hoop               | 31.9      | 62.8    | 56.1    |
| (20-21)       | Radial             | 55.2      | 34.8    | 44.7    |
| (31-41)       | Radial             | 68.9      | 25.1    | 26.3    |
| (30-31)       | ×                  | 65.1      | 24.9    | 38.2    |
| (21-31)       | Radial             | 62.3      | 33.2    | 29.6    |
deformation texture formed by the HPT process. In the case of sample-A, as observed in the ND experiment, main slip plane of \( \omega \)-phase \( \{11-20\}_\omega \) is parallel to tensile direction (Schmidt factor = 0) or close to parallel. Therefore, slip deformation could not be activated during the tensile test and fractured with brittle manner. In the case of sample-B, the slip plane of most part of material is perpendicular to tensile direction. However, as seen in Table 1, observed plane in radial direction \( \{20-21\}_\omega \) have angle of nearly 45° between slip plane \( \{11-20\}_\omega \). It is suggested that the slip deformation of grains with such orientation is easy to activated during the tensile test. Consequently, slight plastic deformation was observed in the sample B.

4. Summary

By investigating the deformation texture of single high-pressure \( \omega \)-phase by using the neutron diffraction technique, \( \{11-20\}_\omega \sim 0001 > \omega \) seems main slip system of \( \omega \)-phase. The high-pressure \( \omega \)-phase exhibits slight plastic deformation when the tensile direction is perpendicular to shear direction applied in HPT process. This is probably the effect of texture; the slip deformation of some grains easy to activated upon loading.

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Fig. 4 (a) Geometry of the tensile test samples of the HPT-processed pure Ti used in this study. The tensile direction is parallel and perpendicular to the shear direction by the HPT process in the sample-A and sample-B, respectively. (b) The obtained stress-strain curves of the sample-A and sample-B.