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Orientation relationship between α-phase and high-pressure ω-phase of pure group IV transition metals

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The deformation texture of the high-pressure ω-phase of pure group IV metals was investigated using neutron diffraction analysis. The $\langle 0001\rangle_\omega$ direction gradually aligned with the shear direction with an increase in the shear strain introduced by the high-pressure torsion process. The deformation texture suggested that the main slip system of the ω-phase was $\{11\text{-}20\}\langle 0001\rangle_\omega$. We experimentally observed the orientation relationships of the mechanically stabilized ω-phase with respect to the reverse-transformed α-phase, and determined that the relationship was similar to that proposed by Silcock and Rabinkin.

Keywords: Texture; Orientation relationship; Phase transformations; Neutron diffraction; Severe plastic deformation

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; this pressure-induced α-to-ω transformation was first observed by Jamieson [1]. The α- to ω-phase transition pressure for Zr at ambient temperature has been reported to be ~2–6 GPa [2,3]. A number of orientation relationships between the α- and ω-phases have been proposed previously. Silcock was the first to propose the orientation relationships between ω-phase precipitates and the α-phase matrix on the basis of X-ray observations of Ti–V, Ti–Mo and Ti–Cr alloys [4]: $(0001)_\alpha \parallel (11\text{-}20)_\omega$ and $[11\text{-}20]_\alpha \parallel [0001]_\omega$. This mechanism assumes a direct α- to ω-phase transformation. Rabinkin et al. reported a mechanism to account for the α- to ω-phase transformation; this mechanism is similar to the one suggested by Silcock in that the basal plane of the α-phase is parallel to the prismatic plane of the ω-phase [5]. Such orientation relationships have been confirmed in shock-loaded Ti [6]. Usikov et al. have also reported orientation relationships, which were deduced by considering the β-phase as an intermediate state [7]: $(0001)_\alpha \parallel (01\text{-}11)_\omega$ and $[10\text{-}10]_\alpha \parallel [\text{-}2\text{-}131]_\omega$. Furthermore, other orientation relationships, namely $(0001)_\alpha \parallel (\text{-}1101)_\omega$ and $[\text{-}1101]_\alpha \parallel [\text{-}211\text{-}3]_\omega$, have been observed in shock-loaded pure Zr [8]. Recently, Trinkle et al. postulated that for TAO-1, $(0001)_\alpha \parallel (0\text{-}111)_\omega$ and $[11\text{-}20]_\alpha \parallel [01\text{-}11]_\omega$ are energetically favorable mechanisms for the α- to ω-phase transformation; these were deduced from ab initio calculations [9] and have not been confirmed experimentally. They also showed that the mechanism corresponding to the relationship proposed by Silcock has a relatively low energy barrier. All of these experimentally observed orientation relationships have been determined by analyzing the orientation of the retained ω-phase and that of the α-phase matrix, meaning that only α- to ω-phase orientation relationships have been investigated so far. Brown et al. have investigated the ω-to-α reverse transformation behavior in shock-loaded pure Zr using an in situ X-ray diffraction technique [10], but could not identify the orientation relationships due to the relatively random crystalline orientation after shock-loading.

Since the α- to ω-phase transformation exhibits hysteresis, the high-pressure ω-phase is partially retained under ambient conditions after the application of a hydrostatic pressure [1,11,12]. The retention of the ω-phase was also observed after deformation through shock loading [13,14], and high-pressure torsion (HPT) [15–17]. Of these, HPT has the advantage in that nearly 100 vol.% of the ω-phase can be retained along with the bulk shape [15,18]. In

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addition, the ω-phase obtained after the HPT process has a strong texture [18]. This fact enables the determination of the mechanical properties of single high-pressure ω-phases, such as elastic constants [19] and slip systems.

Until now, the orientation relationships between the α-phase and the HPT-induced ω-phase were unknown. Furthermore, in general, the α- to ω-phase transformation is a diffusionless one. However, our recent results (to be published elsewhere) suggest that the reverse transformation of the ω-phase formed by the HPT process becomes a diffusion-controlled process. This finding implies that the mechanisms underlying the α- to ω- and ω- to α-phase transformations are different. In this study, by using neutron diffraction (ND) to compare the strong texture of the deformed ω-phase stabilized after the HPT process and that of the reverse-transformed α-phase, the orientation relationships corresponding to the ω- to α-phase reverse transformation were determined.

We have previously investigated the deformation behavior of the high-pressure ω-phase of pure Ti by measuring the texture of the deformed ω-phase using ND analysis [18]. The strong texture of the prismatic plane along the shear direction of the HPT suggested that the main slip plane of the high-pressure ω-phase was the prismatic plane. However, owing to the wavelength of the neutron beam and the lattice constant, the diffraction peaks of the prismatic $\{11\text{-}20\}_\omega$ plane and the pyramidal $\{10\text{-}11\}_\omega$ plane could not be separated. To overcome this problem, we employed pure Zr, which has a larger lattice constant than that of Ti. As a result, the diffraction peaks of the two planes could be separated.

The materials used in this study were commercially pure Zr (99.2%) and Ti (99.9%). First, the Zr and Ti disks—with diameters of 10 and 20 mm, respectively, and a thickness of 0.85 mm—were annealed at 1073 K for 3.6 ks in vacuum, while using sponge Ti as an oxygen getter to homogenize their microstructures. The disks were placed individually between two anvils that form a depression 10 mm in diameter and 0.25 mm in depth, and a torsional strain was applied under high pressure. A compressive pressure, P, of 5 GPa was used for each sample, and the number of revolutions, N, was 10. The rotation speed was 0.2 rpm, and the HPT process was performed at room temperature. The shear strain, γ, introduced by the HPT process was calculated using the expression $\gamma = 2\pi rN/t$, where r is the distance from the disk's center and t is the specimen thickness. The temperature increment during the HPT process for the 10 mm diameter Ti specimen was ~30 °C, suggesting that the effect of the heat generated was negligibly small.

The ND experiments were 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) × 15 (height) mm. The experiments were performed under ambient conditions, using RESA-1 (diffractometer for residual stress analysis). The ND samples consisted of stacks of the HPT-processed disks with a total thickness of 5 mm; the neutron beam was irradiated from the lateral side of the disks. The diffraction patterns were measured along two directions of the incident beam—radial and hoop—at each diffraction angle. The diffraction plane of the sample in the radial (or hoop) direction was parallel (perpendicular) to the direction of the shear applied during the HPT process.

Further, through X-ray diffraction (XRD) analysis, we confirmed that the stabilization of the ω-phase occurred in pure Zr after the HPT process and that the fraction of the stabilized ω-phase increased with N (and γ); this result agrees with those previously reported by another research group [20]. After the HPT process, the volume fraction of the ω-phase was nearly 100%.

Using scanning transmission electron microscopy, we could also confirm that both the high-pressure β-phase and the ω-phase existed in high-purity Zr after the HPT process; this result will be published elsewhere. During the ND experiments, however, the β-phase and the ω-phase could not be distinguished, even when a long-wavelength beam was used. This was because the ω-phase's superlattice structure includes a bcc structure in its interior, and all the diffraction peaks of the β-phase overlap with those of the ω-phase [2,21]. Therefore, the existence of the β-phase was not investigated in this study.

The r (and γ) dependencies of the ND profiles ($2\theta = 36$–$40°$) of the HPT-processed samples are shown in Figure 1. The variation in the total intensity with an increase in r was due to the difference in the beam path length. When the beam was along the radial direction, its path length decreased with an increase in r, resulting in an increase in the total intensity. On the other hand, the path length was nearly independent of r in the hoop direction. The theoretical diffraction angles (solid line: α-phase; dotted line: ω-phase) were calculated from the lattice constants (α-phase (JCPDS Card No. 05-0665): $a = 3.232$ Å, $c = 5.147$ Å; ω-phase (JCPDS Card No. 26-1399): $a = 5.039$ Å, $c = 3.136$ Å) and the neutron beam wavelength (0.164 nm). Regardless of whether the beam was in the radial or hoop direction, the diffraction peak attributable to $\{10\text{-}11\}_\alpha$ was observed. Further, its intensity decreased with an increase in r (and the shear strain, γ); this agreed with the XRD results. Since the XRD spectra contained crystallographic information collected from the entire disk surface, the α-phase XRD peaks probably corresponded to the disk center. Two diffraction peaks of the ω-phase, which corresponded to $\{10\text{-}11\}_\omega$ and $\{11\text{-}20\}_\omega$, were observed in this 2θ region. Irrespective of the direction of the beam, the pyramidal $\{10\text{-}11\}_\omega$ plane was mainly observed near the sample's center ($r = 0$–1 mm). In the radial direction (Fig. 1b), the $\{10\text{-}11\}_\omega$ peak gradually became smaller with increasing strain ($r > 2$ mm), and the deformation texture of the prismatic $\{11\text{-}20\}\omega$ plane evolved. However, the $\{11\text{-}20\}_\omega$ peak was absent in the hoop direction (Fig. 1a). These results indicate the alignment of the ω-phase prismatic plane with the shear

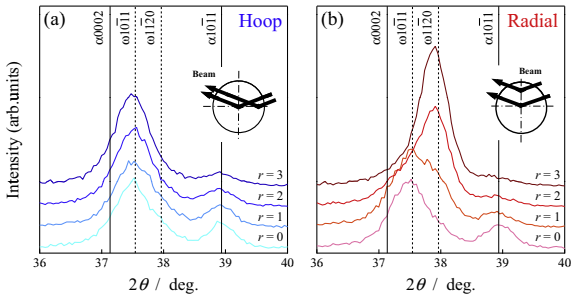

Figure 1. Radial dependence of the ND patterns of the HPT-processed sample of pure Zr when the beam was incident in (a) the hoop direction and (b) the radial direction. The solid and dotted lines indicate the calculated peak positions of the α-phase and the ω-phase, respectively.