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Omega Phase Formation in Shock-Loaded Zirconium

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Abstract

A shock recovery experiment and post-shock mechanical measurements were conducted on high-purity Zr. Mechanical properties of the Zr samples before and after the shock loading are compared and discussed in terms of the substructure evolution during the shock loading. Metastable ω -phase was found in the Zr sample following shock-loading to 7 GPa. A new orientation relationship between the α and ω phases was derived which does not agree with those previously reported in hydrostatic pressure experiments. A mechanism is proposed for the observed $\alpha \rightarrow \omega$ transformation.

Introduction

The pressure-induced phase transformation in Ti and Zr was first reported by Bridgman [1] as a result of his hydrostatic pressure studies of these materials. The nature of this transition was not clear until Jamieson [2] identified the new phase to be the ω -phase whose structure had already been established in Ti and Zr alloys. Since then, an intensive effort has been made to investigate the kinetics, crystallography, and electronic structure variation of this $\alpha \rightarrow \omega$ transition under high hydrostatic and dynamic pressures.

Although the existence of the pressure-induced $\alpha \rightarrow \omega$ phase transformation in Ti, Zr, and Hf is well established, limited experimental data exists concerning the structure/property relationships of zirconium subjected to shock loading. Crystallographically, one of the fundamental problems concerning the transformation, i.e., the orientation relationship (OR) between the α - and ω -phases in Ti and Zr remains a controversial issue. The α - ω OR was first studied by Sargent and Conrad in pressure-treated pure Ti and it was found to be $(0001)_\alpha // (11\bar{2}0)_\omega$ and $[11\bar{2}0]_\alpha // [2\bar{2}01]_\omega$ [3]. A later study by Usikov and Zilbershtein [4] found that the OR of $(0001)_\alpha // (1101)_\omega$ and $[2110]_\alpha // [3033]_\omega$ was present in pressure treated pure Ti as well as Zr which, however, conflicts with the previous result. Moreover, Rabinkin et al. [5] studied the $\alpha \rightarrow \omega$ transition in pure Zr subjected to high pressure soaking up to 10 GPa and obtained an OR similar to that reported by Sargent and Conrad in Ti but different from that reported earlier for Zr [4].

Reviewing the studies concerning the transformation, it appears that little effort has been made to describe the microstructural aspects of the process. It is commonly accepted that the $\alpha \rightarrow \omega$ transformation is of the martensitic (diffusionless) type [5,6,7]. Since this type of

transformation involves a lattice reconstruction that occurs principally through homogeneous shearing plus a discrete amount of dilatation (or contraction), the crystallographic and microstructural aspects of the process are crucial in understanding its mechanism.

A new OR between the α and ω phases was observed in shock-loaded pure Zr in the present investigation which differs from those previously reported in Zr and Ti. A detailed crystallographic and microstructural study using TEM was conducted in addition to the pre- and post-shock mechanical measurements at various strain rates and temperatures. Owing to the length restriction of the proceedings, only the main results and a brief discussion are presented here.

Experimental

High-purity crystal-bar zirconium containing < 50 ppm oxygen was shock-loaded to 7 GPa and soft-recovered using techniques described elsewhere [8]. Cylindrical specimens of 5 mm (height) $\times$ 5 mm (diameter) were machined from both the annealed starting material and the shock-loaded sample for post-shock measurements at different strain rates and temperatures. Samples for TEM examination were subsequently sliced, ground, and polished to a thickness of 100 ~ 150 nm, then twin-jet polished in a perchloric (10%) and methanol (90%) solution until perforation. Thin film samples were examined using a Philips CM30 transmission electron microscope operating at 300 kV.

Results and Discussion

Mechanical behavior

Figure 1 shows the stress-strain response of the compressive tests on some of the pre- and post-shock Zr samples. The stress-strain curves are seen to contain two distinct regions in the plastic regime, i.e., a protracted

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elastic-plastic transition (Regime I), particularly in the annealed materials followed by a nearly linear hardening regime (Regime II). Close examination of the results, not all shown in Fig. 1, reveals that i) the yield stress increases with strain rate and decreases with temperature in both the pre- and post-shock samples and the yield stress of the latter is about 60% higher than that of the former; ii) the decrease of the initial work hardening rate in Regime I is faster in tests of higher strain rate in the pre-shock samples and it is insignificant in the post-shock samples; iii) in contrast with the post-shock sample deformed at 76K, a very rapid elastic-plastic transition to a nearly constant hardening rate is seen in the curve of the pre-shock sample tested under the same conditions.

Figure 1 Stress-strain response of the annealed and shock-loaded Zr samples, (SL - shock-loaded, AN - annealed, LN - liquid nitrogen, RM - room temperature, and 0.001 - strain rate).

Microstructural examination

The typical microstructure of the pre-shocked samples deformed to 20% strain at room temperature is shown in Fig. 2a. Besides the well-developed subgrain structure about half the grains are heavily twinned. All the twins observed in TEM examinations are of the (1102) type seen most frequently at room temperature in Zr [9,10]. A much larger scale of twinning was seen in the samples deformed at 76K and in the shock-loaded samples, Fig. 2b. In addition to the dominant twin, a fair amount of (1122) twinning was also detected. The most active slip system in matrix is $\langle 1120 \rangle$ - $\{1100\}$ system, consistent with previous studies on slip in Zr [11], however, $\langle 0001 \rangle$ - $\{1100\}$ slip system was occasionally seen in some grains.

Figure 2 Typical microstructure of the samples deformed at room temperature (a) and an optical micrograph of the sample deformed at 76K (b).

The latter type dislocation, however, was frequently found within twins, the origin of which is discussed elsewhere [12]. In addition to the high volume fraction of twins in the shock-loaded samples, well-developed microbands were frequently seen in the matrix.

Morphology of the ω -phase

Figures 3a and b show TEM bright and dark-field micrographs of the retained ω -phase in the shock-loaded samples. A large portion of the micrographs consists of the retained ω -phase, with lattice constants $c = 3.136$ and $a = 5.039$ [5,13]. The reverted α -phase is seen at the edge and in the middle as lamellae, as marked in the micrographs. The dark field image was obtained using the $(10\bar{1}3)$ reflection of the α -phase and, hence, the reverted α -lamellae in the ω -phase are lit up. A dense dislocation network is seen in the ω -phase and its

orientation is consistent throughout the ω -phase region (see Fig. 3b). The projection of the dislocations is oriented along the c -axis of the ω -phase and hence they lie on either the prism plane of type I $\{10\bar{1}0\}$ or type II $\{11\bar{2}0\}$.

Figure 3. Retained ω -phase and reverted α -phase in a bright field (a) and a dark field image (b).

Orientation relationship

There are two sets of spots in the selected area diffraction (SAD) pattern from both the α and ω -phase, shown in Fig. 4. Indexing this pattern is straightforward since both lattices are almost on zone, the misorientation of the two zone axes being measured to be $\sim 1.5^\circ$ using CBD techniques. The large rectangular lattice is the $\{10\bar{1}0\}$ plane of the α -phase and the small rhombic lattice is the $\{11\bar{2}3\}$ plane of the ω -phase in reciprocal space. The OR derived from this pattern is $(0001)_\alpha \parallel (1\bar{1}01)_\omega$ and $[11\bar{2}0]_\alpha \parallel [11\bar{2}3]_\omega$. The lattice transformation matrix is

$$\omega_{II} = \begin{pmatrix} 0.8484 \frac{a}{a} & 0.8484 \frac{a}{a} & 0.0561 \frac{c}{c} \\ 0.8484 \frac{a}{a} & 0.8484 \frac{a}{a} & 0.0561 \frac{c}{c} \\ 0.8408 \frac{a}{a} & 0 & 0.8208 \frac{c}{c} \end{pmatrix}$$

where a , c , a , and c are the lattice constants of the α - and ω -phase, respectively, (see Fig. 5a). To confirm the relationship, stereographic projections of the two lattices along the common c -axis are overlapped and rotated to match the poles of the $(0001)_\alpha$ and $(1\bar{1}01)_\omega$ planes. This leads to the coincidences of $[1\bar{5}40]_\alpha$ and $[2\bar{1}13]_\omega$ as well as $[5413]_\alpha$ and $[1120]_\omega$ zone axes. These two instances were also confirmed by TEM examinations. It is noted that this OR is different from those reported previously in hydrostatic pressure-treated Zr [4–5]. Since the OR is obtained in this previous study, were derived from diffraction patterns that were not on zone. It is certain that these relationships and the microscopic details may be safely deduced from the patterns.

Figure 4. SAD pattern showing both $[10\bar{1}0]_\alpha$ zone and $[11\bar{2}3]_\omega$ zone axes.

Transformation mechanism

Though the $[1100]_\alpha$ and $[11\bar{2}3]_\omega$ axes are slightly, approximately 1.5° , misaligned the $(0001)_\alpha$ and $(1\bar{1}01)_\omega$ planes are not only precisely parallel but equally spaced. Consequently, it is reasonable to assume that atomic movement during the transformation is confined to these planes. Fig. 5b shows the overlap of the atomic arrangements of the $(0001)_\alpha$ and $(1\bar{1}01)_\omega$ planes. Close

examination of the atomic positions before and after the transformation shows that the lattice reconstruction can be accomplished principally by simple shearing in and between the planes, in agreement with the assumption above. The shear component along the $[1101]_{\omega}$ direction is about 14% and along the $[1123]_{\omega}$ is only 3%. The latter corresponds to an approximately 2° misorientation between the $[1100]_{\alpha}$ and $[1123]_{\omega}$ axes, which is consistent with the experimentally measured value. It can be seen from Fig. 5b that the relative atomic movement required for the lattice reconstruction is quite small and, thus, the nearest neighbors remain the same and short-range order is retained. The characteristics of this transformation are the same as those of a martensitic transformation, so it is considered to be a martensitic type.

Figure 5 Atomic arrangements of the two lattices in the parallel planes.

It is well known that the introduction of internal structures such as dislocations and twins is an integral part of the martensitic transformation, compensating for the distortion caused by the lattice reconstruction and resulting in a macroscopically undistorted sample. Since shear is principally involved in the $\alpha \rightarrow \omega$ transformation, dislocations may be sufficient to compensate for the distortion resulting from the reconstruction. In fact, a very high density of dislocation networks was observed in the retained ω -phase, see Fig. 3b, while twinning was not seen. The dislocations are of c and a type lying on the prism planes. Reverted α -phase at the edge of and in the interior of the retained ω -phase can be seen as well. Since there are eight possible variants of the reverted α -phase in the interior, lamellae of differently oriented α -phase lamellae are readily seen in Fig. 3.

Summary

The introduction of dislocation cell, twins, microbands, and ω -phase during shock-loading significantly alters the mechanical response of Zr shock-loaded to 7 GPa. The yield strength increases by about 60% and the work hardening rate at room temperature is considerably enhanced.

The $\alpha \rightarrow \omega$ transformation resulting from shock loading was detected and the retained ω -phase was observed. The transformation is of martensitic type and the lattice reconstruction is accomplished, in principle, through shearing that is contained on specific planes. The OR observed is determined to be $(0001)_{\alpha} // (1101)_{\omega}$ and $[1100]_{\alpha} // [1123]_{\omega}$ which differs from those previously reported. A high density of dislocations was observed in the retained ω -phase, which was necessary for compensating the distortion due to the lattice reconstruction. Variants of the reverted α -lamellae were found in the interior of the retained ω -phase.

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