Deformation Response of Zr After Shock-Loading

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DEFORMATION RESPONSE OF Zr AFTER SHOCK-LOADING

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The post-shock stress-strain response and microstructural evolution of Zr shock-loaded to 7 GPa were investigated. A Bauschinger effect in the room temperature reload stress-strain behavior due to shock-loading has been observed following yielding. Deformation twinning is shown to play a more important role than slip during post-shock plastic deformation and work hardening. The work hardening rate of the shock-prestrained specimens is less temperature sensitive than that of annealed Zr. The underlying microstructures responsible for the Bauschinger effect and the differences in work hardening behavior are characterized. A new type of dense dislocation arrangement resulting from phase transformations during shock excursion is discussed.

INTRODUCTION

Previous studies on the high pressure behavior of Zr have been concentrated on the pressure-induced \( \alpha \rightarrow \omega \) transformation \[1,2,3\]. The defect structures formed during shock-loading and their effect on the stress-strain response upon reloading, however, remain largely unknown. In the present investigation we studied the post-shock stress-strain behavior and microstructural evolution in Zr during quasi-static reloading.

It has been shown that deformation twinning during quasi-static loading dictates the plastic deformation in Zr at low temperatures \[4\]. Twinning is expected to play an important part in the mechanical behavior of shock-prestrained Zr since increasing strain rate is known to enhance twinning in metallic materials in a way similar to decreasing temperature \[5\]. Knowledge about the shock-induced microstructure and its effect on twinning and slip is crucial to understanding the post-shock behavior of Zr, which is the primary objective of the present investigation.

EXPERIMENTAL

High-purity crystal-bar zirconium containing < 50 ppm oxygen was shock-loaded to 7 GPa and soft-recovered using techniques described elsewhere \[6\]. The recovered sample was sectioned for detailed transmission electron microscopy (TEM) and post-shock mechanical property measurements. Cylindrical specimens 5 mm (height) \( \times \) 5 mm (diameter) were machined from both the annealed starting material and shock-loaded Zr for mechanical measurements at 298 and 76 K. Samples for TEM examination were subsequently sliced, ground, and polished to a thickness of 100 - 150 \( \mu \)m, then twin-jet polished in a perchloric (10%) and methanol (90%) solution until perforation. Thin film samples were examined using a Philips CM30 TEM operating at 300 kV.

RESULTS AND DISCUSSION

Stress-strain response

Figure 1 shows the stress-strain response of Zr following shock loading (SL, thereafter), offset for the transient strain due to shock-loading. For comparison, results of annealed Zr (AN, thereafter) are also plotted. The sharp knee of the elastic-plastic transition for the AN specimen at 76 K, marked by an arrow in the figure, indicates an yielding by uniform twinning \[4\]. The corresponding transition for SL sample at 76 K is more gradual. At room temperature the SL sample started yielding at a stress lower than the AN sample hardened to an equivalent strain level. Although the SL sample yielded at 76 K at a stress slightly higher than the corresponding stress for the AN sample, the latter stress level corresponds to
deformation via uniform twinning. The nominal stress associated with slip for the AN sample at 76 K is considerably higher than that due solely to twinning [4]. Thus the SL samples are actually at a lower stress in terms of slip at both temperatures during yielding than the AN samples. On the other hand, the reloading stress for the linear stress-strain region beyond yielding for the SL sample, where twinning is the rate-controlling deformation mode [4], is higher than the AN samples, indicating a shock hardening effect. Compared with a large increase with temperature in the AN samples, the work hardening rate is seen to be invariant with temperature in the SL samples.

**FIGURE 1.** Stress-strain response of the annealed (dashed) and shock-loaded (solid) Zr samples at 76 and 298 K.

**Defect structure in as-shocked Zr**

A drastic increase in defect density was seen in the matrix of Zr following shock-loading to 7 GPa. The most notable are dense deformation twins, which are evident using optical microscopy (Fig. 2(a)). The twins are smaller in size and more uniformly distributed in the matrix than those observed in specimens quasi-statically deformed to equivalent strains. TEM examination of the SL specimens revealed the presence of retained \(\omega\)-phase and reverted \(\alpha\)-phase (Fig. 2(b)), formed during the \(\alpha\)-\(\omega\) phase transition in Zr. An unusually high density of dislocations are always present in these two phases or regions where the phase transformation occurred, as a result of the lattice transfer accompanying the phase transformation [3].

Two remarkable dislocation arrangements observed using TEM in the SL specimens are of particular interest. The first is heterogeneous distributed very dense dislocation bands piercing the matrix, see Fig. 3(a). The bands affiliated with different crystallographic orientations are seen to intersect each other. At times, spine-like second phases were also seen in the middle of the bands, as shown in Fig. 3(b). The second dislocation arrangement consists of untangled dislocations distributed homogeneously in the \(\alpha\)-Zr matrix (Fig. 4(a)). While the dislocation density is considerably lower than that in the first arrangement this second structure morphology is spread throughout the grains.

**FIGURE 2.** Optical micrograph showing deformation twins (a) and TEM micrograph showing retained \(\omega\)-phase and reverted \(\alpha\)-phase (b) in the as-shocked Zr.

**Bauschinger effect**

It has been shown that the sharp elastic-plastic transition (yielding) exhibited at 76 K in the AN
sample (Fig. 1) is due to the onset of uniform twinning [4]. At this temperature the critical shear stress for slip is raised considerably by the Peierls stress that is temperature sensitive and thus the shear stress is higher than that for uniform twinning. Consequently, the material yields by twinning rather than slip. The reload curve at the same temperature, however, exhibits a parabolic elastic-plastic transition similar to those observed for slip controlled yielding. After yielding the stress-strain curve exhibits a nearly linear and slightly concave-up shape during reloading, characteristic of work hardening by twinning [4].

It is noteworthy that during reloading at room temperature (298 K) the yielding stress is seen to be significantly lower than that of the AN sample at an equivalent strain. This observation is consistent with a Bauschinger effect in Zr after shock-loading. In other words, yielding of the SL Zr occurs at a lower stress than that required for deformation in a forward direction at an equivalent strain. On the other hand, the flow stress in the twinning-dominated plastic regime beyond yielding is higher in the reloaded samples than in the AN samples. It is therefore concluded that a Bauschinger effect in SL Zr is only evident where slip is the dominant deformation mode, whereas shock-hardening is observed in the plastic regime where twinning dictates.

Microstructural evidence supporting a Bauschinger effect due to slip in Zr is seen in Fig. 4(a). Apparent in the figure is the presence of a large number of untangled dislocations distributed uniformly in the grains. This dislocation arrangement is distinctively different from that seen in the sample deformed to the same plastic strain via quasi-static deformation, see Fig. 4(b). Upon reloading the untangled dislocations start to move at a lower external stress than those in samples quasi-statically deformed to equivalent strain, which have relaxed and are tangled and formed into dislocation walls. The movement of the untangled uniformly distributed dislocations leads to a notable yielding before the onset of uniform twinning because of their fairly high density.

Work hardening

The hardening of Zr after shock-loading can be attributed to two different changes in the matrix microstructure. The first is the homogeneous formation of twins during shock-loading. Compared to twins formed during quasi-static deformation at an equivalent strain these twins appear to be thinner and more stretched. The twin-matrix boundary area per unit volume introduced during shock-loading, therefore, is considerably larger than that formed via quasi-static deformation. Upon reloading new twins intersect existing twins more frequently requiring higher stresses to sustain plastic deformation.

![Figure 3](image-url) Figure 3. TEM micrographs showing dislocation bands in Zr grains resulting from phase transformation during shock excursion (a). The retained high pressure phase, indicated by arrows, is seen in the middle of the band shown in (b).

The second microstructural change is the formation of a high density of dislocations during shock loading via the two distinctive mechanisms discussed previously. The presence of a high pre-existing mobile dislocation substructure reduces the local stress concentration required for twinning.
Consequently, higher external stresses are needed for the formation of twins during reloading. Similar observations have been reported previously \[7,8\].

**FIGURE 4.** Dislocation structures resulting from shock-loading (a) and quasi-static loading (b) in Zr.

Another interesting aspect of the reloading stress-strain response of shock-loaded Zr is the low temperature sensitivity of the work hardening rate compared with that seen for quasi-static loading, see Fig. 1. It is noted that the morphology and distribution of twins formed during shock-loading is similar to that formed at 76 K via quasi-static deformation. On the other hand, twins formed at 298 K via quasi-static deformation appear to be larger and unevenly distributed. The growth of new twins in the SL specimens is impeded by the existing high density of twin/matrix boundaries since, generally speaking, the thickness of a twin is proportional to its length. Continued plastic deformation during reloading thus relies more on nucleation of new twins than growth of existing twins. This effect results in a higher work hardening rate than that seen for quasi-static deformation. Consequently it can be concluded that the blocking action of twin/matrix boundaries is the primary microstructural cause for twinning hardening.

**SUMMARY**

Retained \(\omega\)-phase, reverted \(\alpha\)-phase, and dislocation bands resulting from high pressure phase transformations were found in Zr shock-loaded to 7 GPa. A Bauschinger effect on stress-strain response due to shock-loading has been observed. The dislocation structure responsible for the Bauschinger effect is found to consist of untangle dislocations distributed uniformly throughout the matrix. It is noted that the morphology of deformation twins plays an important role in determining the work hardening rate for Zr in the regime in which twinning is the rate-controlling deformation mode.

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**REFERENCES**