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Exploring the dislocation/twin interactions in zirconium

G.C. Kaschner [a,*], C.N. Tomé [a], R.J. McCabe [b], A. Misra [c], S.C. Vogel [d], D.W. Brown [a]

[a] MST-8, Los Alamos National Laboratory, Los Alamos, NM 87545, USA
[b] MST-6, Los Alamos National Laboratory, Los Alamos, NM 87545, USA
[c] MPA-CINT, Los Alamos National Laboratory, Los Alamos, NM 87545, USA
[d] LANSCE-LC, Los Alamos National Laboratory, Los Alamos, NM 87545, USA

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Abstract

This paper explores the 'barrier' effect that tensile and compressive twins exert upon propagation of dislocations and other twins in zirconium (Zr). We do so by pre-deforming textured Zr at liquid nitrogen to selectively induce either tensile or compressive twins; next we anneal the dislocations while preserving the twinning structure; finally we reload the material at room temperature, where only prism slip, pyramidal slip and tensile twins are active. An analysis of the yield stress upon reload, and of the subsequent hardening response allows us to conclude that the twins play a dominant role in determining the hardening, while dislocations only have a second order effect.
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Keywords: Zirconium; Deformation twinning; Dislocation recovery

1. Introduction

We have been applying a multi-faceted approach over the last several years to elucidate the dominant deformation mechanisms in hexagonal close-packed (HCP) metals [1–3]. The material of interest has been clock-rolled zirconium (Zr), processed to a moderately strong fiber texture, that exhibits strong asymmetry in its mechanical response. The mechanical experiments have included quasi-static uniaxial tension and compression, as well as high rate compression, along different directions with respect to the material's *c*-axis and at various temperatures (76–450 K). These samples have, in turn, been characterized using optical microscopy, neutron diffraction, orientation image microscopy (OIM, also known as automated electron backscatter diffraction or EBSD), and transmission electron microscopy (TEM) [4–9]. Our predictive modeling is based on the visco-plastic self-consistent code (VPSC) and has been modified to accommodate the growing fraction of reoriented matrix commensurate with deformation twinning [10–12].

Building on successes of first capturing the characteristic response of monotonic loading at room temperature and equilibrium liquid nitrogen temperature and then applying those parameters to predict the deformation of beams bent in four-point loading [11], we challenged the models with ever more interesting loading conditions. The predominant twin reorientation (PTR) model keeps track, for each grain of the aggregate, of the increasing fraction of reoriented matrix as twins accommodate shear [11]. This volume fraction increases until a threshold value is met and the entire grain is considered saturated and is assumed to adopt the orientation of the predominant twin system. The limitation of the model lies in the fact that it does not recognize such physical factors as twin morphology and the strength of twin boundaries as obstacles to dislocation motion. We determined that, although the PTR model was sufficiently adept at capturing the nuances of temperature change tests, it would not be capable of predicting the response of samples sufficiently loaded in one orientation to produce a population of deformation twins and then reloaded in another orientation.

This set of loading orientation change tests compelled the development and inclusion in the VPSC code of a new twin model that we term "composite grain" [13]. In this application, twins are assigned a representative morphology and spacing within each grain. Such "composite grain" is idealized as an ellipsoidal inclusion embedded in and interacting with a homogeneous medium. Parameters and criteria [14–16] exist for evaluating the strength of boundaries. Specifically, we are interested in the penetration of twin boundaries by dislocations, and an associated Hall–Petch type of strengthening with accumulat-

* Corresponding author. Tel.: +1 505 665 1179; fax: +1 505 667 8021.
E-mail address: kaschner@lanl.gov (G.C. Kaschner).

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Table 1
Summary of deformation modes identified in clock-rolled Zirconium for through-thickness compression at 76 and 300 K

Test conditions	Twin systems (OIM)	Slip systems (TEM)
4–10% strain, 76 K	Primarily $\{11\bar{2}2\}\langle 11\bar{2}3\rangle$ compressive twins with some $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ tensile twins in grains oriented away from *c*-axis compression. TEM revealed fine twins (100–500 nm wide) not resolved by OIM	Prismatic $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$
25% strain, 76 K	Significant numbers of $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ secondary tensile twins within primary compressive twins	Prismatic $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$
4–24% strain, 300 K	No twins observed with either OIM or TEM	Prismatic $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$ and pyramidal $\{10\bar{1}1\}\langle 11\bar{2}3\rangle$

ing strain. It is the possibility of incorporating these mechanisms in our model that compels us now to investigate the interaction of dislocations with deformation twins in our clock-rolled zirconium plate.

Our first efforts to experimentally characterize dislocation/twin interactions have shown [12] that the earliest few increments of strain (∼3.0%) in uniaxial compression are accommodated by prism slip—even at liquid nitrogen temperatures in the through-thickness loading orientation where deformation twinning of type $\{11\bar{2}2\}$ is generally dominant at moderate strains. The commensurate dislocation population at the twin boundary walls is sufficiently dense so as to obscure efforts to observe single dislocations. This challenged us to design and produce a condition where we could characterize the interactions of – ideally – only a few dislocations as they encounter a twin boundary.

The goal, then, is to create a microstructure with a twin population sufficiently great to be adequately captured in TEM foils. Further, the sample has to be annealed such that the dislocation density is sufficiently recovered but not annealed to the point of recrystallization of the twin structure (as manifested by a detectable change in macroscopic texture). We determined that this could be accomplished by either of two paths: (1) a monumental task of compressing samples in liquid nitrogen, annealing, preparing TEM foils, and characterizing or (2) compress samples in liquid nitrogen and then monitor changes in texture during annealing using *in situ* neutron diffraction. We chose the latter.

An equally significant product of this investigation is to achieve a first opportunity to interrogate and compare the relative magnitude of the effects of dislocation forests versus twin barrier Hall–Petch type effects on the yield stress and hardening. If we anneal a material with a pre-existing deformation twin microstructure to reduce its dislocation density, is the yield strength, upon reload at room temperature, dominated by the accumulated population of forest dislocations? Or, is it predominately influenced by the presence of deformation twin boundaries that act to interrupt and reduce the mean free path of dislocations? By recovering an appreciable fraction of the dislocation forests (via an anneal) yet not recrystallizing the matrix, we are thus able to examine the effects of dislocation/twin boundary interactions in a Hall–Petch framework.

2. Experimental design

The material used in this investigation is from the same plate used in our recent, previous works [9,12–17]. It is high purity zirconium, arc-cast into an ingot that is then knock-down forged and clock-rolled such that the final material has a moderately strong *c*-axis fiber texture normal to the plane of the plate. Interstitial impurity content is sufficiently low that it twins readily and predictably over a range of temperatures and strain rates at various orientations [1–3]. The grains are equiaxed with an average diameter of approximately 15–20 μm.

A comprehensive TEM and OIM characterization of the zirconium used in this study is contained in McCabe et al. [9]. The paper evaluates the effects of texture, temperature, and strain including samples strained in uniaxial compression at 76 K in both through-thickness (TT) and in-plane (IP) orientations. We present a synopsis of the typically observed deformation mechanisms in Tables 1 and 2.

The samples are first deformed in uniaxial compression to a strain of 10% at a strain rate of $0.001\ s^{-1}$ in an equilibrium liquid nitrogen (LN) bath to establish a microstructure that is rich in twins. Samples were loaded in either of two orientations: "TT" orientation is loaded with the compression axis normal to the plane of the plate and induces compression twins; "IP" orientation is loaded with the compression axis parallel to the plane of the plate and induces tensile twins. These loading con-

Table 2
Summary of deformation modes identified in clock-rolled zirconium for in-plane compression at 76 and 300 K

Test conditions	Twin systems (OIM)	Slip systems (TEM)
4–10% strain, 76 K	Tensile twins: primarily $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ type with some $\{11\bar{2}1\}\langle 11\bar{2}6\rangle$ type. TEM revealed fine twins (100–500 nm wide) not resolved by OIM	Prismatic $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$
25% strain, 76 K	Considerable secondary $\{11\bar{2}2\}\langle 11\bar{2}3\rangle$ compressive twins within the primary $\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ tensile twins	Prismatic $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$
4–29% strain, 300 K	$\{10\bar{1}2\}\langle 10\bar{1}1\rangle$ tensile twins. No twins detected in TEM	Prismatic $\{10\bar{1}0\}\langle 11\bar{2}0\rangle$