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Twin boundary interactions with grain boundaries investigated in pure rhenium

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Abstract

The mechanical behavior of pure rhenium was investigated using uniaxial compression tests, transmission electron microscopy and electron backscatter diffraction characterization. The plasticity was characterized by a large amount of twin formation and propagation, including twin transmission across grain boundaries. In-depth analysis of the interactions of $\{11\bar{2}1\}\langle\bar{1}\bar{1}26\rangle$ twins with grain boundaries found that grain boundaries with misorientation angles below $\sim 25°$ allowed twin transmission, while grain boundaries with higher angles did not. Similar to dislocation interactions with grain boundaries, twin transmission was largely dictated by the minimization of the angle between the shear vectors of the incoming and outgoing twins.

Keywords: Twinning; Rhenium; Mechanical behavior; Electron backscatter diffraction; Transmission electron microscopy

1. Introduction

Owing to the restricted number of slip planes, deformation in many hexagonal close-packed (hcp) metals involves twinning mechanisms. Consequently, the mechanical behavior of hcp metals can be governed largely by the interaction of twins with defects. For example, interfaces such as grain boundaries can act as nucleation sites for twins as well as obstacles to twin propagation. An understanding of how these interactions unfold, including the dominant factors dictating their progression, is essential to a fundamental understanding of the mechanical behavior of hcp materials in application.

Re is unusual among hcp materials in that the dominant twining system, when either tensile or compressive stresses are imposed on the sample, is $\{11\bar{2}1\}$ $\langle\bar{1}\bar{1}26\rangle$ [1,2]. It is also unusual among refractory metals in that it has no known ductile to brittle transition. For this reason, as well as its excellent ductility and formability, high-temperature strength, and creep resistance, it is an attractive material in extreme environment applications [3,4]. However, limited availability and difficulty of extracting Re has limited both the application and the knowledge of Re. Characterization of the deformed microstructure of Re has been limited primarily to optical microscopy [3,4] as well as a limited number of transmission electron microscopy (TEM) studies [1,2,5]. These studies reported observations of dislocation plasticity occurring on the basal and prismatic planes, as well as twinning activity on $\{10\bar{1}2\}$, $\{11\bar{2}2\}$, and $\{11\bar{2}1\}$.

Statistical studies using electron backscatter diffraction (EBSD) have investigated the behavior of twinning in deformed Mg and its alloys in relation to microstructural characteristics such as grain size and grain boundary character [6–8]. A consistent finding in these studies is that twinning activity is strongly correlated with grain

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boundary misorientation, with lower-angle grain boundaries encouraging the nucleation and growth of twins. It was also found that correlated twinning, where twins in neighboring grains meet at a boundary, is much more likely to occur at low-angle grain boundaries. As all these studies were done *post mortem* at the resolution of EBSD, it could not be determined whether twins meeting at the boundary arose as a result of simultaneous nucleation and growth into both grains or whether it was an impingement and transmission or nucleation event as a single twin traveled across the boundary. Similar studies in Zr also showed a correlation between misorientation angle and propensity for twinning, though not as strongly as that seen in the Mg studies [9].

Bieler and colleagues [10–12] have published a number of studies investigating individual instances of $\{10\bar{1}2\}$ twin interactions with grain boundaries in α-Ti using EBSD analysis and *in situ* synchrotron characterization. They found that, when a grain is surrounded by other grains that are unfavorably oriented for slip, twin nucleation was dictated by the resolved shear stress. In contrast, when a grain was surrounded by other grains that are favorably oriented for slip, twin nucleation was initiated by dislocation pileups at the grain boundary. The nature of the twin plane and shear vector could be predicted based on maximizing the m' factor, which is given as [13]

$$m' = \cos\kappa\,\cos\psi \quad (1)$$

where κ is the angle between slip plane normal of the incoming dislocation system and the twin plane normal, and ψ is the angle between shear/Burgers vectors of the two systems on either side of a grain boundary.

In the current study, twinning behavior, with an emphasis on twin interactions with grain boundaries, are investigated in pure polycrystalline Re samples deformed under uniaxial compression. Multiple interactions were investigated using EBSD, after which TEM analysis was applied to investigate the interactions at a higher resolution.

2. Experimental methods

The 99.99% pure Re was supplied by Rhenium Alloys, Inc., in the form of a 3-mm-diameter rod. Compression samples 4 mm tall were cut from the rod using electrical discharge machining (EDM). The samples were then annealed for 20 h at 1100 °C in a 50% hydrogen, 50% argon environment. For digital image correlation (DIC) purposes, a speckle pattern was created on the sample surfaces by first spray painting them white, and then lightly spraying them with black spray paint (Fig. 1b). Two samples were strained in compression using a MTS Criterion Model 43 testing machine at a nominal strain rate of $10^{-3}\ s^{-1}$. The compression was recorded for DIC analysis using a 1280 × 1024-pixel Dino-Lite camera at a frame rate of 15 frames per second. DIC analysis of the images was performed in Matlab (analysis details can be found in Ref. [14]).

Samples were prepared for TEM and EBSD analysis using EDM to cut two disks from the center of each compressed rod with an approximate thickness of 150 μm. Polishing of the sample surface was performed using a twin-jet polisher with a 78% ethanol, 11% butoxyethanol, 11% perchloric electrolyte maintained at a temperature of −10 °C. As needed, thinning to electron transparency was achieved using a PIPS II ion mill with a final accelerating voltage of 0.5 keV. Orientation-dependent etching rates resulted in ridging at twin and grain boundaries, which facilitated site-specific focused ion beam (FIB) lift-out samples to be machined from locations of interest. The post-FIB amorphous layer was removed using Ar-ion milling in a FEI NanoMill operated at 900 eV.

TEM analysis was performed using a JEOL 3010 TEM operated at 300 kV, and scanning transmission electron microscopy (STEM) analysis in a FEI Titan instrument, also operated at 300 kV in STEM mode. A camera length of 300 mm was used for diffraction contrast imaging. EBSD scans were taken using an accelerating voltage of 20 keV over 70 × 70 μm regions at a 100-nm step size. No cleanup of the data was done except for the removal of points with a low confidence index value. The large grain-to-twin size ratio required that a small step size be used in relation to the grain size, limiting the size of the scans acquired and reducing the statistical significance of the results.

3. Results and discussion

The engineering stress/engineering strain curve for the compression tests is shown in Fig. 1. The samples showed high rates of work hardening, similar to those reported previously [2,4].

Inverse pole figure (IPF) maps created from the EBSD data and an IPF of the annealed Re are shown in Fig. 2. Areas of poor indexing, represented as black pixels in the maps, are present. These were caused by differential etching rates, dependent on crystal orientation, leading to significant ridging occurring at grain boundaries and shadowing in the EBSD patterns. The decreased indexing rate did not adversely affect the analysis of the scans. The undeformed matrix is composed primarily of grains with the *c*-axis oriented perpendicular to the surface normal and an average grain size of 30 μm. No twins were seen in the pre-deformation microstructure, and the uniform orientations across the grain interiors suggest that there are few dislocations present. As the deformation progresses, the microstructure becomes dominated by deformation twins, with the twinned area of the microstructure increasing from 12.0% of the total area after compression to 5% strain (Fig. 1b) to 19.4% of the total area after compression to 8% strain (Fig. 1c). These twins were all of the same type, $\{11\bar{2}1\}\langle\bar{1}\bar{1}26\rangle$, and had an average width of 700 nm. Multiple locations are evident in the scans where twins meet at the grain boundaries. At other locations, the twins impinge on only one side of the boundary, with no correlated twin