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Influence of irradiation damage on slip transfer across grain boundaries

B. Cui [a], J. Kacher [a], M. McMurtrey [b], G. Was [b], I.M. Robertson [a,c,*]

[a] Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign, 1304 West Green Street, Urbana, IL 61801, USA
[b] Department of Nuclear Engineering and Radiological Sciences, University of Michigan, 1921 Cooley Building, Ann Arbor, MI 48109, USA
[c] Department of Materials Science and Engineering, University of Wisconsin-Madison, 1415 Engineering Drive, Madison, WI 53706, USA

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Abstract

The interaction of lattice dislocations with grain boundaries in proton- and ion-irradiated austenitic stainless steels is investigated. At the macroscale, interactions of dislocation channels with grain boundaries result in slip blockage, slip transmission and slip along the grain boundary with an associated in-plane displacement. At the dislocation level, slip transmission prediction is governed by the strain energy density minimization. However, the importance of the local resolved shear stress increases, in the sense that it must be of sufficient magnitude to propagate the dislocations through the damaged matrix. If this condition is not satisfied, the grain boundary will activate an alternative mechanism to relieve the strain accumulated from accommodation of lattice dislocations and the stress concentration associated with the formation of a dislocation pile-up. One observed relief mechanism involves the nucleation and propagation of a crack along the grain boundary, which is accompanied by an out-of-plane displacement of the boundary.

Keywords: Grain boundary; Irradiation; Slip transfer; Transmission electron microscopy; Austenitic stainless steels

1. Introduction

Deterministic criteria for predicting the process of slip transfer across grain boundaries have been developed and shown to be applicable for unirradiated face-centered cubic (fcc) and hexagonal close packed metals as well as for interfaces between dissimilar metals [1–5]. The criteria are as follows.

(1) The magnitude of the Burgers vector of the dislocations left at the grain boundary $|\mathbf{b}_r|$ following the emission of dislocations should be minimized; i.e., $\mathbf{b}_r = \mathbf{b}_{in} - \mathbf{b}_{out}$, where $\mathbf{b}_{in}$ and $\mathbf{b}_{out}$ refer to the Burgers vector, expressed in the same frame-of-reference, of the incoming and outgoing dislocations, respectively.
(2) The resolved shear stress acting on the possible slip systems in the adjoining grain should be maximized.
(3) The angle between the lines of intersection of the incoming and outgoing slip planes with the grain boundary plane should be minimized.

Of these, the slip system activated by the grain boundary in response to an impinging slip system is determined primarily by the first criterion. That is, the dominant system activated by the grain boundary will minimize the strain energy density accumulation in the grain boundary, but it must experience sufficient shear stress to ensure dislocation motion away from the nucleation site and into the grain. In general, nucleated dislocations propagate away from the grain boundary source easily, suggesting that nucleation is the rate-limiting step in the transfer process.

Irradiation of a metal with energetic particles results in the formation of dislocation loops, stacking-fault tetrahedra and other defect clusters, which serve to harden the matrix [6,7]. The irradiation of an alloy can also induce segregation, which can lead to an increase or decrease in the

* Corresponding author. Address: Department of Materials Science and Engineering, University of Wisconsin-Madison, 1415 Engineering Drive, Madison, WI 53706, USA. Tel.: +1 217 979 9947.
E-mail address: irobertson@wisc.edu (I.M. Robertson).

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concentration of an element from a specific location. For example, in stainless steels neutron-irradiated at low temperatures ($T < 0.3T_m$, where T_m is the melting temperature), radiation-induced segregation results in depletion of Cr from the grain boundary [6,8]. A consequence of this irradiation hardened matrix is a transformation of the deformation mechanism from relatively homogeneous slip in unirradiated materials to the confinement of slip to coarse bands termed dislocation channels, in which the passage of dislocations has altered the defect state and in some cases removed it [9–12]. For example, Briceño et al. have shown how the dislocations create the channels through their interaction with ion-irradiation defects [9]. They found that dislocation motion was impeded by the obstacles, occurred in a fragmented and jerky manner, and the dislocation velocity increased as the channel became more structured and distinct. The passage of multiple dislocations within the channel appears to remove the irradiation defects and leads to the eventual formation of a defect-free channel [10,13]. Kiener et al. [14,15] conducted quantitative *in situ* nanocompression testing in a transmission electron microscope (TEM) on proton-irradiated (100)-oriented single crystal Cu pillars. They showed that the strength of Cu pillars is affected by the interaction between moving dislocations and radiation-induced defects, and size-independent yield strengths are measured for samples as small as ∼400 nm in diameter. For even smaller specimens, the yield strength of the Cu pillars becomes affected by the sample size, as the stress required to operate ever smaller dislocation sources overcomes the obstacle strength posed by the radiation-induced defects. All these studies revealed distinct characteristics of deformation mechanisms in irradiated materials are different from those in unirradiated ones.

Understanding how the dislocations within these channels interact with grain boundaries has important implications for determining how strain is transferred across them in terms of the mechanical properties of irradiated metals [6,8]. The present paper reports the results of a study of slip transfer across grain boundaries in either proton- or heavy-ion-irradiated 304 and 13Cr15Ni stainless steels. At the microscale, the dynamics of the interactions of dislocations with grain boundaries were observed in real time by performing deformation experiments *in situ* by TEM, following irradiation with heavy ions, 1 MeV Kr^+, to a dose of 0.14 displacements per atom (dpa). The primary objective was to ascertain whether and how the presence of a hardened matrix influences the criteria for predicting slip transfer across grain boundaries. To the present authors' knowledge, this study is the first to quantify the slip transfer process in irradiated material. At the macroscale, the interaction of slip bands with grain boundaries was assessed in proton-irradiated (3.2 MeV protons, 5 dpa, 360 °C) 13Cr15Ni stainless steels, using a combination of scanning electron microscopy (SEM) coupled with digital image correlation to determine the strains following mechanical testing at 288 °C. Although the test conditions differ significantly, the dislocation interactions with grain boundaries show similarities. The commonalities enhance the interpretation of the results from both experiments.

2. Experimental procedures

The materials used were a commercial purity 304 (Fe–18.3Cr–8.5Ni–1.38Mn–0.65Si–0.04C by weight) and a 13Cr15Ni (Fe–13.41Cr–15.04Ni–1.03Mn–0.1Si–0.016C) austenitic stainless steel. The geometrical dimensions of the straining stage TEM samples were typically $11.5 \times 2.5 \times 0.2$ mm. Two 1-mm-diameter holes were drilled into the ends of the bar for attaching it to the straining stage. The samples were then annealed under vacuum at 1060 °C for 30 min to minimize the density of matrix dislocations and to develop a large grain size. The central section was thinned to electron transparency using a twin jet polisher with a 6% perchloric acid, 39% butanol and 55% methanol electrolyte at −20 °C. The ion irradiation experiments were performed *in situ* in the IVEM-Tandem microscope at Argonne National Laboratory [16,17]. The samples were irradiated at room temperature with 1 MeV Kr^+ to a dose of 3×10^{17} ions m^{-2}. This ion energy and dose was selected to give a reasonable defect density throughout the foil [9,18]. It was reported that the irradiation-induced defects in austenitic stainless steel are dislocation loops and "black dots" (defect clusters with a diameter <2 nm) for both proton [12,19] and heavy ion [9,18] irradiated samples. Using the TRIM simulation code [20], it was estimated that this ion dose is equivalent to a damage level of 0.14 dpa for a 100-nm-thick foil. The TRIM simulation suggested that 96% of the Kr ions pass through the foil, resulting in negligible ion implantation in the sample. The deformation experiments were performed *in situ* in either the IVEM microscope at Argonne National Laboratory or a JEOL 2010 LaB_6 TEM operating at 200 keV. The *in situ* TEM straining was accomplished using a single-tilt heat/strain stage (Gatan Model 672, Gatan Inc., Warrendale, PA) allowing samples to be deformed *in situ* under tension.

Microstructures were examined in the TEM using two-beam bright-field imaging conditions, and selected-area electron diffraction was used to obtain crystallographic information. The dynamic interactions between matrix dislocations and grain boundaries were recorded as videos, using a CCD camera with a recording rate of 10 fps. The recorded video together with still-frame micrographs were used to determine the active slip systems. Using the conventional g.b invisibility condition, the Burgers vector of the incoming and outgoing dislocations were determined. The character of the dislocations and their slip planes were identified using trace analysis techniques. Grain boundary misorientation (i.e., the axis/angle pair) was calculated using sets of Kikuchi patterns acquired at different tilts on either side of the grain boundary and following the procedure outlined by Edington [21]. The grain boundary misorientation was used to form the transformation matrix T,