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ScienceDirect

Acta Materialia 65 (2014) 393–399

Acta MATERIALIA

www.elsevier.com/locate/actamat

Inhomogeneous deformation behavior in intercrystalline regions in polycrystalline Ni

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Received 10 September 2013; received in revised form 1 November 2013; accepted 1 November 2013
Available online 5 December 2013

Abstract

We report on three-dimensional spatially resolved X-ray microdiffraction measurements of inhomogeneous deformation within individual polycrystalline grains. These measurements provide new insights into the role of grain boundaries. Particularly striking is the qualitatively different mechanical behavior of subgrain volumes near grain boundaries and triple junctions compared to bulk-grain mechanical behavior. These differences are studied by characterizing the evolution of the local orientation distribution in a polycrystalline Ni sample using polychromatic synchrotron X-ray microdiffraction. Dependence of deformation on grain boundary types and triple junctions is determined. Quantitative distinctions in deformation behavior between low-angle boundaries, special low-energy high-angle boundaries, general high-angle boundaries and triple junctions are characterized in terms of spatially resolved misorientation development and extensions of grain boundary effects. In general, larger lattice misorientations are observed near boundaries with higher grain boundary energy. Special high-coincidence grain boundaries exhibit smaller deformation misorientations than general high-angle grain boundaries. These observations are consistent with the observed increases in ductility in grain boundary engineered materials.

Keywords: Plastic deformation; Grain boundaries; X-ray diffraction

1. Introduction

Polycrystalline materials are distinguished by their network of misoriented anisotropic grains and grain boundaries. This complex network significantly alters plastic deformation, and identification of differences in grain boundary behavior is central to efforts at grain boundary engineering and property modification [1,2]. This information is essential to guide the development of realistic computational models for deformation and for understanding the underlying features of polycrystalline deformation [3–5]. X-ray microdiffraction measurements from single grains have revealed average grain rotations that are inconsistent with widely used deformation models, and thus have raised intriguing questions that go to the heart of our ignorance about polycrystalline deformation [6]. In particular, identifying the difference in behaviors between different grains and grain boundaries is necessary to improve the constitutive equations in deformation models to describe the behavior of polycrystals realistically.

Although there are many unknown aspects of polycrystalline deformation, there is compelling evidence for the importance of grain boundaries with respect to mechanical properties [7,8]. The basic driving force for size-dependent deformation behavior is believed to arise from the interaction of dislocations with grain boundaries. A good example of the importance of grain structure is the improved mechanical properties of materials with small grains predicted by the Hall–Petch relation. Improvement in fracture toughness and an increase in the brittle–ductile transition temperature are associated with small grain size [9]. Superior mechanical properties and superplasticity of nanocrystalline materials are largely attributed to grain

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1359-6454/$36.00
http://dx.doi.org/10.1016/j.actamat.2013.11.008

boundary effects [9]. At the same time, damage mechanisms such as cracking are often initiated at the intercrystalline regions [10].

Mesoscale models based on discrete dislocation plasticity [11,12], strain-gradient plasticity [13,14] and continuum plasticity [15] to calculate the collective behavior of large numbers of dislocations in polycrystalline systems are under rapid development, largely because of the growth of supercomputers and enhancements in computational techniques. The thrust of mesoscopic models is to calculate the collective behavior of large numbers of dislocations to determine the evolution of deformation microstructures and their effects on the material properties. A common feature of these models is an attempt to relate stress rate to strain rate (including both dilatation and rotation) for a given material state and boundary conditions through a set of constitutive equations. The significance of size-dependent modeling is its ability to predict the variation of stress and strain states across interfaces and within individual crystals. This emphasis recognizes the need to incorporate the experimental observation of the formation of deformation microstructures in the mesoscale range of submicron to hundreds of micrometers [16,17].

Our present knowledge of deformation microstructure has been largely obtained by detailed, but qualitative, electron microscopy [18,19]. TEM studies on samples with tilt/twin boundaries reveal specific dislocation and disclination activity down to the atomic level; however, structural relaxation and the lack of 3-D dislocation networks in thin-foil samples precludes capture of the true complexity of deformation inhomogeneity in bulk polycrystals. Orientation imaging microscopy (OIM) with scanning electron microscopy is a widely used technique to determine the microtexture and orientation distributions of sample surfaces [20–22]. OIM is time-efficient; however, the angular resolution is limited to ~0.1–1.0°, depending on the sample quality and experimental setup [23]. The need for a smooth surface also restricts studies to small deformation [23]. Electron microscopy techniques, though powerful, cannot study buried interfaces in bulk materials.

Advances in synchrotron X-ray sources and optics have enabled the development of a new class of instrumentation called 3-D X-ray crystal microscopes [24]. The submicron-resolution 3-D X-ray Laue microdiffraction [25,26] developed at the Argonne Photon Source (APS) beamline 34-ID, with a volumetric spatial resolution of $\sim 0.5 \times 0.5 \times 1\ \mu m^3$, is particularly suitable for the investigation of deformation mechanisms at the mesoscopic level. The angular resolution is ~0.02° for undeformed samples and typically ~0.1–0.2° for deformed samples. Local rotations within grains and misorientations across individual grain boundaries and triple junctions can all be non-destructively measured quantitatively with high precision in three dimensions. In this work, we begin to directly address questions about the near-boundary behavior of materials by making measurements of subgrain behavior, including information about the local environment.

2. Experimental technique

The X-ray microdiffraction experiment was designed to characterize the behavior near individual grain boundaries in a Ni polycrystalline sample and was performed with the 3-D X-ray crystal microscope on station 34-ID-E at the APS. The 3-D X-ray Laue microdiffraction technique does not yet have the resolution to resolve individual dislocations. Instead, the lattice rotations caused by collective dislocation motions in individual volume elements are measured [27]. The rotation matrix ΔG between the two volume elements with crystallographic orientations g_1 and g_2 was first determined by

$$\Delta G = g_1 g_2^{-1} \tag{1}$$

Each of the rotation matrices ΔG^i [28] equivalent to ΔG based on cubic symmetry was then deduced by

$$\Delta G^i = O_C^m g_1 g_2^{-1} O_C^n \tag{2}$$

where O_C^m and O_C^n represent the 24 independent symmetry operators for the cubic point group.

The misorientation angle ω and rotation axis UVW between two volume elements are defined by

$$\cos\omega = \min\left[\left(\frac{\Delta G^i_{11} + \Delta G^i_{22} + \Delta G^i_{33} - 1}{2}\right)\right]$$
$$U : V : W = \Delta G^i_{32} - \Delta G^i_{23} : \Delta G^i_{13} - \Delta G^i_{31} : \Delta G^i_{21} - \Delta G^i_{12} \tag{3}$$

Full orientation tensors for each volume element can be determined by the microbeam Laue technique, and misorientations and common plane sharing between two adjacent volumes can be calculated with high precision with its 3-D penetration ability. Unambiguous determination of the grain boundary geometry can be made, which is impossible with electron microscopy. This capability allows us to quantify the relationship between grain boundaries and plastic deformation effectively.

The purpose of the measurements is to provide new insights with regard to the importance of neighboring grains and the effect of particular local grain boundary types on deformation mechanisms. These include low-angle boundaries and boundaries with highly ordered structures which possess low-energy states. They are denoted as coincidence site lattices (CSLs), with Σ-values <29 (see Randle [29] for a definition of CSL). Twin boundaries ($\Sigma = 3$) show the lowest energy among all CSLs.

3. Experiment

A 99.97% pure Ni sample with an average grain size of ~30 μm was studied. The polycrystalline material was annealed in vacuum at 800 °C for 4 h for strain relief. A tensile sample, with a tensile length of 10 mm and a thickness of 0.5 mm, was fabricated by electron discharge. The sample surface was mechanically polished and electrochemically polished with a 3:1 ratio of hydrochloric acid and sulfuric acid. Microindents 30 μm in width were marked on the two ends of the tensile axis of the sample