



Full length article

Experimental quantification of mechanically induced boundary migration in nanocrystalline copper films

Paul F. Rottmann^a, Kevin J. Hemker^{a, b, *}^a Department of Materials Science & Engineering, Johns Hopkins University, Baltimore, MD, 21218, USA^b Department of Mechanical Engineering, Johns Hopkins University, Baltimore, MD, 21218, USA

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ABSTRACT

A new experimental technique is presented that combines *in situ* straining with transmission electron microscope-based automated crystal orientation mapping to document microstructural evolution with nanoscale resolution at sequential stages of deformation. Orientation maps of freestanding annealed nanocrystalline Cu films have been collected, and the resultant datasets provide direct measures of grain size, shape and orientation as well as local grain boundary character and position at various stages of applied strain. Numerous examples of stress-driven grain boundary and twin boundary migration were recorded and studied. Detailed analysis of the misorientation of mobile and immobile grain boundaries provided clear experimental evidence that a broad range of grain boundaries are susceptible to stress-assisted migration; no general correlation between grain boundary misorientation and mobility was detected. Incoherent $\Sigma 3$ boundaries were observed to be significantly more mobile than coherent $\Sigma 3$ twin boundaries. Nevertheless, deformation twins were observed to nucleate and grow from grain boundaries and triple points.

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1. Introduction

It is broadly understood that for polycrystalline metals, yield strength scales with grain size [1] and that mechanical behavior can be tailored by carefully controlling the grain size of a microstructure. Because of this, nanocrystalline metals hold significant promise due to their exceptionally high strength, but they are limited by a lack of ductility. Wang et al. highlighted this tradeoff between strength and ductility [2], and the holy grail in nanocrystalline materials research has become the search for ways to increase ductility without significant loss of strength. One potential avenue to this goal was uncovered when Gianola et al. [3] observed that stress-assisted room temperature grain boundary (GB) migration can impart nanocrystalline Al with significant ductility albeit with modest reductions in strength. When dislocation plasticity is mitigated and stress-assisted GB migration is active, shear stresses can and do couple to high-angle grain boundaries causing them to migrate, which leads to significantly higher tensile ductility [4–6]. There has been much progress in recent years in determining

the conditions under which stress-coupled GB migration is active; stress-coupled GB migration and attendant stress-assisted grain growth have been observed in a wide range of nanocrystalline metals under various deformation conditions: Al [5,7–12], Cu [13–20], Mg [21], Ni [16,22–25], Pt [26], and a few alloys [27,28].

Mechanistic studies of stress-coupled grain boundary migration in nanocrystalline metals and alloys fall under three categories: *ex situ* experiments, simulations, and *in situ* experiments. *Ex situ* experiments have been used to determine whether stress-coupled GB migration is active by measuring the grain size distribution of samples before and after straining and noting if there is a statistical difference. In these experiments, which have been conducted across large ranges of temperature and strain rate, the extent of the coarsening of the microstructure is attributed to stress-assisted migration of boundaries. These experiments have been conducted on many different materials [5,7–18,21–28], and stress-coupled GB migration has been observed for both fcc and hcp crystal structures. *Ex situ* experiments have also provided key insights that have led to better understanding of stress-coupled GB migration. There are numerous studies that have observed and quantified the migration of single, well-characterized GBs within a bicrystalline specimen at elevated temperatures [29–36]. These studies have shown that mobility correlates with misorientation angle for low-angle 100

* Corresponding author. Department of Materials Science & Engineering, Johns Hopkins University, Baltimore, MD, 21218, USA.

E-mail address: hemker@jhu.edu (K.J. Hemker).

boundaries in Al [36] and that certain CSL-type boundaries do not necessarily couple to shear stresses in the same way as random, high-angle grain boundaries [35].

Observations of mechanical grain growth at cryogenic temperatures [13], high rates [21], and low homologous temperatures [26] all point to the importance of stress in promoting GB migration. Moreover, *ex situ* experiments performed by Rupert et al. employing local stress and strain concentrators demonstrated that grain growth in nanocrystalline Al correlates with the global imposed shear stress rather than strain or principle stresses [12]. In order to better describe the mechanism of stress-coupled GB migration in particular, and the deformation behavior of nanocrystalline materials in general, it is desirable to know whether there are certain types of boundaries that are significantly more mobile than others and to measure the relative velocity at which different boundaries move. Previous experiments on bicrystals and well-characterized flat tilt and grain boundaries provide clear insight, see for example [35], but the number of flat boundaries that have been studied is rather modest. Polycrystalline specimens possess a much greater variety of grain boundaries, but detailed experimental observations of grain growth are hard to obtain owing to the complexity of the microstructure and the need for nanoscale spatial resolution.

Computational simulations, especially molecular dynamics (MD), do provide an opportunity to visualize atomic-scale mechanisms and. MD simulations have been used to study specific boundaries and to suggest potential mechanisms for how stress-coupled GB migration operates. For example, MD simulations of [001] symmetrical tilt boundaries in Cu motivated Cahn et al. to propose a unified approach to the motion of grain boundaries [37–39]. These MD simulations, which were performed for high-angle tilt boundaries that cannot simply be represented as an array of edge dislocations, pointed to the coordinated motion of clusters of atoms as the mechanism by which these boundaries migrate in response to an applied shear. This study was subsequently expanded to account for general grain boundaries by other researchers [40–42]. Results from these simulations also suggest that GB migration is not governed by dislocation migration or diffusive processes. Other MD simulations have sought to quantify the relationship between the energy required for a GB to couple to shear stress and migrate and temperature [43]. MD simulations employing a synthetic driving force have been used to determine how the mobility of a boundary varies as a function of its character in metals like Al, Au, Cu, and Ni [44–47]. These simulations suggest that the mobility of coherent $\Sigma 3$ boundaries is lower than other boundaries but did not find a direct correlation of mobility with GB misorientation, energy, or free volume. More recently, simulations have been performed that observe the migration of CSL-type grain boundaries located in polycrystalline networks [48].

Taken as a whole the MD simulations have advanced to the point that atomic-scale characterization of specific boundaries has been conducted for a variety of metals; however, there are limitations on the types of grain boundaries that can be studied using simulations. Generally, the simulated grain boundaries are limited to straight, defect-free, CSL boundaries with no effects from triple points or other boundaries. Experiments would be very helpful in substantiating the findings of the MD simulations and for observing the behavior of a wide range of curved boundaries with shapes, misorientations, and characters that are typical of polycrystalline microstructures, but *ex situ* snapshots do not provide this level of detail.

In situ experiments performed by straining nanocrystalline thin foils in a transmission electron microscope (TEM) do allow one to

observe the sample while it is deforming. The first direct observation of stress-driven GB migration *in situ* was performed by Jin et al. in ultrafine-grained Al [49]. Legros et al. studied stress-coupled GB migration with TEM-based *in situ* straining and bright field video recording [7,8,41,50]. This technique allowed them to accurately measure the grain boundary velocity and show that it was too high to be explained by diffusive processes; however, since only a few grains were clearly illuminated, they were not able to obtain a full 2D representation of the microstructure or information on the character of the grain boundaries.

The introduction of SEM-based electron backscatter diffraction (EBSD) has revolutionized microstructural characterization by enabling rapid collection and indexing of Kikuchi diffraction patterns and the formation of orientation maps. Use of EBSD for *in situ* studies of nanocrystalline microstructures is challenged by spatial resolution limits and by the fact that EBSD only maps the surface grains. Transmission Kikuchi Diffraction (TKD) addresses some of these issues and can be used to collect orientation maps with 2–3 nm resolution [51]. TEM-based automated crystal orientation mapping (ACOM), developed by NanoMEGAS, uses selected area diffraction patterns to determine crystal orientation and allows for rapid collection of diffraction patterns (>200 per second) and reliable indexing of the phase and orientation of the crystal with a spatial resolution of 2 nm [52,53]. This allows rapid collection of 2D maps that contain a wealth of information about the microstructure including grain size, grain boundary character, and texture. Additionally, the orientation maps can be uploaded into existing EBSD analysis software. By combining this technique with *in situ* straining it is possible to observe the overall microstructure throughout deformation and to characterize mobility of specific grain boundaries. This technique has been utilized by Kobler et al. to observe the evolution of a deforming nc Au and Pd thin films [54–57]; evidence of deformation twinning and stress-assisted grain growth was noted. This technique was also used by Mompieu et al. to measure the rotation of grains undergoing stress-driven GB migration [58].

This manuscript outlines a study that focused on applying TEM ACOM to nanocrystalline Cu films to directly observe deformation mechanisms and quantify the effect of local microstructure on them. Specifically, the goal of this work is to better understand nanocrystalline deformation mechanisms such as stress-coupled GB migration and twin boundary migration by characterizing the mobility of specific boundaries.

2. Experimental details

2.1. Specimen fabrication

Freestanding *ex situ* and *in situ* thin film tensile specimens were produced using the lift-off procedure outlined in Fig. 1. Polished 4" silicon wafers were patterned with an array of dog-bone shaped plateaus using Futuress NR9-3000PY negative photoresist and UV photolithography. The fabrication procedure produced photoresist layers 1.5 μm thick with near-vertical side walls, which assured no connectivity between the film on the photoresist and on the wafer. This allowed for consistently smooth edges around the specimens to be retained throughout lift-off. A blanket film of copper (99.999% pure) was deposited over the whole wafer at a rate of 1.5 $\text{\AA}/\text{s}$ using electron beam vapor deposition to a final thickness of approximately 200 nm and 60 nm for *ex situ* and *in situ* specimens, respectively. Film thicknesses were verified using contact profilometry. For the *ex situ* specimens, a polypropylene loading frame was then attached to the specimen using Araldite fast-setting two

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