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A comparison of grain boundary evolution during grain growth in fcc metals

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Abstract

Grain growth of Cu and Ni thin films, subjected to in situ annealing within a transmission electron microscope, has been quantified using a precession-enhanced electron diffraction technique. The orientation of each grain and its misorientation with respect to its neighboring grains were calculated. The Cu underwent grain growth that maintained a monomodal grain size distribution, with its low-angle grain boundaries being consumed, and the Ni exhibited grain size distributions in stages, from monomodal to bimodal to monomodal. The onset of Ni's abnormal grain growth was accompanied by a sharp increase in the Σ3 and Σ9 boundary fractions, which is attributed to simulation predictions of their increased mobility. These Σ3 and Σ9 fractions then dropped to their room temperature values during the third stage of grain growth. In addition to the Σ3 and Σ9 boundaries, the Σ5 and Σ7 boundaries also underwent an increase in total boundary fraction with increasing temperature in both metals.
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1. Introduction

The strength, toughness, corrosion resistance, electrical properties and optical properties of nanocrystalline materials are frequently grain size dependent [1–4]. The stability of these properties is of concern, since these fine grain sizes are associated with an inherently high driving force to coarsen [1,2,5]. Extending the processing and application of these fine grain materials requires validation of modeling predictions. Elucidating microstructural evolution is currently one of the central tasks of materials science [3,5,6].

Examples of microstructural instability in nanocrystalline metals include abnormal grain growth at low temperatures [7], as well as abnormal grain growth under cyclic loads [8]. Since grain boundary properties, such as energy and mobility, play a key role in determining the growth of grains, there has been a long-standing interest in these topics. Recent microstructural modeling that explicitly includes the variation of boundary properties with boundary type [9–13] has drawn even more interest to the topic. These studies utilize simple models for the variation in boundary properties with macroscopic boundary geometry, and they would benefit greatly from a more complete description of the property variations.

The conventional concept of grain growth, derived from coarse-grained polycrystalline studies [14], is that the process is driven by the reduction of the total grain boundary area in the material. The underlying mechanism involves curvature-driven grain boundary migration, where the motion of the grain boundaries is towards the center of their curvature [6,11]. Normal grain growth occurs continuously, with grain boundary migration distributed uniformly over the interfacial network. This allows the grain size distribution to undergo self-similar growth behavior [15–17]. Normal grain growth occurs when there are no opposing energies to curvature driven growth. This description leads to a simple relationship between the grain boundary migration velocity, V, the interface mobility, M, and the driving force, $P(V)$ [6]. Alternatively, abnormal

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grain growth may occur when the coarsening of some grains is suppressed or augmented by a variety of effects, such as grain boundary grooving, precipitate drag, significant distribution of grain boundary energies and substrate interactions [15–17]. This causes the growth of a specific orientation to be favored, typically a low-energy, high-symmetry plane.

The microstructural instabilities of nanocrystalline metals have been observed through grain growth, including abnormal grain growth, at temperatures below 600 K in Au [18–20], Cu [19], Pd [21] and Ni [22]. Previous observations of abnormal grain growth have been attributed to the initial microstructure, including the distribution of grain sizes, misorientations, grain boundary energies and mobilities, triple junction mobilities, impurities, microstrains, and topological features such as curvature and the number of boundary faces [23,24]. In addition, for thin films, the formation of grooves at grain boundaries has been noted to occur and to contribute to the stagnation of grain growth when the grain size is comparable to the film thickness [25–28]. There has also been considerable theoretical attention focused on abnormal grain growth [29,30]. Most of the models propose that the initial grain size distribution is not uniform, but contains at least one large grain [31]. The models analyze possible growth mechanisms that could have created this large grain. Recently, Rios and Glicksman [24,32] found that abnormal grain growth can occur even when a uniform grain size distribution is present if grain growth is restricted by a pinning force.

While there is some understanding of how boundary properties vary, the variation in boundary properties has not yet been fully elucidated [33,34]. A number of experimental and theoretical studies in the past decade have shown that polycrystalline materials develop an anisotropic grain boundary character distribution during grain growth [35,36]. Results from experiments and simulations suggest that there is an inverse relationship between the relative energy of a grain boundary and its total area in the polycrystal. While it is difficult to measure the frequency with which boundaries are eliminated, it is possible to determine which grain boundaries are shrinking and which are growing. Holm et al. [36] found, through atomistic modeling, that the boundary structure is the dominant influence on boundary energy for materials with the same crystal structure. For some boundaries, they observed a large number of configurations that minimize to approximately the same low-energy value [36]. Low-energy boundaries, such as low coincidence site lattice (CSL) boundaries, are more likely to grow [35].

Theoretical models for the misorientation distribution function predict that the population of special grain boundaries in polycrystals ranges from 20% to 60% [37]. Such a large fraction of special boundaries is rationalized by the annealing textures and existence of local clusters of special boundaries, as discussed by Randle et al. [38]. Despite the numerous computational and experimental studies referenced above, very little of the actual grain boundary migration is understood. In large part, this is because of the difficulty in experimentally observing grain boundary migration. Typically, migration is inferred by characterizing the change in the microstructure after discrete annealing treatments. This study implements a stepwise annealing schedule so that the motion of individual grain boundaries can be tracked as the grain boundaries evolve during annealing. This allows the tracking of not only the grain size, but also the evolution of specific boundary types and the overall texture of the film as it responds to the increase in temperature.

Previous experimental studies [39,40] on grain boundary character distributions in low stacking fault energy (SFE) face-centered cubic (fcc) materials found that the distribution of CSL boundaries show a strong prevalence of $\Sigma 3^n$ (where $n = 1, 2, 3$) boundaries. As for fcc metals, there have been several computational studies [41,42] investigating the structure and energy of these and other grain boundaries. The evolution of the CSL boundary distributions has been found to be influenced by the processing conditions [43] and has been discussed in terms of the relative contributions of both the kinetic and geometric influences [44]. The main objective of this study is to experimentally assess the relative proportions of low-Σ CSL boundaries in fcc metals to elucidate the role of special boundaries in microstructural evolution.

The designed study is unique in that we are able to determine the specific orientation of each grain, as well as the misorientation across each grain boundary. To date, the nanoscale size regime of grain growth has not been systematically studied because of the limitations of scanning electron microscopy (SEM) electron backscatter diffraction (EBSD) techniques when quantifying grains that are $<$50 nm. This is because the spatial resolution of traditional EBSD scans is governed by the SEM electron optics. This work uses precession-enhanced diffraction orientation analysis in the transmission electron microscope (TEM) to generate pseudo-kinematic electron diffraction conditions, allowing grain orientations to be captured to sizes approaching 5 nm. This allows the program to probe a length scale an order of magnitude smaller to elucidate how the grain type (special boundaries, twins or high-angle boundaries) in nanocrystalline Cu and Ni contributes and evolves during grain growth.

Nanocrystalline thin films are optimal to investigate these outstanding questions because of their high grain boundary curvature and because they possess a high surface area-to-volume ratio. Normal grain growth usually ceases when the grain size becomes comparable to the smallest specimen dimension, in this case the thickness of the thin film [11]. Because of this, the grain boundary energies, along with possible grooving and surface energy effects, will dominate the growth mechanisms since some grains will grow quickly to reach a diameter that is much larger than the film thickness. Hence, the majority of the grain growth will be two-dimensional [45].

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