

Grain Boundary Evolution of Face-Centered Cubic Metals during *in-situ* TEM Annealing

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A metal's grain structure influences a range of material properties such as strength, toughness, and corrosion resistance. Hence, understanding and controlling the evolution of grain sizes is essential for materials engineering. This growth is governed by the grain boundary character, which includes curvature, energy and mobility.

A significant amount of excess enthalpy is associated with the grain boundaries of nanocrystalline materials. In order to reduce this enthalpy, microstructural transformations occur through either normal or abnormal grain growth [1]. The observations of abnormal grain growth have been attributed to the initial microstructure, including the distribution of grain sizes, misorientations, grain boundary energies and mobilities [1].

In this work, an *in situ* TEM step-wise annealing schedule up to 600°C was employed to track individual grains and grain boundaries in 40 nm thick Cu and Ni films. The grain boundary character was quantified using precession-enhanced electron diffraction orientation analysis via the NanoMEGAS ASTAR platform. This allowed the tracking of not only the grain size but also the evolution of specific boundary types and the overall texture of the film. -While it is not possible to measure the frequency with which boundaries are eliminated, it is possible to determine which grain boundaries shrunk or grew based on relative boundary fractions. This provides insight into how face-centered-cubic (FCC) metal grains grow and in particular, which boundaries may facilitate abnormal grain growth. Despite numerous computational and experimental studies [2-4], very little actual grain boundary migration is understood because of the difficulty in experimentally observing grain boundary migration.

The Cu film exhibited normal grain growth, as seen in figure 1(a). In contrast, the Ni film showed the onset of normal-to-abnormal-to-self similar grain growth, figure 1(b). The onset of abnormal grain growth accompanied an increase in the $\Sigma 3$ and $\Sigma 9$ boundaries, figure 2. The high mobility of $\Sigma 3$ caused the boundaries to migrate out of the system by moving to the surface of the film. Once these boundaries were no longer present in the film, the abnormal grain growth ceased.

A Cu-rich Cu(Ni) alloy film was sputter-deposited and *in situ* annealed in the TEM using the procedure above. The film exhibited abnormal grain growth. Using atom probe tomography, the role of grain boundary segregation is used to describe this effect in the Cu-rich film.

References:

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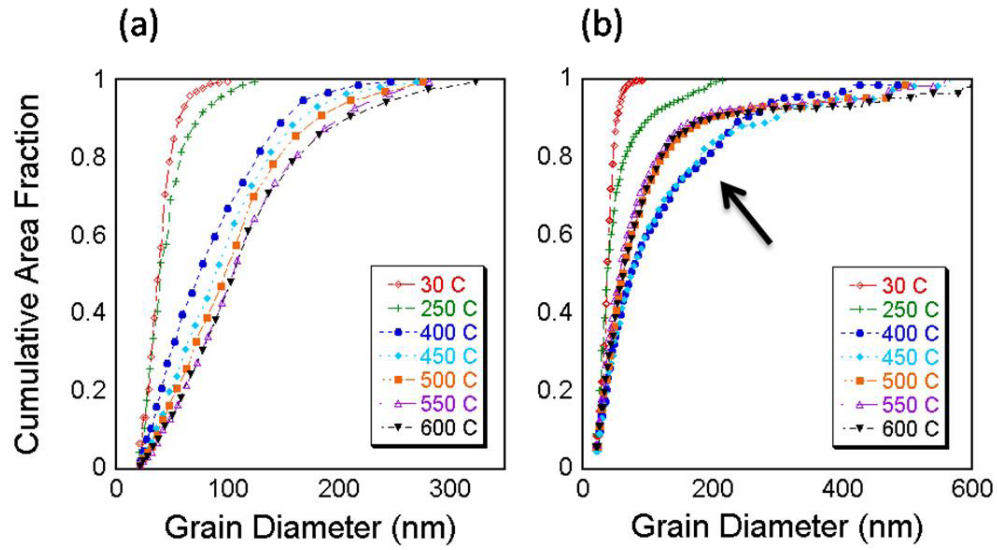


Figure 1. Cumulative distribution function grain growth plots for (a) Cu and (b) Ni. The arrow indicates the onset of abnormal grain growth

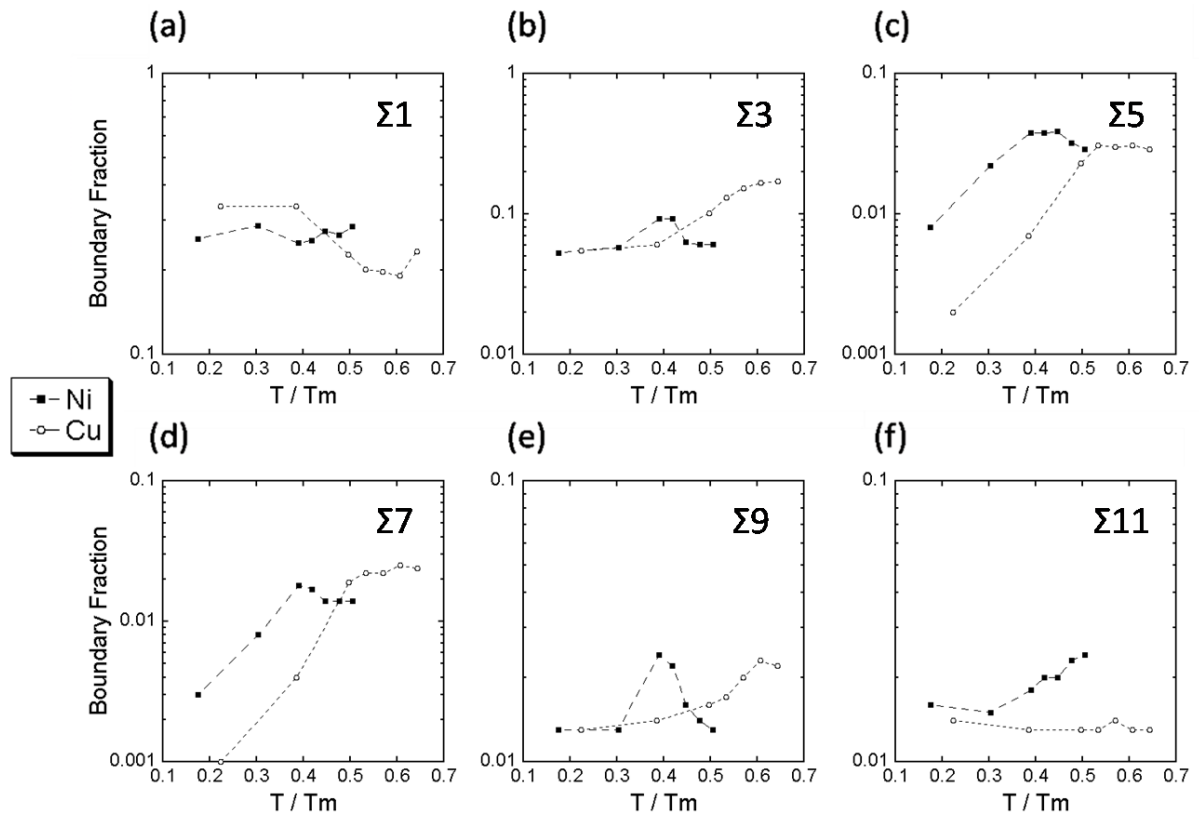


Figure 2. Coincidence site lattice boundary evolution with homologous temperature (a) $\Sigma 1$, (b) $\Sigma 3$, (c) $\Sigma 5$, (d) $\Sigma 7$, (e) $\Sigma 9$, and (f) $\Sigma 11$