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Evidence of Cryogenic Indentation-Induced Grain Growth in Highly Twinned Nanocrystalline Copper

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Grain growth is a product of a driving force and boundary mobility. These driving forces can originate from stored deformation energy, grain boundary energy, surface energy, elastic energy, or chemical and/or thermal gradients [1]. The boundary mobility is representative of the kinetics necessary to allow the system to respond to the applied driving force. A lack of sufficient mobility and/or driving force can prevent a system from reaching the microstructural configuration with the lowest energy. Since the mobility is commonly related to atomic diffusion, temperatures of approximately one-half or higher of the homologous temperature are typically required for substantial grain growth. During grain growth, if the grain size distribution remains self-similar, it is referred to as normal growth. In some cases, however, a single grain or an aggregate of grains can coarsen at a rate faster than others in the microstructure and is referred to as abnormal grain growth.

As the grain size of a material approaches the nanometer regime, the microstructure can exhibit peculiar grain growth behavior. For example, nanocrystalline metals have been shown to undergo both moderate, widespread coarsening as well as dramatic abnormal grain growth as a result of tensile deformation [2], high-cycle fatigue [3], low-cycle fatigue [4], and indentation [5] at moderate to low homologous temperatures. In fine-grained Cu, samples that are left at room temperature have been shown to display abnormal coarsening. Zhang *et al.* [5] previously reported transmission electron microscopy (TEM) studies where abnormal grain growth at room temperature occurred under *in-situ* indentation. In their report, the starting grain size was 40 nm. After indentation, certain grains achieved sizes of nearly 300 nm. Even more surprising was a series of *ex-situ* cryogenic indentations at -190°C where the grains grew up to 700 nm. At temperatures of a few hundred °C, typical boundary velocities for Cu are on the order of 10^{-9} m/s [1]; at -190°C one might expect grain boundary migration to occur only on geologic timescales. To date, the ability for grains to grow under cryogenic indentation, where temperatures are too low for reasonable mobility, is not well understood.

In this research, a series of nanocrystalline Cu films with a high density of twin boundaries ($\Sigma3$) were subjected to indentation while submerged in liquid nitrogen at 77 K. Subsequent quantification using precession-enhanced electron diffraction in the TEM determined the crystallographic texture and grain-to-grain misorientation. The grain boundary misorientations were quantified in order to understand which grain boundary types are present after cryogenic mechanically-induced grain growth.

Three distinct regions were identified, as seen in Figure 1: (i) undeformed, which preserved the parent microstructure, (ii) pile-up (the area of mass build up around the sides of the indent) and (iii) under the indent. As shown in Figure 2, the grains in the undeformed film retained a high density of $\Sigma3$ boundary twins, whereas the pile-up region showed grain coarsening, prevalent $\Sigma7$ subgrain orientations and an order of magnitude decrease in $\Sigma3$ twin boundaries. Coupling these experimental results with prior simulations [6], a detwinning mechanism is believed to be a contributing mechanism that allowed the grains to coarsen at significantly low homologous temperature, where diffusion-based mobility would be inactive.

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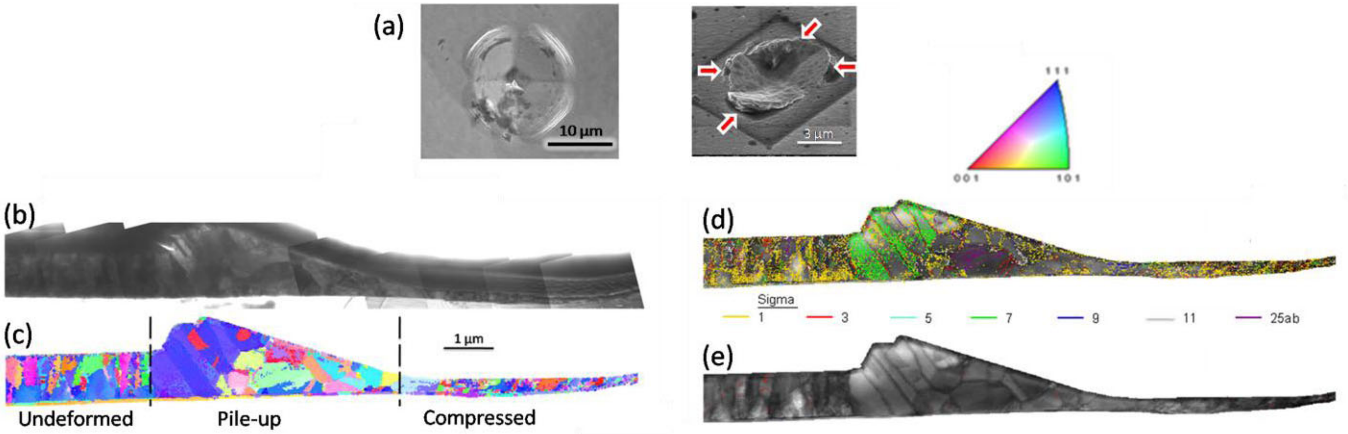

Figure 1. Indented nanotwinned Cu film in (a) SEM image of indent with arrows showing site specific foil lift-out locations (b) TEM bright field image (c) inverse pole figure orientation map (d) image quality map with CSL boundaries highlighted (e) image quality map with twin boundaries highlighted in red.

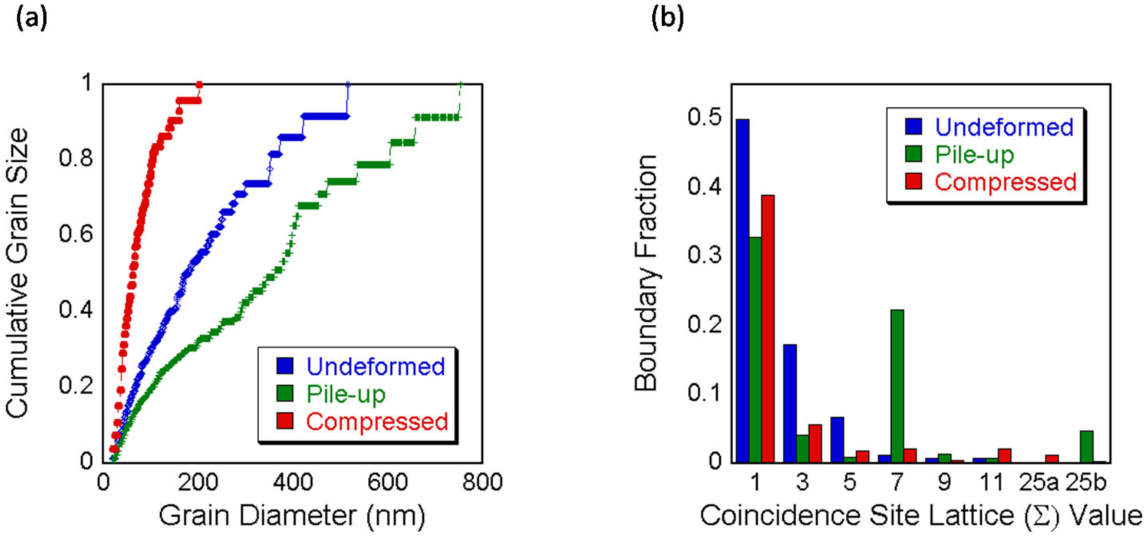

Figure 2. Region-specific response of nanotwinned Cu to mechanical indentation at cryogenic temperatures (a) grain size evolution and (b) CSL boundary evolution