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In Situ Analysis of the Fracture Behavior of Nanocrystalline Copper Using Precession-Assisted Crystal Orientation Mapping.

Paul Rottmann[1], Kevin Hemker[1]

[1.] Johns Hopkins University, Department of Materials Science and Engineering, Baltimore, MD, USA

In recent years nanocrystalline metals with average grain sizes of less than 100 nm have been introduced into a growing number of applications. Nanocrystalline materials differ greatly in many respects from their coarse-grained counterparts. They can be fabricated through vapor-phase deposition (e.g. sputtering, evaporation) or electrodeposition to produce complex parts for small-scale applications, and they are used extensively in micro electromechanical systems (MEMS) and microprocessors. Nanocrystalline materials possess many unique properties, such as fatigue limit [1], deformation mechanisms [2], and magnetic properties [3]. Understanding the physical properties of these materials is imperative if they are to be used as critical components of many applications. This study focuses on the fracture of nanocrystalline copper when subjected to tensile loading.

Freestanding copper thin films were fabricated by electron beam vapor deposition at a rate of 1.5Å/s to a final thickness of 65 nm. After being freed from the silicon wafer with an acetone lift-off procedure the films were attached to a straining grid. The as-deposited grain size was 28.7 ± 14.4 nm. Each film was annealed at 300°C under vacuum (10^{-6} Torr) for 1 hour, producing a final grain size of 62.2 ± 51.2 nm. This resulted in a microstructure with equiaxed grains containing numerous twin boundaries, and the majority of grains were larger than the thickness of the film. The films were characterized using precession-assisted crystal orientation mapping (PACOM) [4]. Utilizing PACOM is advantageous as compared to conventional TEM microscopy, because it allows for full mapping of the crystal orientation at each point. This produces high-resolution maps in which information about grain size, grain boundary character, and grain boundary morphology is readily available and quantifiable. For these samples, the experiments were conducted by straining each film using a Gatan single-tilt straining holder (Model 654) to strain the film until a crack began to propagate across the gage section. Once a crack began to move the straining was stopped and a PACOM scan taken. The scans for this study were taken with a step size of 3.6 nm with a nominal probe size of 2.2 nm. After each scan the specimen was strained further to propagate the crack by a few hundred nanometers, and a subsequent PACOM scan was taken. This process was repeated to produce maps of a given area as it is subject to strain.

Figure 1 shows results from one such experiment. An initial scan, before crack propagation, and the final scan, taken after the crack had propagated 2.4 μm through the sample are shown in Figures 1a and 1b, respectively. The color in each of these pictures represents the out-of-plane orientation at each point (see inverse pole figure legend in Figure 1), and the grayscale indicates the image quality, or confidence, in the selected orientation. The eventual crack path is indicated by a dashed line in Figure 1a. As the film was strained, a number of changes in the local microstructure were observed around the crack tip. Figure 1c shows the evolution of a large grain (outlined by a white box in Figure 1a) as the crack front proceeds. Initially, the grain has a single orientation with a twin toward the right side of the grain. As the crack begins to propagate through the grain, new twin boundaries are formed (marked by dashed lines in Figures 1b and 1c) in the grain. The crack precedes transgranularly across this large grain, not following any of the twin boundaries. By analyzing the crack path, it was determined that the crack primarily propagated intergranularly, along high-angle grain boundaries, (Figure 1d) with the notable exception that Σ3 twin boundaries were never found in the crack path even though Σ3 boundaries comprised 15.3 percent of all boundaries. This indicates that Σ3 boundaries in nanocrystalline copper are more resistant to fracture than other grain boundaries with random misorientations.

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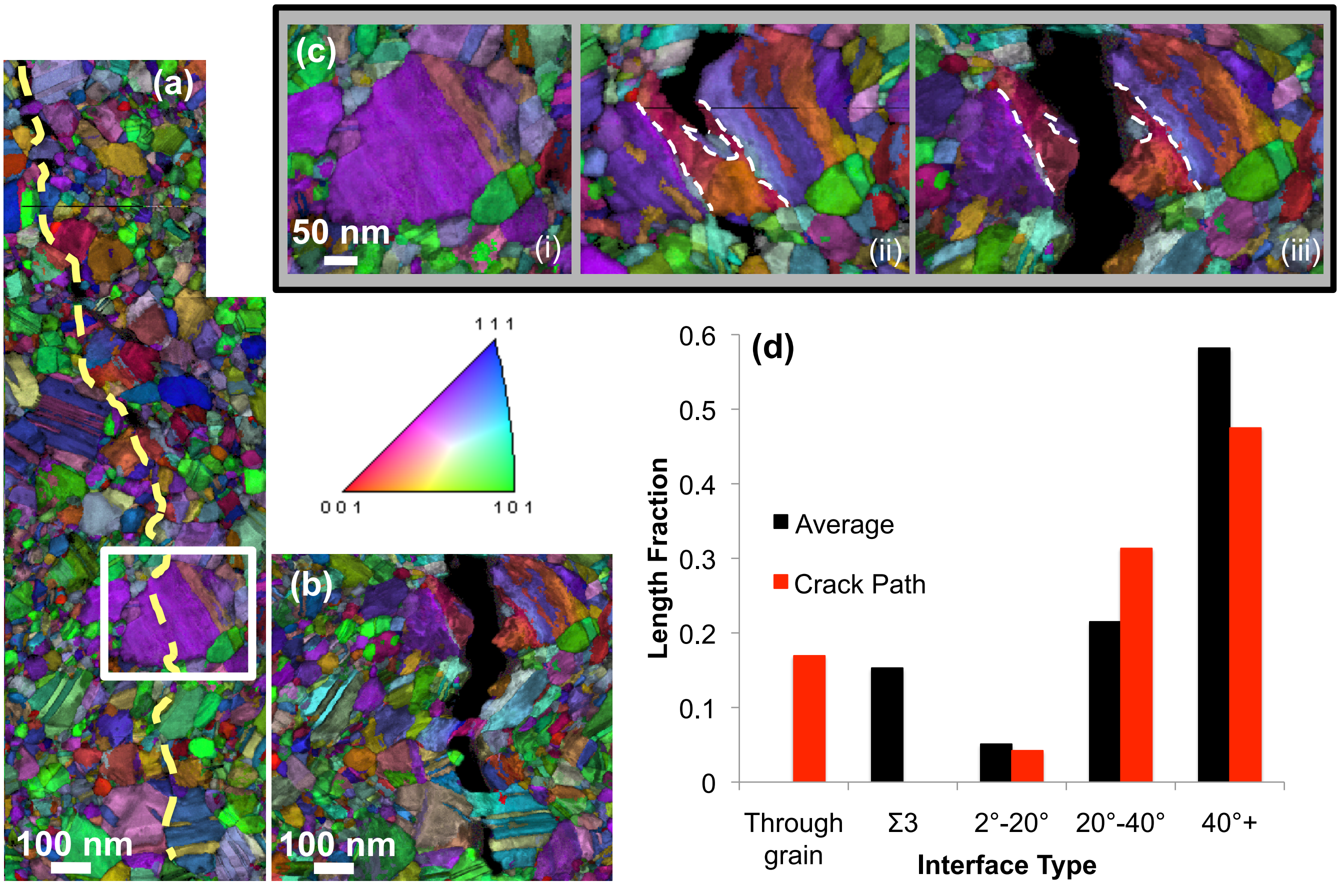

Figure 1. Orientation map of specimen area prior to (a) and after (b) crack propagation. Color corresponds to crystal orientation and grayscale indicates image quality. Eventual crack path is marked by a dashed line. (c) Magnified images of a grain as the crack propagates through it. Specimen is further strained from images (i) – (iii). Twin boundaries nucleated during straining are marked with a dashed line. (d) Graph comparing the average grain boundary misorientation of the specimen to the path followed by the crack.