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Investigating Stress-Assisted Grain Growth in Nanocrystalline Materials Using in-situ Transmission Kikuchi Diffraction

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Observations of strained nanocrystalline materials have shown that discontinuous grain growth can occur at room temperature with as little as 2% strain [1]. Post-deformation analyses using transmission electron microscopy (TEM) effectively demonstrate that certain grains grow significantly, causing a transition from nanocrystalline mechanisms to microcrystalline ductility. This process has been called "stress-assisted grain growth" and has been explained by the preferential migration of stress-coupled grain boundaries. Additional studies have also proposed that the formation of nanotwins can result in the disassociation of boundaries into new segments, which in turn can become more mobile resulting in discontinuous grain growth [2]. Understanding the nanocrystalline processes that alter material properties at room temperature is vitally important and has, until now, required detailed TEM observations of the resulting microstructure and indirect inferences regarding the dominant mechanisms.

The recent development of transmission Kikuchi diffraction (TKD) in the scanning electron microscope (SEM) has opened up the field of nanomaterials characterization to a much wider audience [3, 4]. The advantages in resolution over conventional electron backscatter diffraction have resulted in a wide range of applications of TKD across the materials and Earth sciences [5]. However, to date TKD has not been applied to the study of dynamic processes, utilizing in-situ deformation or heating stages. In this paper, for the first time, we present results from a prototype in-situ tensile deformation stage that has been custom modified for use with TKD, enabling the investigation of discontinuous processes on the nanoscale, such as stress-assisted grain growth.

Nanocrystalline copper and aluminum samples were deposited with a range of grain sizes and thicknesses, and these were then separated from their substrate and floated onto TEM-compatible push-to-pull (PTP) holders (Hysitron Inc., USA). A focused ion beam SEM was used to shape the film into a suitable dog-bone configuration across the PTP holder, so as to enable in-situ analysis in transmission. The sample and PTP holder were then mounted on a custom-modified Hysitron PI-85L in-situ nanoindentor, designed for tensile testing of thin film samples with simultaneous TKD mapping. The experiments were carried out using a Carl Zeiss Ultra Plus field emission gun SEM equipped with an Oxford Instruments AZtec EBSD and TKD system. The tensile tests were carried out using standard TKD conditions (typically 30 kV accelerating voltage, 5-10 nA beam current, step sizes ~ 5 nm and mapping speeds ~ 66 Hz), with loading up to ~ 500 uN. In all cases in-chamber plasma cleaning was essential in order to enable multiple scans of the same area of the sample throughout the experiment without prohibitive contamination. In the example presented here, a nanocrystalline Cu film with a bimodal grain size and a thickness of 55-75 nm was deformed up to failure (at 450 uN) and mapped 8 times using TKD at different loads during the experiment.

The results show the potential of this in-situ approach for the characterisation of nanostructural changes throughout a deformation experiment using TKD. In this example we show how the TKD mapping was able to track the (relative lack of) changes in the grain structure: the eventual failure (and probable region of significant deformation) occurred off to the side of the observed region. Figure 1 shows a series of TKD maps collected during the experiment: each map took approximately 50 minutes to acquire, and the sample remained loaded throughout. The system was sufficiently stable to enable accurate quantification of grain-scale changes, and we will show further examples in which nanoscale changes are effectively captured using this approach.

We believe that this in-situ TKD technique can become an effective tool for understanding the complex nature of discontinuous stress-assisted grain growth, allowing us to identify the exact nature of the grains and boundaries that are most affected by this mechanism. This can lead to further breakthroughs in our understanding of the response of nanomaterials to deformation at room temperature.

References:

[1] D.S. Gianola et al., Acta Materialia **54** (2006) p. 2253–2263.
[2] X.-M. Luo et al., Nature Communications 5:3021 (2014)
[3] R.R. Keller and R.H. Geiss, J. Microscopy, **245** (2012), p. 245-251.
[4] P.W. Trimby, Ultramicroscopy, **120** (2012), p. 16-24.
[5] G. Sneddon et al., Materials Science and Engineering R: Reports, **110** (2016), p. 1-12.
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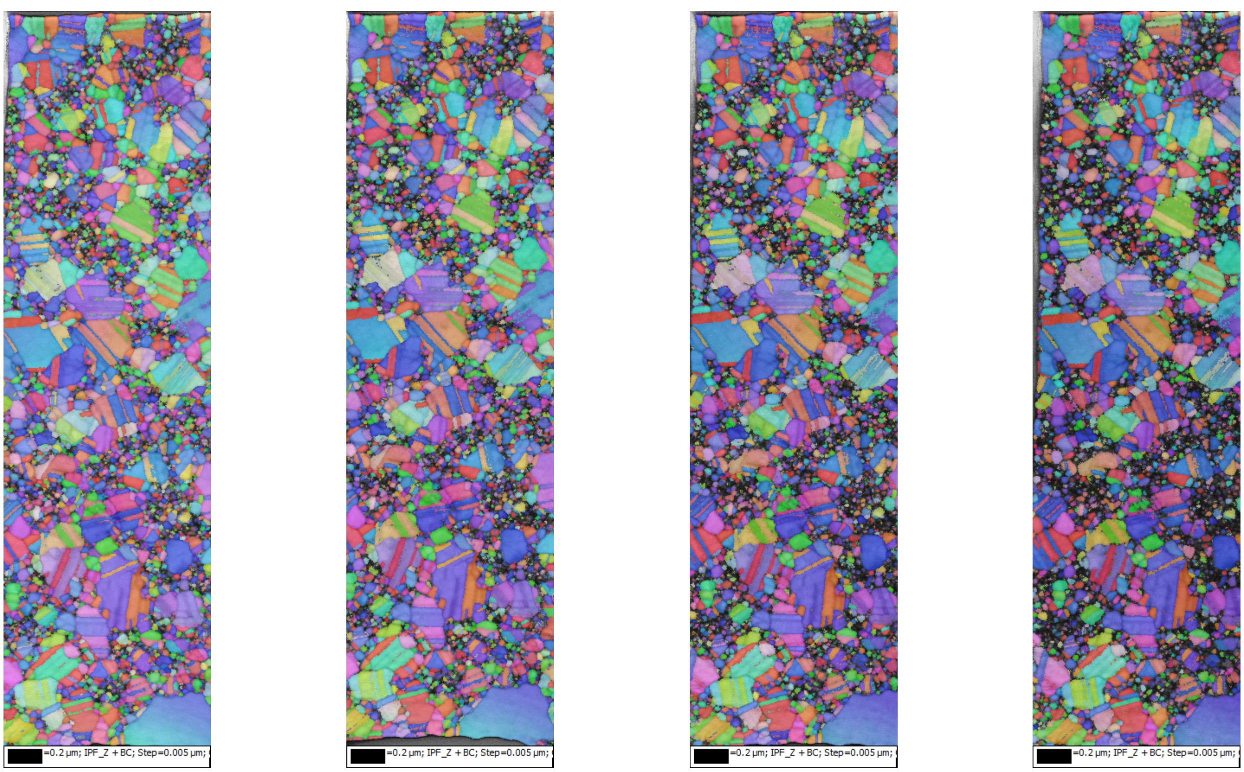

Before experiment | 25 uN load | 100 uN load | 200 uN load

Figure 1. TKD orientation maps (inverse pole figure color scheme superimposed on pattern quality) of a nanocrystalline Cu film at different stages of a tensile deformation experiment. Scale bars mark 200 nm

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