Orientation mapping of nanoscale deformation processes using transmission Kikuchi diffraction

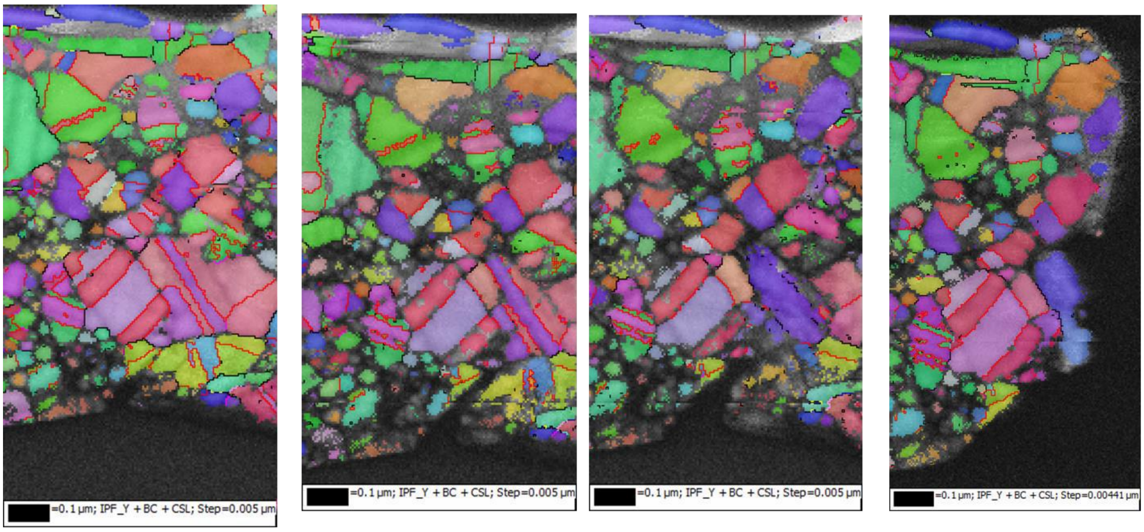
Sneddon, G.[1]

[1] University of Sydney, Australia

Nanostructured materials often behave differently from their bulk counterparts, with different deformation mechanisms becoming available or dominant. Post deformation transmission electron microscopy studies have demonstrated that some nanocrystalline grains can grow significantly under load, termed "stress-assisted grain growth" [1]. However, orientation mapping techniques (such as transmission Kikuchi diffraction (TKD)) conducted in situ during the deformation, can provide more insight into the deformation mechanisms involved and their relationship to the types of grains and grain boundaries involved. TKD is a relatively new technique in the scanning electron microscope (SEM) for the characterisation of nanoscale materials, with the improvement in resolution over conventional electron backscatter diffraction allowing nanocrystalline materials to be more readily characterised in the SEM. It has already been used for a wide range of materials [2] although to date, no other groups have utilised it for dynamic processes, such as in-situ deformation.

Several different materials were tested, including nanocrystalline copper and surface mechanical attrition treated (SMAT) stainless steels, with different preparation methods use for different sample types. Nanocrystalline copper films were deposited on PMMA coated silicon wafers, separated from the substrate and floated onto a push-to-pull (PtP) device (Hysitron Inc., USA). A focussed ion beam (FIB) SEM was used remove material to leave a dog-bone shaped specimen across the testing of the PtP device. The stainless steel specimens were lifted out from the surface of the bulk material and secured on each side of the PtP device by using ion-beam assisted deposition of platinum, followed by further specimen thinning to obtain a suitable sample thickness for TKD. In each case, the specimen and PtP device 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. Standard TKD conditions (30 keV accelerating voltage, 5-20 nA beam current and step size 5 nm) were used for the tensile tests, testing the specimens until failure with TKD scans taken at intermediate steps. In chamber plasma cleaning was used in all cases to ensure multiple scans of the same area could be taken without excessive contamination.

The example provided shows a nanocrystalline Cu film with a bimodal grain size and 75 nm thickness deformed to failure, with TKD maps at four different load steps shown. The clearest change is the detwinning ahead of the crack tip, although some grain rotation and grain growth were also observed. These results show the potential of TKD, when coupled with in situ deformation experiments, to follow changes in the grain structure during the experiment. This should enable us to identify the types of grains and grain boundaries that are most likely to change and, potentially, to link this to the deformation mechanisms.



0 uN   60 uN   112 uN   115 uN

Figure 1 - TKD maps of a single nanocrystalline copper specimen at different loads.

[1] D.S. Gianola et al., Acta Materialia 54 (2006) p. 2253 - 2263.

[2] G. Sneddon et al., Materials Science and Engineering R: Reports, 110 (2016), p.1-12.