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Coupling Quantitative Dislocation Analysis with In Situ Loading Techniques: New Insight into Deformation Mechanisms

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The vast majority of our understanding about the deformation mechanisms in nanocrystalline materials is limited to information gained from experimental and theoretical characterization of FCC materials. Related behavior in nanocrystalline BCC materials is not as frequently studied, and thus outstanding questions remain regarding deformation regimes and Hall-Petch trends. Through the use of coupled in-situ TEM tensile testing and quantitative dislocation density analysis via precession electron diffraction, a study of deformation in nanocrystalline iron films was performed.

Free-standing nanocrystalline iron films were analyzed using *in situ* TEM straining. The films were prepared by either sputter deposition [1] or pulsed laser deposition onto 1cm x 1cm <100> NaCl substrates using a 99.9% pure iron target. *In situ* tensile experiments were performed using a Gatan heating-straining holder (Gatan, Inc). Each specimen was pulled in tension at low magnification (50x) in bright field TEM until a crack initiated, and at higher magnification (30kx) afterward. Images were captured using a Gatan on-axis CCD camera operating with an exposure time of 0.05 seconds. Automated crystallographic orientation maps were acquired intermittently during the TEM tensile test using a NanoMEGAS DigiSTAR™ precession unit and ASTAR™ analysis software [2]. The data collected by the NanoMEGAS system was used to measure grain orientation information and to generate quantitative dislocation density values based on the Nye tensor dislocation theory [3]. This method supplies a lower-bound estimate of the density of dislocations required to accommodate a measured curvature in the lattice.

Deformation at the crack tip was accommodated by dislocation motion, grain rotation, and grain growth, however twinning was not observed. Interestingly, the concurrent nature of the grain rotation and dislocation motion indicates that grain rotation occurs at fairly large grain sizes and there is no sharp transition from dislocation-mediated to grain boundary sliding mechanisms as grain size is decreased in BCC iron. These results have implications for understanding deformation modes in nanocrystalline materials at the dislocation level, and in particular, give new and additional insight into the deformation of nanocrystalline metals [4].

References:

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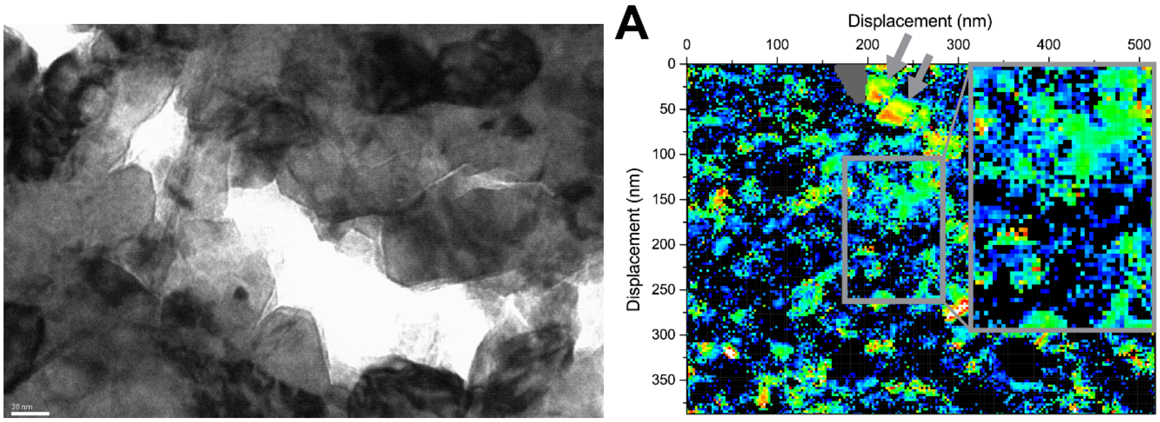

Figure 1. (Left) Crack propagation in nanocrystalline iron during in situ TEM tensile testing; (Right) Dislocation density map generated from precession electron diffraction data.

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