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103 – A Comparative Investigation Between Transmission Kikuchi Diffraction (TKD) and Precession Electron Diffraction (PED)

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Transmission Kikuchi Diffraction (TKD) in the SEM [1] and Precession Electron Diffraction (PED) in the TEM [2] are methods used to achieve higher spatial resolution for grain misorientation and grain size mapping as well as grain texture quantification. Using equivalent samples, a comparison of these techniques to these types of grain characteristic measurements has been undertaken.

In a nanocrystalline Cu specimen, the TKD analysis indexed more grains with a higher confidence than PED, Figure 1(a). The origin of the difference is explain by how the diffraction patterns are captured. In TKD, the diffraction events are derived from within a few to ten nanometers of the exiting surface. In this case, the diffraction is then limited (typically) from a single grain that satisfies the Kikuchi diffraction event if other scattering events may have occurred above the exiting grain. In PED, the diffraction events are an integration through the entirety of the foil, thus the collected patterns can be a convolution if more than one grain's diffraction pattern overlaps. Nonetheless, when the grains did not overlap, PED's spatial resolution for mapping the smallest grains was more evident as compared to TKD.

Though both methods could index twin structures in the Cu foil, PED appears to be more sensitive to identifying twin structures via its template-matching algorithm than equivalent twins captured by TKD, as shown by the circled regions with the solid arrows in Figure 1(a). This could be a result of the inclination plane of the twin coupled with shared bands between the twin and parent yielding incorrect indexing in the TKD condition. Interestingly, when twins were close to a {001} zone axis of a grain, PED did not reveal the twin but was observed for TKD, *i.e.* the dashed arrow in the boxed region in Figure 1(a).

A comparison of the pattern collection as a function of foil thickness was also undertaken. TKD was able to index diffraction patterns in foils up to ~ 350 nm in thickness whereas PED pattern recognition was limited to ~ 140 nm for the Cu foil, Figure 1(b). Though the TKD scans were done at 20 keV and the PED at 200 keV, suggesting a peculiar contradiction in that higher keV would enable deeper electron penetration, the difference in pattern detection as a function of foil thickness is explained again in the origins of the diffraction events themselves. TKD patterns originate from inelastic scattered electrons that satisfy the Bragg condition for electrons exiting the bottom surface of the foil. If these electrons are able to escape the foil in this diffraction condition to yield a sufficient detected intensity, they will provide a TDK pattern. In contrast, PED patterns originate from pseudo-kinematical scattered electrons. If these electrons undergo multiple scattering events with continual penetration into the volume of the material, their ability to satisfy the Bragg condition is lost and no pattern is detected.

In summary, both TKD and PED provide complementary means of quantifying nanoscale grain characteristics. Rather than being competitive techniques to each other, each method has particular strengths and weaknesses dependent on the sample type, foil thickness, and spatial resolution needed.



Figure 1. Figure 1: (a) TKD and PED map of a nanocrystalline Cu foil. The circled and solid arrows identify regions where PED revealed a higher spatial detection of twins whereas the boxed and dashed arrow identifies a region where TKD identified a twin that is absent in the PED scan. Each scan was done at a 5 nm step size. (b) A profile of collected Cu grains from the same region between TKD and PED as a function of foil thickness (measured by EELS). Note the further detection of the patterns for the TKD for thicker foils.

References

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