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D-STEM Combined with Precession Microscopy for Nanoscale Crystal Orientation and Phase Mapping

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An electron diffraction technique called Diffraction Scanning Transmission Electron Microscopy (D-STEM) has been developed in a TEM/STEM instrument to obtain spot electron diffraction patterns from nanostructures, as small as ~3 nm. The electron ray path enables the formation of a 1-2 nm near-parallel probe, which is used to obtain bright-field/ dark-field STEM images (Fig.1). The beam can be controlled and accurately positioned on the STEM image, while sharp spot diffraction patterns can be recorded on the CCD camera (Fig.2). The details of the technique are mentioned elsewhere [1]. When integrated with Digistar^{TM} NanoMEGAS, the D-STEM technique is very powerful for automated crystal orientation and phase mapping.

However, for grains oriented off-axis with respect to the beam direction and regions with a high density of defects, the D-STEM technique alone is still limited, thereby affecting the collection of sufficient data for statistical analysis. Here, we have overcome this limitation by combining, for the first time, D-STEM with the ASTAR^{TM} precession system from NanoMEGAS. The combination of these two techniques allows us to: (i) obtain crystal orientation from off-axis grains, even when located in regions with a high density of defects, because of the resulting near-kinematical diffraction patterns, and (ii) achieve rapid (at least ten times faster than the original D-STEM technique) collection of grain orientation and phase mapping from nanoscale/sub-micron grains.

Figs 3-4 show the application of this technique. In Fig. 3, near-kinematical diffraction patterns have been used to generate an orientation map from multiple copper interconnect 120 nm lines which have been overlaid on a reconstructed bright field image. The copper grains do not exhibit the (111) orientation typically observed in wider lines. Fig. 4 shows a reconstructed image containing phase and reliability information obtained by the D-STEM type lens configuration and precession electron microscopy of a nanocrystalline nickel film. Both *hcp* and *fcc* phase grains are visible in the pulsed laser deposited film. A careful analysis shows that the Ni film contains 6% *hcp* phase.

References

[1] K.J. Ganesh et al, Microsc. Microanal. 16(5) 2010, 614.

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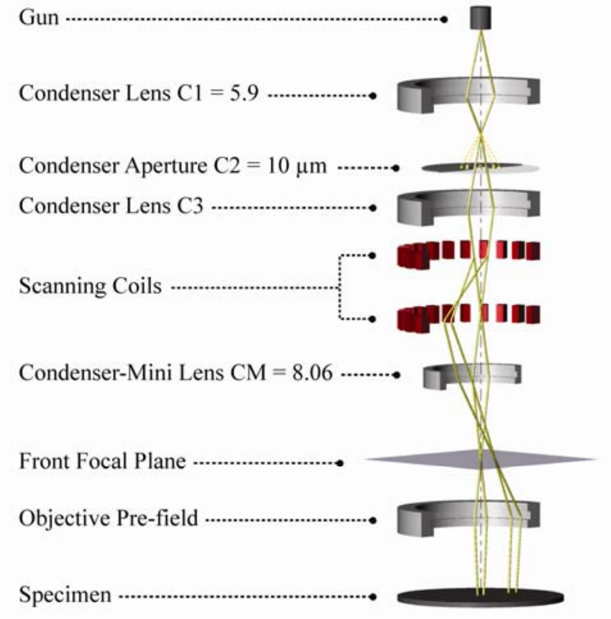

Figure 1: Schematic representation of ray-paths in the D-STEM configuration. The dotted lines depict the rays blocked from entering C3 by the condenser aperture (C2). The strength of the lens is measured in V. Consider the source size to be larger than the illuminated area on the sample [1].

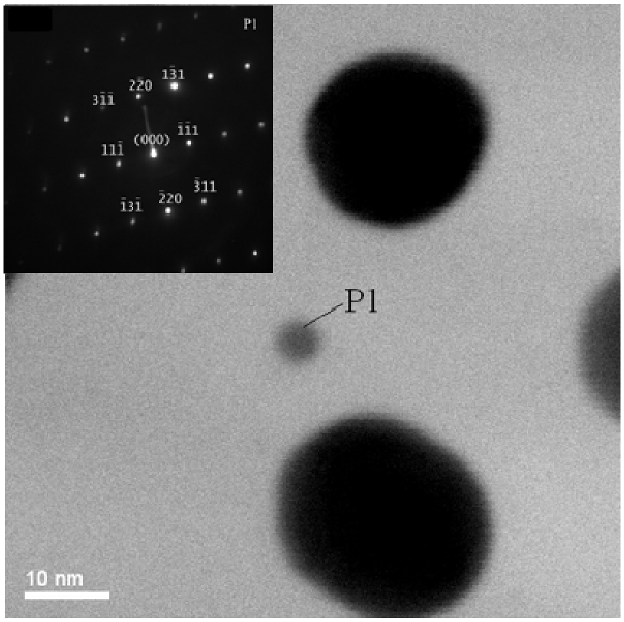

Figure 2: Bright-field STEM image of silver nanoparticles. Inset shows the diffraction pattern acquired by D-STEM of the nanoparticle P1 (~ 4 nm in size) along the [112] beam direction [1].

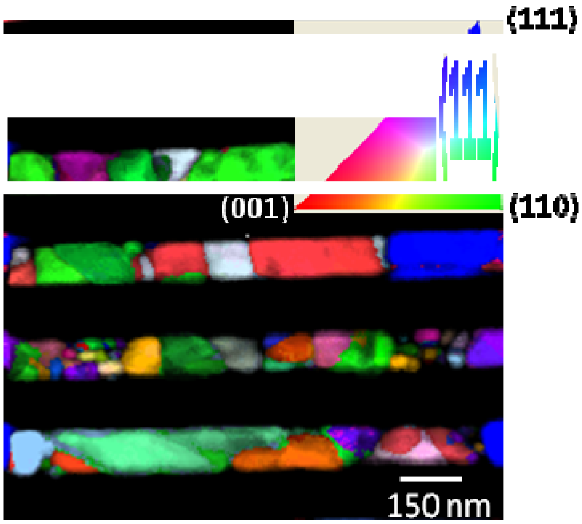

Figure 3: Orientation map of copper interconnect lines obtained using D-STEM and the precession system ASTAR. The inset shows color codes for orientations.

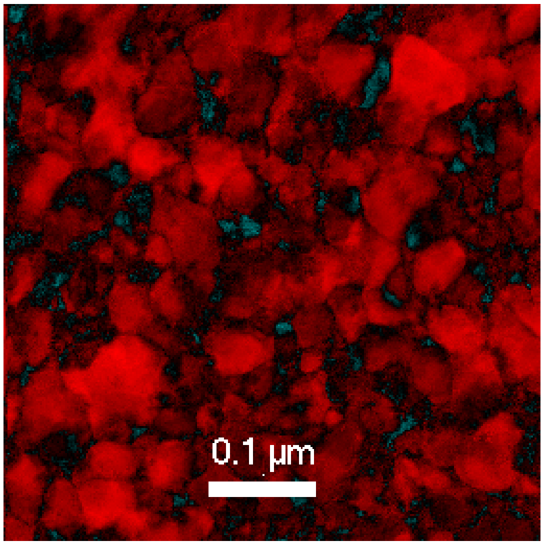

Figure 4: Combined phase and reliability map from a 100 nm thick Ni nanocrystalline film, deposited via pulsed laser deposition, showing both *hcp* (blue) and *fcc* (red) phase grains.