New Method of Electron Diffraction for Characterization of Nanomaterials using the Scanning Electron Microscope

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ABSTRACT

A new method is described for obtaining and analyzing the crystallographic structure and orientation of nanoparticles and ultrathin films using conventional electron backscatter diffraction (EBSD) equipment in the scanning electron microscope (SEM). Electron-transparent samples of nanoparticles, thin films or nanowires, which can be suspended on transparent carbon film substrates, were positioned immediately below the pole piece of the SEM by use of a custom made holder. This allowed electron diffraction patterns to be captured in transmission from nanometer-scale areas on the samples using a conventional EBSD camera. Such an arrangement obviates the need for a transmission electron microscope (TEM). The resulting diffraction patterns were displayed with EBSD software on a computer screen. In this fashion, we obtained diffraction patterns from nanoparticles as small as 20 nm in diameter and from nanowires with widths as small as 30 nm, as shown in figure 1. Patterns were also obtained from free-standing thin film metal samples sandwiched in copper grids and from thin metal samples deposited on silicon nitride window substrates. When coupled with an energy dispersive spectrometer (EDS) for elemental analysis, this diffraction technique provides a tool for phase ID of nanometer-sized particles. In thin film samples, orientation mapping was facilitated by scanning the electron beam in the same manner that is used in reflection EBSD. With this mode of transmitted electron diffraction we observed improvements in pattern contrast, signal to noise ratio, and lateral spatial resolution compared to the same factors observed with conventional EBSD. These improvements are thought to be due in part to a strongly reduced contribution from diffuse scattering and can be qualitatively explained using Monte Carlo scattering simulations.

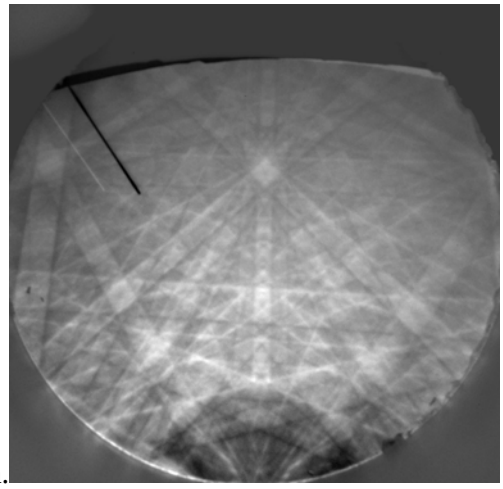
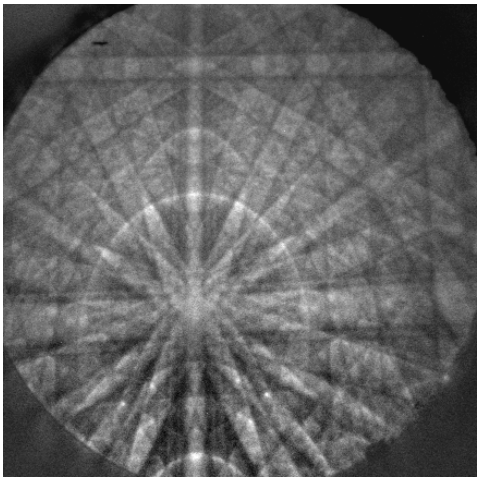
a.  b. 

FIGURE 1. Electron diffraction patterns taken in transmission using a standard EBSD camera in an SEM. Figure 1a is from a 20 nm diameter particle of Al_2O_3 and figure 1b is from a 30 nm diameter GaN nanowire. Both patterns were taken at 20 kV.