High Throughput Automated Crystal Orientation and Phase Mapping of Nanoparticles from HREM - TEM Images

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As the need for research on novel nanostructured material increases, reliable characterization of their structures, sizes, and orientations is becoming very important. Transmission electron microscopy (TEM) is an ideal technique to characterize such nanoparticles through electron diffraction (ED) and high resolution imaging (HREM). However, TEM is still a technique where HREM images and equivalent ED diffraction patterns must be studied individually for every nanocrystal in order to study its structure and orientation.

An automated technique for crystal phase and orientation mapping of polycrystalline materials in a TEM has recently been developed [1]. The proposed tool (ASTAR) works as follows: ED spot patterns are collected with a CCD camera (speed 175 frames/sec) while the sample area of interest is scanned by the electron beam; local nanoparticle crystal orientations and/or phases are revealed by template matching (via image cross-correlation) of obtained experimental electron diffraction spot patterns with pre-calculated simulated templates. Fewer than 2000 simulated templates are enough to obtain an 1° angular resolution while orientation maps can be obtained with record speed of a few minutes (Fig. 1). Those phase/orientation maps in TEM are equivalent to EBSD (electron backscattering diffraction) maps obtained with Scanning Electron Microscopes (SEMs) with generally much lower resolution and speed.

This paper illustrates the application extension of this technique to rapidly characterize HREM images. Instead of generating ED patterns by beam scanning as in [1], selected FFT patterns from pre-selected areas of HREM images are used. In the example shown in Fig. 2, an HREM image containing PbSe nanoparticles in various orientations has been obtained with an FEI Titan 300 kV Cs corrected (0.07 nm resolution) HRTEM [2]. FFT from several particle locations have been compared with simulated PbSe templates in all possible orientations via cross-correlation and experimental crystal orientations have been obtained. We may anticipate that the ASTAR technique can be effectively used as a very useful and rapid phase/orientation fingerprinting technique for nanocrystals.

References

- [1] E. Rauch, M. Veron, J. Portillo, D. Bultreys, Y. Maniette, and S. Nicolopoulos, *Microscopy and Microanalysis*, November 8, 2008, (European Edition)
- [2] HREM courtesy Dr. M. Cheney (SiMaP Grenoble, Dr. Odille Robbe-UTS Lille, France)

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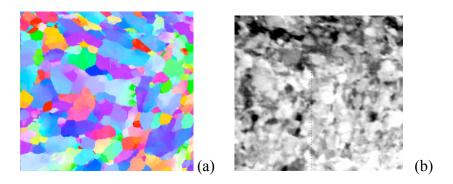


Fig. 1: ASTAR TEM orientation map of hyper-deformed copper (e=0.8). Map area obtained with a scanning matrix of 400 x 300 with 14 nm step (a), same area in bright field map (b).

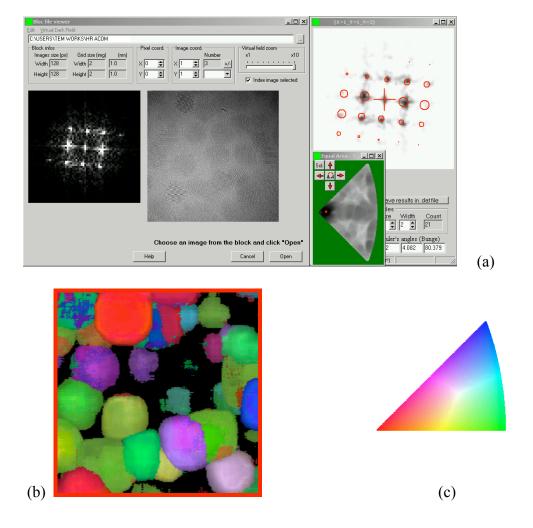


Fig. 2: (a) HREM image of various PbTe nanoparticles (middle) at various orientations and example of FFT with overlaid best fit from simulated template (right) and corresponding correlation map for that orientation (lower row), (b) orientation map of PbTe nanoparticles, with colour code as given in the (c) stereographic triangle.