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Automated Crystal Orientation and Phase Mapping of Iron Oxide Nano-Crystals in a Transmission Electron Microscope

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The development of novel materials for micro- and nano-electronics requires reliable characterizations of structures, sizes, and orientations of thin polycrystalline films and ensembles of nanocrystals. The electron backscatter diffraction technique (EBSD) in a scanning electron microscope (SEM) [1] is often employed for structural characterization of this kind of materials. EBSD in SEM is based on backscattered Kikuchi lines and, therefore, highly sensitive to the plastic deformation state of the crystals as well as to structural damage or contamination of the crystal surfaces.

Electron diffraction in a transmission electron microscope (TEM), on the other hand, delivers a significantly higher spatial resolution as compared to EBSD in a SEM and is also less sensitive to the plastic deformation state and the surface of nanocrystals. In short, crystal orientation and structure mapping by this technique is intrinsically more reliable than by its EBSD/SEM counterpart in cases of small crystallite sizes and foil thicknesses.

An automated technique for the crystal phase and orientation mapping of polycrystalline materials in a TEM has recently been developed [2,3]. This technique is based on template matching of experimental electron diffraction spot patterns to their pre-calculated theoretical counterparts. Very promising results have so far been obtained with this technique for precipitates in heavily deformed austenitic stainless steels. It has also been demonstrated that precession [4] of the primary electron beam around the optical axis of the microscope during the recording of the diffraction patterns improves the reliability of this technique significantly [5]. This is because more reflections are excited in precession electron diffraction spot patterns [5]. For nanocrystal sizes of below about 50 nm, the intensities of these reflections are nearly kinematical (i.e. proportional to the square of the structure factors) [6]. Such precession electron diffraction patterns are, therefore, very useful for structural fingerprinting of nanocrystals in a TEM [7].

This paper illustrates the application of this technique to a mixture of iron oxide nano-crystals of magnetite (Fe_3O_4, Fd3m, a = 0.832 nm) and maghemite (gamma-Fe_2O_3, $P4_132$, a = 0.833 nm). While the crystal orientation and phase mapping was performed at a JEOL 3010 TEM equipped with NanoMEGAS' ASTAR system in Grenoble [5], complementary high-resolution phase contrast imaging of individual nanocrystals in major zone axis orientations of this mixture was performed at an FEI Tecnai G^2 F20 ST TEM at Portland State University (PSU). Both microscopes are equipped with a "Spinning Star" electron precession add-on from the NanoMEGAS company. (PSU serves as the first demonstration site of this Belgium based company in the Americas and the "Spinning Star" can be demonstrated there on request).

The procedure of orientation and crystal phase mapping comprises the automated collection of electron diffraction patterns on an external digital camera while scanning the area of interest with a nanometer-sized electron beam, followed by automatic off-line data processing [2,3]. Typical results are shown in Figure 1. Thousands of electron diffraction patterns have been recorded and analyzed automatically in order to produce the crystal orientation map of the magnetite phase on the right hand side of Figure 1.

The spatial resolution of the orientation and crystal phase maps is variable and depends on the primary electron beam step size. In the particular case of this study, the step size was set to 10 nm and the primary electron beam had a diameter of approximately 20 nm. Technical details of the mapping technique are described elsewhere [2,3,5]. This technique complements high-resolution TEM-image based structural fingerprinting [6,7] and structural electron crystallography because the orientation maps can be used advantageously for the selection of individual nanocrystals that are oriented close to major zone axes [8].

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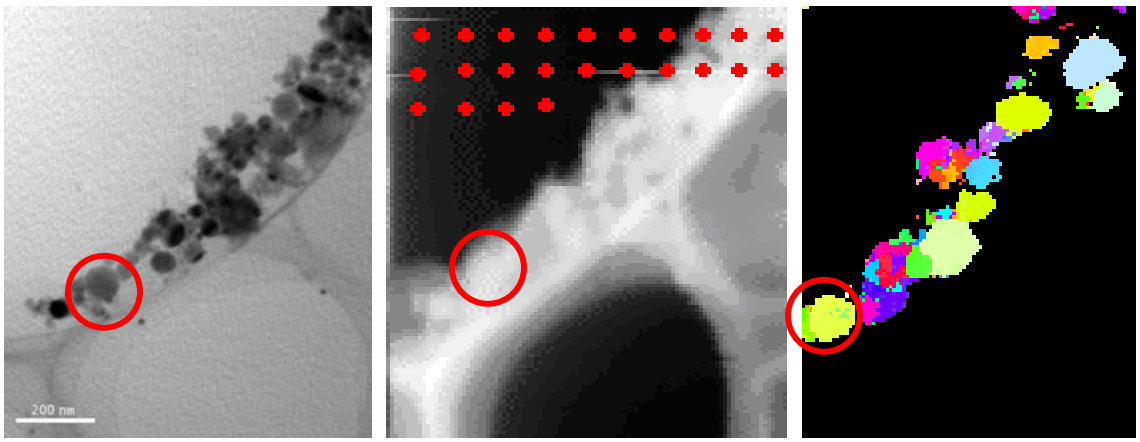

Figure 1: Iron oxide nano-crystals in a transmission electron microscope: (**left**) bright field image, (**middle**) virtual bright map which represents the intensity fluctuations of the primary electron beam in diffraction patterns (Note: The red dots illustrate the mapping procedure showing the positions where diffraction patterns were automatically recorded), and (**right**) crystal orientation map.

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