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Electron Diffraction Based Tilt Angle Measurements in Electron Tomography

Misa Hayashida[1], Marek Malac[1,2], Mike Bergen[1] Peng Li[1], Ray Egerton[1,2]

1. National Institute of Advanced Industrial Science and Technology (AIST), 1-1-1, Higashi, Tsukuba, Ibaraki 305-8565, Japan
2. National Institute of Nanotechnology, 11421 Saskatchewan Drive, Edmonton, Canada
3. Department of Physics, University of Alberta, T6G 2E1, Edmonton, Canada

Electron tomography can provide three dimensional (3D) visualization of nanoscale objects in a transmission electron microscope (TEM) from a tilt series of projected images. The accuracy of the 3D reconstruction depends on quality of input parameters, including accurate alignment in real space and accuracy of tilt and azimuth angle measurements. Here we present an electron diffraction-based in-situ method that allows accurate measurement of the relative tilt (α) and azimuth (β) angles.

Experimental data were collected in a Hitachi HF 3300 TEM / STEM instrument with a cold field emission gun operated at 300 kV. The rod-shaped samples that allow collection of full ±90° tilt range were prepared in a Hitachi NB 5000 dual beam instrument [1]. The sample used for the experiment comprised a few-layer thick ordered array of silver nanoparticles with sub-5 nm diameter deposited on Si <001>. On amorphous carbon deposited on the top of the sample, some fiducial markers were fabricated by electron beam induced deposition for accurate alignment [2]. The accurate measurement of α and β relies on recording a diffraction pattern (DP), in addition to an image, at each tilt α, which is easily achieved using a computer instrument control known as Maestro [3]. The data were collected in 3° increments. Figure 1 schematically shows the experimental set up.

Individual DPs, collected at each α, were aligned over the entire tilt range so that diffraction pattern features, such as Kikuchi lines, are continuous throughout the tilt range, as shown in Figure 2. The relative shift between subsequent DPs is equivalent to change in relative angle between the incident beam and the sample. To facilitate measurement of the each shift, the individual DPs were converted to sinogram by Radon transform. The presence of Kikuchi lines in DPs results in bright peaks in sinograms. The angle of the Kikuchi lines and their distance from center of the DPs appear as the *x* and *y* value respectively of the corresponding peak in the sinogram. Using the angle and distance of Kikuchi lines, the shift among individual DPs was calculated and the DPs over the entire tilt series can be stitched together as shown in Figure 2.

Low-index zone axis, for example [010] and [0-11], were used as in-situ calibration of the tilt per pixel. The known 90° tilt between [010] and [0-11] poles on stitched DP divided by their distance in pixels gives the °/pix calibration. In the example 512x512 pix DPs with large field of view shown here, each pixel is about ~0.04°. The displacement between individual DPs is then converted to tilt (α) and azimuth (β) relative (for example) to the first DP. Since the projected images and DPs at each tilt were collected without change of the sample stage tilt, the measurement provides values of α and β at which an image was collected.

Figure 3 shows the differences between tilt angles displayed by the TEM hardware and measured α. For comparison, the differences between displayed tilt angle and calculated tilt angles by using markers' position are also shown. The main reason of difference between the marker and diffraction methods is likely to be sample drift, to which the marker method is sensitive but the diffraction method is not.

References:
[1] T. Yaguchi et. al. Ultramicroscopy 108 (2008) 1603.
[2] M. Hayashida et. al, (Submitted to Ultramicroscopy)
[3] M. Bergen et. al. Microscopy and Microanalysis 19 (S2), 1394-1395 (2013).

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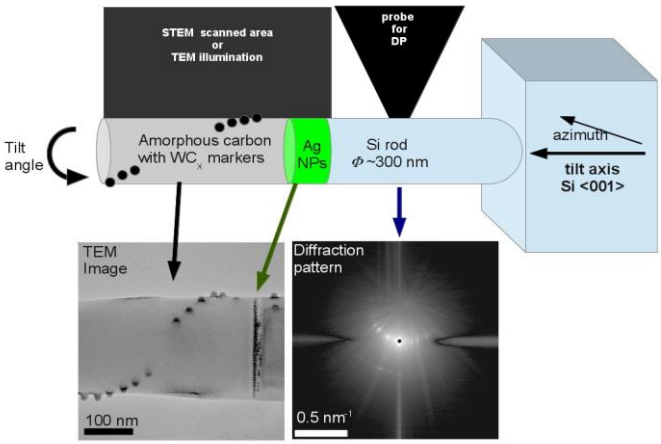

Figure 1 Schematic of tilt (α) and azimuth angle (β) measurement using a diffraction method on a rod-shaped sample.

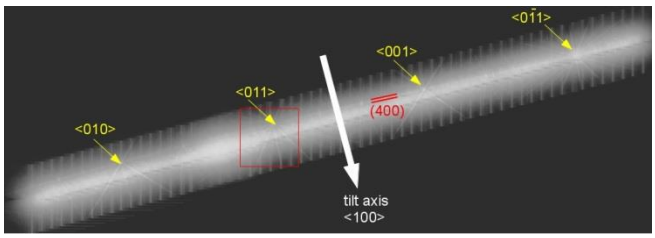

Figure 2 A portion of stitched DPs over a 183° tilt range, showing zone axis used for calibration and (400) Kikuchi band. The DPs were collectedwith 3° increment. The zone axes usedfor calibration are marked in yellow.

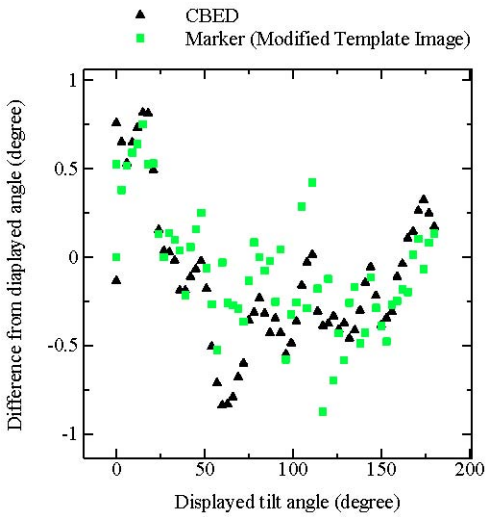

Figure 3 The difference (error) between measured tilt angles and the nominal value shown by sample holder hardware. The difference between the nominal tilt value displayed by the sample holder and the value measured by diffractionis displayed as triangles. The difference between the nominal tilt value and the tilt angles measured by marker methodare displayed as squares.

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