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Local Thickness Measurement in TEM

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Thickness measurement of nanoscale objects is critical in many applications but when the thickness to be measured falls below about 10 nm, the usual methods of conventional transmission electron microscopy (CTEM) are of limited use. For example, convergent-beam electron diffraction (CBED) can only be applied to thicker crystalline specimens while thickness measured by electron energy-loss spectroscopy (EELS) can be distorted by surface-plasmon contributions [1]. Moreover, it is desirable to measure thickness in microscopes that are not equipped with an energy filter or energy-loss spectrometer. Here we demonstrate an improvement of the mass-thickness method so that it can be applied to thin samples in a CTEM without an energy filter. The mass-thickness method [2,3] relates the contrast, defined as $\log(I_0/I_{tr})$, where I_0 is the incident electron intensity and I_{tr} the intensity of transmitted electrons, to the object weight per unit area. In its traditional form, it is difficult to measure I_0 and I_{tr} in very thin objects with sufficient accuracy to assess local sample thickness [3]; often beam current stability is not sufficient to perform accurate sequential measurements of I_0 and I_{tr}. But when the intensities I_0 and I_{tr} are measured from the same digital image, mass thickness can be measured locally in both crystalline and amorphous samples, provided the contrast aperture is large enough.

Commercial holey carbon films were used as test samples and the experiment was performed in a Hitachi HF-3300 CFEG-TEM with Gatan Tridiem EELS spectrometer at 300 kV acceleration voltage. Figure 1 shows an electron micrograph of a holey carbon film recorded using an Ultrascan 1000 slow-scan CCD camera. Although the image was acquired without an objective aperture, contrast is visible between the vacuum and carbon film, arising from the limited acceptance angle of the objective lens (about 104 mrad in our microscope). Two neighboring areas with the same size, one in vacuum and one in the carbon film were selected to measure the incident intensity I_0 and transmission intensity I_{tr}, using DigitalMicrograph to sum all counts in the selected region of interest. The area size can be chosen arbitrarily by adjusting the microscope magnification providing the counting statistics are sufficient.

Figure 2 shows the 300 keV electron transmission (I_{tr}/I_0) in amorphous carbon as a function of collection semiangle β and EELS-measured sample thickness. These scattering-contrast images were obtained with four different objective apertures and with no objective aperture. The thicknesses were also measured by low-loss EELS, using a collection semiangle of 104 mrad (no objective aperture). Our measurements correspond well to 80- keV data by Egerton [4]. The transmission starts to saturate around 18.3 mrad which is very close to the characteristic angle θ_0 of elastic scattering as measured by Reimer [5]. Figure 3 shows the dependence of natural-logarithm $\ln(I_0/I_{tr})$ on film thickness as function of collection semiangle. The linear relationships reveal that the transmission follows an exponential absorption law with increasing thickness. Our

measurements agree with earlier measurement on thicker carbon films at 100 keV reported in [6]. As seen for amorphous materials in Fig. 2 and Fig. 3, the sensitivity of the method increases as collection angle decreases. In crystalline materials, the smallest collection angle that can be used is determined by the need to include a sufficient number of Bragg beams. Once the curve of several samples of known mass thickness has been prepared [3], the arbitrary measured $\ln(I_0/I_{tr})$ can be converted to mass thickness with the corresponding collection angle.

References

[1] F. Wang, R. Egerton, M. Malac, Ultramicroscopy 109 (2009) 1245.
[2] E. Zeitler and G.F. Bahr, *J. Appl. Phys*. 30 (1959) 940.
[3] I. Pozsgai, *Ultramicroscopy*, 68 (1997) 69.
[4] R.F. Egerton, *Phil. Mag.* 31 (1975) 199.
[5] L. Reimer, K.H. Sommer, *Z. Naturforsch.* A 23 (1968) 1569.
[6] L. Reimer, H. Kohl, *Transmission Electron Microscopy*, 5th Edition, Springer, 2008, p.198.

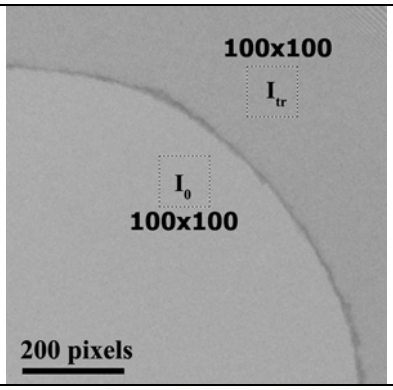

Fig. 1. A digital micrograph of holey carbon film showing areas used for measurement of incident intensity (I_0) and transmission intensity (I_{tr}). The contrast between vacuum and carbon arises from objective lens acting as an angle-limiting aperture.

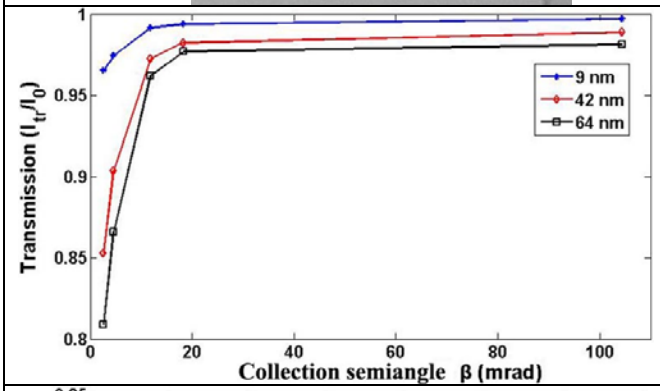

Fig. 2. 300 keV electron transmission in amorphous carbon as a function of collection semiangle β for different sample thickness. The nominal sample thickness was determined by low loss EELS measurement with a collection semiangle about 104 mrad.

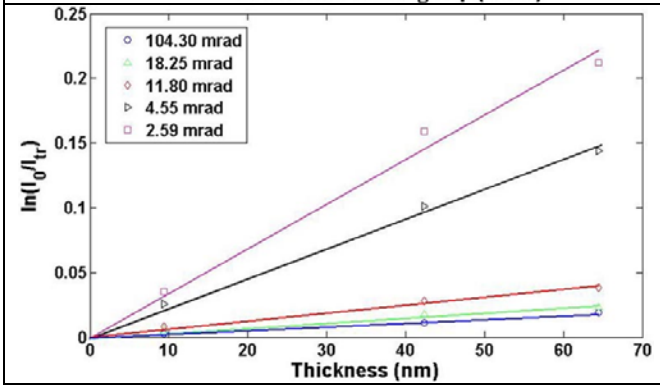

Fig. 3. Semilogarithmic plot of I_0/I_{tr} of carbon film as a function of thickness for different collection semiangle (different objective apertures). The nominal sample thickness was determined by low loss EELS measurement with a collection semiangle about 104 mrad.

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