Spectroscopy in STEM/TEM

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Density measurement in thin layers by electron energy loss spectroscopy (EELS)

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Mechanical, optical, and electrical properties of thin films depend strongly on their density. In view of commercial applications the companies have a major interest in methods for measurement of the density even in ultrathin layers. A density measurement method with high lateral resolution could help to optimize the parameters of deposition processes and to tailor the thin film properties for specific applications.

Our method is based on the acquisition of energy filtered images in the low loss region as well as of an element distribution map using core loss edges. We demonstrate the method at thin tetrahedral amorphous carbon layers prepared with an intentional density gradient in an Oerlikon Balzers PVD production system on polished tungsten carbide substrate.

The "normal" mass thickness contrast in TEM depends on the product of specimen density ρ and thickness t. It is the basic idea of the method to uncouple these two parameters using EELS techniques. Getting high lateral resolution we use an imaging filter for the measurements (Energy Filtered TEM technique). The thickness can be measured as multiples of the mean free path λ for inelastic scattering by comparison of the portions of elastically and inelastically scattered electrons resulting in a thickness map $I_{ThickMap}(x,y)$. The relation between λ and ρ is given by Iakoubovski et al. [1] (see equation (1) on second page).

For the uncoupling of thickness and density we need additional information. This is given by the relation between the element-specific EELS signal (here carbon K-edge) I_C and thickness as well as density (cp. equation (2)). Unfortunately, the proportionality of I_C and thickness t is only valid up to about $\lambda/3$ because of inelastic multiple scattering. We correct this influence by an additional term (polynomial with purely mathematical background and dependence on the experimental setup) and obtain formula (3) for the spatial-resolved density distribution.

Figures 1 and 2 demonstrate the color-encoded results obtained at a cross section of a ca. 1 µm thick carbon layer as mentioned above. Figure 1a shows an example of the thickness map measured on a FIB prepared cross-section of the carbon layer. Figure 1b illustrates the carbon map already corrected regarding the multiple inelastic scattering. Figure 2 shows the two-dimensional result of the density calculation pixel by pixel. For a better understanding two intensity profiles are shown along the line parallel to the substrate surface (white arrow) as well as perpendicular to it (blue arrow). There is a significant difference between them: The line of best fit for the profile parallel to the surface has the slope zero, i.e. there is no systematic change of the density in this direction. The fluctuations at the beginning are believed to be a result of the substrate roughness.

On the other hand, the profile perpendicular to the substrate surface indicates a significant dependence of the carbon layer density on the distance from the substrate which can be approximated by polynomial of 2nd degree as labeled in Figure 2.

The confirmation of the deposition induced expected density gradient in the cross section of the thin carbon layer shows that the method is suitable for the measurement of density fluctuations with a lateral resolution typical for transmission electron microscopy.

1. K. Iakoubovskii, K. Mitsuishi, Y. Nakayama, and K. Furuya, Microsc. Research Techn. **71** (2008), p. 626
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Equations.

$$I_{ThickMap}(x,y) \propto \frac{t(x,y)}{\lambda(x,y)} \propto \left[\rho(x,y)\right]^{0.3} \cdot t(x,y) \qquad (1)$$

$$I_C(x,y) \propto \rho(x,y) \cdot t(x,y) \qquad (2)$$

$$\rho(x,y) \propto \sqrt[0.7]{\frac{I_C}{I_{ThickMap}\left[1+0.42\cdot {}^{t}\!/\!_{\lambda} - 1.34\left({}^{t}\!/\!_{\lambda}\right)^2 + 0.45\left({}^{t}\!/\!_{\lambda}\right)^3\right]}} \qquad (3)$$

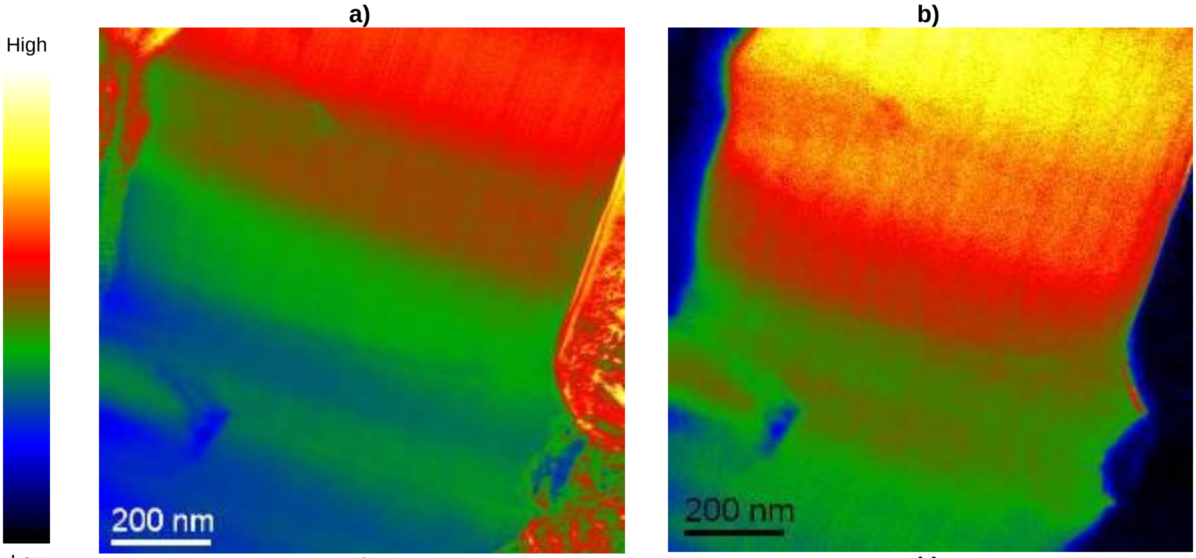

Figure 1. EFTEM results obtained on a cross-section of the carbon layer. a) Thickness distribution *t/λ*. b) Carbon distribution (net intensity of the C K-edge).

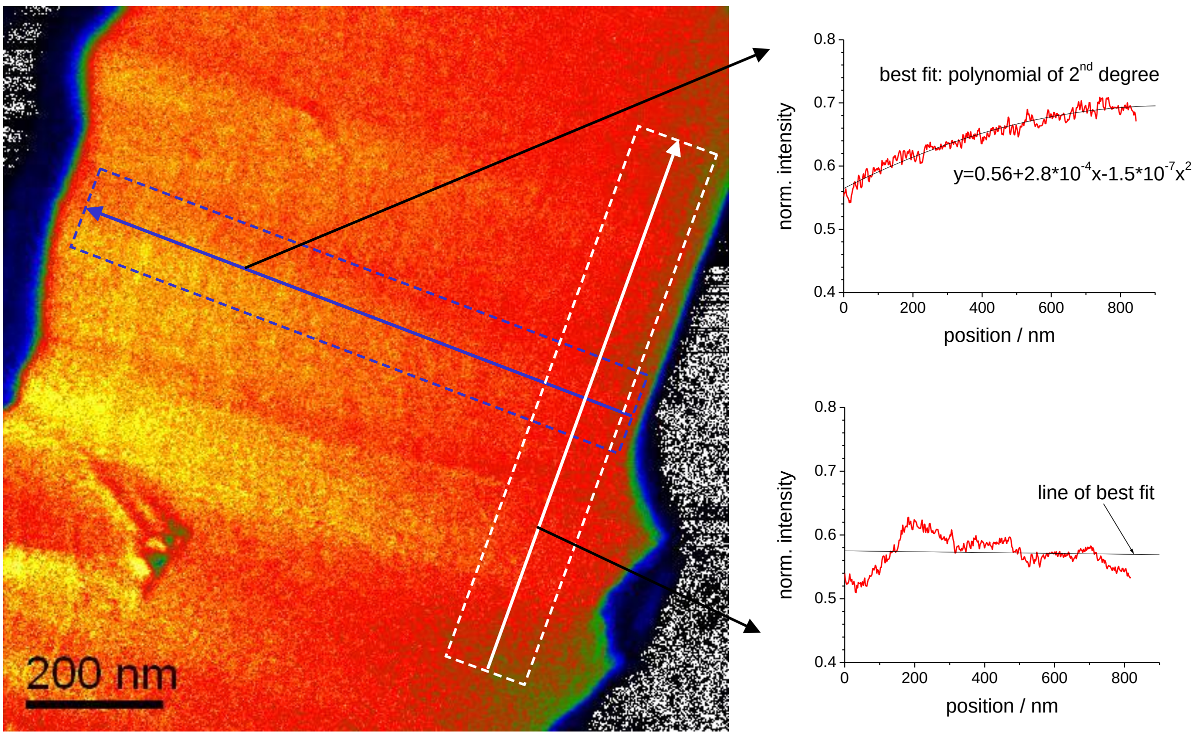

Figure 2. Density distribution derived from the EFTEM results shown in Fig. 1