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Contamination and Charging of Amorphous Thin Films Suitable as Phase Plates for Phase-Contrast Transmission Electron Microscopy

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Contamination and charging of the specimen is a common issue in electron microscopy and can lead to disturbing artifacts in transmission electron microscopy (TEM) and especially in scanning (S)TEM, where a focused electron beam is impinging on the sample [1]. However, these effects can also be utilized beneficially, e.g., in electron-beam induced deposition or in hole-free phase plate (HFPP) TEM [2,3]. To better understand the underlying mechanisms of contamination and charging, we studied amorphous thin films under focused electron-beam illumination with high intensity using electron-energy loss spectroscopy (EELS), HFPP imaging and secondary electron (SE) detection. The experiments provide insight in the behavior of contamination buildup, allow a clear separation between contamination and charging effects and give a possible explanation for the origin of the HFPP image contrast. We also investigated different techniques to reduce or even prevent contamination.

The experiments were performed using a Hitachi HF-3300 microscope at an electron energy of 300 keV allowing EELS, HFPP imaging and the detection of SEs. In a subsequent analysis of the data, the SE trace, the relative film thickness (t/λ, with λ the mean free path for plasmon scattering) and the phase shift (PS) generated by contamination or charging was obtained. We systematically studied amorphous carbon (aC) thin films fabricated by electron-beam evaporation (E-Beam aC) and Thread evaporation (Thread aC), amorphous Si substrates (Plano, NTUS100A05Q33A) and thin films made from metallic glass alloys such as $Pd_{77.5}Cu_6Si_{16.5}$ (PCS).

Experiments on untreated thin films conducted shortly after insertion in the microscope show a strong increase in t/λ upon focused electron-beam illumination as a function of the electron dose (Figure 1). The thickness increase can be attributed to the electron-beam induced deposition of a carbonaceous contamination layer originating from adsorbed hydrocarbon molecules with surface diffusion playing a major role. Further analysis using low-loss and C-K edge EELS reveal, that the deposited contamination layer is similar to graphitic carbon. The PS induced by such a contamination layer can be well described solely by the PS caused by the additional material deposited on the thin film suggesting that charging of the contamination layer is not present. Different measures such as UV cleaning, in-situ heating or beam showering can inhibit the formation of contamination layers [4].

Experiments performed on thin films which do not show a thickness increase and thus are not prone to contamination reveal that charging may still be present. Figure 2 displays the results obtained for an amorphous Si thin film with a nominal thickness of 5 nm at temperatures between room temperature (RT) and 300°C. While the t/λ evolution is flat for the entire temperature range (Figure 2a) indicating that contamination is prevented, the measured PS increases with decreasing temperature (Figure 2b). The PS has a negative sign corresponding to a negatively charged area on the thin film. A negative PS was also observed for other thin films not showing contamination. Possible processes causing the charging of a thin film are the generation of SEs leaving a positive charge in insulators, the trapping of charges in surfaces layers or electron-stimulated desorption of surface layers leading to a work-function change. The localized charges are screened within a distance depending on the (di)electrical properties of the thin film material, e.g., the conductance. Although the overall net charge of the thin film is always zero, electrostatic fields emerge in the adjacent vacuum causing the detected PS.

References:

[1] J J Hren, Ultramicroscopy **3** (1979), 91-95.
[2] M Malac *et al*., Ultramicroscopy **118** (2012), 77-89.
[3] R Danev *et al*., PNAS **111** (2014), 15635-15640.
[4] S Hettler *et al*., Micron (2017), accepted.
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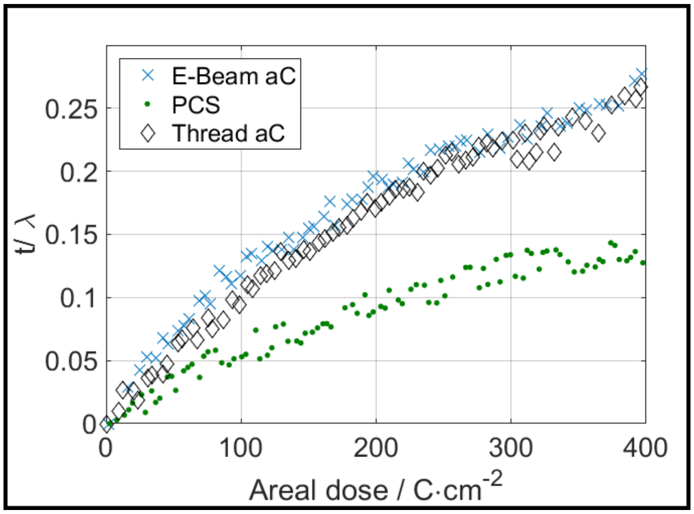

Figure 1. Evolution of t/λ during the buildup of a contamination layer under focused electron-beam illumination for an E-Beam aC, Thread aC and a PCS thin film. The PCS thin film was pumped overnight which allowed more adsorbates to desorb and lead to the slower t/λ increase compared to the aC thin films.

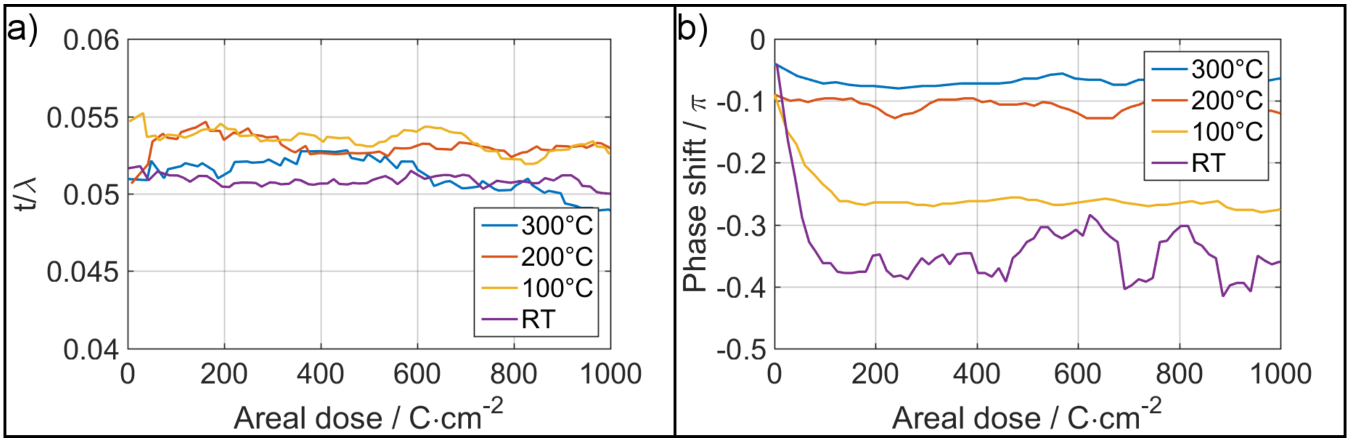

Figure 2. Analysis of an amorphous Si thin film after UV cleaning and heating to 275°C overnight. (a) The thin film exhibits a flat t/λ evolution for the entire temperature range. (b) The induced PS has a negative sign and the PS increases with decreasing temperature.