Application Note
Ultra-Thin Carbon Support Films for Improved STEM-EELS Analysis of Nanoparticles

related instruments: Leica EM ACE600
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Recent developments in aberration corrected transmission electron microscopes as well as further improvements in monochromaters and spectrometers have pushed the attainable energy resolution for EELS to 100 meV and beyond. STEM-EELS of individual nanomaterials can be challenging due the necessity of a support film. For such experiments, it is of the outmost importance to use carbon films of the highest quality both in terms of uniformity and cleanliness. It has previously been reported that many commercially available grids contain a number of graphitic structures and a large amount of hydrocarbon molecules which makes analysis particularly difficult. Thickness variations and imperfections in the carbon support film will result in inaccurate background subtraction that can preclude quantitative or even qualitative EELS analysis. Carbon support films should be as thin as possible to eliminate a strong distortion in the low loss region because of the strong carbon edge. Therefore 3 nm ultra-thin carbon films are essential to minimize the signal from the substrate during EELS data acquisition.

Images 1a and 1b show the dispersion of CdSe quantum plates onto 3 nm carbon film deposited on a Quantifoil (see application note 'Ultra-Thin Carbon Films'). The ultra-thin carbon films can also be deposited on holey carbon films, but in order to show the decreased thickness a Quantifoil is used as a reference. Imaging the quantum plates on the ultra-thin carbon film is clearly beneficial. The uniform intensity of the ultra-thin carbon film reflects its uniform thickness. Although carbon films prepared by the EM ACE600 are of high quality, often other factors will determine how well the specimen will behave in the microscope. In this particular case, mobile hydrocarbons diffused across the sample surface to the area of interest where they locally decomposed and polymerized under the electron beam. This can be clearly observed in image 1c. The scanning probe polymerized the diffusing hydrocarbons in the region of interest. This carbon build-up results in poor signal strength or even causes intense Ck edges in the EELS spectrum that obscures signal from other elements. Especially for thin specimens the contribution of contamination build up is significant because they are a large fraction of the total projected mass thickness. Specimen contamination is complex and has several sources. In cases where the specimen itself acts as a local source of hydrocarbon some post treatments can be performed in order to reduce hydrocarbon contaminants. A mild plasma treatment (use a low percentage of oxygen and a low power) can be used to remove contaminants as long it does not affect the sample or the integrity of the carbon film. A better approach to eliminate contaminants in this particular case is a bake out at 60 degrees Celsius at high vacuum (5x10⁻⁷ mbar) for several hours. The principle here is to desorb the contaminants by mild heating in high vacuum and limit outgassing from the specimen during analysis once it is introduced in the microscope. After a high vacuum heat treatment, the contamination issue was nearly eliminated and the specimen was suitable for STEM-EELS experiments requiring long acquisition times. Because of the limited thickness of the quantum plates, EELS analysis was performed at 80 kV and a thickness map of the plates was obtained easily.

In conclusion, both the high vacuum heating procedure and long-term stability at low kV show the full potential of these ultra-thin carbon films. Moreover the procedure is completely reproducible and accurate due to the adaptive carbon thread coating resulting in a straightforward analysis.

Images acquired by Eva Bladt, Nicolas Gauquelin, Jo Verbeeck and Sara Bals (EMAT, University of Antwerp)
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