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Advanced Monochromated Low-voltage EELS and EFTEM

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The advantages of using lower acceleration voltage for imaging in order to reduce sample damage and typically improve contrast for biological materials have been known for many years. However, the distinct benefits for EFTEM imaging and EELS have only begun to be exploited due to the recent ability to use monochromated electron sources. With Cs corrected TEM's now able to obtain Ångstrom level resolution at < 80 keV [1] opens up the ability to investigate a wider range of materials and bio-materials problems. The combination of Cs-correction and an electron source monochromator yields a further step along the path to an instrument that performs closer to the ideal microscope; the expansion of the temporal envelope function of the contrast transfer function by reducing the delta E is a direct benefit that can lead to improved resolution [2].

The extreme benefits of electron monochromator integration are for applications that especially depend on energy resolution such as spectroscopy (EELS) and advanced contrast mechanisms such as "atomic scale" energy filtered imaging (EFTEM), the increase of the electron scattering cross-section at lower electron voltages being perhaps the biggest benefit for filtered imaging and spectroscopy performance. This opens up new opportunities to extract highly localized structural information from transition metal oxides with negligible interference from instrumental broadening. In addition to high-energy resolution, the high isochromaticity extends over a large field of view. This feature is necessary for accurate spectrum imaging, where a stack of images is taken with small energy window ~at 0.1 eV! It is also critically important when doing zero-loss, energy-filtered imaging at high magnification. To determine the isochromaticity, the shift of the center of the zero-loss peak was measured over the field of view at a TEM magnification of 16kX, resulting in an object field of 1.63 x 1.63 mm^2. The resulting energy deviation over the field of view was less than 0.1 eV. In the development process to produce an electron source with all the benefits of both cold and thermal emitters the incorporation of a monochromator on a thermal source is one of the mechanisms of choice to minimize the energy spread.

The energy spread of Harvard's Zeiss Libra TEM field emission source is reduced by the incorporation of an Omega monochromator from 600 meV to 100 meV at 200 kV, 70 meV at 80 kV and after tuning the instrument to perform at an accelerating voltage of 40 kV we obtain ~ 50 meV FWHM energy spread with suitable remaining brightness for high-resolution imaging (Figure 1). (Our FWHM value for 40 kV is approximate due to the dispersion limit of our energy filter). We present results of our investigations and application of using advanced EELS and EFTEM on various materials systems, such as photonic nanowires and biomaterials incorporated with nano-materials such as Au and Ag nanomarkers for cancer research and acting as markers for labeling.

The extreme energy resolution of the in-column filter of this instrument allows us insight in materials classes previously excluded from high-resolution analysis due to their beam-sensitivity, including light element materials which can now be investigated at an unprecedented contrast and spatial resolution.

References
[1] D. C. Bell, C.J. Russo, G. Benner, “Sub-Ångstrom Low-Voltage Performance of a Monochromated, Aberration-Corrected Transmission Electron Microscope”, Microsc. Microanal. 16, 386-392 (2010)
[2] P.W. Hawkes and J.C.H. Spence *Science of Microscopy* Springer (2007)

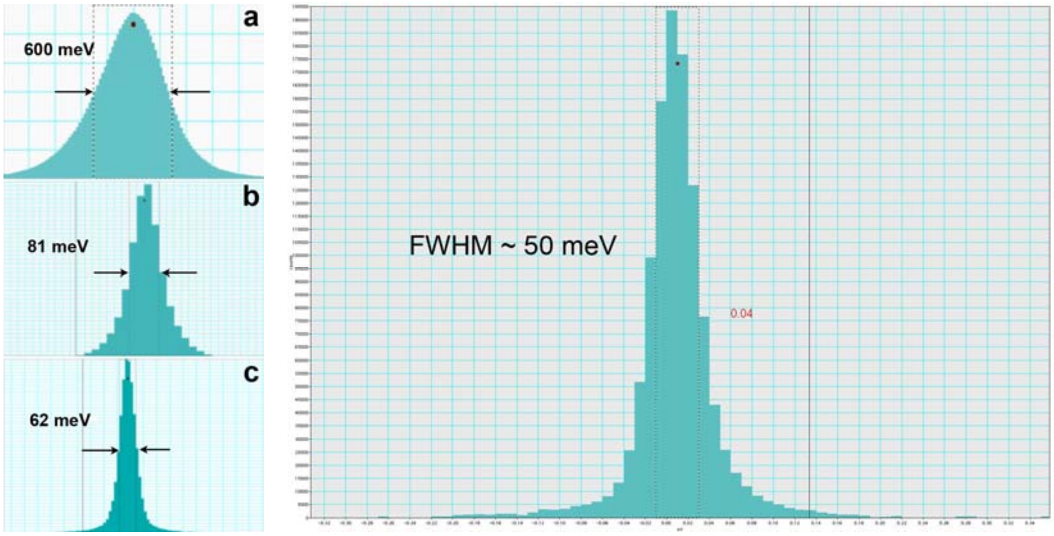

Figure 1. Left, Zero-loss peak showing energy spread with Zeiss-CEOS monochromator, a) non-monochromated b) monochromated (1µm slit) at 200 keV c) monochromated (1µm slit) at 80 keV. Right, Zero-loss peak showing the energy spread of the electron source with monochromator at 40 kV.

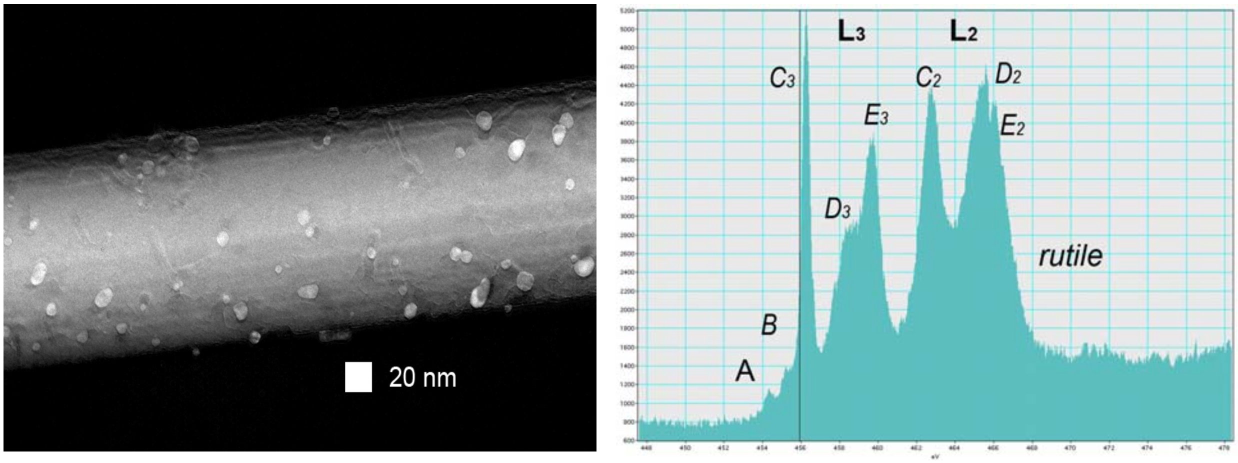

Figure 2. Left, high-resolution, low-loss EELS thickness difference map of Al clad nanowire confirming that the gold nano- particles occur only the surface. Right, extreme EELS energy resolution, obtained at 80 keV from a sample of TiO_2. Prepeaks A and B only appear with monochromator. They exist only in the rutile and anatase structure. The shape of D3 and E3 peaks correspond to rutile.