Type of presentation: Oral

IT-5-O-1653 New EM signals made accessible by sub-20 meV resolution EELS

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Nion's High Energy Resolution Monochromated EELS-STEM (HERMES) instrument [1] is able to combine Scanning Transmission Electron Microscopy (STEM) spatial resolution of a 1-10 Å with 12-50 meV Electron Energy Loss Spectroscopy (EELS) energy resolution. These capabilities promise to make new signals available in analytical EM, and thereby revolutionize it even more than aberration correction has revolutionized EM imaging. Here we explore two new signals: spatially-resolved phonon spectroscopy, and the detection of very light elements by energy-filtered imaging of electrons scattered to high angles.

Fig. 1 illustrates how important signals have up to now been "hidden in plain sight" – obscured by a broad EELS zero loss peak (ZLP). The solid green spectrum was recorded with the beam passing through the monochromator but the energy-selecting slit retracted. The full-width at half-maximum (FWHM) of the ZLP is ~250 meV. The red (line) spectrum was recorded with the slit in, in 0.1 s, and shows FWHM of 14 meV.

The blue (x1000) spectrum in Fig. 1 was recorded from a ~2 nm Ø area of SiO_2. The optical phonon peak visible at 140 meV energy loss is in good agreement with the energy of the strongest feature in infrared spectra of SiO_2, at 1100 cm^{-1}. (To convert cm^{-1} to meV, divide by 8.)

Fig. 2 demonstrates that some phonon signals can be spatially resolved with a resolution of a few nm, and hopefully better in the future. The phonon intensity decays close to zero within ~3 nm inside the Si and there is also an initial sharp intensity drop-off at the SiO_2–vacuum interface. There is also a long tail stretching tens of nm outside the sample, which suggests that damage-free phonon spectroscopy may be possible with an aloof electron beam.

Imaging phonons in compounds containing light elements such as H should allow the spatial distribution of the compounds to be mapped. It may, however, also be possible to image the light elements in a more general way, by using the fact that electrons scattered incoherently by atomic nuclei to high angles (Rutherford scattering) transfer small amounts of energy to the recoiling nuclei, inversely proportional to their mass.

Fig. 3 shows proof-of-principle energy-filtered high-angle dark field (EFHADF) mapping of light vs. heavy atoms: 60 keV spectrum-image data from Au particles supported on an amorphous carbon foil ~20 nm thick, next to a hole in the foil. Energy window B is centred on the ZLP (±10 meV) and the corresponding image 3(b) shows mainly Au particles. Energy window C is placed over energy losses of 85±10 meV, and image 3(c) shows only carbon. The fact that we are able to image only the carbon shows that we now have sufficient energy discrimination to map very light elements such as H and Li [2].

[1] OL Krivanek et al., Microscopy 62 (2013) 3-21.
[2] TC Lovejoy et al., M&M meeting (2014, Hartford).
[3] We are grateful for the use of LeRoy Eyring Center facilities at ASU.

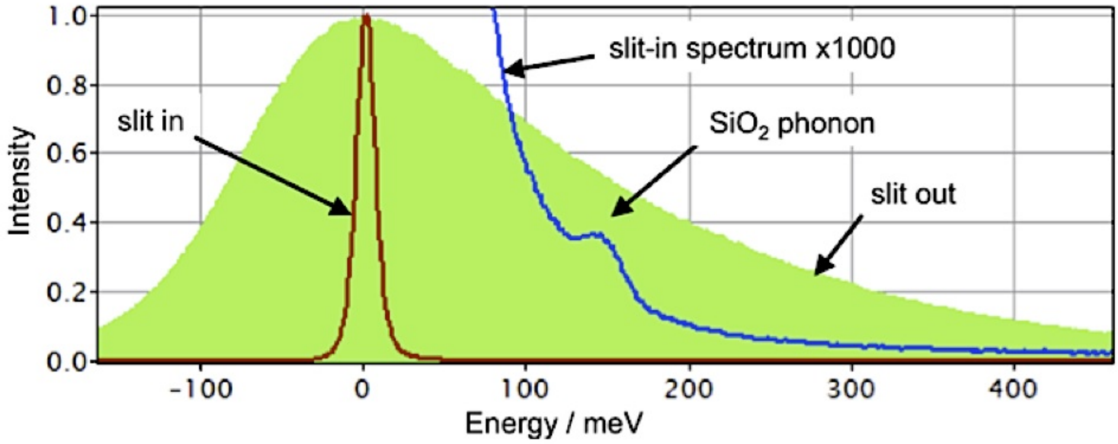

Fig. 1: EEL spectra recorded under various conditions by Nion HERMES at 60 keV. Solid (green) spectrum: monochromator slit out; red spectrum: slit in; blue (x1000) spectrum: slit in, electron probe on SiO_2, acquisition time 10 s, beam current ~10 pA, probe convergence angle ±12 mrad, collection angle ±12 mrad

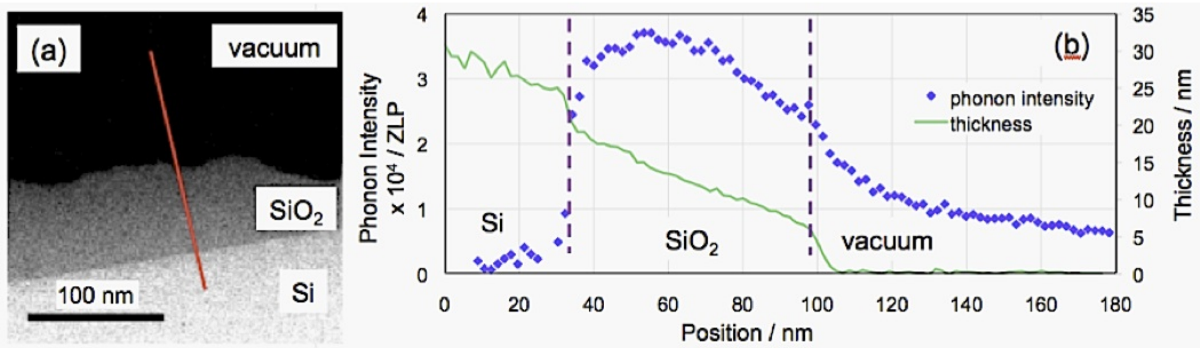

Fig. 2: a) HAADF image of a Si-SiO_2 cross-section; b) profile of SiO_2 phonon intensity and sample thickness along the red line in (a). The SiO_2 phonon intensity was measured from a series of 100 spectra in 10 s each, normalized by the ZLP. The sample thickness was determined from spectra of all energy losses up to 180 eV, recorded separately.

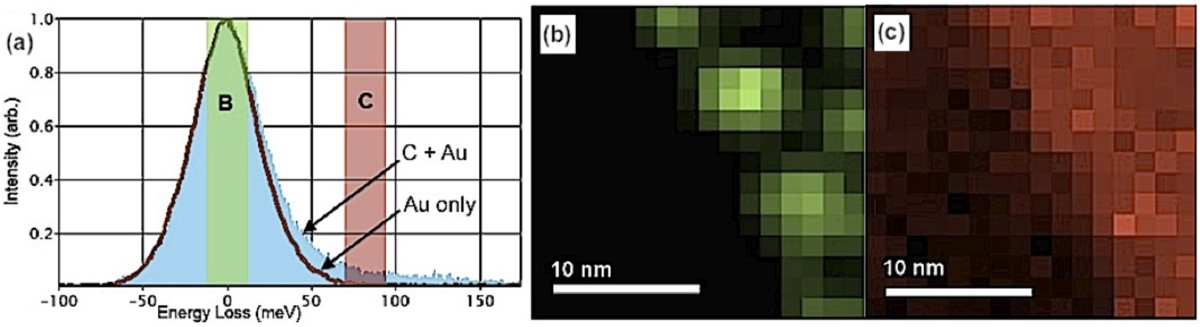

Fig. 3: EFHADF recoil mapping of Au on am. carbon: a) EEL spectra from a Au particle and from carbon film; b) image formed with window B showing only Au; c) image formed with window C showing only carbon. The angles admitted into the spectrometer were 120±30 mrad, and scattering from carbon nuclei was expected to give a broad peak centered on 40 meV.