Exploring signals made accessible by sub-20 meV resolution EELS

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The high energy resolution monochromated EELS-STEM (HERMES) instrument we have developed [1] can attain an energy resolution of 12 meV full-width at half-maximum (FWHM) of the zero loss peak (ZLP) in electron energy-loss spectra (EELS) obtained at 60 keV primary energy (Fig. 1), and further improvements are expected in the future [2]. In routine operation, we are achieving 15 meV resolution in spectra acquired in one second or longer acquisition time. We have also been able to improve the rate of the ZLP decay such that tail intensities of $<4x10^{-4}$ of the ZLP maximum have been recorded at energy losses as small as 100 meV.

HERMES is thus approaching the fractional energy resolution ($\Delta E/E_o$) of the Boersch-Geiger monochromated EELS instrument, which reached 3 meV energy resolution at 30 keV [3], and it may approach the B-G absolute energy resolution in the future. At the same time, it provides several new capabilities:

Spatial resolution and flexibility: HERMES can obtain spectra at the full energy resolution from sample areas as small as a few Å and as large as 10 μm, and attain an angular resolution of ~1 μrad (using a camera length setting of ~1 km).

Angular acceptance: HERMES can attain good energy resolution with entrance acceptance angles of ±30 mrad and greater.

Brightness: HERMES uses a bright cold field emission gun, and can therefore extract appreciable signals from Å-sized areas.

The above capabilities promise to make new signals available for analysis, and we have begun to explore them. Up to now, the signals have been "hidden in plain sight": obscured by a broad zero loss EELS peak. Here we provide examples of our progress in two areas: phonon spectroscopy, and energy analysis of HAADF scattering.

We have observed optical phonons in a range of materials, at energies spanning from 70 to 180 meV. The green (right side) spectrum in Fig. 2 shows a phonon peak from an SiO_2 passivation layer in a MOSFET device, collected at 60 keV from an area about 100 nm in diameter, for forward scattering (ß ~ ±30 μrad). The energy resolution was worsened to about 25 meV to admit a stronger signal through the energy-selecting slit and there was an asymmetry in the ZLP, most likely caused by charging at the slit, which we are presently addressing. The phonon peak at 140 meV is in good agreement with the energy of the strongest feature normally observed in infrared spectra of SiO_2, at 1100 cm^{-1}. (To convert cm^{-1} to meV, divide by 8.)

Electrons scattered incoherently into HAADF (high angle annular dark field) detectors transfer a small amount of energy to the scattering nucleus. HERMES promises to allow such energy losses to be studied experimentally. Fig. 3 shows spectrum-image data collected at 60 keV from Au particles supported on amorphous carbon about 20 nm thick, next to a hole in the sample. The line (red) spectrum shown in (A) is from Au only; the solid (blue) spectrum is from a carbon area with a contribution from neighboring Au particles and shows a tail due to Rutherford scattering from carbon nuclei. Energy window B is centered on the ZLP and the corresponding image shows mainly Au particles. Energy window C is placed over energy losses of 85±10 meV, and shows only carbon. The scattering angles admitted into the spectrometer were 120±30 mrad and Rutherford scattering from carbon was expected to give a broad peak centered on 40 meV. The fact that we are able to image only the carbon shows that we now have sufficient energy discrimination to selectively image very light elements such as H and Li by energy-filtered HAADF [4], and also to separate incoherent and coherent high angle scattering in elements up to about carbon.

In summary, high spatial resolution studies of phonons and other very low loss EELS signals have now become possible. They may well revolutionize STEM-EELS just as much as aberration correction has revolutionized EM imaging.

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References

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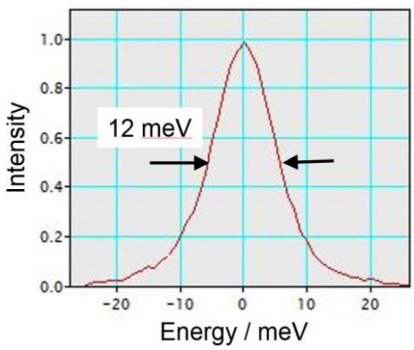

FIG. 1. EELS Zero Loss Peak (ZLP) recorded by HERMES in 0.25 sec. E_o = 60 keV.

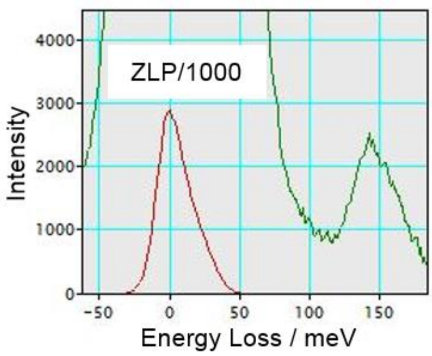

FIG. 2. Optical phonon in SiO_2. ZLP tail is reduced to 1/3000 at 100 meV.

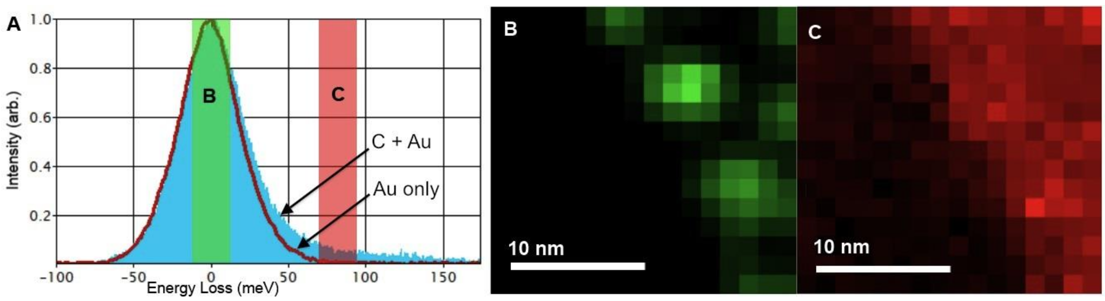

FIG. 3. Energy-filtered HAADF imaging of Au particles on amorphous carbon. A (line): spectrum from Au particles; A (solid) spectrum from carbon plus Au; B: image formed with an energy window of 0±10 meV; C: image formed with an energy window of 85±10 meV.