

Advances in Ultra-High Energy Resolution STEM-EELS

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The Ultra-High Energy Resolution Monochromated EELS-STEM (U-HERMES™) system developed by Nion combines a dispersing-undispersing ground-potential monochromator [1], a bright cold-field-emission gun, an advanced aberration corrector, and a new EEL spectrometer. The latest version of the system allows 5 meV energy resolution EELS and has achieved 1.07 Å spatial resolution at the sample at 30kV when monochromating [2], and it greatly extends the capabilities of vibrational spectroscopy in the EM, introduced 4 years ago [3]. U-HERMES™ has so far been used for: damage-free identification of different bonds including hydrogen bonds in guanine [4]; probing atomic vibrations at surfaces and edges of nano-objects with nm-level spatial resolution [5]; achieving sub-nm spatial resolution in images obtained with dark-field EELS vibrational signals [6]; nanoscale mapping of phonon dispersion curves [7]; nanoscale temperature determination by electron energy gain spectroscopy [8]; identification of different isotopes by vibrational spectroscopy in the EM [9]; and vibrational spectroscopy of ice [10].

A key challenge for EELS in the EM is to improve the energy resolution to 5 meV and better, as needed for vibrational spectroscopy of organic compounds in which different C-C, C-N etc. vibrational modes are resolved. This required a new electron energy loss spectrometer, with an energy resolution that matches the Nion ultra-high energy resolution monochromator. The monochromator produces a diffraction-limited spectrum at the energy-selecting slit and a diffraction-limited probe at the sample. This type of performance has not been attained by regular spectrometers, whose energy resolution is typically limited by instabilities. The monochromator's electron optics is equivalent to two spectrometers arranged back-to-back, with the first half of the monochromator producing a spectrum in the monochromator midplane and the second half cancelling the energy dispersion. If a single half were used as a stand-alone, diffraction-limited spectrometer (as it was in fact used during the monochromator bring-up tests [11]), it would achieve higher energy resolution than regular spectrometers.

Our new spectrometer (Fig. 1) has been named Iris, after the Greek goddess of the rainbow. It consists of a pre-prism aberration-correcting lens assembly, an energy-dispersing prism, a post-prism aberration-correcting assembly, a movable beam trap, a spectrum-magnifying lens assembly, and final detectors. As in the Nion monochromator (and also in Nion aberration correctors) all the coils of each multipole are linked in series and powered by a single power supply, which minimizes parasitic dipole deflections compared to designs in which the individual poles are powered by separate power supplies. Also like in the monochromator, the energy-dispersing prism uses a "C-shape" magnetic circuit with a compact coil winding designed for minimum heat production, but whereas the monochromator uses two prisms with gradient $n=0.5$ and one $n=0$ parallel-face prism, the spectrometer's prism has a gradient of $n=0.25$. Other shared features include a multipole assembly in front of the prism, which combines flexible optical coupling into the prism with the ability to adjust geometric aberrations up to 4th order separately from chromatic aberrations, a dispersion-magnifying lens assembly that includes layers with strong quadrupoles and no higher-order multipoles (i.e., similar to the projector lenses of an EM, which do not correct aberrations), and a mechanical construction that uses the outer housing of the multipoles as the principal mechanical element and thereby maximizes the spectrometer's rigidity. New features include: 16-pole multipole layers to correct aberrations up to fifth order with adjustable orientation; a motorized beam trap

which is moved close to the beam axis when the spectrometer operates at very high energy dispersion, and away from the optic axis when it operates with low energy dispersion, projecting a spectrum of a wide energy range onto the detector; and double-winding, constant-power quadrupoles in the magnifying lens assembly that allow the energy dispersion to be varied without changing the heat load in the spectrometer, minimizing thermal drift. The detection chamber has four ports on it, making it possible to use a range of detectors optimized for detection speed, single electron sensitivity, wide dynamic range, etc. The entire spectrometer and post-sample column feature double- or triple- mu-metal shielding and rigid mechanical construction to reduce the effects of environmental disturbances. Switching from vibrational spectroscopy to elemental mapping by core-loss EELS is as simple as selecting a new dispersion and taking out the monochromator slit (done with one click).

Figure 2 shows 5 meV FWHM (full width at half-maximum) of a zero loss peak (ZLP) acquired at 30 keV (at 60 keV, we reach 6 meV FWHM). The spectrum was acquired with 100 ms acquisition time (single-shot) and 20 mrad collection angle at the sample. Good energy resolution with long acquisitions times is critical when working with spectra that lack sharp reference peaks needed for post-acquisition alignment of short exposures, e.g. in dark field EELS work. The first Iris spectrometers have recently been installed in user laboratories, and results on real samples will be reported at the meeting.

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[4] P Rez et al., *Nat. Comms.* **7** (2016) 10945.

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[6] C Dwyer et al., *Phys. Rev. Lett.* **117** (2016) 256101.

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[8] JC Idrobo et al., *Phys. Rev. Lett.* *accepted* (2018).

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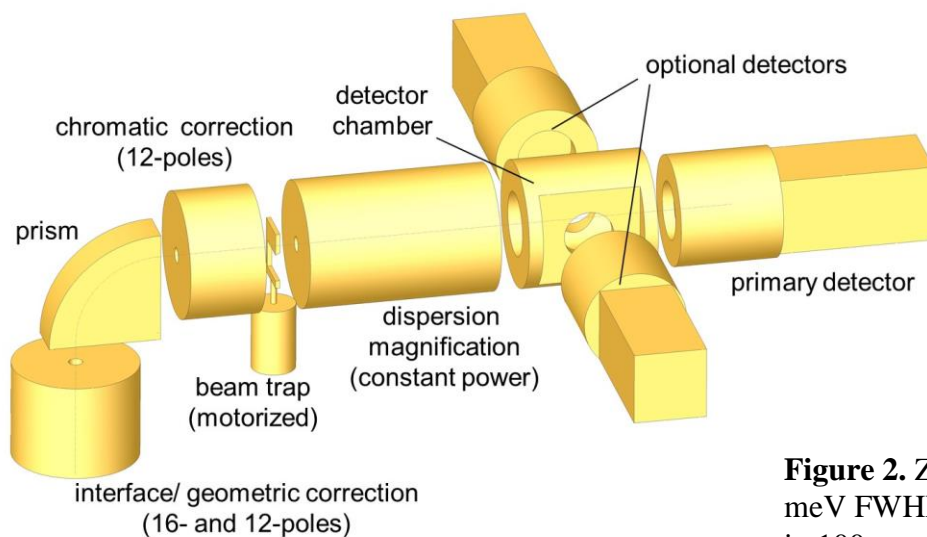


Figure 1. Schematic overview of the new Nion “Iris” spectrometer.

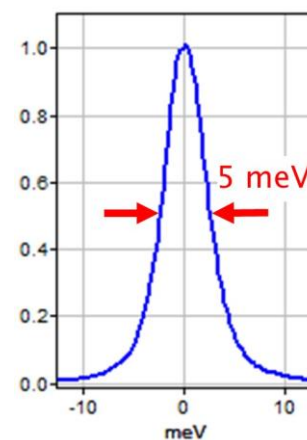


Figure 2. Zero Loss Peak (ZLP) with 5.0 meV FWHM acquired at 30kV in vacuum in 100ms with $\beta/2=20$ mrad, and energy dispersion at the spectrometer detector of 0.3 meV/channel.