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Evaluating Long-Term Stability and Transient Disturbances of a TEM

Marek Malac[1,2], Robert A. McLeod[1,2], Yoshifumi Taniguchi[3], Mike Bergen[1], David Hoyle[4]

1. National Institute of Nanotechnology (NINT), 11421 Saskatchewan Drive, Edmonton, Canada
2. Department of Physics, University of Alberta, Edmonton, T6G 2E1, Canada
3. Hitachi High-Technologies Corp., 882, Ichige, Hitachinaka-shi, Ibaraki-ken, 312-8504, Japan
4. Hitachi High Technologies Canada, 89 Galaxy Blvd., Rexdale, M9W 6A4, Canada

Modern transmission electron microscopes (TEMs) offer outstanding instrumental stability when placed in adequate laboratory environment [1]. We describe methods to evaluate instrument stability, to identify some of the disturbances, and we show an application of a stabilized instrument to momentum-resolved electron energy loss spectroscopy (EELS). We evaluated stability and disturbances of EELS, sample stage, electron probe, electron holography, scanning TEM and HRTEM imaging performance, and repeatability for a modified Hitachi HF 3300 TEM / scanning TEM with a Gatan Tridiem^{TM} model 863 and double biprism set-up [2].

To acquire data (images or spectra), an instrument must be sufficiently stable to obtain adequate signal-to-noise ratio (SNR) over a period of time where disturbances are below the desired precision of the measurement. The necessary acquisition time is determined by available beam current, cross section of the measured interaction, detector collection efficiency and its DQE and the experiment suitability for summing of multiple acquisitions. Momentum-resolved EELS (*q*EELS) is particularly demanding as the incident beam current is mapped in the momentum-energy (*q*-E) plane leading to low count rate and long acquisition time. To evaluate EELS stability, "streak EELS" is often used, where a post-EEL spectrometer CCD is continuously read out while the energy-dispersed beam illuminates the CCD. An example of a streak EEL spectrum is in Fig. 1a. The data in Fig 1a measures the drift of the zero-loss peak (ZLP) and beam current and the Fourier Transform (FT) of Fig 1a reveals frequencies affecting the ZLP position. Using a Gatan Ultrascan 1000^{TM} camera, the maximum sampling frequency in Fig 1a is ~700 Hz which can detect disturbances up to ~350 Hz. Long-term drift can be characterized by increasing the number of collected spectra or by introducing a delay between individual spectra, as used in Fig 1b. The ZLPs drift shown in Fig 1b was about 38 meV/min about 2 h after the microscope was started and decreased to about 20 meV/min after about 3 h.

To acquire TEM and EELS data from a desired area of a sample, the relative position of the sample and detector must change less than a desired spatial resolution. A convenient way to evaluate such stability is to collect a series of (high resolution) images. The relative position of features in the images can be then evaluated by cross correlation methods. Gold nanoparticles on amorphous Ge support test sample avoids some of the problems encountered when periodic lattice fringes in a crystal are used, but overestimates the sample instabilities as the sample itself undergoes changes due to electron-beam irradiation. Using an Au on Ge sample we obtained sample linear drift 50 pm/min and the random-walk diffusion coefficient 395 pm^2/s [4]. Evaluating the mean displacement gives 92 pm for 5 s and 350 pm for 60 s exposure.

In electron holography, the wavefront phase drift caused by biprism (BP) instabilities and beam tilt drift, projected back to sample plane, are important parameters. They are measured by taking a series of reference holograms, without a sample, and evaluating the scalar phase drift with time [4]. At 0.34 nm fringe spacing we measured BP random walk component as 10 pm^2/s (i.e. 3×10^{-3} rad/s) and the linear BP drift component 0.025 pm/s (i.e. 5×10^{-4} rad/s), projected to the sample plane [4], values indicating good electron holography performance.

An initial indication of instrument stability can be obtained from a series of high resolution images of a single crystal with increasing acquisition time and evaluating the presence of peaks in the FT of the images. Although the peaks in FT are not a measure of instrument resolution [5], their reproducibility is an indication of the TEM stability. A series of 10 images with 1 to 60 s image acquisition time was measured. The 1 s series yielded 10 out of 10 images with 0.1 nm reflections in FT with the detection limit at high spatial frequencies set by shot noise. At 10 s acquisition, 9 out of 10 images show 0.1 nm peaks while a 60 s series 10 out of 10 images show peaks at 0.1 nm or less indicating good long term stability.

Of a further concern is the drift after a change of the lens currents. This is not an issue for lens that use constant-power supply arrangement [6], but is a concern for projector lens when low drift is required, such as for *q*EELS measurements. Fig. 2 shows a valence-loss *q*EELS obtained from a single-crystal *h*BN sample. The incident beam stays within ±2x10^{-5} rad over the 40 s data acquisition with no observable linear drift component and has angular width below 1.5x10^{-4} rad including broadening due to detector point spread function.

Microscope stability and repeatability improves experiment success rates for samples that are radiation sensitive. We described several simple methods for evaluating microscope stability and provided an application example of valence *q*EELS.

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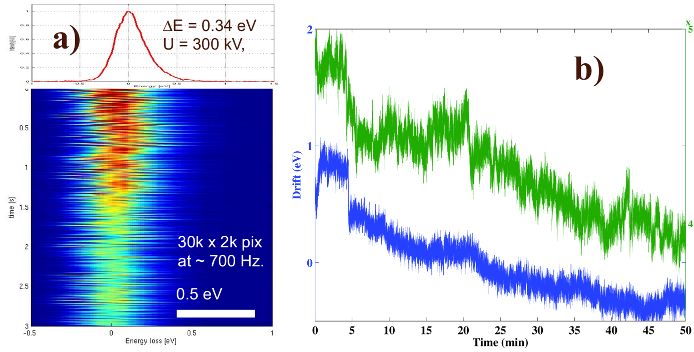

Fig. 1 a) Streak EELS spectrum containing 30,000 EELS spectra collected at 700 Hz over about 40 s period. An example spectrum extracted from the streak EELS is shown at the top. b) Drift of zero-loss peak in EELS (lower blue curve) and beam current change (upper green curve) over a 60 min period ~2 h after microscope was started.

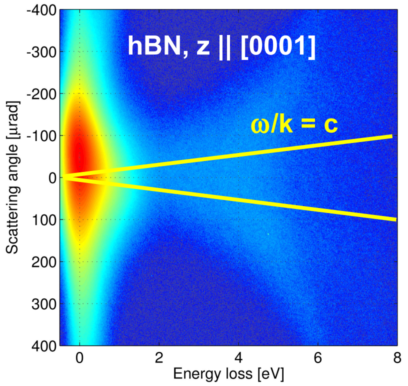

Fig. 2. Example of a momentum-resolved EELS in the valence region of *h*BN sample collected at 300 kV. Logarithmic intensity scale was used. The light line, corresponding to light dispersion in vacuum, is indicated by straight yellow line.