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Post-Specimen C_c Correction Enabled Scanning Confocal Electron Energy Loss Microscopy for High-Throughput 3-D Spectroscopic Imaging of Nanomaterials

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The ever increasing complexity of the 3-D architectures of modern nanodevices drives the demands to image nanoscale features in 3-D. ADF-STEM tilt-series tomography can reconstruct materials with nanometer resolution; however it often requires days to align and reconstruct the tomograms. With the development of aberration correctors where the depth of focus has been reduced to the sub-10nm region, 3-D imaging by depth sectioning becomes a possibility. It potentially allows for a reconstruction of nanomaterials by simply recording a through-focal series. Unfortunately, it has been demonstrated that both ADF-STEM and bright-field scanning confocal electron microscopy (BF-SCEM) have cones of missing information that produce excessive elongation artifacts in the resulting reconstructions. Here, we explore the inelastic counterpart of BF-SCEM—scanning confocal electron energy loss microscopy (SCEELM). This technique is free of the missing-information cone and resulting elongation artifacts. In a microscope without post-specimen chromatic aberration (C_c) correction, this method has a dose efficiency comparable to that of ADF-STEM depth sectioning if valence-loss signals are used. However, the efficiency can be increased by a factor of 10-100 with post-specimen C_c correction by parallel acquisition of SCEELM signals in spectroscopy mode (Fig. 1a). It can potentially enable a rapid and reliable 3-D reconstruction of materials with sub-10 nanometer depth resolution in C_c-corrected confocal instruments.
A key aspect of SCEELM is that it delivers spectroscopic information with depth resolution that is so far not available in any other spatial-resolved EELS techniques. However, in regular aberration-corrected TEMs, SCEELM can only be operated in the energy-filtered mode (termed as EF-SCEM or serial-SCEELM) due to significant chromatic aberration of the post-specimen focusing element. Owing to the difficulty in compensating for chromatic defocus change without affecting the incident electron beam, unlike EF-TEM, it is almost impossible to acquire a meaningful EELS spectrum in a regular TEM in EF-SCEM mode. However, this problem can be solved by post-specimen C_c correction. Fig. 1b shows a meaningful full spectrum across a 600 eV range can be acquired with limited degradation of depth resolution.
For 3-D imaging, as a proof of concept we used a holey carbon film as the test material. We only performed SCEELM depth profiling of the film at a single (x,y) position. The probing depth was changed by moving the piezo-electric stage in z. Fig. 2 shows the images of the electron probe exit waves in the energy filtered mode (serial SCEELM) as the sample moves in and out of focus. Clearly, the elastic probes are not sensitive to the z-position of the sample ($\Delta E = 0$ eV, BF-SCEM). However, the probe peak intensities of the energy-loss images decrease as the sample moves away from the focal point. The intensity drops faster with higher energy losses—less delocalization. In order to acquire SCEELM signal at different energy losses in parallel, a post-specimen C_c corrector need to be used to focus exit waves with different energies to the same imaging plane. A physical pinhole is also needed to improve depth discrimination. Fig. 1c shows the pinhole setup and the parallel SCEELM (Fig. 4d) profiling of a thick carbon film (t/λ=1.4). The energy-loss dependent depth profiles demonstrate that as the energy loss increases, delocalization decreases and the depth profile become narrower [1, 2].

References

[1] HL Xin *et al*, Microscopy and Microanalysis, submitted.

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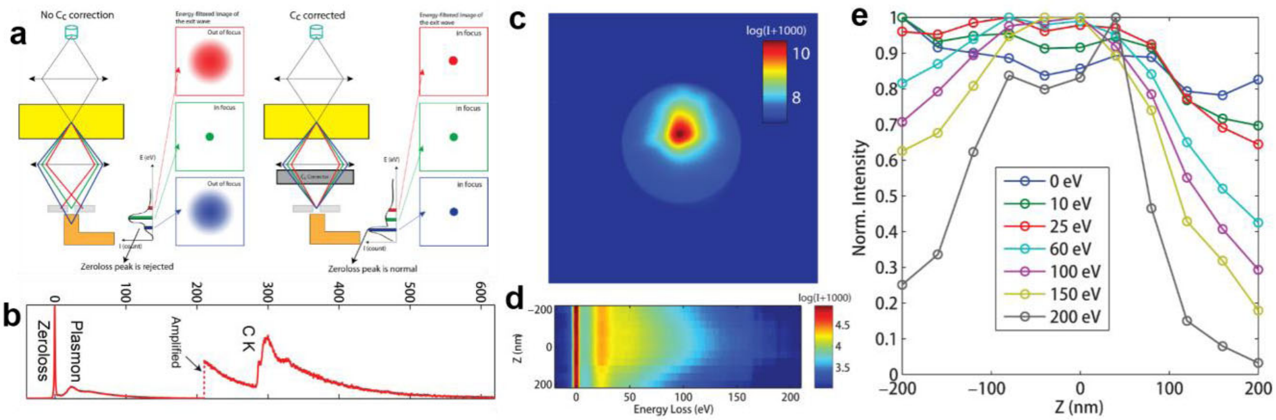

FIG. 1. (a) Schematics for scanning confocal electron energy loss microscopy (SCEELM). (left) typical setup without post-specimen C_c correction; (right) setup in a Cc corrected TEM. Exit waves in a wide energy window are simultaneously in focus. (c-e) Parallel SCEELM (spectroscopy mode + C_c correction + physical pinhole); (c) image of the probe and the pinhole; (d) depth-dependent SCEELM spectra; (e) energy-loss-dependent depth profiles of the carbon film extracted from the spectra in (b).

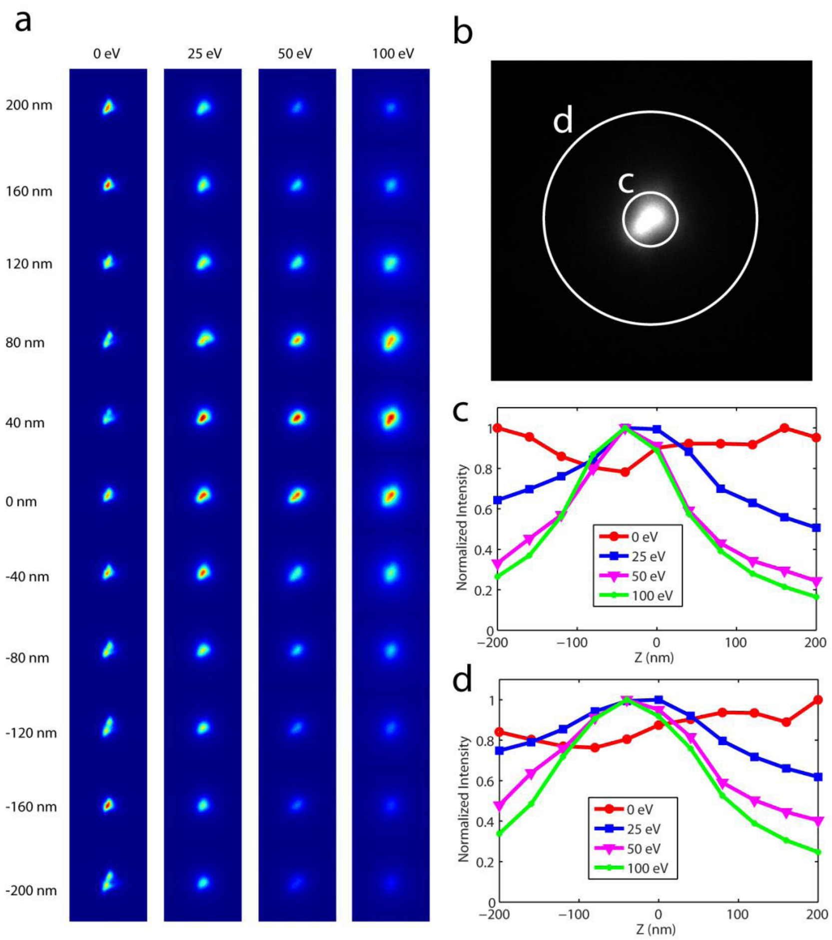

FIG 2. The energy-loss-dependent images of the 300-keV probe in serial-SCEELM (energy-filtered mode) as the sample moves in and out of focus. (a) The images of the probe as a function of the stage *z* position and the energy loss. (b) The schematics showing the two chosen sizes of the digital pinholes. (c and d) The energy loss dependent depth profiles of the carbon film.

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