

Development of a High Electron Energy-loss Spectrometry System for Advanced Scanning Transmission Electron Microscopy

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Electron energy loss spectrometry (EELS) in the scanning transmission electron microscope (S/TEM) has become an essential tool for materials characterization and been widely used both in academia and industries. In addition to elemental identification and quantification, the EELS-based approach can provide insight on chemical bonding status [e.g. 1], which is similar information to that generated by synchrotron-based X-ray absorption spectroscopy (XAS) available at several Department of Energy National Labs. In fact, electrons can interact with matters more strongly than X-ray, materials information especially at high spatial resolution can be obtained more efficiently by EELS in combination with the latest aberration-corrected S/TEM. The energy-loss range obtainable in current EELS systems is typically up to ~3,000 eV, which is still very useful for the analysis of light elements such as carbon and oxygen through their K ionization edges and certain 3d transition elements via their L-edges. However, this limited energy-loss range prohibits the detection of energy-loss characteristic signals from some important heavy elements such as gold, platinum, iridium and tungsten, which can only be provided by synchrotron-based XAS with relatively broader spatial resolution. The limited energy-loss range is mainly due to the extremely poor signal generation at higher energy-loss regions and to lack of appropriate electron optics inducing undesired artifacts in spectrometry, etc. Therefore, we start developing a new high electron energy-loss spectrometry system for advanced S/TEM in collaboration with JEOL (USA and Japan) and CEOS (Germany). In this study, we report initial results of the collaborative project.

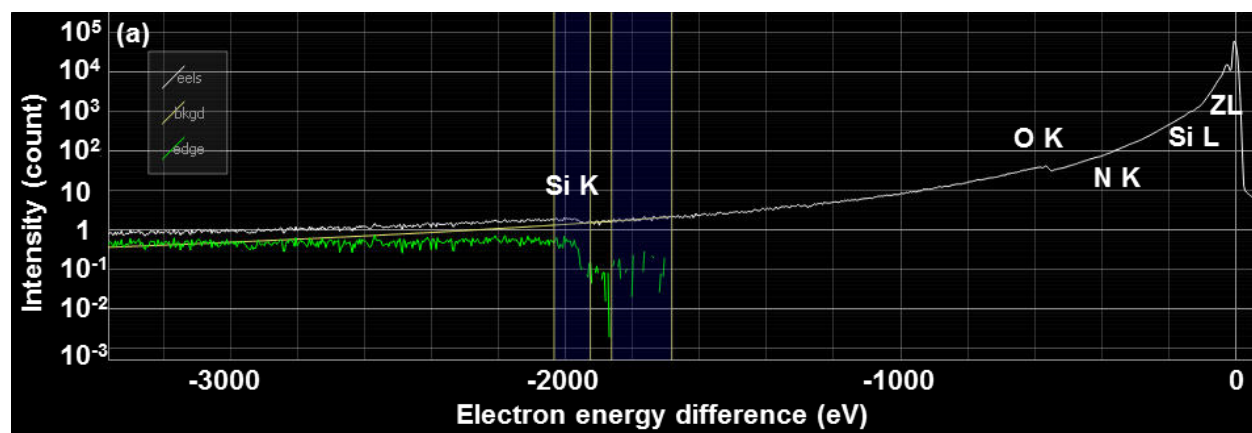
The high energy-loss EELS spectrometry system is based on a new EELS spectrometer CEOS Energy Filtering and Imaging Device (CEFID) [2], in combination with two electron detectors: (1) a commercially available CMOS-based CCD camera TVIPS TemCam-XF416 and (2) a highly sensitive electron counting detector Dectris ELA. The system control and tuning of the CEFID can be performed through the CEOS Panta Rhei platform as well as data acquisition of both STEM and TEM images, and of EELS spectra including spectrum-imaging (SI) datasets.

A set of initial results obtained by STEM-EELS SI from a semiconductor device is shown in Fig. 1. The SI dataset was acquired with 96x96 pixels and 1028 channels for a dwell time of 50 ms (~8 min. for the total acquisition) at 200 keV by using the ELA detector in the CEFID system. Figure 1(a) shows an EELS spectrum in a log scale extracted from a single pixel in the SI dataset. The detector has a very impressive dynamic range, which allows us to acquire the highly intense zero-loss peak and the very weak Si K edge signals at ~1800 eV simultaneously. Furthermore, the very weak Si K edge signals can be acquired for such a short acquisition, 50 ms per pixel, in this system. A zero-loss image and extracted elemental maps including Si L and K edges are shown in Fig.1(b)-(g), respectively. There is discrepancy in contrast between the Si L and Si K edge maps, which is caused by improper background subtraction

for the Si L edge map (at 99 eV) due to complex features (presence of W O and Cu L edges, and plasmon peaks caused by multiple scattering) at the lower loss region. Thus, the Si K edge is a good selection for elemental distribution mapping, which can be acquired with the intense zero-loss signal simultaneously using this new system. It is straightforward to convert those elemental maps to more quantitative atomic density maps in combination with the low-loss signals including the zero-loss peak.

Another SI dataset was acquired from the boxed region shown in Fig. 1(g), the Ti L map. In this acquisition, the spectrometer bias was shifted to ~ 2000 eV in order to record energy-loss signals ranging 3000–6000 eV and the SI dataset was acquired with 26x86 pixels and 1028 channels for the dwell time of 300 ms per pixel. Figure 2(a) shows an EELS spectrum extracted from the SI dataset (4x10 pixel average). Since this is the first attempt to simply shift the energy-loss without any system tuning or optimization, there are some artifacts in the spectrum. However, the Ti K edge is clearly visible (ideally the edge should be located at ~ 5000 eV). The Ti K edge map was extracted from the SI dataset without applying any signal processing but a normal power-law background subtraction, which is shown in Fig. 2(b). According to elemental maps shown in Fig. 1, this Ti enriched part is not pure Ti but a TiN layer. So, it is very impressive to acquire the Ti K edge map for such a short dwell time from the Ti compound layer.

We are currently optimizing the spectrometer settings for higher energy-loss signal acquisition without changing the S/TEM accelerating voltage. This new high energy-loss spectrometry system would effectively transform the advanced analytical electron microscope, making it into the equivalent of a synchrotron XAS facility but with improved spatial resolution [3].



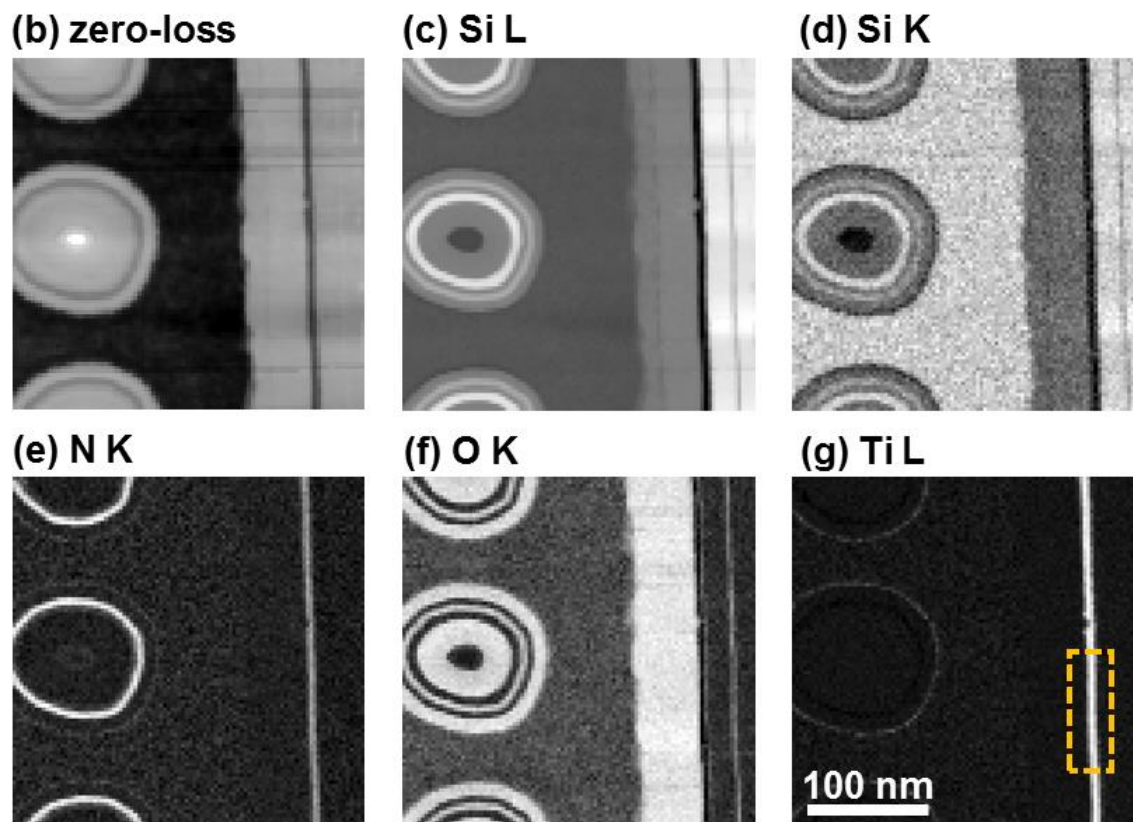


Figure 1: A set of results of a STEM-EELS SI dataset acquired from a semiconductor device specimen by using the CEOS CEFID system in combination with the Dectris ELA detector mounted under a JEOL JEM-ARM200CF instrument at CEOS. (a) An EELS spectrum in a log scale extracted from a single pixel, and signal maps of (b) zero-loss, (c) Si L, (d) Si K, (e) N K, (f) O K and (g) Ti L.

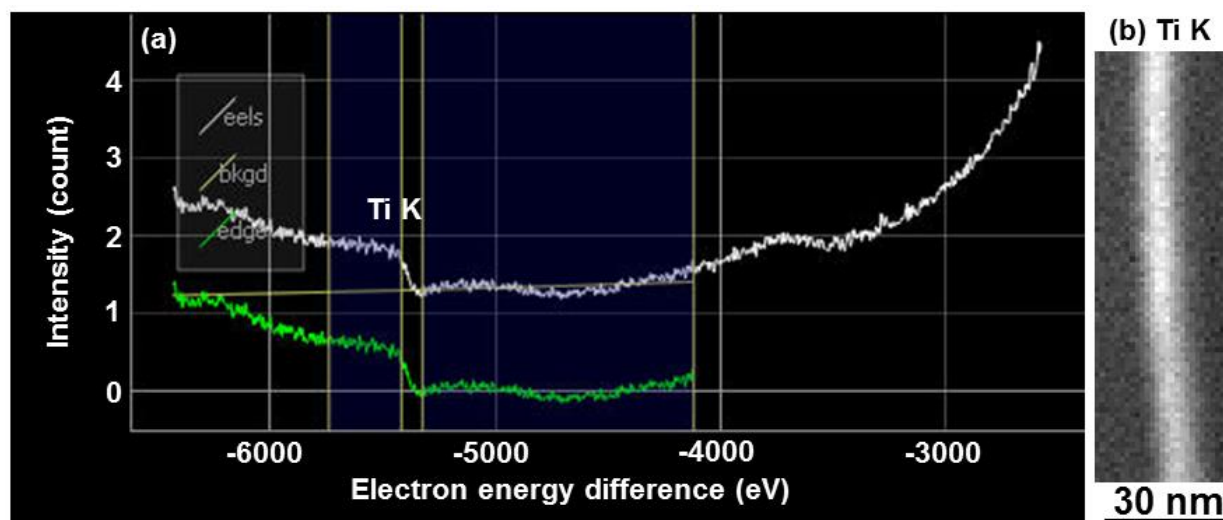


Figure 2: A set of results of a STEM-EELS SI dataset acquired in the vicinity of the TiN layer, (boxed region in Fig. 1g). (a) a high energy-loss EELS spectrum showing the Ti K edge and (b) extracted Ti K edge map.

References:

- [1] RF Egerton in “Electron Energy-Loss Spectroscopy in the Electron Microscope”, 3rd ed, (Springer, NY).
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- [3] The authors wish to acknowledge financial support from the NSF through grants DMR-2018683 and CMMI-2016279.