Microscopy and Microanalysis, **29** (Suppl 1), 2023, 416–417
https://doi.org/10.1093/micmic/ozad067.196

Microscopy AND Microanalysis

Meeting-report

Installation of New Systems for High-Energy Electron Energy-loss Spectrometry in an Aberration-Corrected Scanning Transmission Electron Microscope

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Installation of very new hardware and/or software systems is one of the extremely challenging (and sometimes stressful) tasks not only for users but also for vendors especially if such systems are installed first time after the development. At Lehigh, we had installed the first aberration-corrected scanning/transmission electron microscope (S/TEM) JEOL JEM-2200FS in the US, early 2000s. Recently a newly developed electron energy-loss spectrometry (EELS) system for higher energy (aiming to enhance to 13,000 eV energy-loss range from conventional ~3,000 eV) has been installed first time to an existing aberration-corrected S/TEM JEOL JEM-ARM200CF together with a new scanning generator called MDP. The system development and installation were performed in collaboration with JEOL (USA and Japan) and CEOS (Germany). In this presentation, we will share our experiences learned through this installation to achieve the high energy EELS signals.

The high energy EELS system is based on a new EELS spectrometer CEOS Energy Filtering and Imaging Device (CEFID) [1], in combination with two electron detectors: (1) a commercially available CMOS-based CCD camera TVIPS TemCam-XF416 and (2) a highly sensitive hybrid-pixel electron detector Dectris ELA. The CEFID system can be controlled and tuned through the CEOS Panta Rhei platform, as well as data acquisition of both STEM and TEM images. In addition to the Panta Rhei platform, imaging and spectrometry not only via EELS but also via X-ray energy dispersive spectrometry (XEDS) can be performed in a newly developed JEOL FEMTUS platform.

Figure 1(a) shows a 2D distribution of energy-loss electrons at the energy-dispersion plane in the 3,000-eV view mode, as our initial attempt to acquire higher energy-loss signals. The horizontal field of view indicates a ~3,000 eV energy-loss range and the Ti K edge is visible at ~5,000 eV with a certain energy shift controlled by the liner (drift) tube voltage of the spectrometer. Although energy-loss electrons should appear as a straight band with parallel edges at the energy-dispersion plane, the energy-loss band is no longer parallel around the Ti K edge. In addition, there are several strange lines which could be due to unexpected electron reflections somewhere between the project lens of the instrument and the liner tube of the spectrometer. In order to improve the energy-loss dispersion at higher energy, (1) the spectrometer optics including multipole lenses before and after the liner tube were optimized and (2) an energy-selective beam blocker was inserted in the spectrometer to reduce the unexpected electron reflections. Figure 1(b) shows a 2D-dispersed energy-loss distribution at the dispersion plane in the 6,000-eV view mode acquired after these improvements. The energy-loss electrons appear as a parallel band in the whole dispersion range (6,000 eV) and both the zero-loss peak and Ti K edge can be seen in one spectrum without stitching. It should be noted that the intense line indicated as the extraction voltage is caused by secondary electrons generated at the electron gun area after the first anode for the electron extraction, i.e. the extraction voltage. Furthermore, (3) the post objective-lens optics of the microscope was also optimized for the higher energy-loss detection by tuning the focal point of the projector lens [2].

Figure 2 shows an EELS spectrum acquired from a NiO thin-film specimen in the 6,000eV-mode with a 4,000-eV shift via the liner tube voltage and a 2,000-eV shift via the high-tension. Although the intense peak appears at 7.7 keV due to the secondary electrons generated after the gun-lens excitation, the Ni K edge appears at ~8,300 eV. The spectrometer system as well as the post objective lenses of the microscope are still under optimization for acquisition of higher energy-loss signals. Once those optimizations are completed, however, the 200-keV S/TEM with this spectrometer could produce similar information obtainable by X-ray absorption spectrometry in a synchrotron facility but with improved spatial resolution [3].

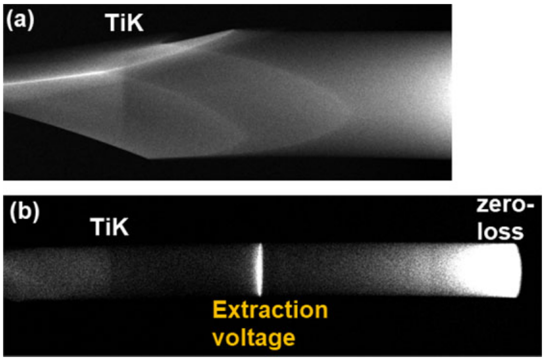

Fig. 1. (a) A 2D-dispersed energy-loss distribution at the dispersion plane in the 3,000-eV view mode, taken by the CEFID system prior to optimizing the system. (b) Another 2D-dispersed energy-loss distribution at the dispersion plane in the 6,000-eV view mode, taken after the spectrometer tuning.

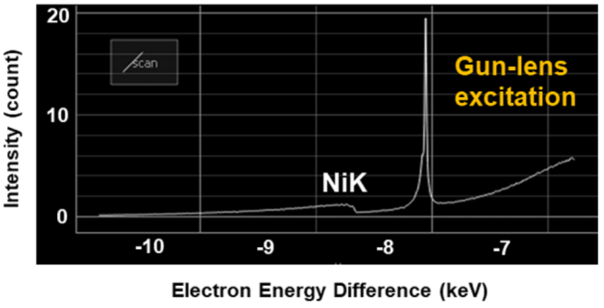

Fig. 2. An EELS spectrum acquired from a NiO thin-film specimen in the 6,000eV-mode with a 4,000-eV shift via the liner tube voltage and a 2,000-eV shift via the high-tension.

References

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3. The authors wish to acknowledge financial support from the NSF through grants DMR-2018683 and CMMI-2016279.