Super-X: Characterization of a new generation EDXS detector

S Fladischer[1], W Grogger[1] and F Hofer[1].

1. Institute for Electron Microscopy and Fine Structure Research & Graz Centre for Electron Microscopy, Graz University of Technology, Graz, Austria.

stefanie.fladischer@felmi-zfe.at

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In recent years Energy-Dispersive X-ray Spectrometry (EDXS) in Transmission Electron Microscopy (TEM) was significantly improved by the introduction of the novel ChemiSTEM technology [1]. At our institute the ChemiSTEM technology is implemented to a Titan³ and comprises a high-brightness Schottky field emission source (X-FEG) in combination with a new EDXS detector system (Super-X design). 4 Silicon Drift Detectors (SDDs) in a windowless design are symmetrically placed around the specimen leading a substantial bigger collection angle and higher detection efficiency.

In this work the performance of the Super-X detector is compared to the performance of a Si(Li) detector attached to a Tecnai F20. In contrast to the Super-X detector, the Si(Li) detector is equipped with an ultrathin polymer window (AP3.3 window from Moxtek). For the following investigations the Tecnai F20 was operated at one single high tension, 200 kV, whereas different high tensions were used at the Titan³, 80 kV, 200 kV and 300 kV.

For the characterization of the different EDXS-TEM systems with respect to energy resolution, signal-to-noise-ratio, stray radiation, collection angle and relative detector efficiency a NiOx specimen was analyzed as described in Ref [2] using a DigitalMicrograph plug-in [3]. EDX spectra were acquired for each system for alpha and beta tilt angle series and for different beam currents. In Figure 1 EDX spectra of a NiOx specimen are shown on the one hand acquired with the Si(Li) detector and on the other hand with the Super-X detector at 80 kV and 300 kV. A beam current of 0,5 nA was used, a specimen area of 0,5 µm² was illuminated and an acquisition time of 100 s was chosen in all three cases. It is observed that the Super-X detector with its bigger collection angle and thus higher collection efficiency acquires an order of magnitude more counts than the Si(Li) detector. Especially the detection efficiency in the low energy region is improved resulting from the windowless design, which favours light element detection. Furthermore, by comparing the spectra of 80 kV and 300 kV the more efficient X-ray generation at lower high tensions can be seen. Additionally, stray radiation of the Mo grid, the Al specimen fixation tool of the high visibility holder in the Titan, and contributions of the pole piece (Fe, Co) are visible.

To determine the detection sensitivity of above mentioned EDXS-TEM systems the NIST SRM 611 standard containing 500 ppm of 61 elements (Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, As, etc.) embedded in glass was used. EDX spectra were acquired for different specimen thicknesses, beam currents and acquisition times. In Figure 2 EDX spectra of the NIST standard are shown for the Si(Li) detector and the Super-X detector for acquisition times of 600 s and 300 s respectively. For all three spectra the beam currents were 1 nA, the specimen thicknesses were about 190 nm and the illuminated specimen areas were 0,1 µm². The trace elements are detectable in all three spectra. Unfortunately different stray radiations superimpose certain trace elements. Using the same acquisition parameters for the different EDXS-TEM systems the measured intensities for vanadium are 2 cps for the Si(Li) detector and for the Super-X 13 cps and 21 cps for 300 kV and 80 kV respectively. Obviously, the detection sensitivity of the Super-X is significantly higher and trace elements can be detected in much shorter times.

In this work the improvements of EDXS systems for TEMs are shown. New possibilities for EDXS are established with the Super-X detector.

References

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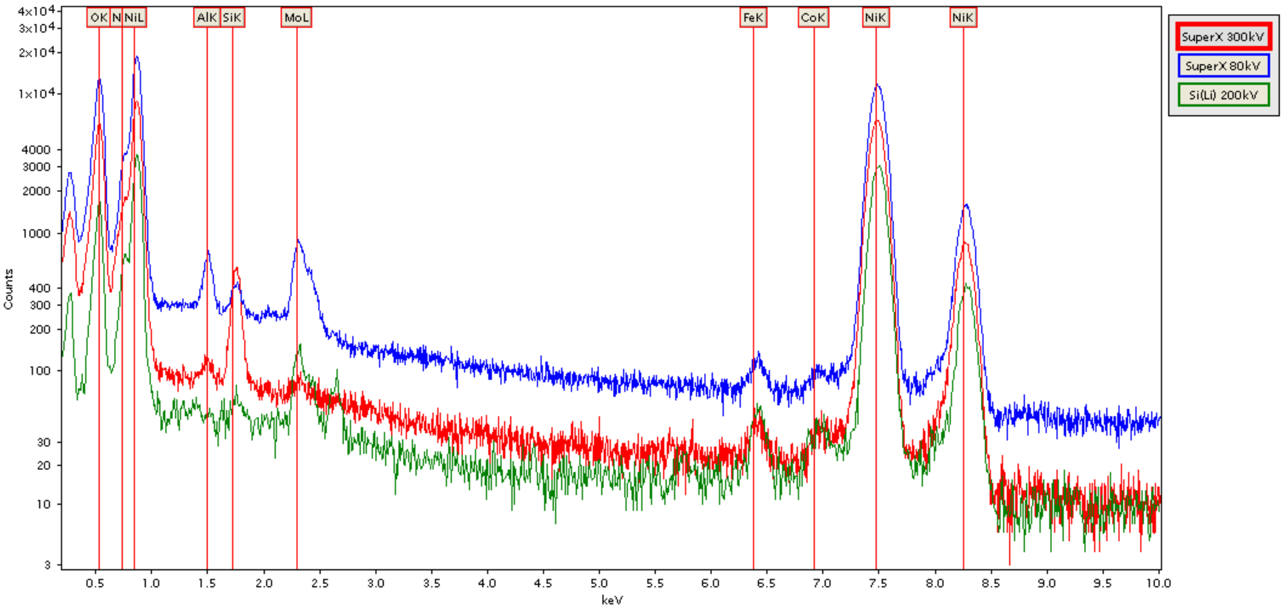

Figure 1. *EDX spectra of a NiOx specimen acquired with the Si(Li) detector (green), the Super-X detector at 80 kV (blue) and 300 kV (red) in logarithmic scale.*

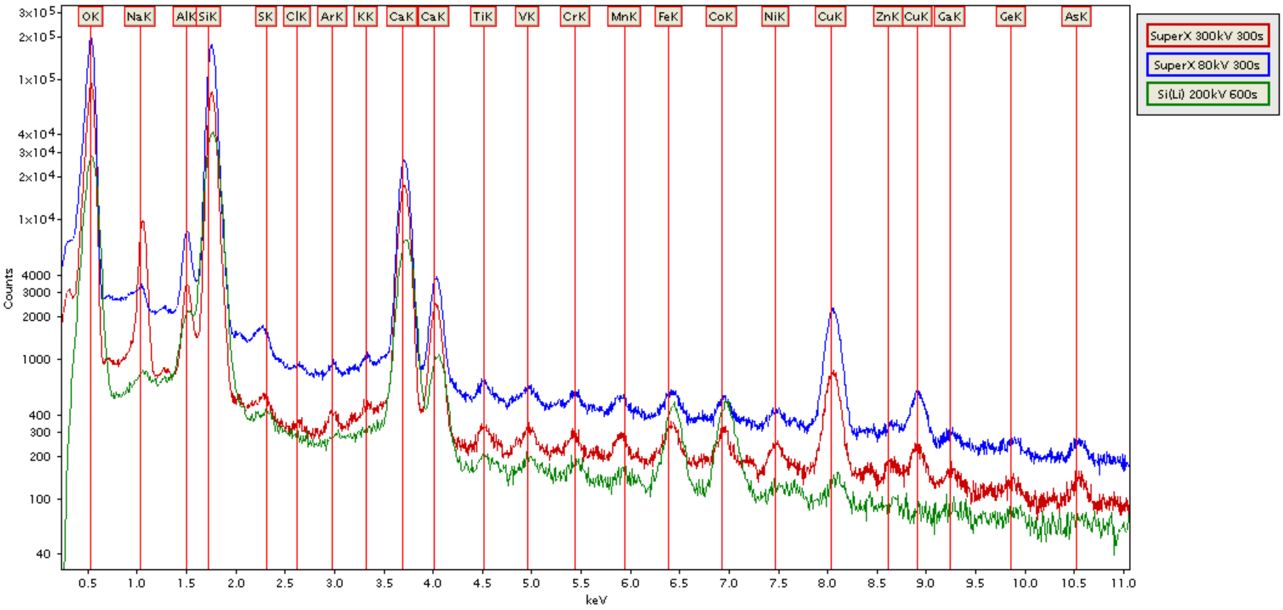

Figure 2. *EDX spectra of the NIST standard acquired with the Si(Li) detector (green) with an acquisition time of 600s, and the Super-X detector at 80 kV (blue) and 300 kV (red) with an acquisition time of 300s in logarithmic scale.*