

## On the Performance of XEDS and EELS in the AEM: 25 Years Later

H.L. Fraser<sup>1</sup>, D.O. Klenov<sup>2</sup>, Y.C. Wang<sup>3</sup>, H. Cheng<sup>3</sup>, N. J. Zaluzec<sup>4</sup>

<sup>1</sup>Dept of Materials Science & CAMM, The Ohio State University, Columbus OH

<sup>2</sup>FEI Company, Achtseweg Noord 5, 5600 KA Eindhoven, The Netherlands

<sup>3</sup>FEI Company, 5320 NE Dawsons Creek Dr., Hillsboro, OR 97124

<sup>4</sup>Electron Microscopy Center, Mat. Science Div, Argonne National Laboratory, Argonne IL

Traditionally, electron energy loss spectroscopy (EELS) is viewed as one of the most efficient means for microanalytical measurements in the analytical electron microscope. This perspective is based not only upon the physics of the signal generation process but also due to the relatively high geometrical signal collection efficiency of EELS relative to that used in conventional x-ray energy dispersive spectrometer (XEDS) geometries. A comparison study of XEDS and EELS was conducted in 1984 which considered the merits of the two spectroscopies for light and medium atomic number materials [1]. Recent developments in silicon drift detectors (SDDs) have facilitated the construction of large solid angle x-ray spectrometers which when combined with the current generation of electron-optical instruments significantly impacts the performance of these analytical technologies [2-3]. In this work we revisit that original study to appraise the changes which have occurred in the intervening 25+ years, paying attention to their application in practical rather than ideal conditions.

To compare the merits of these two spectroscopies, data sets were measured as a function of thickness using two state-of-the-art spectrometer systems on a FEI Tecnai Osiris system. This instrument was equipped with a Super-X silicon drift x-ray detector system having a variable collection solid angle of up to 0.9 sr, as well as a post column high performance FS-1 EEL spectrometer. The specimens studied include: crushed BN, ion-milled stoichiometric NiO, ion-milled MgAl<sub>2</sub>O<sub>4</sub> and electropolished 304 stainless steel. All specimens were mounted in a low background double tilt Be analytical TEM stage and were tilted to achieve non-channelling conditions. Plasma cleaning was used as appropriate to mitigate any hydrocarbon contamination [4]. Measurements were made at 200 kV in STEM mode using a nominal probe diameter of 1 nm with probe currents which varied depending upon the measurement from ~ 0.1 nA to 1.5 nA. For EELS the collection angle ( $\beta$ ) was chosen to be greater than  $\theta_E = \delta E / 2E_0$  for the highest energy edge measured, while the incident angle  $\alpha \sim \beta/2$ . Data acquisition was varied over the range of 10 msec to 10 sec/point depending upon the spectral features being studied. In all cases, XEDS and EELS spectra were acquired simultaneously and for identical acquisition times to assure relevant comparisons. Figure 1 shows a limited selection of the compendium of XEDS/EELS spectra measured as a function of thickness expressed in units of  $t/\lambda$ . Here  $t/\lambda$  was measured using low loss EELS [5].

Thicknesses ranging from  $\sim 0.2 t/\lambda$  to  $\geq 1.5 t/\lambda$  were specifically targeted so as to test the performance in specimens of interest to typical materials science investigations. As expected as specimen thickness is increased all signal levels correspondingly increase. In order to permit comparison, both the XEDS and EELS data shown in Figure 1 have been normalized to the background intensity in their respective spectra. Although this visually suppresses the magnitude of the raw intensity change, instead, this normalization best demonstrates the effects of thickness on sensitivity of the spectral data, which is revealed by the peak (or edge) to background ratio. In EELS, this is manifest by the rapid onset of multiple scattering and its increasingly significant contribution to the background, while in XEDS, it is evidenced by onset of x-ray absorption for the lower energy lines. Both of these trends can be seen by inspection of the individual spectral profiles of Figure 1. Figure 2 and 3 expand on this and plots the variation in the measured Edge/Background (EELS) and Peak/Background (XEDS) for the BN and NiO samples, respectively.

Similar trends were obtained for MgAlO and 304 SS specimens but are omitted here due to space considerations. These figures illustrate the strong degradation of the Edge/Background for EELS due to multiple inelastic scattering, as well as the differential absorption of low energy x-rays. They

also demonstrate that when an optimized x-ray detector technology is employed that the sensitivity of XEDS, as manifest in the Peak/Background ratio not only competes, but for real world samples, can outperform EELS, particularly when  $t/\lambda \geq 0.3$  for higher atomic number materials. EELS on the other hand still surpasses XEDS in energy resolution and for near edge and electronic structure studies. We have not in this work addressed the comparative performance of the two techniques regarding spatial resolution, which will be the subject of a follow up study.

References

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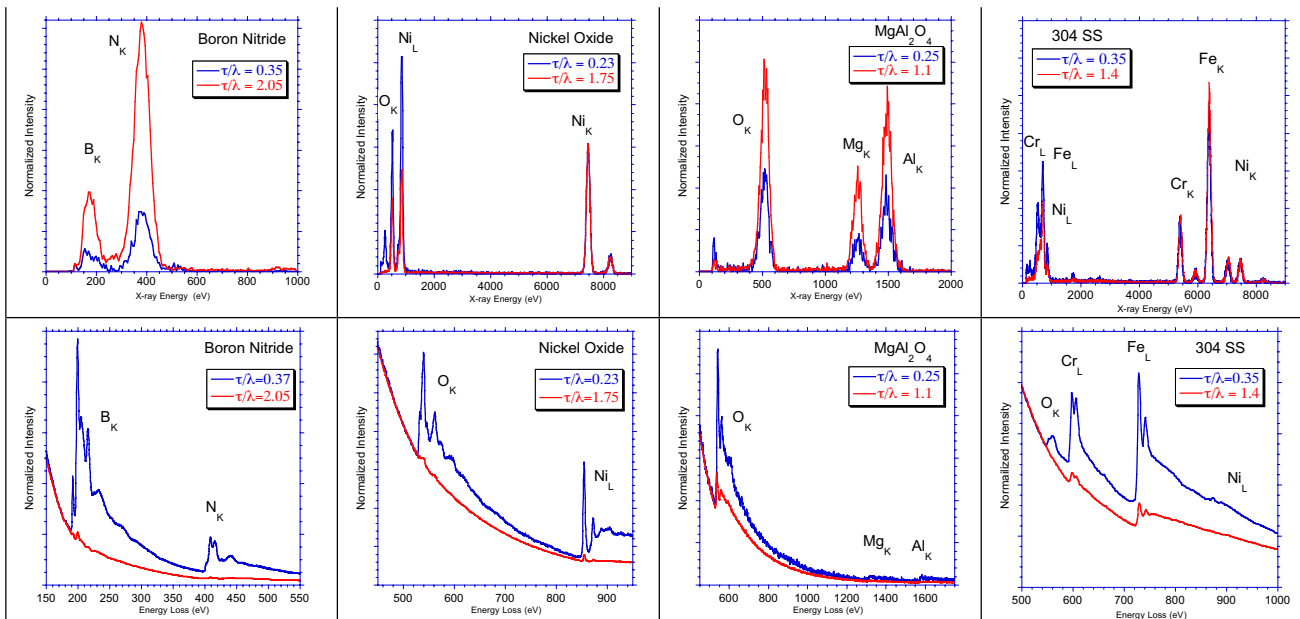


Fig. 1 Comparison of Normalized XEDS and EELS spectra recorded simultaneously for BN, NiO, MgAl<sub>2</sub>O<sub>4</sub> and 304 Stainless Steel. Normalization was to background intensity at a region of interest near the highest intensity edge/peak

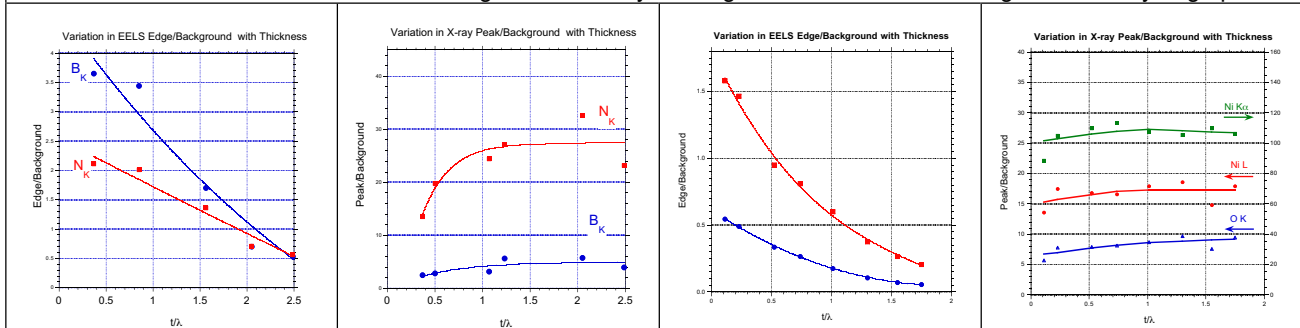


Fig. 2 Experimental measurements for BN vs  $t/\lambda$ , the integration windows for Edges, Peaks & Bg were identical for the respective data sets.

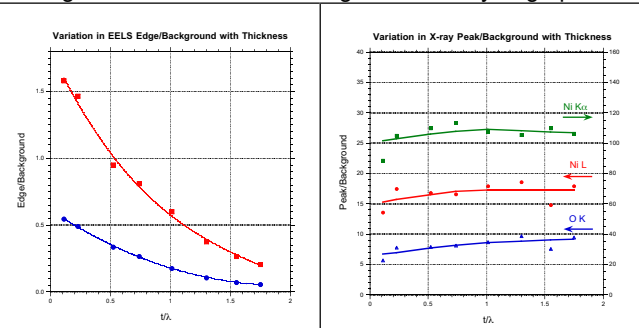


Fig. 3 Experimental measurements for NiO vs  $t/\lambda$ , the integration windows for Edges, Peaks & Bg were identical for the respective data sets.