

Comparison of the detection limits of EDS and EELS in S/TEM

H. Sebastian von Harrach¹, Dmitri O. Klenov¹, Bert Freitag¹, Peter Schlossmacher¹, Peter C. Collins², Hamish L. Fraser³

¹FEI Company, Achtseweg Noord 5, 5600 KA Eindhoven, The Netherlands

²Quad City Manufacturing Laboratory, Rock Island, IL 61201

³Department of Materials Science and Engineering, Ohio State University, OH 43210

The detection limit for analytical techniques has been the subject of many publications. [1] The most general approach [2] is to define the detection limit at 99% confidence limit of detecting a minor element as $I_p - I_b > 3 * \text{SQRT}(2 * I_b)$ where I_p is the total intensity in an energy window for a given element and I_b is the background intensity under the same window. The ionization cross-section of a given element determines the number of inner-shell energy loss events in both EDS and EELS, but there are 3 major differences:

1. the X-ray production for an inner-shell transition competes with the generation of Auger electrons, giving rise to a Z-dependent fluorescence yield factor ($\omega < 1$)
2. the detection efficiency of EELS systems is generally much higher (up to 80% at low count rates) than for EDS systems ($< 1\%$)
3. the peak-to-background ratios ($P/B = I_p - I_b / I_b$) for EELS are generally much lower for EELS than EDS, especially near the detection limit.

These differences make EELS a much more sensitive technique for light elements and EDS for heavy elements. However, with the advent of new detector technology it has been possible to improve the EDS detection efficiency by an order of magnitude [3]. This paper re-examines the detection limits of the two techniques under identical conditions in an FEI 200kV S/TEM equipped with a Super-XTM EDS detector with 0.9 sr collection angle and an EELS spectrometer. The detection efficiency (as a %age of 4π) of the Super-X is compared with Si(Li) detectors in Fig.1; the high efficiency for low X-ray energy (light elements) is due to the absence of a window in front of the detector. The dead time is also much lower for Super-X at the same energy resolution (Fig.1 right)

Fig.2 shows simultaneous spectra acquired on a titanium alloy (0.5%Fe, 3%Cr, 5%V, 5%Mo by wt.) of medium specimen thickness, containing Fe-rich precipitates. All the minor elements in the alloy are visible in the EDS spectra, but not in EELS. The P/B ratio of Ti is 35 in EDS compared to ~ 3 in EELS; for Fe in the precipitates the P/B ratios are 3 for EDS and ~ 0.01 for EELS. In Fig.3 a line scan from the edge of the specimen shows the specimen thickness t varying from about 0.1 to 0.8 times the inelastic mean free path MFP (λ). In the thin regions ($t < 0.3 \lambda$) both the EDS and the EELS spectra show iron signals in the precipitates, but the P/B ratio is very low ($P/B < 0.01$) in the EELS case, making accurate quantifications impossible. For a relatively thick ($t \sim 0.8 \lambda$) region the Fe peak is not visible in EELS signals due to multiple scattering, but is strong in the EDS spectra. The detection limit for EDS is therefore better than for EELS at all specimen thicknesses for all the elements in this alloy.

Further examples will be given for a wide range of Z numbers, for example on a chromium doped TiB₂ WB alloy, for which the detection limits have been determined by both techniques.

References

- [1] J.I. Goldstein, D.B. Williams, G. Cliff in *Principles of AEM*, eds D.C Joy, A.D. Romig, J.I. Goldstein, Plenum Press, New York, 1986, ch.5
- [2] L. Reimer, *Scanning Electron Microscopy*, Springer New York, 1998, p.437.
- [3] H.S. von Harrach et al., *Microsc. Microanal.* 15 (Suppl.2) (2009) 208.

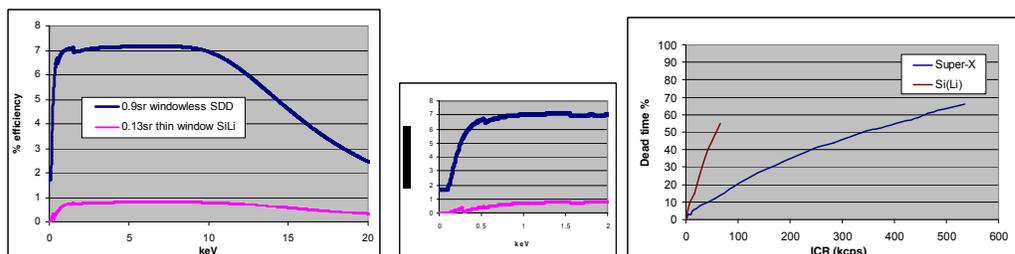


Fig. 1. Left: % detection efficiency of windowless SDD and thin window 0.13sr Si(Li) EDS detectors vs. X-ray energy 0-20keV. Center: 0-2keV. Right: % dead time vs. input count rate ICR for both detectors at 135eV resolution

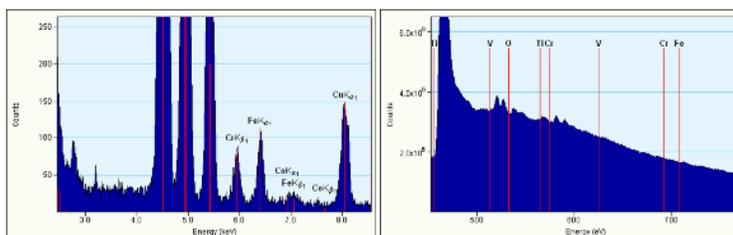


Figure 2 : EDS (left) and EELS spectra of a titanium alloy (0.5%Fe,3%Cr,5%V,5%Mo), thickness $t \sim 0.35 \lambda$, 20 s exposure time, 200kV, 20kcps count rate in EDS.

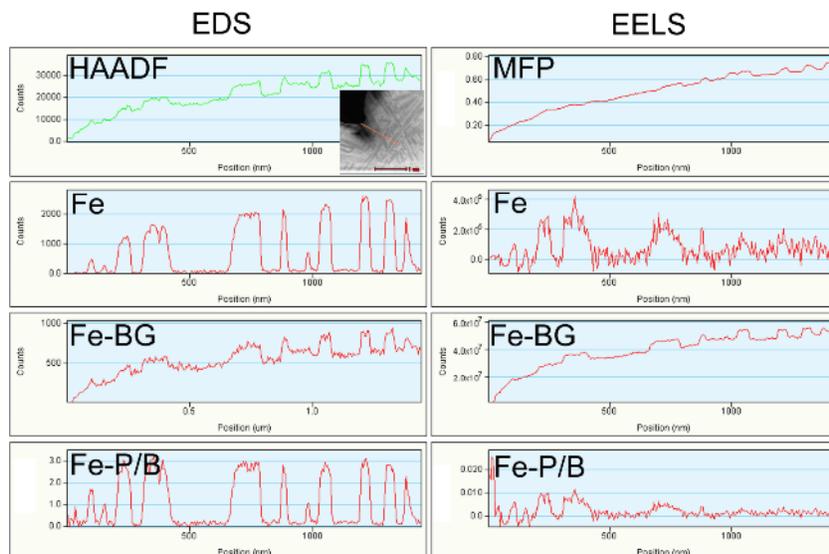


Figure 3 : Simultaneous EDS and EELS line scan across Ti alloy of 0 to 0.8 time MFP thickness showing the change in Fe signals in EDS/EELS. (top left:HAADF image)