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Experiencies and Possibilities with a 200 kV Monochromated (S)TEM

G. Kothleitner, W. Grogger and F. Hofer

Research Institute f. Electron Microscopy (FELMI), Graz University of Technology
Steyrergasse 17, A-8010 Graz, Austria

The energy resolution in electron energy-loss spectra attainable with present-day analytical TEMs is typically limited to around 1 eV, preventing detailed information about the electronic structure of materials to be obtained. For comparison, synchrotron x-ray sources and beam line spectrometers provide a resolution well below 0.1 eV for absorption spectroscopy. The main limitations in TEM EELS stem from the instability in the high tension, the insufficient aberration correction of the spectrometer and the natural energy width of the electron source. To go beyond, FEI / Gatan have recently commercially introduced a monochromated 200 kV (S)TEM aiming at an energy resolution level for EELS which is roughly a factor of 5-10 better [1,2]. The much better energy resolution in this instrument was in part due to improvements in the high voltage supply, the more stable electronics and improved electron optics of the spectrometer but mostly because of the incorporation of a Wien filter monochromator positioned directly after the field emission gun. Improved spectral resolution reveals many more effects that cannot be predicted as precisely as they can be measured and opens up new possibilities in both the analysis of low loss and core loss spectra.

The smaller energy spread of the zero-loss peak is particularly significant for measurements in the low loss region. One problem for thin insulators or semiconductor samples in the TEM is the fact that the dominating elastic signal strongly extends into the energy region of the bandgap (1-10 eV), forming a background that can hardly be estimated and extracted. Furthermore the reliability in the determination of optical properties via Kramers-Kronig analysis can be improved when the intensities are more accurately known [3]. Figure 1 displays two area normalized zero-loss peaks with the monochromator switched on and off, showing the advantage of a narrower zero-loss profile when subtracting low loss intensities.

In core-loss spectroscopy it is well known that the line width of ELNES features are further influenced by intrinsic effects like core hole lifetime broadening and the broadening caused by the finite lifetime of the excited state. First monochromator measurements on selected transition metal oxides [4] confirmed that O-K edges are intrinsically broader compared to L_3-edges, for which the core level widths lie in the range of 0.2-0.4 eV. The observed $2p_{1/2}$ level (L_2) widths on the other hand are significantly larger, which is likely to be due to rapid $L_2L_3M_{45}$ decay processes (Coster-Kronig broadening), rising from Ti to Co. The final state lifetime is less well characterized, but various approaches [5,6,7] predict the damping of the DOS to be within 2 and 6 eV for the first 20 eV. The model followed by Muller et al. is based on experimental measurements of the dielectric function determined from the EELS valence spectra whereas Schattschneider et al [priv. comm.] make use of momentum resolved measurements of the loss function.

First experiences, tests and measurements on material science specimens will be presented to illustrate that with the improved EELS resolution a better understanding of the chemical and electronic properties can be obtained.

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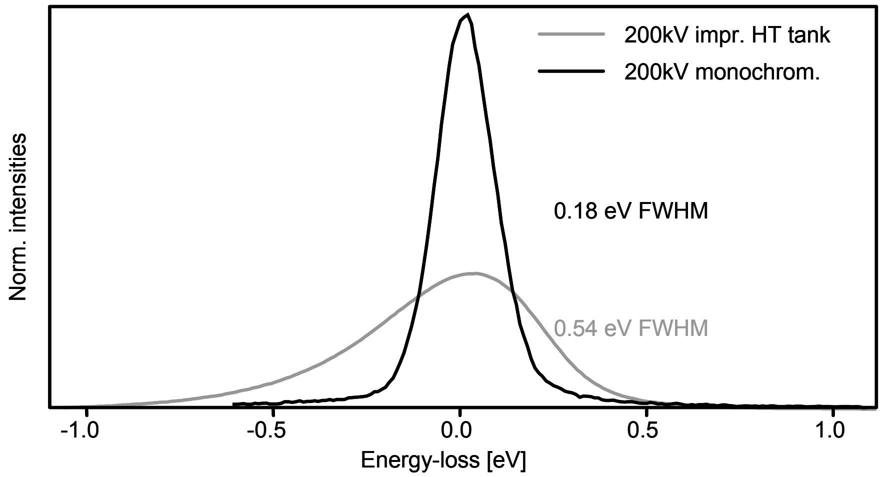

Fig. 1: Comparison of two zero-loss peaks taken with the WIEN filter monochromator switched on and off on a Tecnai T20F as installed on the FELMI. Both spectra have been taken at 200kV, with a gun lens setting of 1kV, an extraction voltage of 3.4 kV and with a 2mm entrance aperture during an exposure time of 1s. The peak areas are normalized and the graph clearly reveals the difficulties associated with the accurate extraction of inelastic intensities at low energy loss.

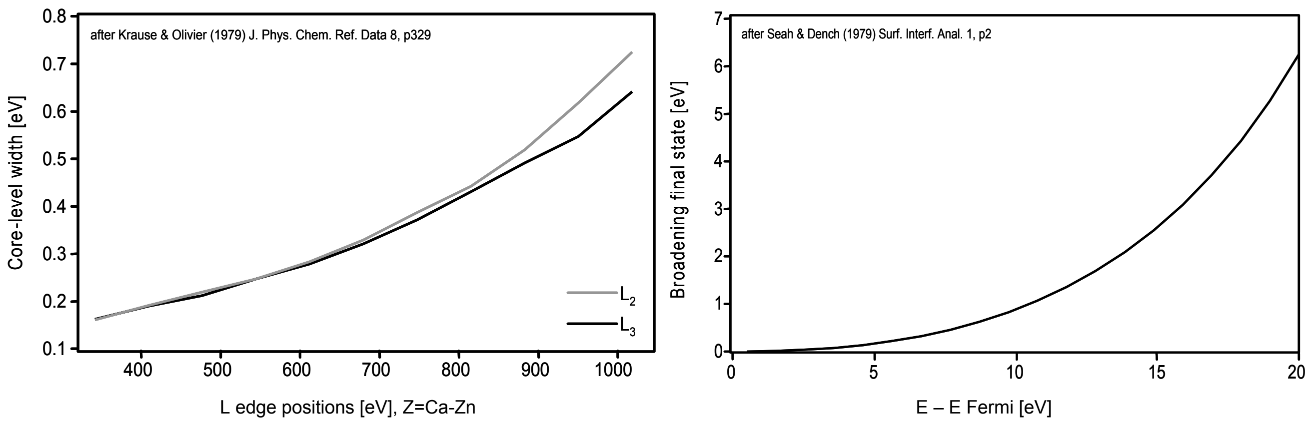

Fig 2: Left) Core level widths as a function of L edge positions for the transition metals after Krause & Olivier (1979). Preliminary measurements seem to indicate that L_2 widths are largely underestimated. Right) Final state broadening estimation based on inelastic mean free paths from Seah and Dench (1979)

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