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INTERMEDIATE VOLTAGE AEM'S: HAVE THEY FULFILLED THEIR POTENTIALS?*

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INTERMEDIATE VOLTAGE AEM'S: HAVE THEY FULFILLED THEIR POTENTIALS ?

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For the last few years analytical developments in intermediate voltage AEM's have remained relatively quiescent. We have instead seen a number of improvements in the basic microscope mainly along the lines of microprocessor controls, automation, and of course improvements image resolution and electron sources. Since the spectroscopic technologies (XEDS, EELS) have seemed to reach a mid-term plateau it is appropriate to consider whether or not we have achieved the expectations in these methodologies that we predicted nearly a decade ago.

In XEDS, there are three topics to consider: signal, sensitivity and quantification. In AEM's employing a conventional electron sources we expected that as a function of accelerating voltage (E_0) we should realize a continuous increase in intensity even though the ionization cross-section decreases with kV and this has been realized (figure 1). It occurs simply due to the fact that the increase in probe current with E_0 is more rapid than the more slowly decreasing ionization cross-section. In cold field emission gun (CFEG) systems where the probe current does not increase with kV, a decrease in signal with E_0 is instead observed. This decrease is in proportion to the variation in x-ray production cross-section, and hence it is generally not of major importance, as in FEG systems the probe current is more than sufficient to produce plentiful x-ray signal. In the most recent AEM's, the x-ray peak to background ratio, and hence the analytical sensitivity, increases with E_0 (figure 2) and reflects the fact that the manufacturers have made improvements to reduce the uncollimated background radiation generated in the illumination system. In the best cases factors of ~ 3-4 (relative to 100 kV) have been measured. There still remains the a few instruments which exhibit uncollimated fluorescing radiation, leading to a pronounced hole count, however, it is a problem which can be solved as the source and solution is well documented.[1] There have been no major breakthroughs in standardless quantification and the use of experimental "k-factors" is well established as the most accurate method for quantification. The next set of improvements is clearly in the area of x-ray detector technology, mainly in the areas of energy resolution and sold angle.

EELS has always suffered from two problems, contamination and thickness. While new technologies[2] exist to mitigate the contamination problem, the only way to overcome the multiple scattering issue is by increasing E_0 or decreasing the sample thickness. The effects on the visibility of a core loss edge in this situation is dramatic (figure 3). This is a direct result of the change in mean free path with E_0 (figure 4). There are, however, other compromises in increasing E_0, most noticeably, a decrease in the energy resolution of some EELS detector systems. While the decrease in resolution does not noticeably affect quantification methodologies, it does impact on using EELS to study the near edge structure (NES). As NES can provide insights into subtle processes this can be a result in a significant loss in capability.

Aside from the "hole count" problem in XEDS, and the loss in resolution in EELS, the next most vexing problem which will begin to limit AEM at intermediate voltages is electron sputtering[3]. This is most pronounced in field emission gun systems, but can be observe on any truly "clean" specimens. In this situation the focused electron probe readily "drills" holes in the specimen as illustrated in figure 5. Here a 300 kV, 1 nm, 1 nA probe was stepped across a grain boundary in 304 SS in 0.5 nm steps at 30sec/pt. The resulting trace shown in this figure is actually a sputtered trench where the electron beam has thinned the specimen. At the end of the track is a hole which was created by holding the probe stationary for ~ 120 sec. The sputtering effect is due to the lower bonding energy of atoms on a surface and with clean specimens and high current probes the effects can be both rapid and detrimental.

Have we reached the point we expected a decade ago? The answer is generally, yes. Were the gains worth the effort? That is still an open question in some analytical regimes. If the specimen is not limited by sputtering then the answer is certainly yes and the 200-400kV AEM does afford better performance and higher sensitivity. However as always as some limitations are overcome, others have taken their place.

References:
1.) N.J. Zaluzec, *Introduction to Analytical Electron Microscopy*, Plenum Press, (1979), Chptr. 4
2.) N.J. Zaluzec et al, "Reactive Gas Plasma .." Microscopy & Microanalysis -97 these proceedings.
3.) C.R. Bradley - Proc. of 46th EMSA Meeting, 1986 pgs 644-646
4.) This work was supported by US. DoE under BES-MS W-31-109-Eng-38 at ANL.

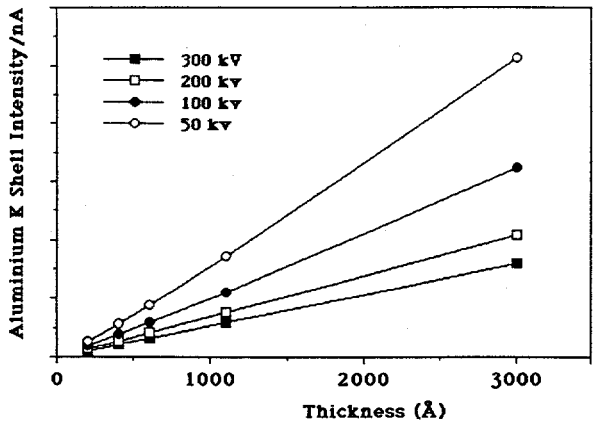

Figure 1.) Expt. variation in intensity of Al Kα as a function of accelerating voltage & thickness

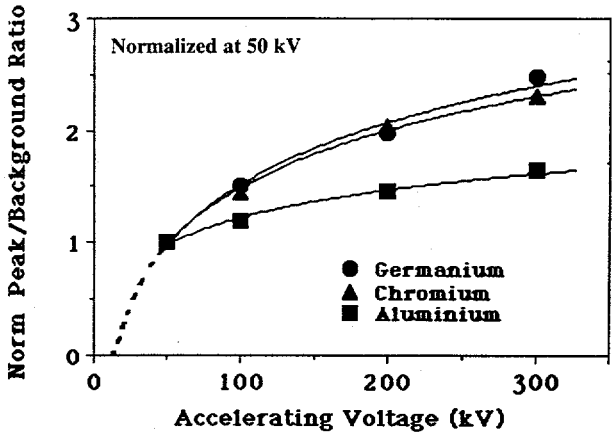

Figure 2.) Experimental P/B ratio in Al, Cr, and Ge as a function of accelerating voltage

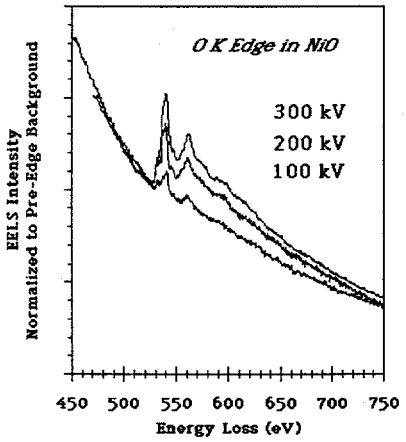

Figure 3.) Experimental change in O K EELS edge in NiO vs. accel. voltage

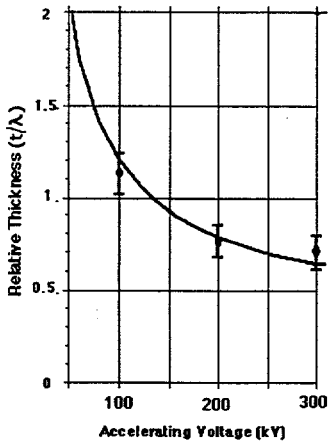

Figure 4.) Comparison of expt. and theoretical variation of mean free path vs accel. voltage

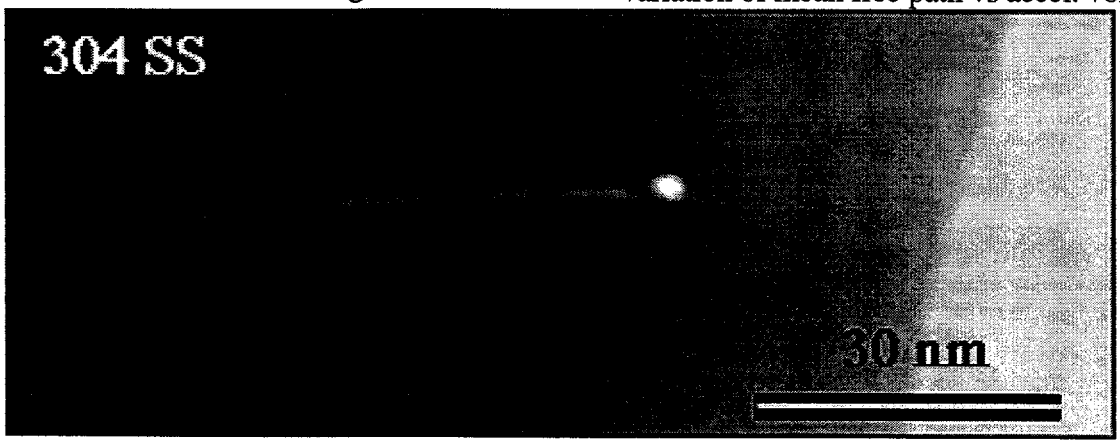

Figure 5.) Trench formed by electron sputtering in 304 SS by a 300 kV 1 nm, 1 nA electron probe.