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# An Assessment of the Pros and Cons of Low Voltage X-ray Analysis in the SEM

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Low voltage analysis is now widely used in many industrial applications. It has two main characteristics. The primary advantages (pros) derive from the strong sensitivity to voltage of the electron beam penetration range into the sample. The depth (Fig. 1) and spatial resolution (Fig. 2, 3) of analysis broadly follow the Bethe relationship where for a beam voltage $E_o$ the range R = $F(E_o)^{5/3}$. For the same x-ray line energy the improvement from 20kV to 5kV is about one order of magnitude (10x). The escape range of the x-rays through the sample is reduced by a similar amount; leading to a large reduction and in many, but not all, cases effectively elimination of the matrix absorption (A) and fluorescence (F) corrections. As a practical consequence light elements are generally more faithfully represented, and sometimes even overly so, in low voltage analyses (Fig.3). The downside is a similarly enhanced sensitivity of the analysis to surface coatings, oxides or contamination; although in practice this problem can be controlled quite well. For micron and sub-micron sized features the geometrical factors may dominate the corrections required for accurate analysis. Restricting the analysis volume to smaller features which are more likely to be single phase meets one of the prime criteria for accurate analysis of heterogenous materials and simplifies interpretation.

The primary disadvantages (cons) of low voltage analysis lie in the characteristics of the x-ray emissions. Firstly, the beam voltage must exceed that of the target x-ray line by at least a factor of 1.3x. This may require the selection of lower energy x-ray lines with different, less favorable and often less well known parameters. Secondly, even with the same x-ray line energy ($E_x$) the peak to background and kcps/nA sensitivity are reduced at lower beam voltages (Fig. 4). This is a fundamental limitation of the method. For a given series of x-ray lines, e.g. K, the problem can be partially generalized on the basis of overvoltage ($U = E_o/E_x$). The characteristics of the different types of x-ray detectors also play a role. A typical EDS system has an energy resolution and a P/B ratio inferior to a WDS system by a factor of ~10x. Although the practical EDS energy resolution improves at low x-ray line energies (e.g. ~60eV at 300eV vs ~130eV at 6kV) the opportunity for improvement with new types of detector combining improvements in both sensitivity and energy resolution is unquestionably substantial.

Complications arise when the two primary characteristics interact. An example is the sensitivity for analysis of small (sub-micron and even nm) surface particles as is typical in root cause analyses of process defects. At lower voltages the beam is more concentrated in the particle, by a mass factor of ~1000x in the previous example, but the P/B is at the same time reduced by a factor of ~10x; for a nett gain in sensitivity of ~100x. However, if light elements are involved (as the $SiO_2$ example in Fig. 3) the relative sensitivity for oxygen analysis with respect to Si gains back ~5x due to greatly reduced differential mass absorption of the O(K) x-ray line along the shorter x-ray escape path and the disproportionate attenuation of the Si signal with beam energy. The relative mass sensitivity for O is therefore ~500x improved. The detailed numbers will depend on the particular experiment but these data are representative of the big gains which can typically be achieved in low voltage analysis; even if they are not quite as large as may at first be promised.

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The balance of corrections is transformed at low beam voltages. The conventional ZAF corrections are replaced with something more like the Cliff-Lorimer model for thin sections in the analytical transmission electron microscope, but with much modified Z factors. Ideally, and in most but not all practical cases, the A and F corrections are reduced to insignificance which greatly simplifies quantization, especially where samples are heterogeneous on a fine scale. However the Z term combining x-ray fluorescence with detector sensitivity becomes much more complex and a strong function of both electron beam and x-ray line energies. Fortunately these issues can be addressed for a particular system with a limited number of standards. A simplification is to fix the beam voltage for quantitative analysis at one value, say 5kV, as we have mostly done. For analysis at 5kV non-conducting samples may require coatings not necessary for imaging at ~1kV. The acute surface sensitivity of 1-10nm and >100x range at a beam energy of 1.5kV requires great care in the preparation, preservation and transfer of sample surfaces. At 5kV continuous coatings <5nm should stabilize the imaging and analysis conditions without adding appreciably to the spectra. At this time there is also a serious lack of data to support quantitative analysis at low beam voltages and we are still working to determine exactly which parameters are important. In principle low voltage analysis should be more sensitive and more precise than the conventional approach; but this is so far probably only realized in selected examples where light elements are analyzed directly.

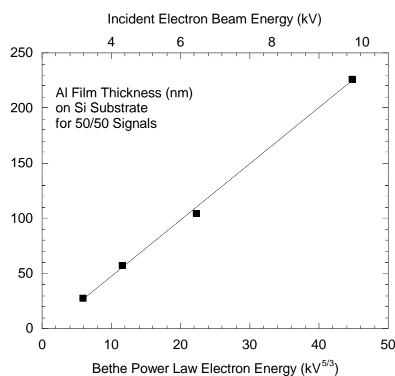



Fig. 1 : Depth resolution Al thin film on Si wafer substrate.

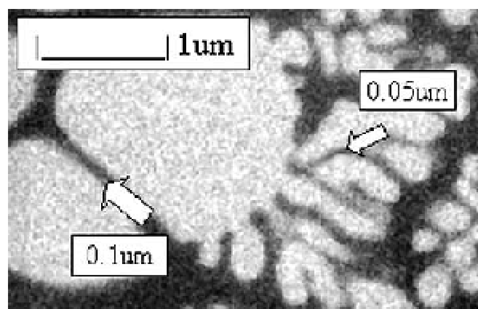
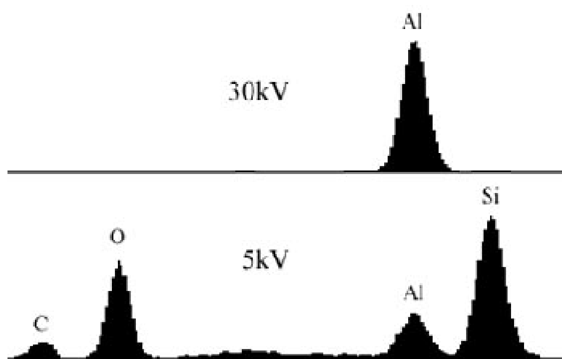



Fig. 2 : High lateral resolution $ZrO_2$ in $SiO_2$ matrix.



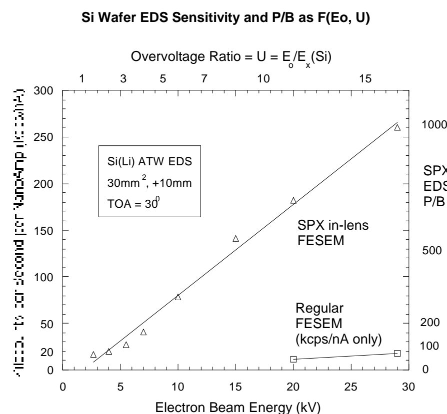




Fig. 3 : $SiO_2$ on Al substrate at 3 and 30kV.

Fig.4 : kcps/nA sensitivity and P/B for Si

Reference : G Cliff and G Lorimer, *J Microscopy*, 103 (1975) 203

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