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Quantitative Microanalysis at Low kV : Precautions and Validation

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A new method for checking for stray radiation, more accurate knowledge of incident beam energy, and improved consistency checks to validate analyses are useful at low kV. Fig 1 shows a large increase in C K, O K and Zr M peaks when a Zr standard is analysed at 5kV. C and O may be due to a carbon coating and superficial oxide layer but how much is stray radiation due to backscattered electrons travelling towards the detector and exciting C and O from the polymer window or dagged surfaces in the collimator and electron trap? Spectrum synthesis affords a "CheckTotal" for validating analytical results [1,2] provided the observed background is bremsstrahlung from the sample alone and there are no backscattered electrons entering the detector. At 5kV it is more difficult to recognise any "hump" due to backscattered electrons because energy losses in the window push it to a region where the bremsstrahlung background is already convex. For the new method, an "electron mirror" was constructed by gluing a 3.2mm diameter steel ball bearing to a 0.8mm thick, 10mm diameter disk of PTFE, mounted on a standard Al specimen stub. A spectrum of Al was acquired at 5kV from the stub. The ball bearing was then charged by exposing to 25kV electrons for one minute then the beam voltage was reduced back to 5kV. The charged ball reflected all electrons and an image of the inside of the JSM 6400 specimen chamber was obtained (fig.2). A spectrum image was recorded using digital beam control with a reduced scan over just the collimator (Fig.3). The C K map in Fig.3(b) shows where electrons hit the dagged walls of the collimator and the Ni L map shows where electrons end up striking the surfaces of Ni-rich magnets used in the electron trap. The sum spectrum in Fig.4 was obtained by integrating over all pixels in the collimator aperture which subtends a solid angle, $\Omega = 0.01$ ster at a TOA=30degrees with the sample. Taking the backscatter coefficient, η, of Al ≈ 0.2, and the livetimes for the Al and "collimator" spectra to be T_{Al} and T_C respectively, then scaling the sum spectrum by the factor $(\eta * \Omega * \mathrm{Sin(TOA)}/\pi) * T_{Al}/T_C$ shows that the total area of the collimator contribution corresponds to less than 0.2% of the Al spectrum.

At 20kV, measurement of the Duanne Hunt limit (DHL) within 100eV is usually adequate to determine incident beam energy. However, at 5kV a change of only 100eV in beam energy will, for example, alter Ca Kα intensity by more than 20% so a much more precise measure of DHL is required. Having confirmed electron trap effectiveness, the continuum shape was studied and showed a difference between high Z and low Z materials. Fig 5 shows how extrapolation methods [3] can produce differences of the order of 200eV depending on atomic number and the energy band used for fitting. However, it appears that with about 10^7 counts in a spectrum and a small fitting interval close to DHL, the limit can be determined within +/-20eV.

References

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[3] R.L.Myklebust, C.E.Fiori and D.E.Newbury, *Microbeam Analysis-1990* (1990), 147
[4] The author gratefully acknowledges Grahame Lawes and Judith Root for assistance with the data acquisition and Peter Duncumb for helpful discussions.

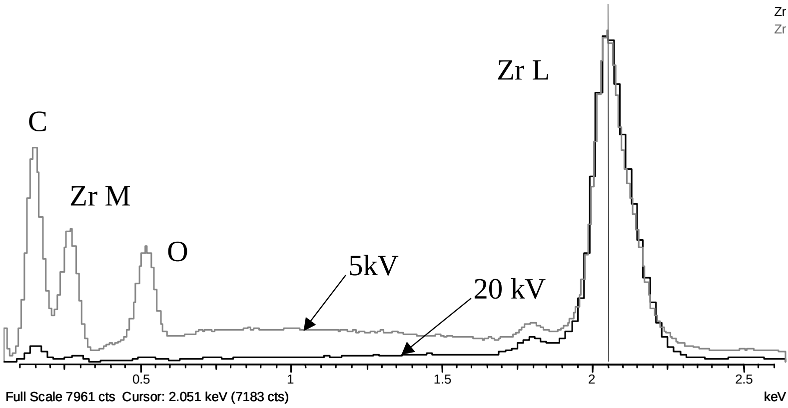


FIG 1: 5kV and 20kV spectra, C-coated Zr std, scaled at ZrL

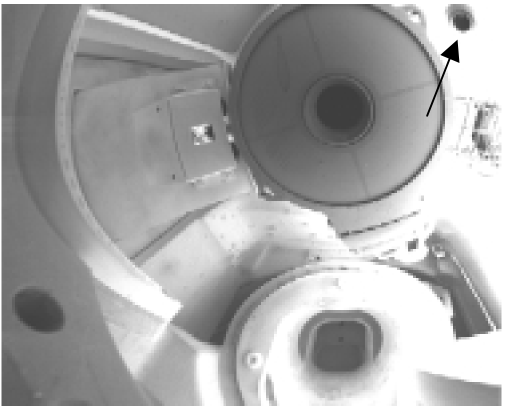


FIG 2: b.s.electron view of chamber

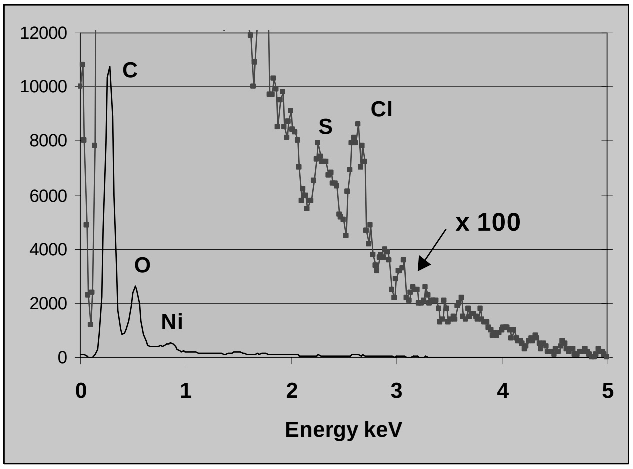


FIG 4: Spectrum obtained by integrating all data over collimator aperture in fig.3.

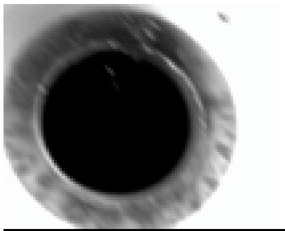
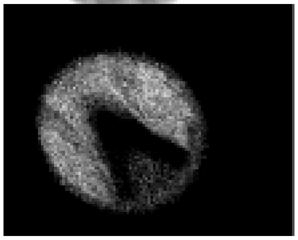
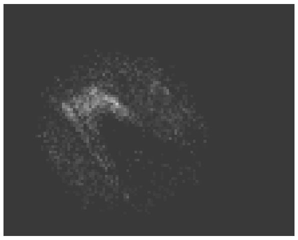


FIG 3: Digital beam scan over EDX collimator which is top right in fig 2.
3(a) S.E. image.



3(b) C K x-ray map



3(c) Ni L x-ray map

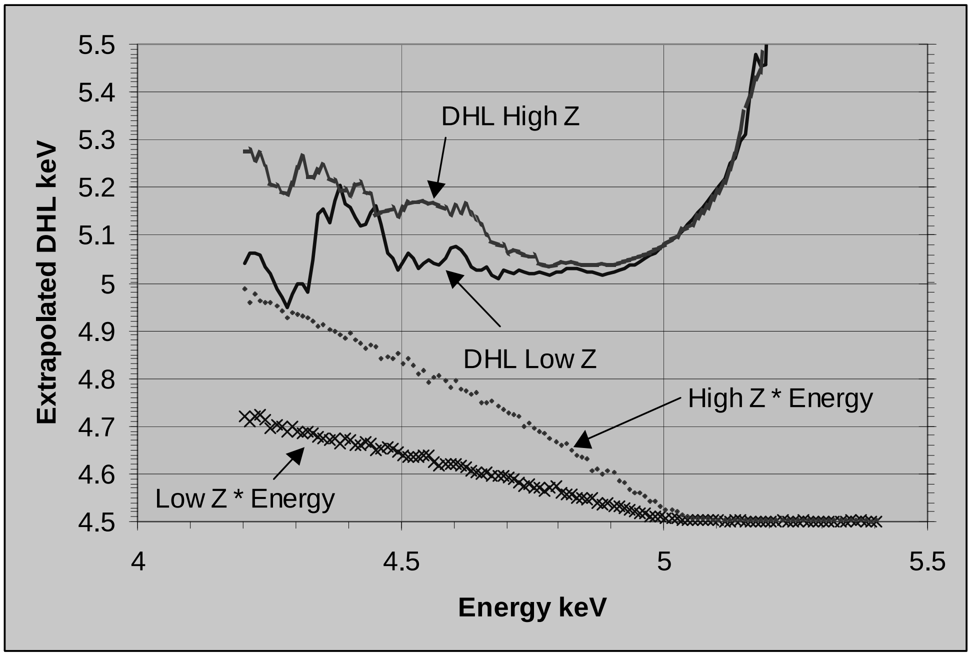


FIG 5: Duanne-Hunt Limit DHL obtained by fitting straight line over 210eV energy range to product (channel count x energy) and extrapolating. Low Z is total of 18 spectra with Z=4-30, total area 10^7 counts. High Z is for 11 spectra, Z=45-83, total $7x10^6$ counts. Extrapolated DHL varies depending on position of 210eV fitting window.