Title

BACKGROUND MODELLING ALTERNATIVES IN ELECTRON ENERGY-LOSS SPECTROSCOPY AND THEIR IMPLICATIONS ON MICROANALYSIS

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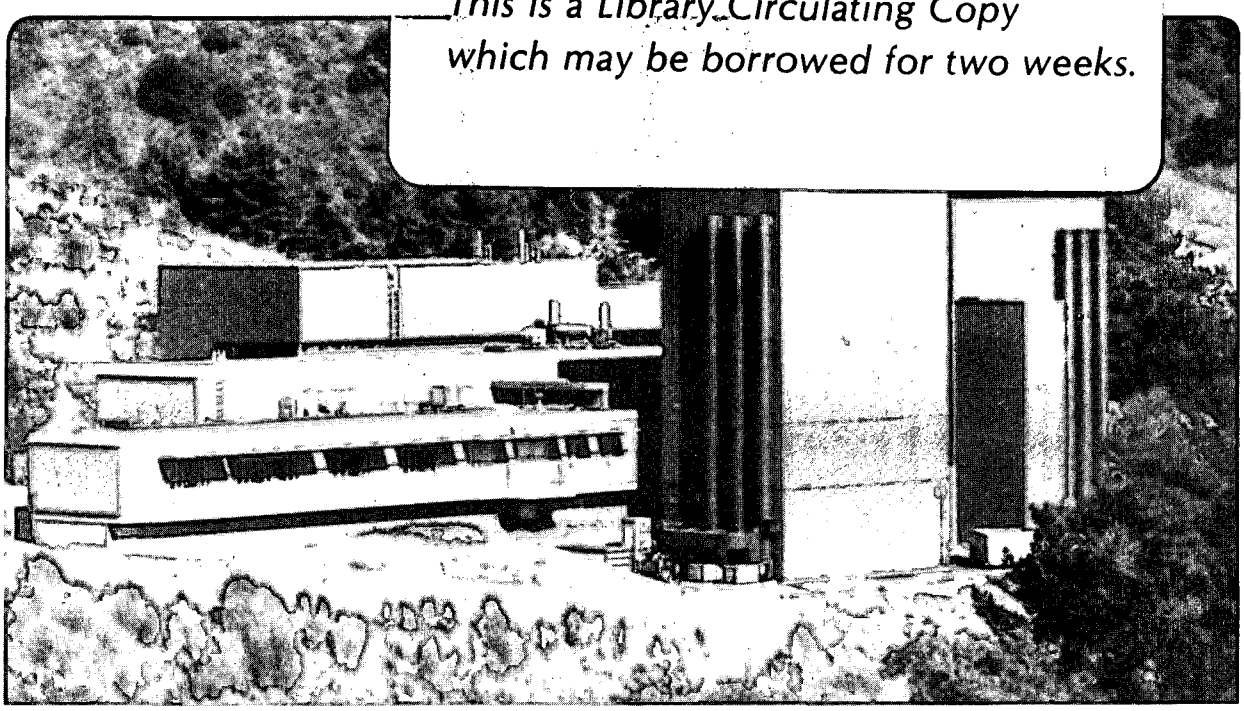

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BACKGROUND MODELLING ALTERNATIVES IN ELECTRON ENERGY-LOSS SPECTROSCOPY AND THEIR IMPLICATIONS ON MICROANALYSIS

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With the advent of parallel detectors, electron energy-loss spectroscopy (EELS) is expected to be employed increasingly in the routine microanalysis of light elements[1]. The quantitation formulae that are used are relatively simple and straightforward and require only a measurement of the integrated core-loss intensity over a particular energy window beyond the ionization edge (Δ_i) and most often, a calculated ionization cross-section. Even though the hydrogenic cross-sections that are used routinely for microanalysis can be a source of error, it is observed that the procedure that largely determines the accuracy of quantification is the removal of the contribution of the background below the ionization edge. Based on empirical observations, an inverse power law function of the form $I = AE^{-r}$, where the exponent 'r' takes values from 2 to 6, is now commonly used for the background[2]. A background fitting region preceding the ionization edge (Δ_b) is chosen, the constants A and r determined by least squares refinement and the background extrapolated beyond the ionization edge for the required energy window (Δ_i). In addition to the fact that Δ_b is often chosen arbitrarily, significant errors are introduced by the extrapolation procedure, a systematic component of which can be due to the variation of the exponent 'r, in the above power law form, with energy[3]. Some efforts to use alternative functions such as higher order polynomials have been observed[4] to give better results, but without any systematic conclusions. Here we summarize our recent efforts to overcome these systematic extrapolation errors and to make the selection of Δ_b less arbitrary.

Several spectra from a variety of high purity binary compounds (BN, TiC, NiO, Fe_3O_4, LiF, etc.) were acquired. A JEOL 200CX TEM operating mostly at 200kV, fitted with a Gatan 607 magnetic sector spectrometer was used for all experiments. Single scattered distributions were obtained by the standard Fourier-log deconvolution procedure[2], but in almost all cases this proved unnecessary as the samples were very thin. Cross-sections were calculated using the modified hydrogenic formulations of Egerton (SIGMAK and SIGMAL)[2]. Processing of the EEL spectra was undertaken using an interactive software package developed here at the NCEM. Versions of the program are being developed both for the IBM PC as well as the DEC mVAX computers.

For each ionization edge, three background fitting regions (Δ^i_b) were selected. For every Δ^i_b, polynomials up to the third order, were fitted to the logarithm of the spectra, i.e. the ln(intensity) vs. ln(energy) curve. Note that the first order polynomial here is the same as the AE^{-r} form. The χ^2 values used in the least squares refinement were weighted using the standard deviation for each data point of the logarithmic curve. For each Δ^i_b then, the optimal order of the polynomial chosen for extrapolation was the one with the minimum χ^2 value. These polynomials of optimal order were extrapolated to include a constant energy window (Δ_i) beneath the ionization edge. A formulation for the calculation of the uncertainties in each of the parameters of the polynomial fit has been derived. The error involved in the extrapolation was calculated as the weighted sum, over Δ_i, of the uncertainties in each of the coefficients used in the fit. The background fitting region that contributed a minimum extrapolation error was then finally chosen for the microanalysis. Table 1 is a summary of the microanalysis of four representative compounds, including the best fitting parameters. This procedure should minimize the systematic extrapolation errors, eliminate the arbitrariness in selecting background fitting regions and make EELS quantification more reproducible[5].

1. R. F. Egerton, SEM/1984/II, 505.
2. R. F. Egerton, EELS in the Electron Microscope, New York: Plenum (1986).
3. D. R. Liu and L. M. Brown, J. Micros. (1987) 147, 37.
4. J. Bentley, G. L. Lehman and P. S. Sklad, Electron Microscopy (1982) 1, 585.
5. This research was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Science Division, U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

TABLE I

Compound	Edge (eV)	Background Fitting Region (Δ_b)	# of Terms N_{opt}	Concentration (at%)
TiC	C-K = 284	190-280	3	48.2
	Ti-L = 456	400-450	4	51.8
Fe_3O_4	O-K = 532	180-510	3	48.1
	Fe-L = 708	640-700	3	51.9
BN	B-K = 188	75-180	4	55.2
	N-K = 401	290-360	2	44.7
NiO	O-K = 532	370-460	2	56.9
	Ni-L= 855	695-785	2	43.1

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