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Quantitative X-Ray Microanalysis in the Analytical Electron Microscope

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The foundations of quantitative x-ray microanalysis in the analytical electron microscope were published in the famous paper of Goldstein *et al.* [1]. The weight fraction of two elements A and B, C_A and C_B, are related to their net x-ray intensities I_A and I_B by this equation:

$$\frac{C_A}{C_B} = K_{A-B}\frac{I_A}{I_B} \quad (1)$$

where K_{A-B} is the well known Cliff – Lorimer K factor. Equation (1) is valid when absorption and fluorescence effects are negligible. K_{A-B} can be determined with the measurement of x-ray net intensities using a standard of known composition or it can be computed using an equation based from first principles that was derived by Goldstein *et al.* [1]. Because of uncertainties on fundamental parameters, especially the ionization cross-sections, experimental K_{A-B} factors are more accurate. Gauvin & L'Espérance [2] have proposed to use the ratio of two experimental K_{A-B} factors obtained at 2 different accelerating voltages, R, in order to determine the C_{nl} parameter of the Bethe equation of the ionization cross sections then allowing computing the K_{A-B} factors, and saving time to perform quantitative x-ray microanalysis. The great advantage of equation (1) is that K_{A-B} is independent of composition when x-rays are generated by primary electrons only, because they do not loose significant energy in the TEM. Therefore, a K_{A-B} factor determined at one composition can be used with another composition. However, K_{A-B} becomes dependant of composition when x-rays are generated by fast secondary electrons (FSE) [3]. Figure [1] shows K^*, the ratio of K_{A-B} computed with and without FSE for the B-N, B-Fe and B-Nd systems at 100 keV. Clearly, when A or B is a light element, the K_{A-B} factor can be very sensitive to composition and equation (1) is no longer valid. An experimental validation of these Monte Carlo simulations is therefore needed. The paper of Goldstein *et al.* [1] presented an equation to compute the beam broadening, b, of primary electrons in the TEM based on a single scattering model. Figure [2] shows b (simulated and computed for a zero beam diameter) as a function of thickness for Al and Fe at 100 keV with their model and by using Monte Carlo simulations. In the practical range of TEM thin foils (20 to 100 nm), the single scattering model is valid. However, Monte Carlo simulations will have to be used to determine the ultimate resolution of microanalysis in the TEM.

References

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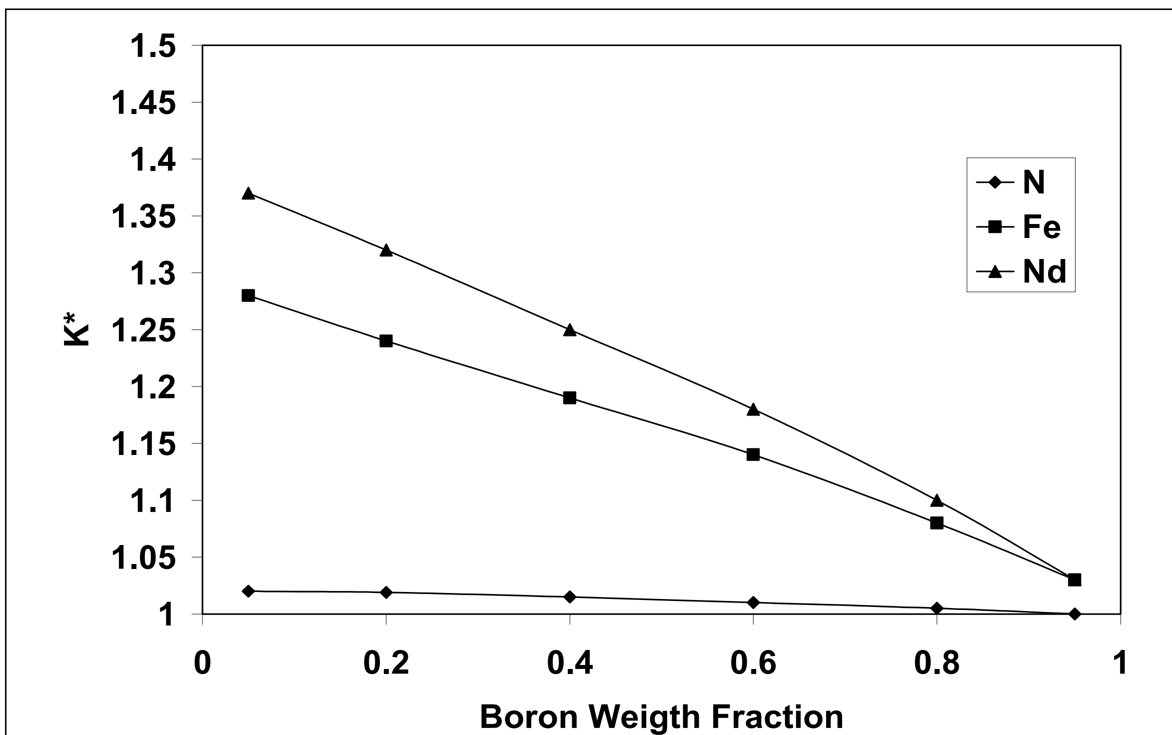

Figure [1] K^*B-X versus B composition at 100 keV and 100 nm of thickness.

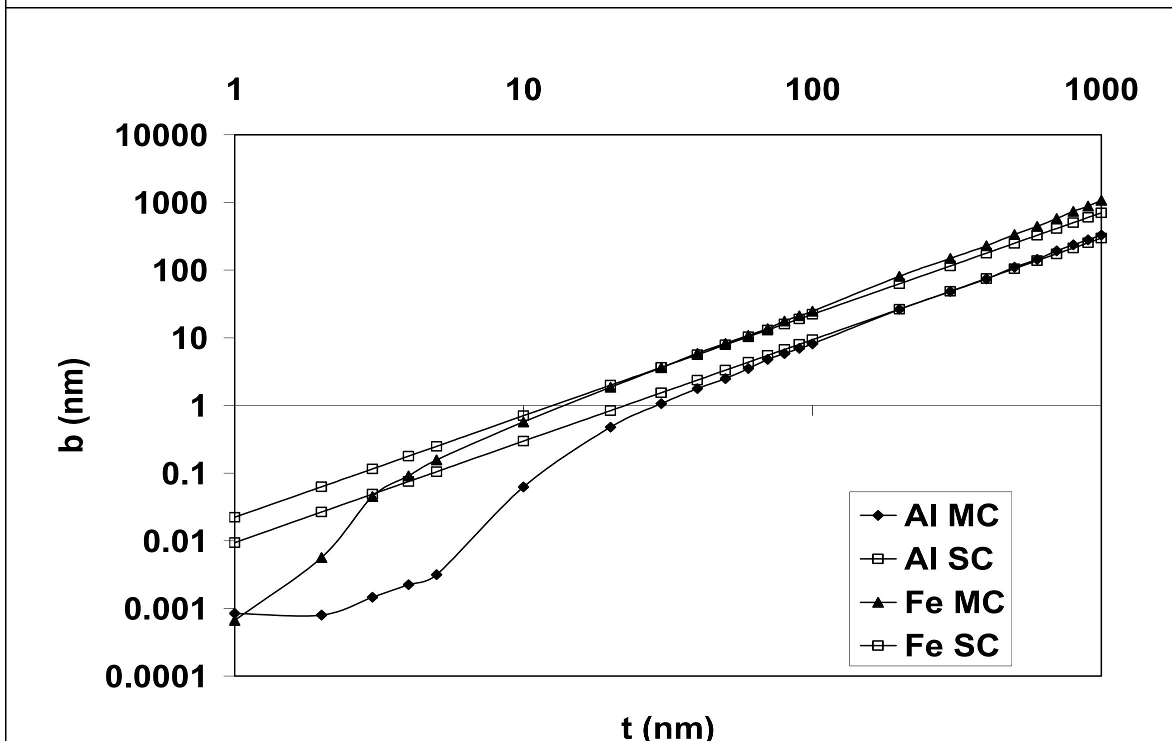

Figure [2] b versus t at 100 keV from Monte Carlo (MC) and single scattering model (SC) of Goldstein *et al.*[1].

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