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Future Trends for Quantitative X-Ray Microanalysis is a Field Emission Scanning Electron Microscope at Low Beam Energies

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Conventional quantitative x-ray microanalysis in the SEM is based on this equation:

$$\frac{C_i}{C_{(i)}} = ZAF\,\frac{I_i}{I_{(i)}} \qquad (1)$$

where I_i is the net intensity of a characteristic x-ray line of element i measured with the specimen of unknown composition, $I_{(i)}$ is the net intensity of the same characteristic line of the same element i measured with a standard of known composition, C_i is the composition of the unknown specimen, $C_{(i)}$ is the known composition of the standard of element i and the factors Z, A and F are respectively the correction factors for atomic number, absorption and fluorescence effects. In order to measure the x-ray intensities, the specimen current must be the same during the measurement of the two intensities for each element i from the unknown and the standard. A disadvantage of the cold field emission gun scanning electron microscope is that the beam current is not stable, making quantitative analysis problematic. A new method of Quantitative analysis is being developed however[1], where normalized ratios of x-ray lines from different elements are taken. For two elements A and B, the ratio f_A is defined as follow:

$$f_A = \frac{I_A}{I_A + I_B} \qquad (3)$$

where I_A and I_B are the net x-ray intensities of the characteristic lines of element A and B. Since f_A is taken from the same spectrum, the unstable beam current is not an issue since the current is the same for all the lines and it disappears in the ratio.

Preliminary analysis of this technique was performed on the Al-Mg binary system. A calibration curve was fabricated using the Monte Carlo simulation program, WinX-Ray[2]. The simulations were run for the whole range of weight fractions of Al and Mg. Figure [1] shows the calibration curve for various accelerating voltages. It is seen that the curve for 5 kV requires less correction than for 20 keV. At lower accelerating voltages, there is less absorption because of the decrease in the interaction volumes. Experimental ratios are obtained and fitted on the curve to find the actual value of C_{Mg} in an Al-Mg binary diffusion couple experiment performed at 400° C. Figure [2] shows the composition profile obtained with this method from x-ray spectra acquired at 10 keV with a Hitachi S-4700. These values are in a very good agreement with those obtained with an electron microprobe. Still in a preliminary stage however, these results show that the new ratio technique for quantitative x-ray microanalysis is feasible.

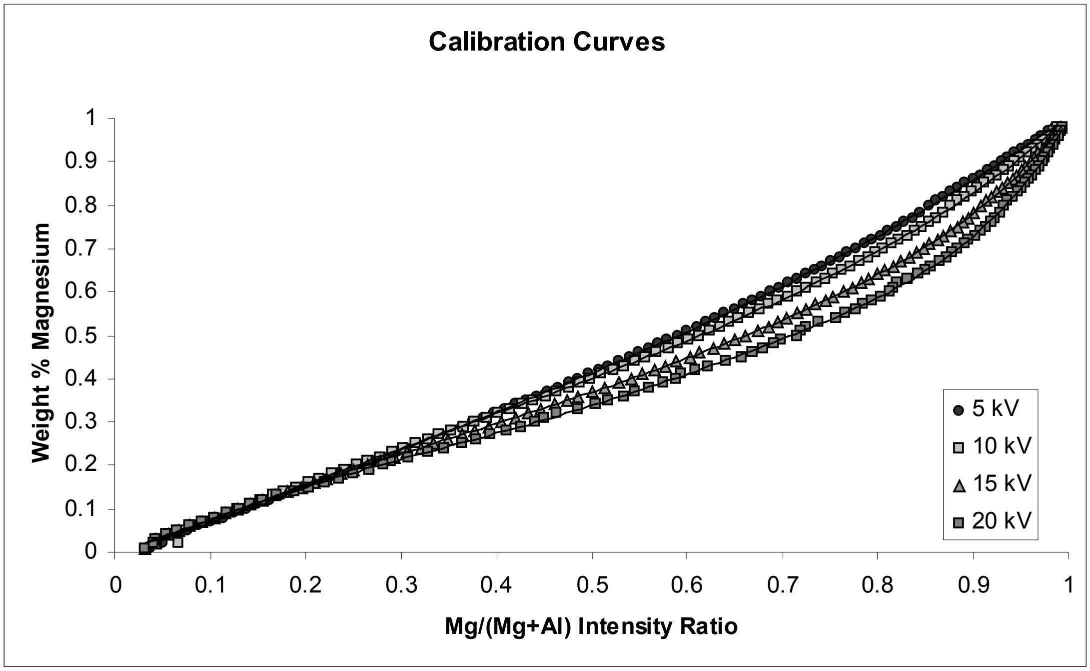

Figure [1] Calibration curve of c_{Mg} versus f_{Mg} in the Al - Mg system computed at 5, 10, 15, 20 keV using the Monte Carlo program Win X-Ray.

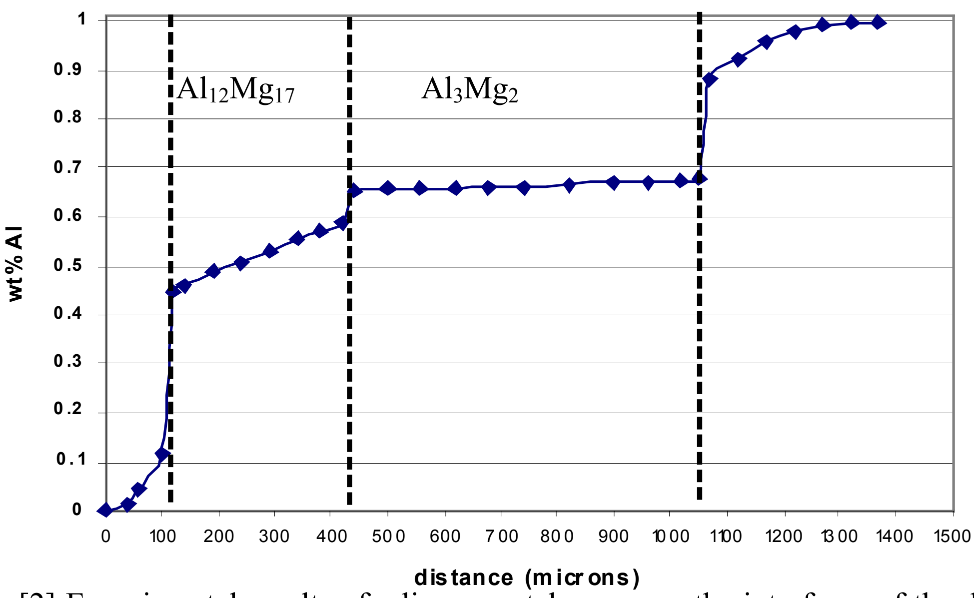

Figure [2] Experimental results of a line scan taken across the interfaces of the diffusion couple. This work was performed at 10 keV.

References

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