Type of presentation: Poster

IT-5-P-2551 Quantitative X-Ray Microanalysis in the Scanning and Transmission Electron Microscopes with the Generalized f-Ratio Method

Demers H.[1], Brodusch N.[1], Trudeau M.[2], Gauvin R.[1]

[1]Department of Mining and Materials Engineering, McGill University, Montreal, Quebec, Canada, [2]Materials Science, Hydro-Québec Research Institute, Varennes, Québec, Canada

Email of the presenting author: hendrix.demers@mail.mcgill.ca

Quantitative x-ray microanalysis of bulk samples is usually obtained by measuring the characteristic x-ray intensities of each element in a sample and in a corresponding standard. The k-ratio of the measured intensities from the unknown material over the standard is related to the concentration using the ZAF or φ(ρz) correction methods. Under optimal conditions, accuracies approaching 1% are possible. However, all the experimental conditions must remain the identical during the sample and standard measurements. This is not possible with a cold-field emission scanning electron microscope (CFE-SEM) where beam current can fluctuate by 5% in its stable regime. To address this issue, a new method was developed using a single spectrum measurement (Horny et al., 2010; Gauvin, 2012). It is similar in approach to the Cliff and Lorimer (1975) ratio method developed for the analytical transmission electron microscope. However, corrections are made for x-rays generated from thick specimens using the ratio of the characteristic x-ray intensities of two elements in the same material. The proposed method utilizes the ratio of the intensity of a characteristic x-ray normalized by the sum of x-ray intensities of all the elements measured for the sample. Uncertainties in the physical parameters of x-ray generation are corrected using a calibration factor that must be previously acquired or calculated. With this method, relative accuracies better than 5% were obtained in a CFE- SEM.

The f-ratio method was generalized to more than two elements. The correction factors are still acquired experimentally relatively to two elements and they do not change with composition. They are obtained from measurement of one known phase. The concentration curves versus f-ratio are obtained by Monte Carlo simulations and the unknown concentrations are calculated from these curves and the measured f-ratios by combination of multi-dimensional interpolation and iterative procedure. An example is shown in Figure 1, where quantitative x-ray maps of ternary Al-Mg-Zn diffusion couple sample were obtained with a CFE- SEM at 5 kV. The generalized f-ratio method was also applied to a thin specimen in the transmission electron microscope. An example is presented in Figure 2, trace element concentration of Fe in a Zr-Nb alloy was determined. The generalized f-ratio method allows the quantification of multi-elements sample in both SEM and TEM. Furthermore, in which condition this method can be applied to heterogeneous sample is currently explored.

References:

P. Horny, E. Lifshin, H. Campbell and R. Gauvin, Microscopy and Microanalysis, 16, 821-830 (2010).

R. Gauvin, Microscopy and Microanalysis, 18, 915-940 (2012).

G. Cliff and G. W. Lorimer, Journal of Micrsocopy, 103, 203-207 (1975).

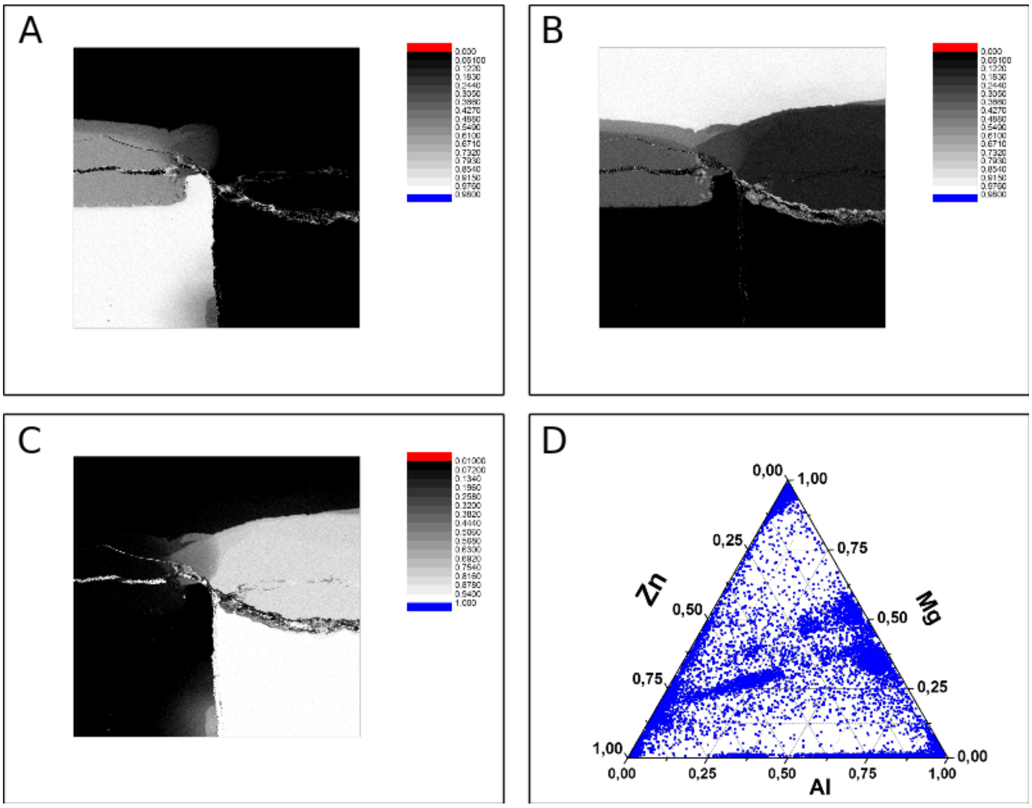

Fig. 1: Quantitative x-ray maps of ternary Al-Mg-Zn sample obtained with the f-ratio method at 5 kV. The weight fractions of each element (A: aluminum, B: magnesium, C: Zinc) are represented by a gray scale: black 0% and white 100%. D A ternary phase diagram was obtained from the x-ray maps.

A

Elements Kα	Λ
Fe-Zr	0.773
Fe-Nb	0.880
Zr-Nb	1.139

B

Elements	Weight %
Fe	0.96 +- 10%
Zr	82.4 +- 2%
Nb	16.6 +- 2%

Fig. 2: X-ray quantification of Zr-Nb-Fe alloy obtained with the f-ratio method in a TEM at 200 kV. A Correction factors measured with 76 wt% Zr, 19 wt% Nb and 5 wt% Fe alloy. B Iron concentration in a Zr-Nb phase was calculated with the f-ratio method and Monte Carlo simulations.