A new tool to treat peak overlaps in electron-probe microanalysis of rare-earth-element L-series X-rays

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Abstract: An empirical method for X-ray data processing is presented for the treatment of peak overlaps in electron-probe microanalysis. The performance of this method has been tested on the complex X-ray L-spectra of compounds containing rare-earth elements. The estimated minimum detection limits are better than 0.1 wt.% even in the presence of strong spectral interferences. The method can be easily included in computer software for on-line quantitative analysis. Most problems of peak interferences can thus be solved routinely with substantial reduction of time spent in the preparation of analytical session.

Key-words: electron microprobe, LiF and PET monochromators, overlap standard, rare-earth-element fluorides, monazite.

Introduction

The L-peak spectra emitted from rare-earth-element (REE) bearing compounds constitute a well-known illustration of spectral interferences in electron-probe microanalysis (EPMA). Two papers, Roeder (1985) and Remond et al. (1989), may be cited as examples of valuable studies devoted to this EPMA application. The complexity of REE L spectra, where at least ten lines are detectable for each element (Remond et al., 1989), may be the source of strong interferences leading to unsolved analytical problems. Owing to their high intensities, the La lines should be preferred to achieve accurate and rapid analysis. However, Table 1 shows that these lines may strongly overlap with one or several lines emitted from accompanying REE. Two types of monochromators are used in REE EPMA: LiF and PET with 2-d spacings of 0.4027 and 0.875 nm, respectively. LiF exhibits good resolution but leads to poor counting rates and the opposite situation holds for PET. The peak-overlap values (%) listed in Table 1 have been measured on REE fluoride specimens (REEF₃ type) with PET.

The peak-overlap correction method briefly described in the next section was included as part of a general quantitative analysis software presented elsewhere (Fialin et al., 1993). Its performance has been already tested on the processing of notorious peak overlaps, such as Nₐα-TiL₁ in TiN and related compounds, and Vₐα-TiK₁β₁ in oxide minerals.

The overlapping peak correction procedure

Basic concepts

The empirical method proposed consists of estimating the contribution of a line, emitted from an element B, which overlaps the target line for analysis emitted by element A. The intensity Iᵢ of the overlapping peak is measured at the Bragg position of the A line on a standard free of A (hereafter termed overlap standard) with the same acquisition parameters used for A. The relative intensity k (k-ratio) of the A line corrected for B overlap is given by:

\[ k = \frac{[I_A - I_{\text{overlap}}] \cdot (C_B/C_A) \cdot (PAP/BAP)}{I_{\text{std}}} \]

where: