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Accuracy and Precision in the EDS-TEM Matrix and Precipitate Quantification in Zr-0.85Nb-0.20Ta Alloy

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When the chemical composition of small precipitates immersed in a matrix is required, the technique of X-ray energy dispersion spectroscopy (EDS) mounted on Transmission Electron Microscopy (TEM) equipment is very useful.

The Cliff-Lorimer quantification method is the most widely used and it relates the X-ray intensities emitted by the chemical elements present in the precipitate, grouped in pairs, with their concentration ratios by means of constants. There are two ways to determine the constants (K factor), the experimental one uses a single phase sample of known composition (standard), and the other uses calculations of first principles. Commercial software uses this last way, but warns the user that the accuracy error can reach ± 20% relative [1].

The present work exposes a procedure to obtain, in an experimental way, the K factors necessary for the chemical composition quantification of phases in a ternary alloy considering all the measurement errors (uncertainty and dispersion in the composition of the standards and in the emitted X-ray intensities). Additional difficulties such as overlapping peaks and the use of L-layer emission are also addressed (see Figure 1).

The calibration process was carried on TEM thin films extracted from two standards (15Zr-46Nb-39Ta and 66Zr-18Nb-16Ta) which chemical composition was measured by electron probe microanalyzer. The application of the procedure for estimation of the K factors and its propagation errors is shown.

The K factors obtained by the experimental procedure are applied to quantify the hcp (α) matrix composition and bcc (β) precipitates of an alloy for nuclear applications Zr-0.85Nb-0.20Ta (% weight). The β phase has a volume fraction fv <0.5% and dimensions between 10 and 250 nm. The α phase was quantified on a TEM thin film and the precipitated phase on an extractive carbon replica (see Figure 2). The results show the propagation and incidence of each error in the quantification of the composition of the phases present.

References:

[1] D.B. Williams and C.B. Carter in “Transmission Electron Microscopy. A Textbook for Materials Science”, (Springer Science+Business Media, LLC, 233 Spring Street, New York, NY 10013, USA, 2009) pp. 639-660.
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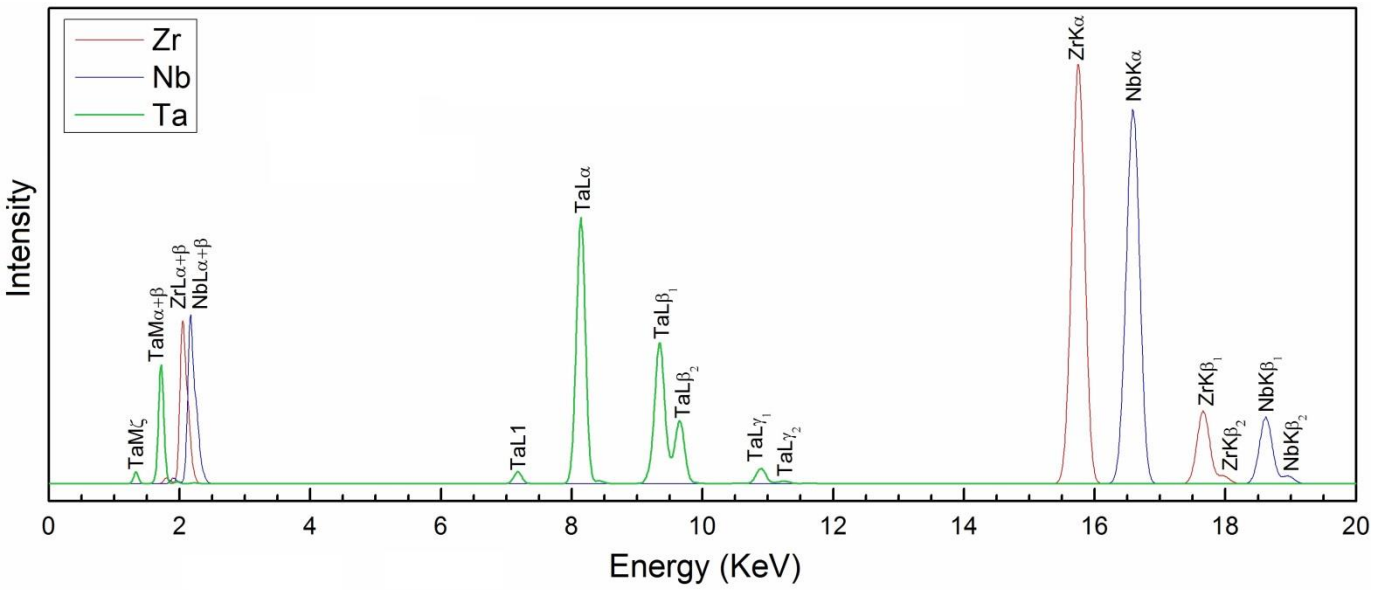

Figure 1. Simulated EDS spectra of pure metals showing the superposition of the X-ray intensity peaks of the L-layer in Zr and Nb preventing the resolution of K factors linked to the L-layer of Ta.

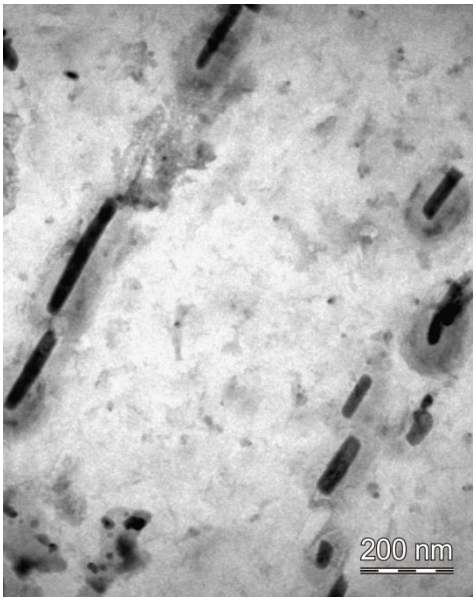

Figure 2. TEM image of β precipitates obtained by an extractive carbon replica in Zr-0.85Nb-0.20Ta alloy (% by weight)..

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