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IT-5-P-2428 Obtaining an accurate quantification of light elements by EDX: K-factors vs. Zeta-factors

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The new energy dispersive X-ray (EDX) technology based on four silicon drift detectors (SDD) with a windowless design provides new possibilities in the field of analytical characterization at nanometer scale. The four detectors are symmetrically arranged with respect to the sample and this unique configuration provides very high collection efficiency, allowing high counting statistics and rapid acquisition of X-ray spectra, line scans and maps. However, new methodologies for the precise quantitative assessment of the elemental composition at nanometer scale are still needed.

Classically, EDX quantification has been carried out using “Cliff-Lorimer” ratio method. This method requires the knowledge of the k-factors and their precise determination is a key point to obtain an accurate quantification. They can be determined theoretically or experimentally, nevertheless, several limitations are found: i) the theoretical k-factors present large uncertainties, ii) the experimental determination of k-factors required multi-element samples with known compositions and iii) X-ray absorption correction may be important for low energy X-ray emissions, especially for light elements, which require the prior knowledge of the specimen mass thickness. To overcome such limitations, a new procedure named “zeta (ζ)-factor” method has been proposed [1]. In this method, the composition and mass thickness are computed simultaneously for each analysis point enabling X-ray absorption correction.

In this work, we present an accurate EDX quantification of various samples containing light elements or elements with low energy X-ray lines using the ζ-factor method. In this regard, a Super-X Tecnai-OSIRIS installed at PFNC-CEA-Grenoble and operating at 200kV has been used. Fig. 1 shows a representative HAADF image of a thin foil of wollastonite (CaSiO3) prepared by FIB, together with the EDX maps of Ca (3.69 keV), Si (1.74 keV) and O (0.53 keV). Individual profiles of the net X-ray counts are extracted from a line scan (see arrow in Fig. 1a) and quantified using k- and ζ- factors (Fig. 2). Quantification using the k-factors gives wrong results as a consequence of the strong absorption of oxygen (Fig. 2b). Conversely, an excellent agreement between the computed and expected results is obtained using the ζ-factor method (Fig. 2d), due to the specimen thickness is determined for each analyzed point (Fig. 2c) and allowing therefore the X-ray absorption correction.

This example clearly illustrates the potential of the ζ-factor method using the new Super-X detector. By measuring composition and mass thickness simultaneously, the ζ -factor method is a very promising tool for quantitative 3D reconstructions.

[1] M Watanabe and DB Williams, Journal of Microscopy 221 (2006) p. 89

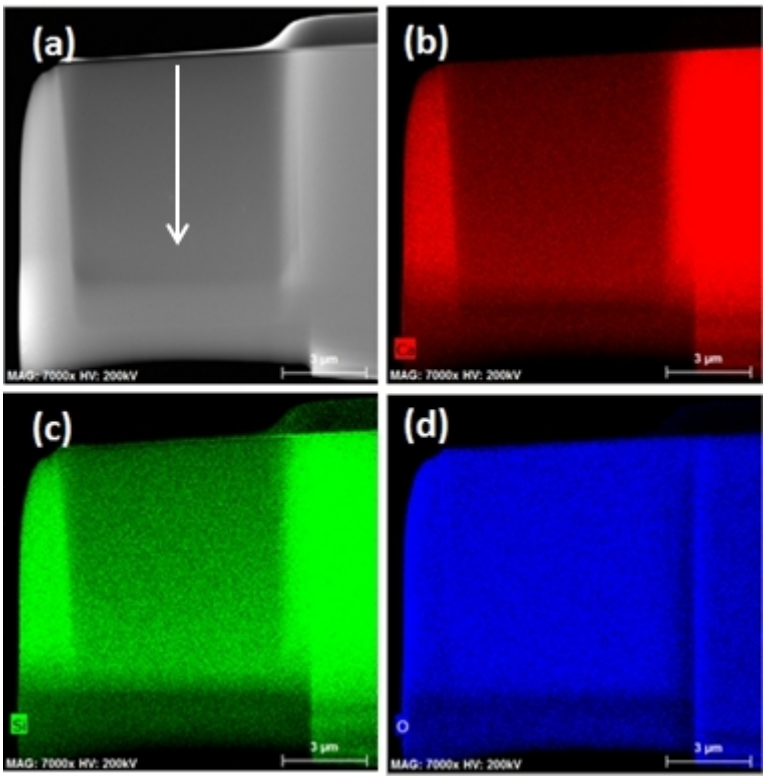

Fig. 1: HAADF-STEM image of wollastinite recorded on a Tecnai-OSIRIS (a) and EDX elemental maps of Ca (b), Si (c) and O (d) using the Super-X detector.

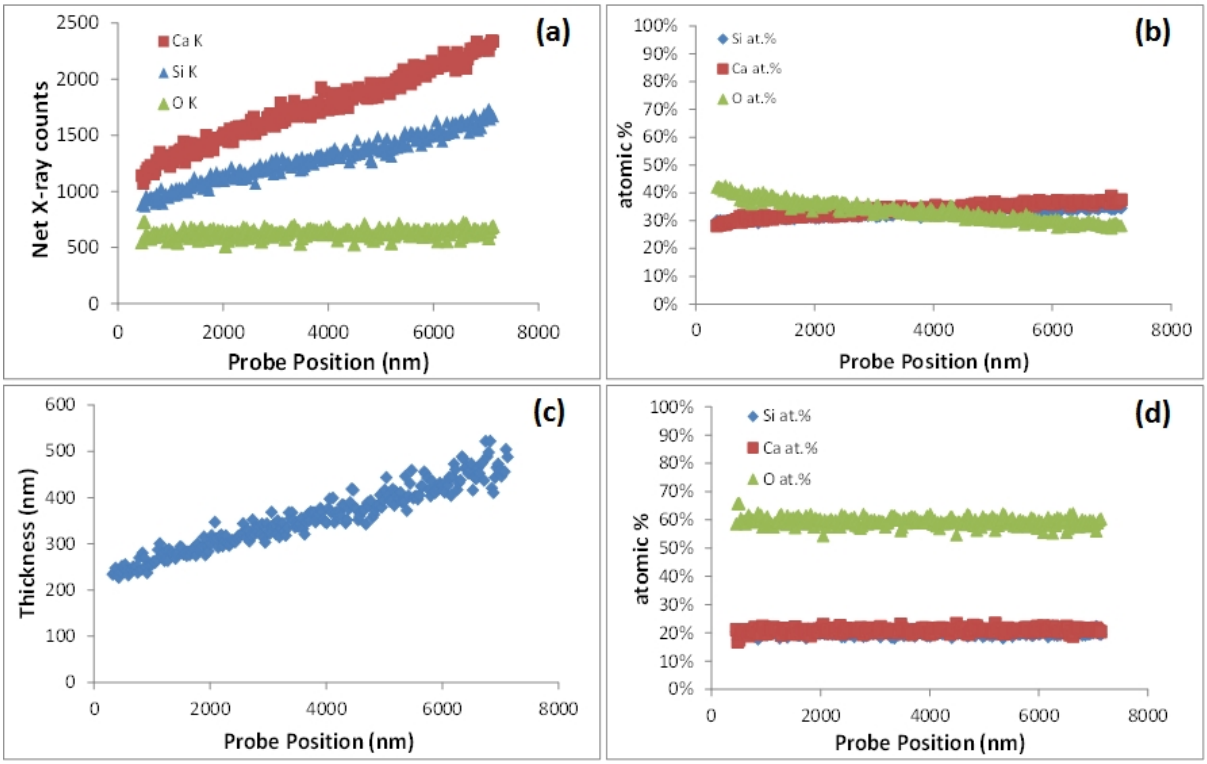

Fig. 2: EDX Line scan extracted from the site marked with an arrow (a). EDX quantification using the k-factor method (b). Thickness measurement obtained by EDX analysis (c) and EDX quantification in atomic percentage using the ζ (zeta)-factor (d).