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High-Resolution Quantification Across Vertical Interfaces using a Monte Carlo Based Reconstruction Approach

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Electron probe microanalysis (EPMA) stands out for its high sensitivity and high accuracy in quantitative measurements of mass coverages of multi-layered structures. Using a perpendicular incident electron beam, the mass coverage of elements in sub-micrometer thick layers can be quantified by analytical models or Monte Carlo simulation based techniques [1-2]. Without any destructive preparation techniques, this technique is limited to buried layers containing elements, which are not present in the surrounding matrix, and to a depth smaller than the absorption path length of the characteristic x-ray lines. To improve depth profile analysis EPMA can be combined with surface removal techniques, such as a focused ion beam instrument, to obtain shallow bevels with well-defined slope (less than 1°) [3]. Due to the small angle, conventional electron microprobes (equipped with a tungsten filament) can deliver quantitative concentration depth profiles despite their larger beam diameter.

With the availability of field emission microprobes (equipped with a Schottky emitter), which deliver smaller beam diameters, sub-micrometer structures (particles, lathes, etc.) can now be measured using conventionally prepared cross sections. Quantification of these features is no longer limited by the beam diameter, but by the x-ray generation volume. Using a low accelerating voltage has the obvious advantage of improving the spatial resolution, but quantification using low energy x-ray lines (Lα and Mα) is challenging due to peak shifts, less proven mass absorption coefficients and a higher influence of contamination [4].

The proposed method aims at improving the spatial resolution by using a reconstruction algorithm. From a set of experimental k-ratios and an initial guess, an iterative method is used to solve the true dimensions of the features of interest as well as their composition. The non-linear optimization compares the experimental k-ratios with predicted k-ratios obtained from Monte Carlo simulations. This procedure is comparable to the established reconstruction algorithms for multi-layered structures [1-2]. In this work, Monte Carlo simulations are used since no analytical model exists for the lateral intensity distribution of x rays.

As an example, a simple case of a Fe-Mn vertical layer (infinite in depth) inside an homogeneous Cu matrix was tested. The feasibility of the reconstruction was assessed using a similar methodology as the one previously used for multi-layered structures [5]. It consists in varying the thickness of the layer and its composition, and evaluating the presence of a single local minimum in the residuals surface (Figure 1). The residuals were calculated from the Euclidean difference between the simulated k-ratios for each thickness-composition combination and a single reference point chosen as a 200 nm thick layer with a composition of 50 wt.% Fe and 50 wt.% Mn. The simulated k-ratios were obtained using the Monte Carlo program PENELOPE [6]. Simulations were performed at 15 kV until the uncertainty (3σ) on the x-ray intensity of all elements was below 1%. A Gaussian-distributed beam with a diameter of 50 nm centred on the layer the was used. The smoothness of the residuals surface and the presence of a significant curvature at the minimum confirm the possibility to quantify a 200 nm thick Fe-Mn layer inside a Cu matrix at 15 kV.

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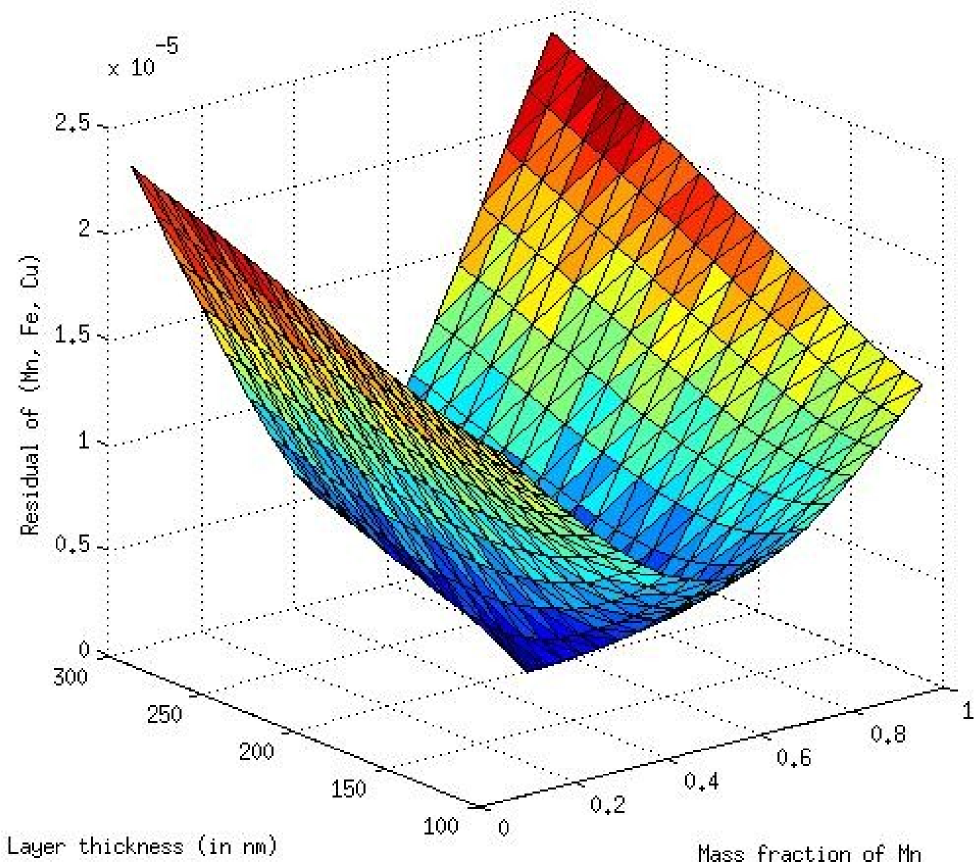

Figure 1: Residual surface obtained from Monte Carlo simulations of a Fe-Mn layer inside a Cu matrix at 15 kV.

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