

## Capability and Uncertainty in Multilayer Quantitative Procedure with Electron Probe Microanalysis

C. Merlet

ISTEEM, CNRS, Université de Montpellier II, Pl. E. Bataillon, 34095 Montpellier cedex 5, France

Electron probe microanalysis (EPMA) has become a well established technique for determining compositions and thicknesses (in the range from 1nm to 2000 nm) of multilayer samples. For the simple case of a thin film deposited on a substrate, this technique is efficient, and the accuracy for the concentration is similar to a bulk specimen. Depending of the instrument, the operator experience, the nature of the film and of the substrate, the uncertainty in the thickness determination can be expected to be less than 10% even when the difference in the atomic number of the film and of the substrate is very large (Fig. 1, Fig. 2). For a multilayer sample, quantitative results require hypothesis concerning the layer description and consequently, the operator experience is crucial in the quality of the results. In addition each defined layer must be homogeneous in depth, and in the majority of cases, when the same element is present in more than one layer, the solution is undetermined without an hypothesis on the concentration of this element. For a buried layer in a multilayer sample, the technique is less and less efficient when the layer thickness decreases and is deeply buried (Fig 3). In some cases, the X-ray lines are completely absorbed by the upper layers and are not detected.

To get reliable results, this technique requires an accurate description of the X-ray depth distribution from which the emitted X-ray intensities are calculated. Intensities can be estimated by two different methods: Monte Carlo simulation [1] and analytical approximations [2,3,4]. The first, although more accurate, is very time consuming, even with the fastest computers available. Moreover, quantitative results are obtained with the help of automatic iterative numerical procedures or with a manual trial and error approach. Consequently, for on line quantification of electron probe measurement only analytical models are used in practice. However, the lack of knowledge about the X-ray depth distribution for stratified samples limits the accuracy attainable with these analytical procedures, mainly when the atomic number between the different layers are largely different. In addition, analytical approximations as well as Monte Carlo simulation require the knowledge of many atomic parameters which describe the electron interaction and the X-ray emission, such as the ionization cross section, mass absorption coefficient, fluorescent yields, and others. Many of these atomic parameters are canceled or do not need to be known accurately by using standard in quantitative microanalysis on bulk sample. However, as shown by the figure 4 as example for the ionization cross section[5], multilayer quantitative analysis require more accurate knowledge of atomic parameters. Similarly, the uncertainty of mass absorption coefficient is less counterbalanced in multilayer than in bulk sample by using standards.

### References

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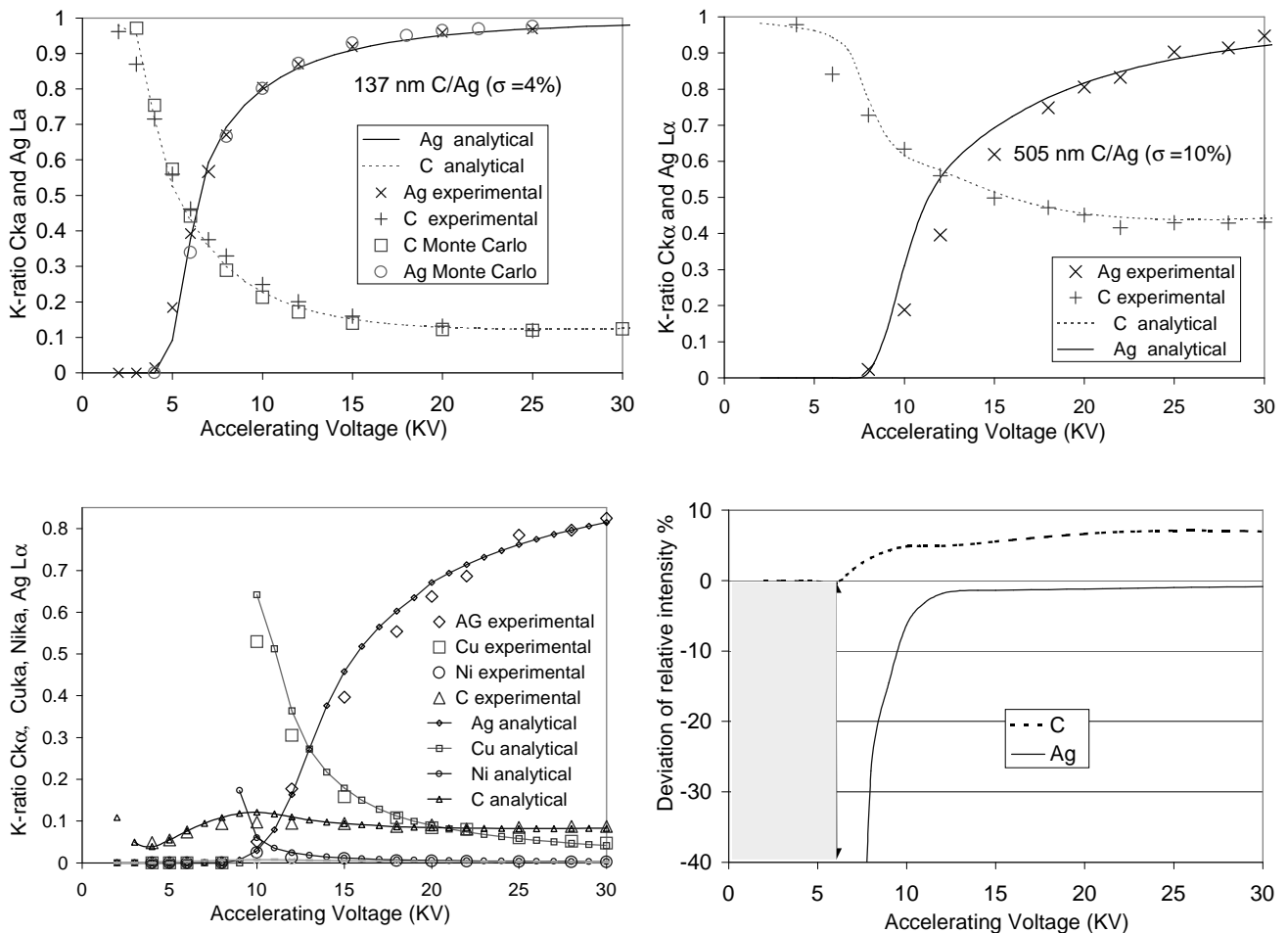


Fig. 1. Relative x-ray intensities for C/Ag specimen (C=137nm) as a function of the electron incident energy, ( x, + ) measurements, ( o, ) Monte Carlo calculation[1], (---) analytical procedure[4]

Fig. 2. Relative x-ray intensities for C/Ag specimen (C=505nm) as a function of the electron incident energy, ( x, + ) measurements, (---) analytical procedure[4]

Fig. 3. Relative x-ray intensities for Ni/Cu/C/Ag multilayer specimen (Ni=5.6nm, Cu=66nm, C=505nm), as a function of the electron incident energy, symbols represent the measurements and the lines represent the result of the analytical procedure[4]

Fig. 4. Deviation of the computation of the relative intensity obtained by using two different ionization cross section model in the analytical procedure, for the 505nm C film deposited onto the Ag substrates.