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The Role of Carbon Film Thickness in Electron Microprobe Analysis: A Comment

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Abstract

In electron-probe analysis non-conducting specimens are usually coated with a conducting film of carbon. This film can affect the accuracy of quantitative analyses, and specifications are given for conditions under which the effect is important and for conditions under which it can be neglected.

The article by Kerrick, Eminhizer, and Villaume (1973) has provided a revision and clarification of the correction formula for film-thickness as published by Sweatman and Long (1969), and it has substantiated the validity of the formula by direct experimental evidence. However, numerical values mentioned for possible X-ray intensity errors due to film-thickness differences provoke some comment, although this is not intended to detract from the value of the authors' work.

Figure 1 shows values for the film-thickness correction in the atomic number ranges usually examined by $K\alpha$, $L\alpha$ or $M\alpha$ radiation. The plots were derived from the expression

$$I_f/I_0 = f$$

with

$$f = [1-8\cdot 3.10^5\ \rho z(V_0^2 - V_c^2)] \cdot \exp(-\mu\rho z \operatorname{cosec}\theta) \qquad (1)$$

which are, in content, identical to the relationships given by Kerrick *et al* (1973). I_f and I_0 are the intensities with and without a coating, respectively. V_0 and V_c are the respective acceleration and critical excitation-potentials for the analyzed element (both in kV). θ is the X-ray take-off angle. The film mass thickness ρz is expressed in g/cm², and μ is the mass absorption coefficient of the film material for the measured radiation. Designating the standard by the superscript ST and the specimen by SP one sees that the correction factor F that must be applied to a measured intensity value follows from $I_0^{SP}/I_0^{ST} = F\cdot(I_f^{SP}/I_f^{ST})$ to be $F = f^{ST}/f^{SP}$.

In the cases where there is no film on the standard, F simplifies to $1/f^{SP}$. In practical terms this latter condition applies also to thickness differences ($\Delta\rho z$) between specimen and standard; $\Delta\rho z$ is then inserted into formula (1) instead of ρz. The factor 1/f has been plotted in Figure 1 for a carbon film 200 Å thick (or for 200 Å difference in thickness, the specimen being coated more heavily). 200 Å films usually provide electrical conductivity high enough to prevent charging effects. The carbon density was taken to be 1.3 g/cm³, as suggested by Kerrick *et al* (1973). The mass-absorption coefficients were calculated after Kelly (1966).

Figure 1 illustrates the following points: (1) Corrections (or errors if corrections are not applied) do not exceed 1 percent at acceleration potentials of more than 20 kV, and they are less than 2 percent at more than 15 kV. This applies to elements heavier than Na and to cases where there is a 200 Å difference in coating thickness between specimen and standard. If the thickness difference can be reduced to 100 Å, corrections at 15 kV are also less than 1 percent, and errors of 1 percent are usually within the limits of precision of the electron-probe analyzer. (2) X-ray absorption in the coating film has only a small effect on the magnitude of the correction, electron absorption being much the stronger influence. X-ray attenuation is worth considering for elements with atomic numbers of less than 15, but there is no case where the contribution of X-ray absorption

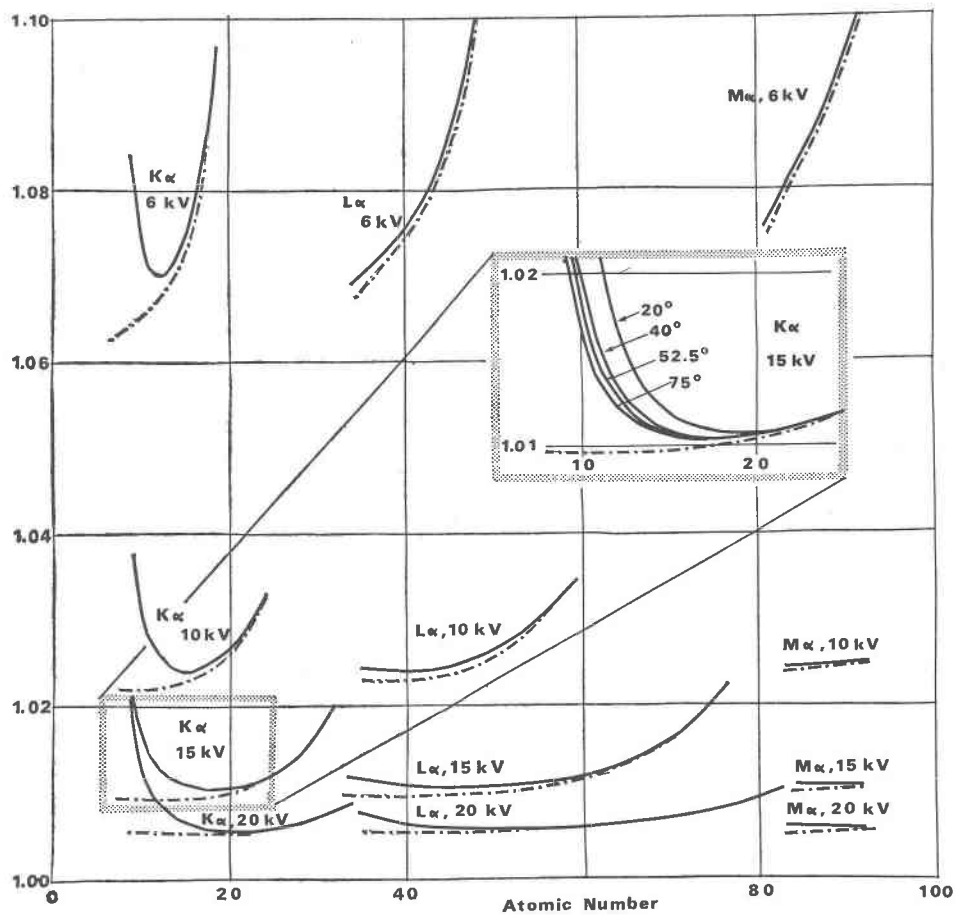

FIG. 1. Plot of correction factor (1/f) for 200 Å carbon film. 75° take-off angle except for conditions plotted in inset. Dash-dotted lines are for electron absorption only (*i.e.*, leaving out exponential term for X-ray absorption in equation (1)).

to the total correction is greater than 1 percent for elements heavier than Na, provided the X-ray take-off angle is greater than 40° and the film-thickness difference not more than 200 Å. (3) Corrections are always significant at acceleration potentials of 10 kV or less. They are very important when analyzing elements with characteristic lines in the long wavelength region, *e.g.*, elements with atomic numbers less than 11 (Na). Controlling the film thicknesses on specimens and standards to close tolerances is an essential requirement for accurate analyses in these cases.

It may be concluded that carbon films have a negligible effect on measured X-ray intensity-ratios in a great number of cases commonly encountered in quantitative electron-probe analysis. However, if quantitative measurements are attempted at very low accelerating potentials (less than about 10 kV) or for very low atomic number elements (less than about 11, Na), good control of the film thickness is important.

References

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