ELECTRON MICROPROBE ANALYSIS AT LOW OPERATING VOLTAGE: DISCUSSION

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ABSTRACT

Recently Desborough *et al.* (1971a, b) proposed using an unusually low accelerating voltage of 6 kV for quantitative microprobe analysis of minerals for Mg, Al, Si, and S, in order to minimise X–ray absorption effects. Calculations presented here show that it is difficult if not impossible to control the thickness of the conducting coating accurately enough to avoid significant analytical errors at 6 kV.

Desborough *et al.* (1971a, b) proposed that quantitative microprobe analysis of minerals could be simplified by using a low voltage to accelerate the incident electron beam. Experiments on Mg, Al, Si, and S in a range of samples showed that plots of X–ray count–rate versus concentration in weight percent were much more nearly linear at 6 kV than at the commonly used accelerating voltage of 15 kV. This is because the depth of penetration of the electrons is strongly dependent on the accelerating voltage, and if the depth is reduced, emerging X–rays have a shorter path in the specimen and are absorbed less.

However, the shallow depth of penetration at 6 kV has a disadvantage not discussed by these authors. Electrically non–conducting specimens have to be given a conducting surface coating, and since incident electrons lose energy in this layer, less energy is available for X–ray generation in the specimen. If the resulting loss of X–ray intensity were constant it would be unimportant, but the following calculation shows that it is probably impossible to maintain a sufficiently constant coating thickness to avoid significant errors at 6 kV.

The percentage intensity loss (ΔI) can be calculated from the equation (Reed, 1964): $\Delta I = 8.3 \, \rho x / (V_o^2 - V_c^2)$, where x is the coating thickness in nm, and ρ is the density of the coating in g/cm^2; V_o is the accelerating voltage, and V_c is the critical excitation potential of the X–ray line concerned, both in kV. Carbon is the commonest coating material. Sweatman and Long (1969) found $\rho = 1.3$ for evaporated carbon. Substituting a typical thickness: $x = 20$ nm., and $V_o = 6$ kV, the calculated values of ΔI for the elements considered by

Desborough *et al.* lie between 6.3 percent and 7.2 percent. It follows that for X–ray intensity measurements accurate to ±0.5 percent, the variation in x must not exceed ±7 percent, or ±1.4 nm.

Such constancy of coating thickness requires very good control of the evaporation process, and may be unattainable in practice, especially considering that the fraction of evaporated carbon atoms that stick to the surface varies with the coated material. This suggests that the best analytical accuracy is obtained with a compromise accelerating voltage, probably in the range 10–15 kV, chosen to reduce the effect of coating thickness, while avoiding excessive absorption. The problem of the absorption correction is not serious, since corrections can be calculated with good accuracy even for higher accelerating voltages, as shown by Sweatman and Long (1969).

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