DOI: 10.1017/S1431927605501983
Microsc Microanal 11(Suppl 2), 2005

Is the Energy Loss of Scattered Primary Electrons a Further Complication with X-ray Microanalysis in VPSEM?

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X-ray microanalysis in variable pressure scanning electron microscopes (VPSEM) is complicated by the processes unique to the variable pressure environment that occur between the electrons, ions, detectors and sample. Careful attention has been paid to primary beam electron scatter and its negative effects on the spatial resolution of the x-ray microanalysis, with various options reported to minimize this effect [1,2]. Secondary electron emission and gas amplification is also considered well understood although the mechanisms behind various image contrasts remain uncertain [3,4].

The imaging and microanalysis of insulating samples give rise to a further range of interesting phenomena that are charge related. At the restricted chamber pressures that most VPSEM operate at, these charging effects can be also minimized with care. Despite these, and other studies, the collection of quantitative x-ray data by EDS in VPSEM remains problematic [5].

One aspect of these processes that has received little attention is the question of the magnitude of the energy loss of the primary electron beam resulting from the initial scattering to the sample. To measure this potential effect, spectra from a range of copper samples have been collected at an accelerating voltage (11.5 kV) just above the Ka excitation energy under different operating conditions. With such a low overvoltage ratio the intensity of the Ka peak relative to the strongly excited La peak is expected to be very sensitive to variations of primary beam electron energy, as has been shown in studies of charging phenomena [6].

The results for variation of the Cu L/K ratio with accelerating voltage at our threshold chamber gas pressure of 0.2 torr are coherent and provide a calibration (fig. 1). The Cu L/K data then collected against chamber gas pressure at a constant accelerating voltage show a strong and linear relationship (fig. 2). Under the extreme experimental condition of a gas chamber pressure of 10 torr an extrapolated energy loss of 400v has been recorded.

The result is significant and from a simple perspective the effect will affect data reduction of x-ray data sets collected under high chamber gas pressure. Perhaps more importantly the result must be understood as a nett measurement representing the minimum energy loss. The energy loss must increase radially if the loss is scattering related. An alternative explanation for this energy loss is that it is a retardation effect of a positive space charge above the sample. In this case it should not result in a progressively increasing energy loss radially away from the primary beam axis.

A series of Cu annuli are being prepared to provide an experimental resolution. Under constant conditions the Cu L/K ratio is predicted to decrease as the radius of the annulus is increased, ie progressively more highly scattered primary beam electrons generate the Cu x-ray spectrum.

The data illustrate a further complexity inherent within x-ray data collected under variable pressure operating conditions.

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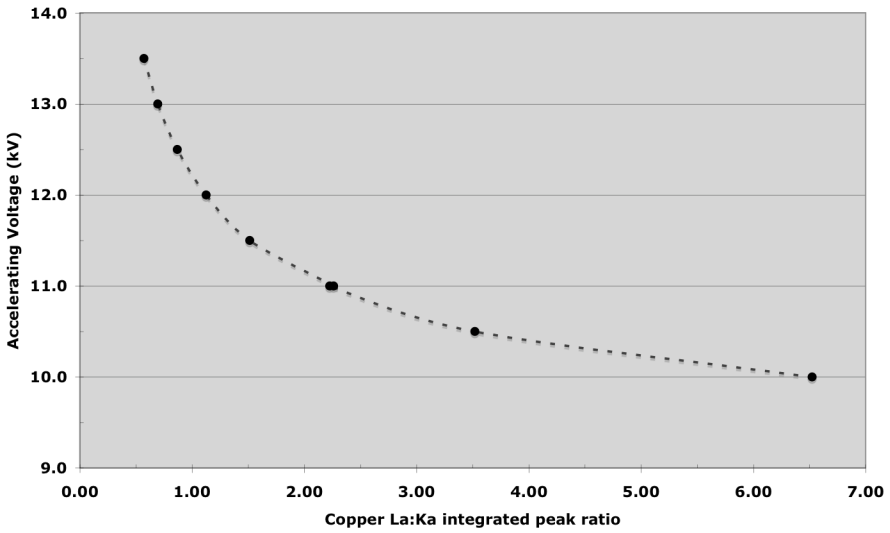

Figure 1: Measured variation in copper La:Ka integrated X-ray peak ratios with a range of accelerating voltage. The analytical conditions were chamber pressure 0.3 torr H_20, WD 8.2 mm, GSE detector bias 676v, using an Oxford Instruments ISIS system with a Be-windowed EDS.

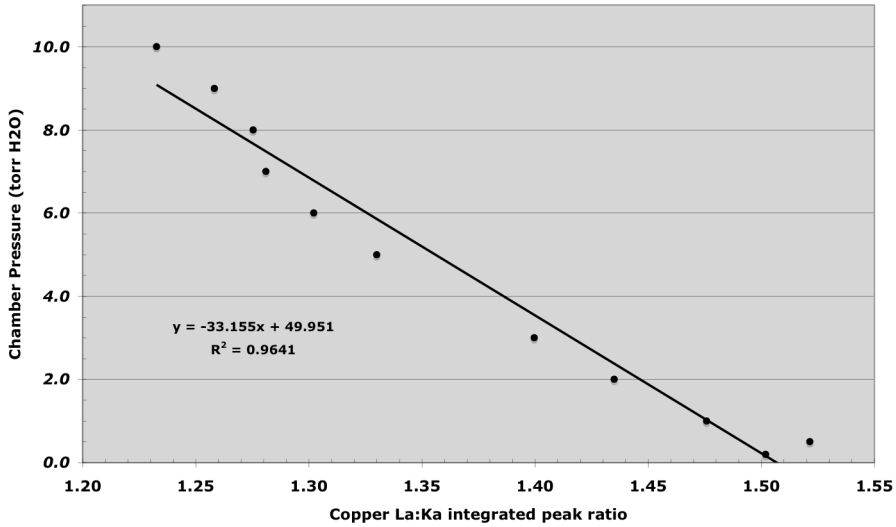

Figure 2: Measured variation in copper La:Ka integrated X-ray peak ratios with a range of chamber gas pressure at constant accelerating voltage (11.5 kV). Analytical conditions and sample as per figure 1.

https://doi.org/10.1017/S1431927605501983 Published online by Cambridge University Press