

X-ray Microanalysis of Insulators in a Variable Pressure Environment

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In a low vacuum environment, electric fields generated by ionized gas molecules and sub-surface trapped charge (Q) can alter the primary electron landing energy (ϵ^{DHL}). Consequent artifacts in x-ray microanalysis can be alleviated by working under conditions whereby the net electric field (\underline{E}) is dominated by the component produced by gaseous ions ($\underline{E}^{\text{ION}}$), and excess ions are rapidly removed *via* efficient ion neutralization routes. Such conditions can be attained over a wide of microscope operating parameters simply by employment of appropriate sample-electrode geometries.

In a variable pressure/environmental SEM, the electric field at each point (x,y,z) in the space between the sample and the pole piece typically consists of three distinct components:

$$\underline{E}(x,y,z) = \underline{E}^{\text{E}}(x,y,z) + \underline{E}^{\text{Q}}(x,y,z) + \underline{E}^{\text{ION}}(x,y,z) \quad (1)$$

where \underline{E}^{E} is the field generated by a biased electrode (ie, the electron collector of a gaseous electron detector [1]) and \underline{E}^{Q} is the field produced by Q. If the specimen is an uncoated insulator, ϵ^{DHL} and the maximum bremsstrahlung x-ray energy (the Duane-Hunt limit, DHL) are given by:

$$\epsilon^{\text{DHL}} = eV^{\text{A}} + e(\Delta V^{\text{E}} + \Delta V^{\text{Q}} + \Delta V^{\text{ION}}) = eV^{\text{A}} + e\Delta V \quad (2)$$

where e is the charge of an electron, V^{A} is the primary electron accelerating voltage, and ΔV is the net potential difference between the pole piece and the sample surface corresponding to \underline{E}^{E} , \underline{E}^{Q} and $\underline{E}^{\text{ION}}$. The sign of ΔV^{Q} is determined by the net polarity of sub-surface charge, as in the case of high vacuum SEM [2,3]. However, in contrast to high vacuum SEM, ΔV^{ION} can cause a significant increase in ϵ^{DHL} , alter the overvoltage and compromise x-ray quantification procedures. Such increases in ϵ^{DHL} are illustrated by the energy-dispersive x-ray spectra shown in Fig. 1, acquired as a function of electrode bias (V^{E}) and gas pressure (P). The data clearly illustrate that ϵ^{DHL} scales with V^{E} and P. This behavior is attributed to the influence of V^{E} and P on \underline{E}^{E} , $\underline{E}^{\text{ION}}$ and \underline{E}^{Q} , and consequent effects of ΔV on ϵ^{DHL} (see Eqn. 2). We will present a detailed model of ϵ^{DHL} behavior in a low vacuum environment, based on knowledge of the polarity of ΔV^{Q} and ΔV^{ION} , obtained from simultaneous measurements of x-ray spectra and Q-induced changes in the SE emission current.

From a practical viewpoint, it is desirable to eliminate the changes in ϵ^{DHL} caused by ΔV without imposition of restrictions on operating parameters such as V^{A} , V^{E} , working distance and gas pressure. On the basis of the aforementioned model, this can be achieved if: (i) ΔV^{E} is minimized, (ii) $\Delta V^{\text{ION}} > \Delta V^{\text{Q}}$, and (iii) excess ions are rapidly neutralized so that: $\Delta V^{\text{ION}} + \Delta V^{\text{Q}} \approx 0$. In a low vacuum environment such conditions can be attained simply by employment of appropriate sample-electrode geometries in the specimen chamber. Equipotentials calculated for two-dimensional representations of two such geometries are shown in Fig. 2. The effectiveness of these geometries in reducing ΔV -induced ϵ^{DHL} shifts is demonstrated by the corresponding x-ray spectra also shown in Fig. 2. These results will be explained using the abovementioned model. [4]

References

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 [3] L. Reimer, *Scanning Electron Microscopy*, Springer, Berlin, 1984.
 [4] This work was sponsored by EPSRC (grant number GR/M90139) and FEI corporation.

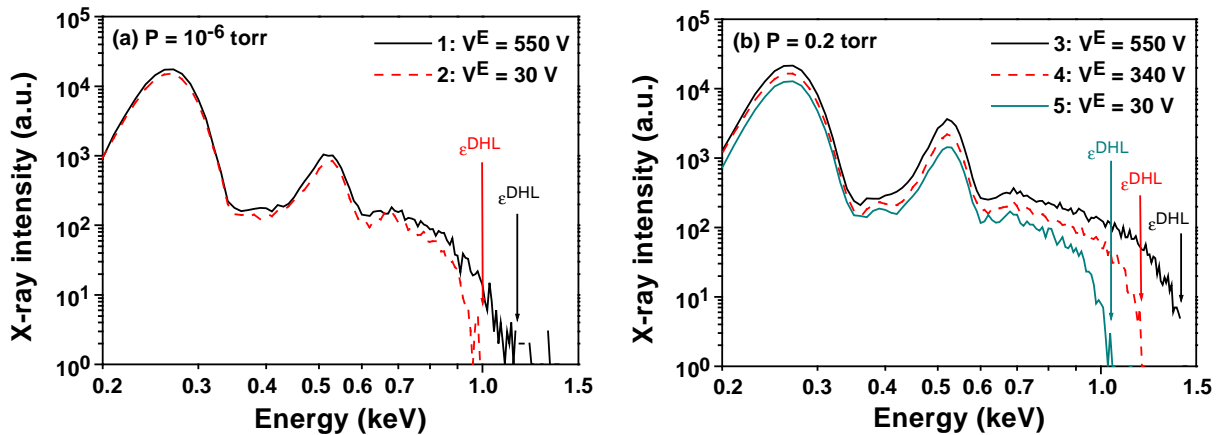


FIG. 1. Energy-dispersive x-ray spectra acquired consecutively from the same region of mica, in the order shown in the figures, as a function of electrode bias (V^E) and water vapor pressure (P): (a) $P = 10^{-6}$ torr, (b) $P = 0.2$ torr. The data show that ϵ^{DHL} scales with V^E and P [$V^A = 1$ kV, working distance = 10 mm, electrode-sample separation = 4.5 mm, horizontal field width = 130 microns].

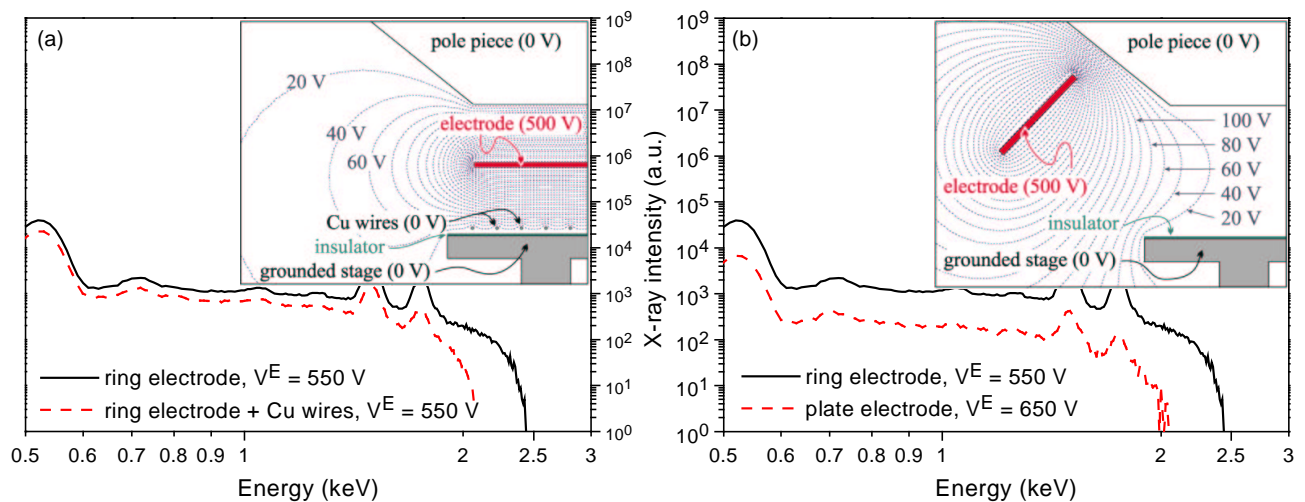


FIG. 2. X-ray spectra of mica showing differences between ϵ^{DHL} in data acquired when a ring electrode was placed 4.5 mm above the sample (“standard” geometry) and: (a) an array of grounded Cu wires was placed 0.55 mm above the specimen, or (b) the ring electrode was replaced with an off-axis plate electrode. [$V^A = 2$ kV, $P = 0.5$ torr, working distance = 10 mm, horizontal field width = 130 microns]. The insets show the electric equipotentials (broken lines) calculated for simplified two-dimensional representations of the abovementioned geometries [$V^E = 500$ V]. The spectra show that employment of these geometries serves to reduce Duane-Hunt shifts caused by gaseous ions.