X-ray Microanalysis of Insulators in a Variable Pressure Environment

M. Toth*, J.P. Craven*, M.R. Phillips**, B.L. Thiel* and A.M. Donald*

* Polymers and Colloids Group, Cavendish Laboratory, University of Cambridge, Madingley Road, Cambridge, CB3 0HE, U.K.
** Microstructural Analysis Unit, University of Technology Sydney, Broadway, NSW 2007, Australia

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 (~E) is dominated by the component produced by gaseous ions (~E_{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:

\[ \mathbf{E}(x,y,z) = \mathbf{E}(x,y,z) + \mathbf{E}^Q(x,y,z) + \mathbf{E}^{ION}(x,y,z) \] (1)

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

\[ eDHL = eV^A + e(\Delta V^E + \Delta V^Q + \Delta V^{ION}) = eV^A + e\Delta V \] (2)

where \( e \) is the charge of an electron, \( V^A \) is the primary electron accelerating voltage, and \( \Delta V \) is the net potential difference between the pole piece and the sample surface corresponding to \( \mathbf{E}^E \), \( \mathbf{E}^Q \) and \( \mathbf{E}^{ION} \). The sign of \( \Delta V^Q \) 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, \( \Delta V^{ION} \) can cause a significant increase in \( eDHL \), alter the overvoltage and compromise x-ray quantification procedures. Such increases in \( eDHL \) 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 \( eDHL \) scales with \( V^E \) and P. This behavior is attributed to the influence of \( V^E \) and P on \( \mathbf{E}^E \), \( \mathbf{E}^{ION} \) and \( \mathbf{E}^Q \), and consequent effects of \( \Delta V \) on \( eDHL \) (see Eqn. 2). We will present a detailed model of \( eDHL \) behavior in a low vacuum environment, based on knowledge of the polarity of \( \Delta V^E \) and \( \Delta V^{ION} \), 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 \( eDHL \) caused by \( \Delta V \) 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) \( \Delta V^E \) is minimized, (ii) \( \Delta V^{ION} > \Delta V^Q \), and (iii) excess ions are rapidly neutralized so that: \( \Delta V^{ION} + \Delta V^Q \approx 0 \). 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 \( \Delta V \)-induced \( eDHL \) 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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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 (VE) and water vapor pressure (P): (a) P = 10⁻⁶ torr, (b) P = 0.2 torr. The data show that ε_DHL scales with VE and P [VE = 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 [VE = 500 V]. The spectra show that employment of these geometries serves to reduce Duane-Hunt shifts caused by gaseous ions.
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