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# 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 ($\varepsilon^{DHL}$). 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 ($\underline{E}^{ION}$), 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:

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

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

$$\varepsilon^{DHL} = eV^{A} + e(\Delta V^{E} + \Delta V^{Q} + \Delta V^{ION}) = eV^{A} + e\Delta V \qquad (2)$$

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 $\underline{E}^{E}$, $\underline{E}^{Q}$ and $\underline{E}^{ION}$. 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 $\varepsilon^{DHL}$, alter the overvoltage and compromise x-ray quantification procedures. Such increases in $\varepsilon^{DHL}$ 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 $\varepsilon^{DHL}$ scales with $V^{E}$ and P. This behavior is attributed to the influence of $V^{E}$ and P on $\underline{E}^{E}$, $\underline{E}^{ION}$ and $\underline{E}^{Q}$, and consequent effects of $\Delta V$ on $\varepsilon^{DHL}$ (see Eqn. 2). We will present a detailed model of $\varepsilon^{DHL}$ behavior in a low vacuum environment, based on knowledge of the polarity of $\Delta V^{Q}$ 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 $\varepsilon^{DHL}$ 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 $\varepsilon^{DHL}$ 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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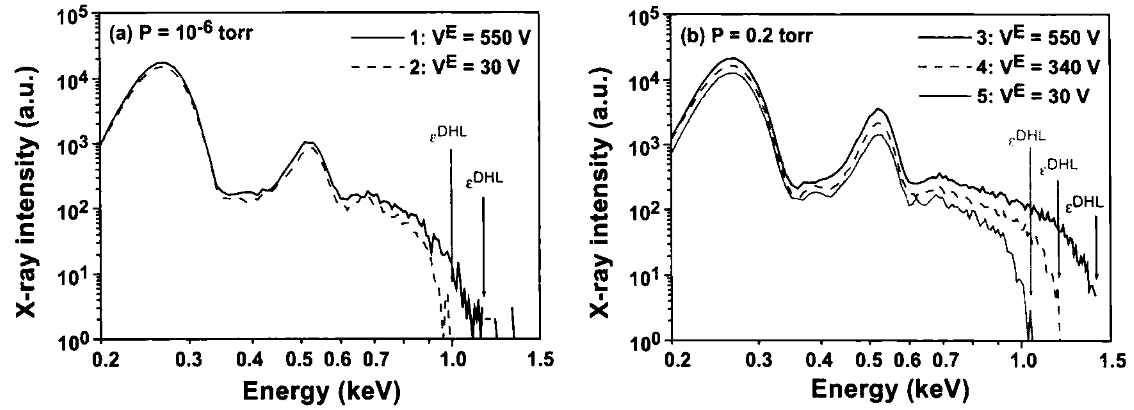

FIG. 1. Energy-dispersive x-ray spectra acquired consecutively from the same region of mica, in the order shown in the figures, as a function of electrode bias ($V^E$) and water vapor pressure (P): (a) P = $10^{-6}$ torr, (b) P = 0.2 torr. The data show that $\varepsilon^{DHL}$ scales with $V^E$ and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

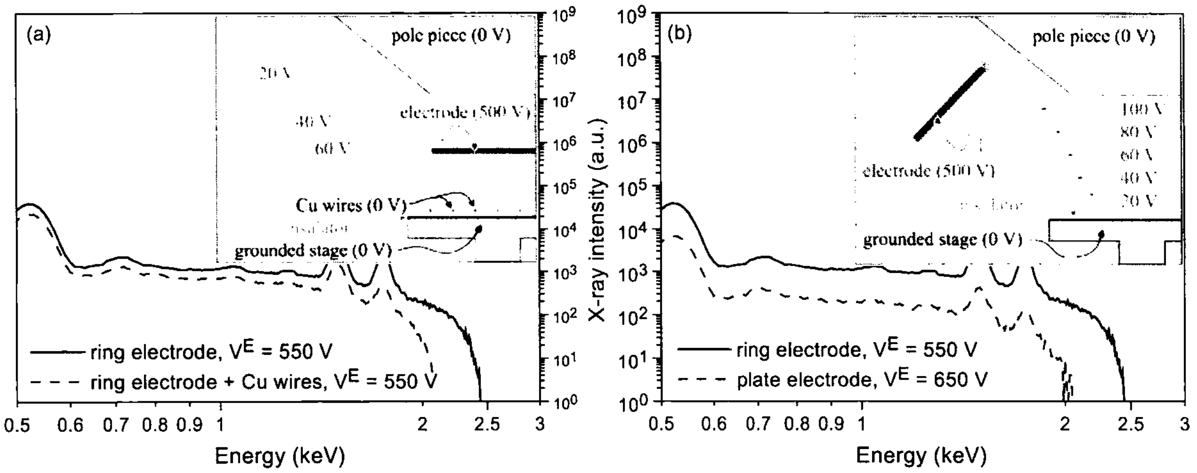

FIG. 2. X-ray spectra of mica showing differences between $\varepsilon^{DHL}$ in data acquired when a ring electrode was placed 4.5 mm above the sample ("standard" geometry) and: (a) an array of grounded Cu wires was placed 0.55 mm above the specimen, or (b) the ring electrode was replaced with an off-axis 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.

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