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Residual Surface Potentials in the Variable Pressure/ Environmental Scanning Electron Microscope

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The recent rapid development and enthusiastic uptake of variable pressure/ environmental electron microscope technology, underlines the importance of understanding the processes of electron irradiation of poorly conducting materials in ionized gaseous environments. In the conventional high vacuum operating mode ($<10^{-4}$ Pa) of a Scanning Electron Microscope (SEM), electron beam induced charging of poorly conducting specimens is minimized using a thin grounded conductive coating, which provides an electrostatic barrier, preventing deflection and/ or retarding of the incident electron beam. The coating also provides a protective physical barrier between the specimen and residual gases in the experimental chamber. The high vacuum constraint is removed in the more recently developed Variable Pressure/ Environmental SEM (VP/ ESEM), which operates with a low pressure (typically a few hundred Pa) of an environmental gas in the specimen chamber. [1] The secondary electrons resulting from electron irradiation ionize a proportion of the environmental gas in the vicinity of the specimen surface. These environmental ions balance excess charge at the surface allowing the imaging and microanalysis of poorly conducting materials without coating. [2,3]. The specimen surface is exposed to the environmental gases and ions, and the electric fields are not terminated at the surface, as is the case with a grounded conductive coating.

Significant subsurface charging may still occur in poorly conducting materials, despite the charging of the specimen surface being minimized using grounded conductive coatings in conventional high vacuum mode, and environmental ions in environmental mode. A small fraction ($<10^{-6}$) of the incident electrons are trapped at irradiation induced and/ or pre-existing defects resulting in a highly localized electric field that induces electro-migration of mobile charged defect species within the irradiated specimen micro-volume. [4,5]

Electron beam irradiation in a SEM under conventional high vacuum conditions has previously been shown to induce modification of grounded coated poorly conducting specimens due to the subsurface trapped charge induced electric field. In this work, silicon dioxide polymorphs have been chosen as the test specimens, because they have a wide band gap and are therefore susceptible to charging induced effects. In addition, electron irradiation induced changes in the microstructure of silicon dioxide polymorphs are well characterized. [5-7] The irradiation damage is associated with characteristic reproducible substantial volume changes in the surface regions of these specimens. Thus the irradiated regions can unambiguously be located for subsequent investigation. [6,7]

The influence of irradiation induced electric fields and residual potentials on silicon dioxide polymorphs in a VP/ ESEM, has been investigated using a combination of scanning probe microscopy techniques. Electric Force Microscopy (EFM) and Surface Potential Microscopy/ Kelvin Probe Microscopy have been used to directly map the induced electric field gradient and residual surface potential respectively in specimens irradiated in an *FEI* XL30 and Quanta ESEMs (W gun). The electric fields and potentials induced in the specimens are associated with the trapping of charge,

and the emission of secondary electrons, in particular. The incident electron beam penetrates into the specimen and a small proportion of charge is trapped at pre-existing or irradiation induced defects. Thus irradiation induces a negative charge distribution within the irradiated micro-volume of specimen, which extends down to the maximum electron penetration depth. In contrast, there is a positive charge distribution localized in the surface region where the electron beam is incident. This positive charge distribution, which extends to a depth of a few nanometers into the specimen, is due to the emission of secondary electrons. In a Variable Pressure or Environmental SEM, an additional component of positive charging is provided by the incident environmental gas ions trapped at the surface. The residual potential is the net sum of the contributions from the potentials associated with the various regions of charge distribution induced in the specimen by electron beam-specimen interaction in an ionized gaseous environment.

The influence of electron beam energy and hence electron penetration depth and irradiation exposure, have been investigated. The investigations show that irradiation induced effects in environmental mode differ from, but are consistent with those observed in the conventional high vacuum limit. In addition the influence of specific variable pressure/ environmental parameters including the secondary electron detector bias, and environmental gas species have also been investigated.

This work gives insight into the possible interactions between the electron beam, the environmental gas ions and poorly conducting specimens. It is essential to assess the influence of the electron beam probe and environmental ions to ensure accurate microanalysis. The observed structural/ chemical modifications (including enhanced desorption), electromigration of mobile charged defect species (including the environmental ions) within the irradiated microvolume [5] and the field and potential induced influences on specimen electron interaction in an VP/ESEM have important consequences for the analysis and interpretation of data including images.

References

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