Letters

The Effect of Glow Discharge on Insulators as observed in the Scanning Electron Microscope

We wish to report that the secondary electron emission of certain insulators is affected by prior glow discharge treatment. This observation is made in the scanning electron microscope, and the change in emission is found to be dependent on the gas used in the discharge.

The glow discharge was produced by applying a 50 Hz alternating current high tension (5 kV) between high purity aluminium ring electrodes in a gas at a pressure range of 0.01 to 0.1 torr and was sustained for a time of 15 min. The material located in the positive column of the discharge, was shielded from direct line of sight of the electrodes ensuring that it was not subjected to high energy bombardment [1]. Two types of vacuum system were used: an unbaked, oil-diffusion pumped system having a residual pressure of 10^{-5} torr and an oil-free, getter-ion pumped stainless steel ultra-high vacuum system which received a 10 h bake at 250° C to achieve a residual pressure of 10^{-9} torr. In the ultra-high vacuum system, mass-spectrometric analysis indicated that the total hydrocarbon partial pressure was in the low 10^{-11} torr range. The effect of glow discharge treatment on the secondary electron emission of the insulators was the same for both types of system.

One half of the specimen area was masked from the discharge by an earthed metal mask; this enabled direct comparison of the effect of the discharge to be made with an area that had been hidden from the discharge. After the discharge, the material was transferred to the scanning electron microscope ("Stereoscan", Cambridge Scientific Instruments) and a difference in emission between the masked and the exposed sides was observed, as in fig. 1. When an air or oxygen discharge is used the emission from the area subjected to the discharge is enhanced; when the gas is nitrogen, hydrogen, argon or ammonia the emission is decreased. The insulators were observed in the scanning electron microscope without prior metallising and the effect could be seen clearly at low magnifications.

Figure 1 Accelerating voltage 20 kV. Soda-lime glass' brighter side exposed to air glow discharge for 15 min (× 18).

The effect has been observed on a variety of insulating materials including soda-lime glass, borosilicate glass, single crystal magnesium oxide, diamond, silicon single crystal, synthetic silica and natural single crystal quartz. In some materials, notably magnesium oxide and synthetic silica, the effect in the microscope can be seen for a few seconds only. This is because these materials charge up considerably under the electron beam, and the resultant increase in collected electron current swamps the smaller differences between the glow discharged and the non-glow discharged surfaces. This problem is present to some extent in all cases and often makes photographic recording of the effect extremely difficult.

With the exception of diamond, the effect could be seen only when the microscope was operated in the secondary electron collection mode. In diamond the effect could also be detected in the back-scattered primary electron collection mode, but only when all the condenser lenses were turned off, i.e. under intense primary beam bombardment.

The emission difference could be still observed on substrates that had been standing in laboratory atmosphere for many hours (in some cases many days) after the glow discharge had been performed. The disappearance of the effect could be hastened by baking the material in air at 150° C for a number of hours.

With silicon (*n*-type with the (1 1 1) plane parallel to the surface) there appears to be a definite dependence on resistivity. The effect could not be seen on silicon single crystal of resistivity of 10^{-2} Ω cm and although it was just visible on silicon of resistivity 10^{+2} it could plainly be seen when the resistivity was 10^{+4} Ω cm.

With all the materials the effect could be seen at all values of the accelerating voltage of the beam (from 1 to 20 kV) and the existence of the effect was independent of the orientation of the materials with respect to the electron beam.

In the positive column of the glow discharge the specimen is bombarded by low energy positive ions and electrons, by excited atoms and molecules and by various radiations. Although it is known that some disturbance of the electrical properties of insulator materials takes place [2], in the particular case of soda-lime glass no net electric charge could be detected to a limit of 10^{-15} C mm^{-2} by electrometer probe techniques [3].

In the discharge the number of surface atoms in excited energy states is changed. A possible explanation for the altered emission is that the secondary electron emission changes as the number of electrons in excited states change. The predominant mechanism for the energy transfer from the plasma to the surface atoms will be dependent on the material.

Under the influence of the discharge and the presence of excited atoms, gas atoms are sorbed into the glass surface [4]. This is supported by our observations that changes in the ellipticity of glass surfaces occur after glow discharge exposure, which are interpreted in terms of compositional changes of the surface layers. In the particular case of glass (and other oxygen containing insulators) a possible explanation for the altered emission is that when the sorbed atoms are electron acceptors (e.g. oxygen) the number of electrons in excited energy states is increased by a process of electron transfer from the doubly-ionised oxygen ions of the glass to the sorbed oxygen atoms. These electrons are then more easily emitted under the action of the electron beam in the microscope leading to enhanced emission. A decrease in the number of electrons in excited energy states compared with the untreated glass will occur when the sorbed gas atoms (e.g. nitrogen, hydrogen, argon) displace oxygen from the glass [5].

The above explanation can account qualitatively for some of the characteristics of the effect. Its persistence is in accordance with the expected long lifetimes of electrons in excited states, and also the decay of the effect by heating is expected, as heating would restore the normal population of electrons in excited states in the glass. The dependence of the change in emission on the resistivity of silicon (which has an oxide layer of about 2 nm thickness) is caused by the effect of resistivity on the time of occupation of excited states. That surface charge effects can be induced by a gas discharge on semiconductors and silicon, and that relaxation of these effects may take many hours has been demonstrated by Estrup [6].

The above explanation cannot account explicitly for the difference in emission from diamond after glow discharge, but an energy transfer from the plasma to the atoms of the surface such as suggested by Dauchot *et al* [7] may be applicable. They have observed that electrons could be raised to excited energy states in zinc oxide by bombardment with thermally-excited or plasma-excited molecules of carbon dioxide, and they deduced that the effect was due to energy transfer from neutral, vibrationally-excited carbon dioxide molecules to the electrons of zinc oxide.

The work of this letter may be of importance in the behaviour of ionisation devices. In the operation of an ion gauge, particularly if used as a pump, gas will be sorbed into the glass envelope and electrode structure. Our work shows that the electron emissivity of glass (stimulated by electron beam energies down to 1 keV) depends on the nature of the gas sorbed. If during gauge operation the glass envelope is bombarded by electrons then the yield of secondary electrons will depend on the nature of the gas in which the gauge was pumped down. This variable number of additional electrons could lead to a variable number of positive ions, i.e. to a variable indicated pressure.

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On the Estimation of Dislocation Densities in Deformed Metals from Small Angle Scattering Data

The dislocations produced in a specimen by deformation can be studied by means of double Bragg X-ray and neutron scatterings [1, 5]. The dislocation density can be estimated, if the distribution in tilt angles of the subgrains is known. Two kinds of substructure models have been used in the interpretation of the small angle measurements. In the first model it has been assumed that the subgrain normals are uniformly distributed over a cone of small half-angle δ [4, 5]. The other model is based on the assumption that the distribution of the normals obeys a Gaussian curve [1, 3, 4].

The dislocation density, D, can be estimated by equation [2]

$$D = \frac{\alpha^2}{b^2}, \tag{1}$$

where b is the Burgers vector and α is the mean angle between two neighbouring subgrains.

If the subgrains are distributed about the mean position in the form of a Gaussian distribution, then [2]

$$\alpha = \frac{\int_{-\infty}^{+\infty}\int_{-\infty}^{+\infty} |\,\theta - \phi\,|\, \mathrm{e}^{-k^2(\theta^2+\phi^2)}\, \mathrm{d}\theta \mathrm{d}\phi}{\int_{-\infty}^{+\infty}\int_{-\infty}^{+\infty} \mathrm{e}^{-k^2(\theta^2+\phi^2)}\, \mathrm{d}\theta \mathrm{d}\phi} = \frac{J}{J_0} \tag{2}$$

The value of α has been solved graphically by Gay, Hirsch and Kelly [2].

It may readily be shown that

$$J = \frac{\sqrt{2\pi}}{k^3} \tag{3a}$$

$$J_0 = \frac{\pi}{k^2} \tag{3b}$$

whence

$$\alpha = \frac{\sqrt{2}}{\sqrt{\pi}}\frac{1}{k} = 0.45\,\epsilon_{\mathrm{m}}, \tag{4}$$

where

$$\epsilon_{\mathrm{m}} = \int_{-\infty}^{+\infty} \mathrm{e}^{-k^2\epsilon^2}\, \mathrm{d}\epsilon = \frac{\sqrt{\pi}}{k}\,.$$

Finally, we can write

$$D = \frac{\alpha^2}{b^2} = \frac{2}{\pi k^2 b^2} = \frac{2{\epsilon_{\mathrm{m}}}^2}{\pi^2 b^2}\,. \tag{5}$$

When the subgrain normals are uniformly distributed over a cone of half-angle δ, we get, immediately, on the basis of equations 1 and 2

$$D = \frac{4}{9}\frac{\delta^2}{b^2} \tag{6}$$

which is given also by Taglauer [5].

The values of the parameters k and δ in equations 5 and 6 can be determined by the small angle measurements. Thus, it is possible to estimate the dislocation density in deformed specimens [1, 3, 5].

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