INVESTIGATION OF INSULATOR CHARGING PROPERTIES WITH A SCANNING ELECTRON MICROSCOPE: DOSE AND CURRENT DENSITY EFFECTS

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Abstract

Fundamental aspect of the charging properties of insulators is investigated with a Scanning Electron Microscope, by meaning the amount of trapped charges and the Secondary Electron Emission yield as a function of dose and current densities, in a wide energy range (~200 eV to 40 keV).

Three charging regimes are observed, depending on the current density. At low current densities (J ≈ 2.10^4 pA/cm^2) a self-regulated regime is obtained, characterised by the formation of a static space charge build up which prevents any further charge accumulation. At intermediate current densities (2.10^4 < J < 7.10^6 pA/cm^2) a small fraction (a few %) of incoming electrons is continuously absorbed in proportion to the injected dose. The consequence of this invasion is an internal stress which affects the life time of the material; this is the ageing regime. At high current densities strong relaxations occur, producing an irreversible damaging of the material; this is the degradation regime. These relaxations are probably at the origin of the partial discharges observed in insulators at a pC level.

Keywords: Insulators; Charging regime; SEM.

1. Introduction

Mobility and trapping [1,2] of charges have been extensively studied over the last two decades either by specific methods [3] or in association with microanalytical techniques [4]. In particular, the characterisation of insulating materials by Scanning Electron Microscopy is being performed for some years by using the so-called "Mirror Method" [5]. Here, a new methodology is developed which allows a more wide investigation of the charging properties by studying the Secondary Electron Emission (SEE) yield δ in a large energy range, as a function of the primary electron current density and electron dose. This methodology has revealed the existence of three charging regimes which condition the insulating capabilities and the life time of materials: the self-regulated regime, the ageing regime and the degradation regime.

2. Experimental

The SEM used in these experiments is equipped with:
(i) a column aligned by computer control, delivering an electron beam whose energy is ranging from 100 eV to several tens of keV and intensity from pA to some tens of nA or more. These characteristics allow positive and negative charging to be investigated in a wide energy and current density domain.
(ii) two low noise detectors. The first one measures the induced current (I IC) produced in the metal sample holder and the second one collects the current (I SB) produced by secondary and backscattered electrons.

The complementarity of the two detectors imposes:
\[ I_{IC} + I_{SB} = I_0 \]  
where I_0 is the primary electron beam current. The SEE yield (including backscattered electrons) δ is then given by:
\[ \delta = \frac{I_{SB}}{I_0} = 1 - \frac{I_{IC}}{I_0} \]

To ensure the complementarity of the two detectors, the secondary and backscattered electron detector must be positively polarised. By applying a bias voltage greater than 100 V on it, complementarity of the two currents is achieved at the precision of the measurements (few %). The positive bias voltage has also the advantage to prevent electrons to come back to the sample surface when it is positively charged.

Under these conditions, it is possible to study the variation of δ with the trapped charge and current density values with a good accuracy. Experiments are carried out by defocusing the electron beam of the SEM in the spot mode and irradiating the sample surface with a sequence of short electron shots. I IC and I SB are measured in the course of each shot. Assuming that during a shot...
charges are trapped uniformly at the sample surface on an area of diameter $a$, the surface potential $V_S$ produced by the amount $Q_T$ of trapped charges can be approximately express as:

$$V_S = \frac{K Q_T}{2\pi \varepsilon_0 a}$$

(3)

where $K = \frac{2}{1 + \varepsilon_r}$, $\varepsilon_r$ being the relative permittivity of the material.

Taking $a = 100 \mu$m in the spot mode and $Q_T = \pm 1 \text{pC}$ one obtains $V_S = \pm 72 \text{V}$ for silica ($\varepsilon_r = 4$). This high surface potential value will certainly produce a sharp variation of $\delta$ during the shot. Then, it is clear that an accurate study of the trapped charge dependence of $\delta$ requires severe experimental conditions. Typically each electron shot delivers 0.1 pC on an area of 500 µm diameter.

An example of the response of amorphous silica to the first 0.2 pC shot of 500 eV electrons is shown in figure 1. The induced current being negative, the sample is positively charged. From the amount of trapped charges $Q_T = \pm 0.481 \text{pC}$ a surface potential of 7 V is deduced. The fact that $I_{IC}$ is constant in this case, indicates that the SEE yield $\delta$ has not changed appreciably during the shot.

![Figure 1](image)

\textbf{Figure 1} : Induced and secondary currents responses of amorphous silica to 0.2 pC electron shot at 500 eV with a current density $J = 12000 \text{pA/cm}^2$.

Therefore the value $\delta_0 = 3.45$ deduced from the measurement corresponds to the intrinsic SEE yield of the uncharged material bombarded with 500 eV electrons.

3. Charging regimes of insulators

Materials studied here are thick alumina and amorphous silica samples for which conduction current is completely negligible. It results that the induced charge

$$Q_{IC} = \int_{0}^{\tau} I_{IC} \, dt$$

(4)

where $\tau$ is the dwell time of the primary beam, is just the opposite of the trapped charge amount $Q_T = -Q_{IC}$.

Three charging regimes have been observed.

3.1. The self-regulated regime

Keeping constant the conditions mentioned in figure 1 (electron energy 500 eV and $J = 12000 \text{pA/cm}^2$), the induced current $I_{IC}$ decreases shot after shot as the injected dose increases. It results a decrease of $\delta$ which tends to unity. This evolution of $\delta$ is due to the progressive build-up of a positive trapped charge layer producing a higher and higher positive potential beneath the surface, which prevents electrons to escape.

The variation of the SEE yield $\delta$ with the injected charge density ($\text{pC/cm}^2$) is represented in figure 2 for polycrystalline $\alpha$-Al$_2$O$_3$ in the energy range where $\delta \geq 1$.

![Figure 2](image)

\textbf{Figure 2} : Variation of the SEE yield $\delta$ with the injected density of charges. In both cases the current density was about 6000 $\text{pA/cm}^2$. Numbers below arrows indicate the surface voltage $V_S$ when the self-regulated regime is reached ($\delta = 1$).

Once the value $\delta = 1$ is reached, a stationary state is achieved, characterised by a trapped charge amount and a surface potential which do
not vary any more with the irradiation dose. This is the self-regulated regime. For example, the self-regulated regime is obtained in α-Al₂O₃ irradiated with 600 eV electron energy (figure 2) for a charging density of 600 pC/cm² which corresponds to a net trapped charge concentration far below 1 ppm beneath the surface.

**Figure 3**: Current density dependence of the SEE yield $\delta$, in the self-regulated, ageing and degradation regime.

It is noticed in figure 2 that this regime is reached for a dose and surface potential which are getting closer to zero as the primary electron energy $E_p$ gets closer to the cross-over energy $E_2$ for which $\delta_0 = 1$. In the present case, the cross over energy $E_2$ was found to be about 2600 eV for alumina.

In a similar way, when $E_p > E_2$ that is $\delta \leq 1$ (a-SiO₂ in figure 2), $\delta$ increases progressively and tends to unity (self-regulated regime) as a negative trapped charge layer is built-up, producing a negative potential beneath the surface, which favours electrons to be emitted. An important feature to be mentioned is that in no case the energy $E_p + eV_s$ of primary electrons impinging on the surface equals the cross-over energy $E_2$ in the self-regulated regime, as it is often stated in the literature. In the self-regulated regime a positive / negative static space charge is built up which prevents any further charge accumulation.

### 3.2. The ageing regime

The self-regulated regime ($\delta = 1$) is maintained independent of the injected dose, provided the current density is less than about 2.10⁴ pA/cm². At higher current densities $\delta$ stabilises at values typically few % less than unity, whatever the energy of incident electrons in the range 200-6000 eV. For example in a-SiO₂ (figure 3) $\delta$ lies in the interval 0.85-0.98 for $10^4 \leq J \leq 7.10^6$.

The fact that $\delta$ stabilises at a value less than unity means that electrons are continuously absorbed, in proportion to the irradiation dose, even in the energy domain $E < E_2$ where the initial charging of the material is positive ($\delta_0 > 1$). This is the ageing regime. As in this regime a small fraction of primary electrons is continuously absorbed in proportion to the injected dose, it is concluded that the surface potential remains well above the negative voltage of primary electrons. Clearly, absorbed electrons do not accumulate at the surface where they would produce a giant negative potential repelling the incident beam, but spread deeply and deeply inside the material as irradiation proceeds. The insulator is literally invaded by electrons. The consequence of this invasion is an internal stress which affects the lifetime of the material. This is why this regime is called the ageing regime.

### 3.3. The degradation regime

At very high current densities, typically greater than few millions pA/cm² in a-SiO₂, strong electron relaxations are observed in the course of irradiation. Burst of electrons are suddenly released from the material, giving rise to high ISB peak intensities and depleted IIC intensities. These quasi-periodic relaxations are at the origin of an irreversible damaging process characterised by the molecular decomposition of the material. It is noticed that the charge involved in those electron relaxations is of the same magnitude as the sub-pC partial discharges observed by J. Sarjeant et al. [6] in composite ceramics. So, it is believed that the partial discharges are triggered by electron relaxation processes.

### 4. Conclusion

The fact that the charge regime depends upon the current density is a major feature which has to be taken into account in the design of devices where high energy electrons flux can interact with insulating materials.

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References
