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Dependence of secondary electron emission on surface charging in sapphire and polycrystalline alumina: Evaluation of the effective cross sections for recombination and trapping

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ABSTRACT

The evolution of the secondary electron emission from sapphire and polycrystalline alumina during electron irradiation, achieved in a scanning electron microscope at room temperature, is derived from the measurement of the induced and the secondary electron currents. The semi-logarithmic plot of the secondary electron emission yield versus the surface density of trapped charges displays a plateau followed by a linear variation. For positive charging, the slope of the linear part, whose value is of about 10^{-9} cm^2, is independent of the primary electron energy, the microstructure and the impurities. It is interpreted as an effective microscopic cross section for electron–hole recombination. For negative charging of sapphire, the slope is associated with an effective electron trapping cross section close to 10^{-11} cm^2, which can be assigned to the dominant impurity trap. These effective values reflect the multiple interactions leading to the accumulation of charges. The yield corresponding to the plateau is controlled by the initial density of impurity traps. A charge transport and trapping >model, based on simplifying assumptions, confirms qualitatively these inferences.

1. Introduction

The electron irradiation of insulators creates electron–hole pairs along the slowing down path of primary electrons. Some of the created and injected charges, once thermalized, can be trapped in defects. Concomitantly, a fraction of the free electrons can be emitted if they do not experience trapping or recombination during their transport to the surface. The evolution of the secondary electron emission (SEE) can therefore be traced back to the charging processes [1–9]. Hence, the SEE has been the subject of numerous studies motivated, for instance, by the development of materials able to limit the loss of linearity in electron multipliers [7], the electrostatic charging of spacecraft components [8], the spacer charging in field emission display [9] or to prevent the surface flashover in r. f. windows of klystron [5].

Experimental studies [2–5] have investigated the temporal variation of the net accumulated charge, the surface potential and the SEE for different incident electron energies and current densities in various insulators. It has been shown that the time evolution of the SEE, when irradiation is performed with low current densities, reaches a steady state, i.e., when the flux of electrons corresponding to primary current is balanced by the SEE one [1–3]. Simulation studies [10–12] have provided insights about the time evolution of the SEE, the temporal and spatial distributions of holes and electrons, the electric field and the surface potential. However, agreement of simulations with experiments still remains a challenge as it is hampered by the interplay of the charging mechanisms. In addition, the uncertainties on the values of relevant intrinsic parameters, such as the microscopic cross sections for recombination and trapping constitute a further hurdle. For instance, the suggested values of these quantities, obtained from experimental measurements in SiO_2, can vary within several orders of magnitude [13,14].

The purpose of this work is to provide experimental values of the effective recombination and trapping cross sections in alumina of various microstructures and impurity contents. They will be derived from a framework, which involves a correlation between the SEE and the trapped charge density as well as a charge transport and trapping >model that is based on simplifying assumptions. This will be made possible by achieving the irradiations (at room

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temperature), via the beam of a scanning electron microscope (SEM), under specific conditions that reduces the complexity of the charging analysis. To this end, the induced current method, completed by the measurement of the secondary electron (SE) current [1–3], will be used with a defocused beam and very low current in order to minimize the overlap of the electron slowing down cascades.

2. Materials and experimental method

2.1. Materials

Alumina materials of different microstructures (single crystals and polycrystals) and purities were investigated. Two types of α-alumina single crystals taken from a Verneuil-grown sapphire rod were considered. The first, manufactured from powder of very low impurity level (about 15 ppm) was provided by PI-KEM Co. (U.K.). The second, manufactured by RSA Co. (F.), contains about 400 ppm of various elements where silicon (290 ppm) is the dominant impurity. These samples ($1.2 \times 1.2 \times 0.2\ cm^3$) were polished to flat mirror surface polish, using successively finer grades of diamond pastes down to 1 μm. In order to anneal the defects induced by machining and polishing, a thermal treatment in air at 1773 K for 4 h was performed. The polycrystalline samples (1.6 cm diameter and 0.2 cm thickness) were processed, at E.N.S.M. of Saint-Etienne (F.), by sintering, from a powder containing about 150 ppm of different impurities, supplied by CRICERAM (F.). Solid state sintering, near theoretical density, was carried out in air and a mean grain diameter, d, of 4.5 μm was achieved [2].

Before electron irradiation, all the samples were washed in an alcohol ultrasonic bath and then thermally cleaned at 663 K, for three hours in vacuum (5×10^{-5} Pa) within the SEM chamber; to prevent any surface contamination that can alters the SEE [15].

2.2. Experimental method for the measurement of the SEE yield

The experimental set up and method, which were previously described in details [1–3], will be succinctly reviewed.

2.2.1. Experimental set up

The experiments are performed using a SEM (LEO 440), specially equipped (Fig. 1) to measure the SE current via the detector and to inject a controlled amount of charges, Q_{inj}, under a large spectrum of experimental conditions:

- energy of incident electrons, E_P varying from 300 to 40000 eV;
- current beam intensity, I_P, varying from a few pA to a few μA;
- irradiated area with a diameter, Φ, varying from a few nm (focused beam) and few hundred μm (defocused beam);
- injection time, t_{inj}, varying from 10^{-3} to 1 s.

To probe a zone representative of the material, we use a defocused beam over an area of 560 μm diameter [16]. The experiment are performed at room temperature with $I_P = 100$ pA that gives a primary current density $J_P = 4 \times 10^4\ pA/cm^2$.

2.2.2. The induced current measurement method

The induced current measurement (ICM) method is based on the measurement of the induced current I_{ind}, produced by the variation of the induced charges Q_{ind} (in the sample holder) due to the trapped charges in the sample Q_T [17]. As a result, the amount of the net trapped charges is:

$$Q_T(t) = -\frac{1}{K} Q_{ind}(t) = -\frac{1}{K} \int_0^t I_{ind}(t)\ \ dt \tag{1}$$

where K is the influence coefficient found close to 1 [2].

In the case of positive (negative) charging, the surface potential attracts (repels) low energy emitted secondary electrons which can return to the sample surface. This additional source of incoming electrons can be avoided by applying a sufficient positive voltage ($V_C = 100$ V) to the secondary electron collector (Fig. 1) allowing the measurement of the SE current I_{SE}. Accordingly, the amount of the emitted charges $Q_{SE}(t)$ is:

$$Q_{SE}(t) = \int_0^t I_{SE}(t)\ \ dt \tag{2}$$

The charge conservation law implies that the sum of $Q_{ind}(t)$ and $Q_{SE}(t)$ is equal to the amount of injected charge $Q_{inj}(t)$, which is given by $\int_0^t I_P(t)\ \ dt$.

The currents are amplified (Fig. 1) with a rise-time of 300 μs (the injection time being higher than this value and set at 25.5 and 50 ms) and a multiplication factor of 10^9 V/A. The primary electron beam current I_P (adjusted in a Faraday cage before experiments) and the current I_{SE} are always positive whereas I_{ind} can be positive or negative depending on the SEE yield, Y, which is equal to I_{SE}/I_P. In fact, if I_{SE} is higher than I_P ($Y > 1$), the sample charges positively and I_{ind} is negative. On the other hand, if $Y < 1$, the sample charges negatively and I_{ind} is positive. With low primary current density, positive charging takes place when incident electron energy E_P lies between the two crossover E_{PI} and E_{PII} (for which Y is equal to 1) and negative charging occurs either at low energies ($E_P < E_{PI}$) or high energies ($E_P > E_{PII}$).

2.2.3. The SEE yield measurement

The measurements of the foregoing currents $I_{SE}(t)$ and $I_{ind}(t)$ give means to deduce the total SEE yield, $Y(t) = I_{SE}(t)/I_P(t)$. Furthermore, since the conditions for the current conservation are met ($I_P(t) = I_{ind}(t) + I_{SE}(t)$) as the SE detector is sufficiently biased, $Y(t)$ can be rewritten as:

$$Y(t) = \frac{I_{SE}(t)}{I_{ind}(t) + I_{SE}(t)} \tag{3}$$

The total SEE yield Y comprises the true SEE yield, δ, and the backscattered one, η. The backscattered coefficient η is considered constant (about 0.15 for alumina [18]). The reproducibility of the SEE yield was checked by at least triplicate measurements of the currents by performing the irradiations over well separated zones. The deduced SEE yield was found reasonably reproducible, within about 5%.

3. Experimental results

3.1. Correlation between the SEE yield and the surface density of trapped charges

3.1.1. Evolution of SEE and trapped charges with irradiation time

It is customary to represent the SEE yield, Y, as a function of the irradiation time, t [1–3]. An example is shown in Fig. 2 that was obtained in the case of positive charging in sapphire RSA. As irradiation proceeds, the SEE yield decreases from the value of the uncharged material Y_0 to $Y_{st} = 1$ corresponding to the steady state. The decay part of the time evolution of SEE yield $Y(t)$ can be fitted by an exponential decay law:

$$Y(t) = Y_{st} + (Y_0 - Y_{st})\ \ \exp\left(\frac{-t}{\tau}\right) \tag{4}$$

This fit can give the time constant τ of the charging process.

The information gained from the above description can be completed by the time evolution of the trapped charges, Q_T, given below in Fig. 3. The maximum quantity of trapped charges Q_{st}, equal to $-(1/K)\int_0^{t_{inj}} I_{ind}(t)\ \ dt$ (*cf.* Eq. (1)), is reached asymptotically at