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NOVEL DIELECTRICS CHARACTERIZATION USING SEM

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ABSTRACT

Materials stress under such irradiation can be found in space craft and industrial electronics. This pa introduces the use of a scanning electron microscope (SEM) to evaluate the insulation property of insulat An SEM may be used not only to observe a surface image but also to provide a fine electron beam for charg an uncoated-insulator surface at once. The distribution of electric field created by the surface charging can developed by a simple model. The increase of electric field at the surface may exceed a critical value whe surface breakdown/flashover occurs. An insulation property is evaluated by varying the duration charging/electron bombardment which is needed to initiate a treeing-formation (hereinafter time to flasho treeing/TTF). In this paper, under a certain SEM's energy and magnification, SiO_2 addition into a high pu MgO has resulted in different TTF. Therefore, this method can be used to evaluate the insulation property insulators which are exposed in electron beam environment.

Keywords: Breakdown, SEM, Flashover, MgO.

1. INTRODUCTION

The ability of insulators to withstand a high voltage is of great importance knowledge in modern technology. The phenomenon involves surface charging, discharging and flashover (dielectric breakdown), resulting the instrument damage and material degradation. Flashover phenomenon has been studied for many years and it is believed that a flashover is initiated from triple junction of metal, insulator and vacuum [1]. A number of experimental [2,3] and theoreritical works [4,5] stressed the leading role of the surface charge accumulation on the flashover. Electron bombardment is often used to make charge accumulation on the surface of insulator. Electrons produced in an SEM are possible to be controlled in their parameters: the implantation depth and the dose rate by adjusting, respectively, the primary beam energy E_0 and the primary beam current I_0.

The use of an SEM for investigating surface charging and discharging was reported [4]. Subsequent breakdown has been observed by measuring secondary electron and specimen current. It should be emphasized that breakdown is confined near the dielectric (subsurface) and is not dielectric to metal or metal to metal. The incubation of an accumulated charge at submerged layer until occurring discharge (breakdown) was used to evaluate insulation property in space craft dielectrics (i.e. Kapton, Milar, and Teflon).

Later, another group [5] observed optically-visible flashover (tree like structure) when a wide-band-gap polycrystalline Y_2O_3 sample was first charged with a beam of 30 keV and then discharged with a low beam energy of 3 keV. The accepted idea of this observation is that the flashover is due to the space charge destabilization under low energy electron irradiation. So far, the use of an SEM is limited to find a simple explanation of charging and discharging process on the surface of insulator/dielectric.

Since an optically-visible flashover treeing was observed on the surface of high-purity wide- band-gap polycrystalline magnesia (MgO), it is possible to evaluate the insulation property of insulators in term of withstanding a flashover appearance [6]. A flashover occurs due to the increase of electric field created that exceeds a critical value. This agrees with theoretically time dependent of charge density, $\rho(t)$ on an insulator surface under electron irradiation [7]. Therefore, it is of great interest to use *TTF* to evaluate any wide band gap insulator under electron beam irradiation.

2. THE EXTENDED PERIOD OF SCANNING TO INITIATE FLASHOVER TREEING

2.1 Theoretical Analysis

When an electron beam of an SEM scans a surface of an uncoated-insulator sample, a rectangular charged area is created. The distribution of electric field at the surface can be represented by a model of a rectangular charge area (Fig. 1). The electric field *E* at a point *P*(*x*,*y*), where *P*(0,0) is the center of the charged area, can be deduced from its component *dE*. Taking into account a contribution of a charge element *dq* of a small strip $dr_x dr_y$ at a point (x_q, y_q) and by applying Coulomb's law, the field component $dE(x,y,t)$ is given:

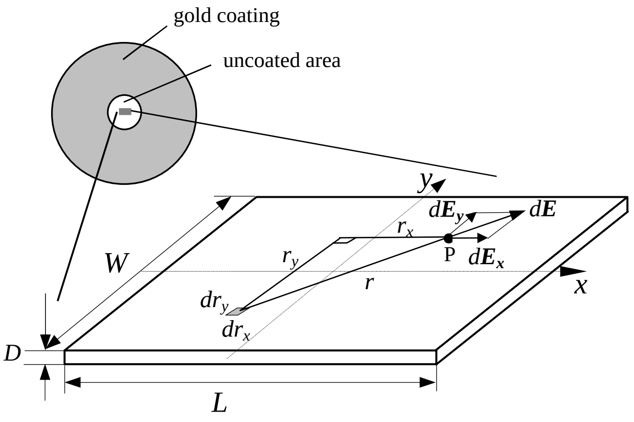

Fig 1. A model of a rectangular charged area

$$dE\,(x,y,t) = \frac{1}{4\pi\varepsilon}\frac{dq}{r^2} = \frac{1}{4\pi\varepsilon}\frac{\rho(t)Ddr_x dr_y}{r_x^2 + r_y^2} \tag{1}$$

where $\rho(t)$ is charge density of the rectangular charged area created by the electron beam bombardment at a certain time *t*, *D* is a depth of the electron beam impinges the surface and *r* is the distance between *P* and the charge element where $r_x = x - x_q$ and $r_y = y - y_q$. The charge density $\rho(t)$ is given by [7] as

$$\rho(t) = \frac{J_0\delta}{D}t \tag{2}$$

Where δ is secondary electron emission coefficient and is defined as

$$\delta = \frac{n_{SE}}{n_B} \tag{3}$$

n_{SE} is the number of secondary electrons emitted from a sample bombarded by n_B, and J_0 is the current density at the surface given as [8]

$$J_0 = J_c E_0 \alpha^2 k^{-1} T^{-1} \tag{4}$$

Where
J_c : emission current density [A/m^2]
E_0 : electron beam energy [keV] = e V_0 ;
e : electron charge = 1.60219x10^{-19} C,
V_0: accelerating voltage [kV]
a : beam divergence at final aperture of an SEM
k : Boltzman constant = 1.38062 x 10^{-23} JK^{-1}
T : operating temperature of tungsten [K]

The emission current density, Jc is expressed by Richardson equation:

$$J_c = A_c T^2 \exp(-E_w / kT) \tag{5}$$

Where A_c = 120 A/cm^{2}K^2 is the constant for all thermionic emitters and E_w is the work function of the filament material.

2.2 The Proposed Flashover Treeing Mechanism

Many of surface breakdown theories have been reported. It is already well understood that initiation of a surface breakdown is begun by the emission of electrons from a triple junction (electrode-dielectric-vacuum). [1] The complete breakdown is because of production of an electron cascade along the surfaces of the insulator, or a secondary electron emission avalanche. There are two groups observed the surface breakdown for an insulator under electron beam irradiation. The first group [4] states the incubation of an accumulated charge at submerged layer leads to a flashover arc (propagating subsurface discharge). Another group [5] has observed that a surface breakdown is lead by space charge destabilization. A "mirror" effect of low beam energy of 3 keV with lowering the SEM's magnification destabilizes the space charge created by high electron beam energy of 30 keV.

In this proposed charging and discharging mechanism breakdown it is first explained the experimental method to produce a surface breakdown and then followed by showing the flashover treeing phenomena resulted from various experimental conditions.

The scanning of a high electron beam of 25 keV allows the formation of a rectangular charge area on the surface of an uncoated insulator. When the beam interacts with the sample, some of the charge injected by the beam is emitted in the form of the backscattered and secondary electrons. The scanned area accumulates charge and its surface potential rises. From the above analysis, integrating Eq. (1) over all strips composing the scanned area, the distribution of electric field after scanning for extended period can be obtained [6]. The electric field takes its highest value at the edge of the scanned area, reduces outward from the scanned area and gets to be zero at the center. Further extended period of scanning, at a certain time, the charge region may undergo a surface breakdown. Figure 2 shows the sequences of visible-optically flashover in the form of treeing (tree-like structure) for high purity (99.99%) polycrystalline magnesia (MgO). Some typical of flashover treeings which can be formed has been reported elsewhere [9].

Extended period of scanning to produce flashover treeing has been performed with other high electron energies of 20 and 30 keV. Figure 3 shows the relation between *TTF* and primary beam energy. The *TTF* tends to increase with lowering electron beam energy. However, flashover treeing was difficult to occur at low electron beam energy. Since surface potential exists, the scanning beam with low energy is reflected on the equipotential and gives an image of the microscope chamber (Fig. 4). Electrons from the primary beam can not reach the sample surface. Consequently, electric field created on the surface can not further increase to exceed a critical value of breakdown even in extending period of scanning.

Fig 2. Flashover treeing produced at the surface of a high purity polycrystalline MgO. The sample was charged by electron beam of 25 keV with magnification of 5000X. The extended period of charging (i.e. time to flashover treeing) was about 7 minutes.

The effect of varying charge area by changing the magnification of the SEM on *TTF* at 25 keV primary beam energy was observed. Increasing the area may increase the extended period of scanning to perform a treeing (Fig. 5). This phenomenon can be explained as follows. Though the secondary electron emission constant at primary beam of 25 keV has not been reported, it might be predicted [10] to be still greater than 1. It means that the charging can be expected to be positive. The distribution of electric field gives its highest value at the edge of the scanned area. Therefore, the subsurface region around the edge behaves as a positive electrode and the subsurface region around the center as a negative electrode (less positive charge). On the other hand, the region above the edge behaves as a negative electrode and the positive one at the center because of the less negative charge. Potential different between two electrodes created on the surface may accelerate some secondary electron emitted somewhere at the edge toward the center. During the propagation the accelerated electrons strike the surface producing additional electrons by tertiary emission. Continuation of this process results in a cascade along the surface within the scanned area that develops into a tertiary electron emission avalanche. This avalanche, in turn, can lead to a complete flashover treeing. Therefore, when the scanned area is reduced, lowering *TTF* is due to the reducing the critical value of electric field to experience breakdown.

The initiation of a flashover treeing is begun from the four possibility regions: (*1*) a grain boundary of the long edge of the rectangular scanned area, (*2*) at grain of the long edge, (*3*) at the grain boundary of the short border, (*4*) at the grain of the short edge. Figure 6 shows some three possibilities of treeing initiation. Observation was carried out for 30 identical samples of polycrystalline MgO.[11] The number of treeing initiation from long edge was found 1.4 times higher than that from short edge at grain, and 1.6 times higher at grain boundary. This result shows that the treeing tends to be initiated more easily from long border region. This result agrees with the above electric field analysis that the region at the middle of long edge has the shortest distance to the center and behaves as a critical place to initiate a treeing. It is also clear that the number of treeing initiation at grain boundary is higher than that at the grain. It is revealed that the grain boundary allows the higher concentration of contaminants than the grain. Therefore, it could be considered that the grain boundary at the long edge of scanned area worked as the most critical place of treeing initiation.

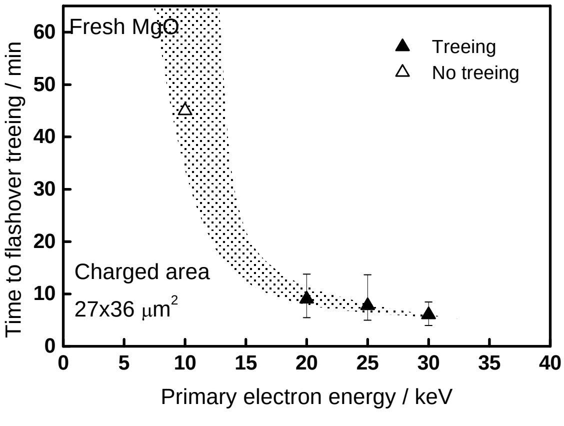

Fig 3. Time to flashover treeing versus primary beam energy. Electron mirror effect might cause no flashover at low beam energy.

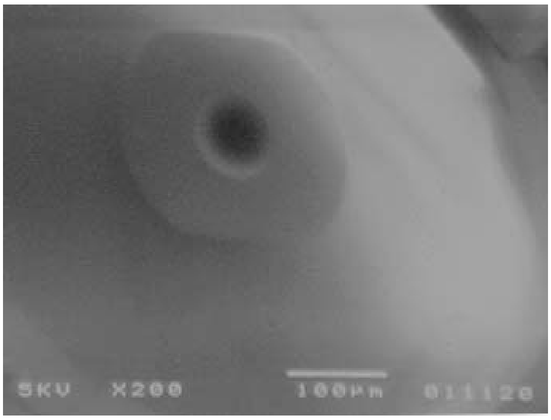

Fig 4. The uncoated MgO was charged by 5 kV. Surface potential becomes to act as an electron mirror, so the scanning beam is reflected off the sample to scan on walls of the camber.

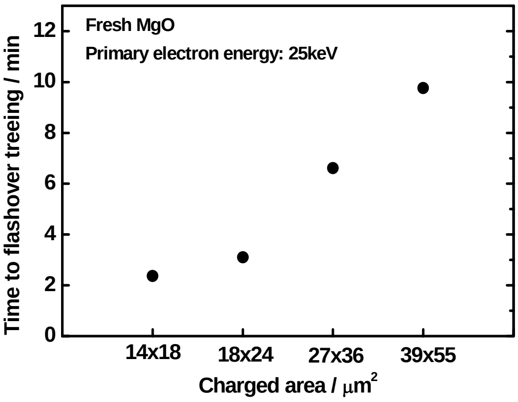

Fig 5. Time to flashover treeing as a function of charged area.

3. TIME TO FLASHOVER TREEING OF VARIOUS INSULATORS UNDER ELECTRON BEAM BOMBARDMENT

From the extended period of scanning to initiate a flashover treeing, it is of great interest to use a parameter *TTF* to evaluate insulation property of insulators in order to withstand flashover treeing appearance under electron beam bombardment in vacuum. Here it is important to note that in order to have the optimum result the evaluation of one kind of insulator was carried out by observing 10 identical samples. Since experiment is time consuming, care should be taken to maintain the identically of samples. The primary electron beam energy should be also properly chosen to avoid the effect of electron mirror as well as the scanned area.

In the case of high purity polycrystalline MgO, such SEM's conditions have been chosen: 25 keV electron beam energy, a scanned area of 27 x 36 μm^2 (magnified by 5000X), a working distance 25 mm, a final aperture of the SEM 200 μm in diameter, a vacuum chamber pressure 7 x 10^{-4} Pa, and a tilting angle 0°.

It was reported [11] that exposing the fresh sintered high purity polycrystalline MgO in air for 5 and 10 days reduces the *TTF* from 7 minutes to 4.5 and 4 minutes, respectively. X-ray photoelectron spectroscopy (XPS) analysis has shown existence of contaminant in the form of carboxyl-, hydrocarbon- and carbide-like species on the sample surface. The peak intensity of those phases' increase by extended period of exposing in air as well as the binding energy was shifted. It can be predicted that the presence of the contaminants at the MgO surface are involved during exposing in air. The shifts of binding energy and the changes of peak intensity imply that there are compositional changes by chemical reaction at the surface. Therefore, the changes influenced to the *TTF* under the electron beam bombardment.

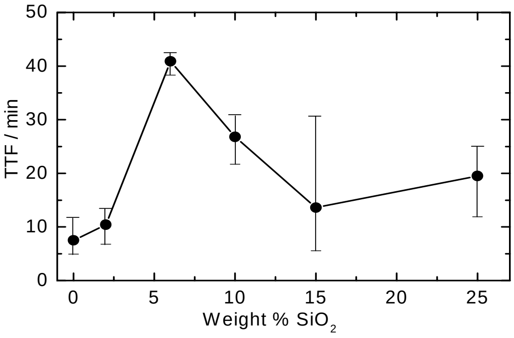

Fig 6. Time to flashover treeing of SiO_2 added MgO.

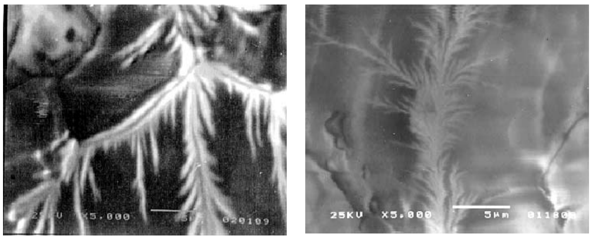

Fig 7. Some possibilities of treeing initiation at the surface of high purity polycrystalline MgO under 25 keV electron beam energy.

Flashovers have also been produced on various weight percent SiO_2 added MgO [12]. Figure 7 shows the results of any *TTF* obtained by adding SiO_2 to MgO. It was found that the noticeable change is found for SiO_2 addition. The *TTF* was found to increase lightly for 2 wt% additions, increase sharply for 6wt% additions that reached the maximum value 41 minutes and then decreased for further addition. These phenomena might be explained by the atomic distribution on the sample surface, since morphology and molecular composition influence to the electrical strength [13]. From the observation of the compositional maps of SiO_2 added MgO, the distribution of Si was spread evenly on the surface. This condition was found for all investigated SiO_2 additions. The existence of Si in all surface space might be considered to be better to improve the insulation property of MgO. However, further addition of SiO_2 over 6 wt% addition was found to reduce the *TTF*. The reduced TTF might be attributable to the influence of further formation of Mg_2SiO_4 phase.

4. CONCLUSIONS

Extended period of scanning for uncoated insulators has been carried out. It was found that the extended period to flashover treeing (*TTF*) is different for different kind of sample. It was seen that the appearance of treeing in various weight percentage of SiO_2 added into MgO samples shows noticeable change in *TTF* under electron beam bombardment. Therefore, this method may be used to evaluate insulation property in term of withstanding flashover treeing appearance under electron beam bombardment.

5. REFERENCES

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