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Flashover Treeing of Magnesia Surface under Electron Beam Bombardment in Vacuum

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A sample of polycrystalline magnesia (MgO) placed in vacuum was charged by an electron beam of 25 keV employing a scanning electron microscope (SEM). The electron beam created a rectangular charged area on the sample surface. After a certain time of bombardment a flashover treeing appeared inside the charged area. The treeing was initiated at the edge and spread into the center of the charged area. From an electrostatic analysis and considering an electric field distribution shape, the electric field took its highest value at the edge of the charged area. It could be understood that the increase of the field exceeded a critical value and triggered a flashover treeing. Time to flashover treeing appearance of fresh and aged samples was also investigated. The fresh sample surface behaved more resistive to flashover. It could be understood that the presence of contaminants accelerated the treeing appearance.

Key words: Magnesia, Breakdown, SEM, Treeing, Aging

1. INTRODUCTION

Failure phenomena of solid dielectric under environmental stresses such as plasma collision and UV irradiation have occurred on spacecraft.[1] The phenomena involve surface charging, discharging and flashover (dielectric breakdown), resulting the instrument damage and material degradation. Surface flashover phenomenon has been studied for many years and it is believed that a flashover is initiated from triple junction of electrode, insulator and vacuum.[2] Surface charging and discharging characteristics in vacuum has been reported by Yamano et al[3] and Okubo et al,[4] respectively. Many researchers are trying to find simple explanations of surface breakdown in solid dielectrics for the 21st century.[5]

In the facing of the development of space station, reusable space transportation system becomes important. Therefore, the safe and reliable launch vehicle material is needed. The material required is not only for high temperature operation but also for resisting discharge events in a space plasma environment. Magnesia, as a classical wide-band-gap insulator of >7.8 eV and one of the best insulators,[6] meets the requirements. Magnesia with its high melting point of 2800°C, low linear expansion coefficient of 13.5×10^{-6} K^{-1} and high dielectric strength of over 200 kV/mm is a promising material for the above purpose.

The present authors already reported that a flashover treeing appeared on the MgO surface under the bombardment of electron beam accelerated by 25 keV in vacuum.[7] This paper will present the further study of treeing appearance related to its initiation, the effect of grain boundary and aging in environmental air.

2. EXPERIMENTAL PROCEDURE

2.1 Sample preparation

High purity MgO powder (99.95%) was calcined at 850°C for 4 h, pressed in a 1 cm diameter die under pressure of 130 MPa and sintered at 1650°C for 7.5 h. The obtained sample had 0.85 mm thickness, 8.5 mm diameter, the average density of 3.32 g/cm^3 and average grain size of 10 μm. The sample was covered by sputtered gold metal and remained a small uncoated area of 500 μm diameter at the center of the sample surface. Three kinds of samples were prepared in order to evaluate aging effects: fresh sample (bombarded soon after sintering) and samples aged at room temperature for 5 and 10 days in air.

2.2 Experimental arrangement

Figure 1 shows the experimental arrangement for bombardment. A SEM (JEOL, T220A) was employed in

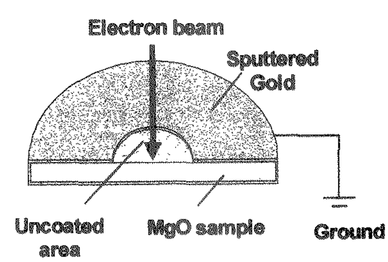

Fig.1. Experimental setup. The electron beam bombarded the center of uncoated area of the sample.

this experiment not only to observe the image of the sample surface but also to provide a fine electron beam for charging the surface at once. The sample was placed inside the SEM vacuum chamber ($4x10^{-7}$ Pa). The SEM was operated at 25 kV of the accelerating voltage and the produced electron probe was scanned on the area of 27.00x36.00 μm^2 in the uncoated area until flashover treeing occurred.

3. RESULTS AND DISCUSSION

3.1 Treeing initiation

Bombarding a fresh MgO sample surface for 7 min resulted a flashover treeing on the charged area (see Fig. 2). The treeing was initiated at the edge and propagated toward the center of the charged area. Figure 3 shows the another SEM photograph taken after a treeing appeared and then the magnification was reduced from x5,000 to x2,000. A dark shadow at the center indicates the charged area of 27.00x36.00 μm^2. It is seen that the treeing appeared inside the charged area in the form of a rectangular shape. From electrostatic analysis described by authors,[7] the calculated electric field distribution on the rectangular charged area follows the shape as indicated in Fig. 4 (the inset shows the position of the charged area). Abscissa indicates the location which follows the cross section view of AB and CD, where AB is viewed from the center of the long line borders of the charged area and CD is for the short one. Center of the charged area is represented by zero. Ordinate is the electric field in arbitrary unit.

From the electric field distribution analysis, the electric field takes its highest value at the edge of the charged area, reduces outward from the charged area and gets to be zero at the center. The highest electric field was found at the center of the long border (cross section of AB).

Miller[2] reviewed that continuation process of collision and producing electrons along an insulator surface developed into so called a secondary electron emission avalanche (SEEA). The SEEA, in turn, may lead to a surface breakdown. The process of flashover treeing initiation on MgO surface can be explained as follows. The value of electric field at any point depends on the duration time of electron beam bombardment.[7] The increase of the electric field causes increase in the field electron emission.[8] Accordingly, in the rectangular charged area, the field emission takes its highest value at the edge where one or some electrons are liberated. The velocity of the electrons is accelerated by the electric field. Its collision with insulator surface allows additional electrons to be liberated. Since the charged area of MgO is positively charged,[9] the electrons move toward the center of the charged area. This process continues and finally results in electron avalanches.

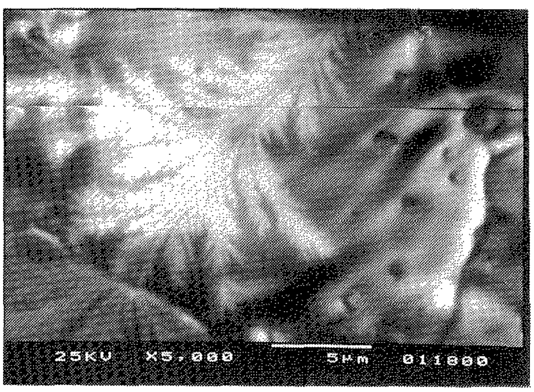

Fig.2. SEM photograph of flashover treeing on the surface of MgO sample after the bombardment of electron accelerated by 25 keV for 7 min.

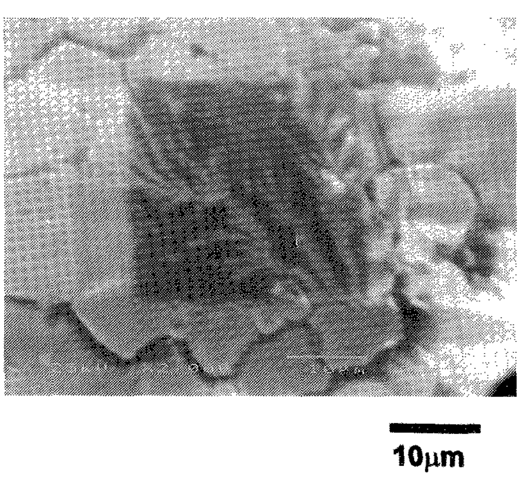

Fig. 3. SEM photograph of charged area and treeing. Treeing is initiateed at the edge of the charged area. Magnification was reduced from x5,000 to x2,000.

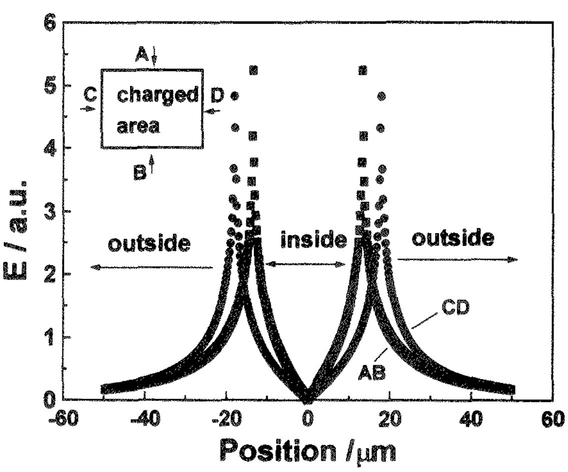

Fig.4. Calculated shape of electric field distribution on the sample surface bombarded by electron beam. A rectangular area at the surface is the charged area.

3.2 Effect of grain boundary

Figure 5 shows the flashover treeing propagates through grain boundaries when there are one or more grain boundaries crossing at the edge of the charged area. The treeing was initiated at the edge and spread through the grain boundary. It branched into three parts when it came to the triple point.

Figure 6 shows the number of treeing initiation per unit length. Figure 6(b) is an illustration of a treeing initiated at grain boundary of long border. There are 4 categories involved:

1. Initiation at the grain boundary of the long border,
2. Initiation at the grain of the long border,
3. Initiation at the grain boundary of the short border,
4. Initiation at the grain of the short border.

The number of treeing initiation on long border was 1.4 times higher than that on short border at grain, and 1.6 times higher at grain boundary. This result shows that the treeing tends to be initiated more easily on long border of the charged area and corresponds to the result of electric field analysis mentioned above. It is also clear that the number of treeing initiation at grain boundary is higher than that at grain.

It has been predicted that the existence of contaminants at the MgO surface accelerates the flashover treeing appearance.[7] 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 edge of charged area worked as a critical place of treeing initiation.

3.3 Effect of aging in air

Figure 7 shows the cumulative number of samples with each time to flashover treeing appearance. It can be seen that aging in air at room temperature gave the shift of flashover treeing appearance. On the fresh samples, the 50% of flashover treeing appearance was about 440 s, while for the samples of 5 and 10 days aging the 50% of the treeing appeared at 280 and 235 s, respectively.

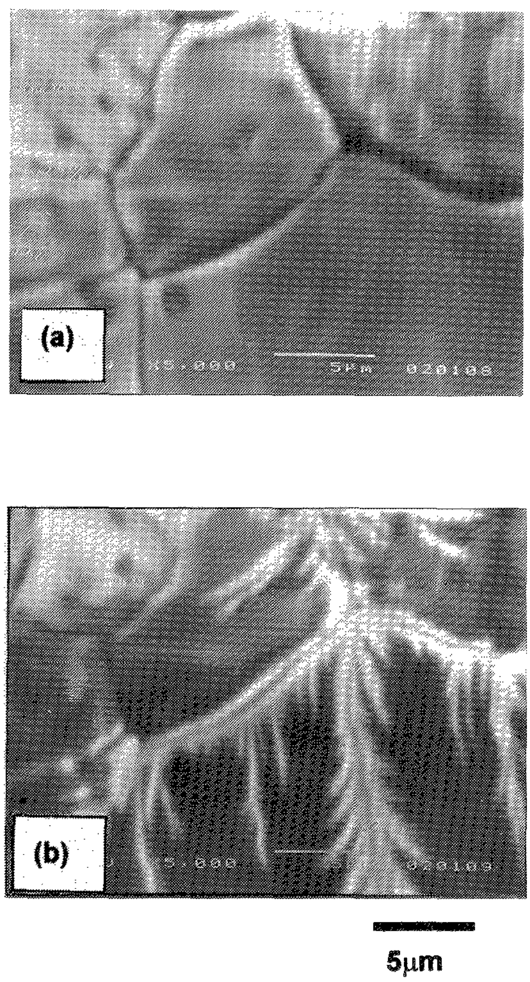

Fig.5. Effect of grain boundary on treeing propagation: (a) before and (b) after flashover.

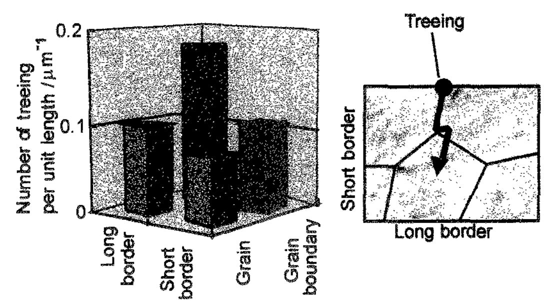

Fig.6. Number of flashover treeing related to its initiation place for 30 investigated samples.

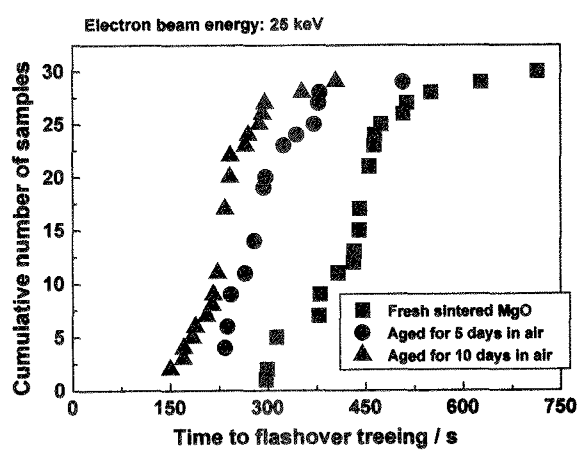

Fig.7. The cumulative number of flashover samples as function of appearance time between the fresh and aged samples.

From a surface analysis using X-ray photoelectron spectroscopy (XPS), the presence of contaminants are shown by the appearance of carbon 1s spectra of each sample: fresh, 5 days aging and 10 days aging in air (see Fig.8). The carbon 1s peaks were categorized into three groups:

- group of carboxyl-like species,
- hydrocarbon-like species, and
- carbide-like species.

The major peaks are shown by carbon 1s spectra of the group of carboxyl-like species and then followed by the hydrocarbon-like species and carbide-like species. In the carboxyl-like species between three samples, the carbon 1s binding energies are shifted from higher binding energy of 291.4 eV for the fresh sample to the lower ones of 290.8 eV and 290.3 eV for the 5 and 10 days aged samples, respectively. The peak intensity increased by increasing the aging time in air: 8300 for the fresh sample, 9400 for the 5 days aging and 10500 for the 10 days aging, on the other hand, the spectra of carbon 1s of the hydrocarbon-like are relatively constant for their binding energy of around 285.6 eV and peak intensities.

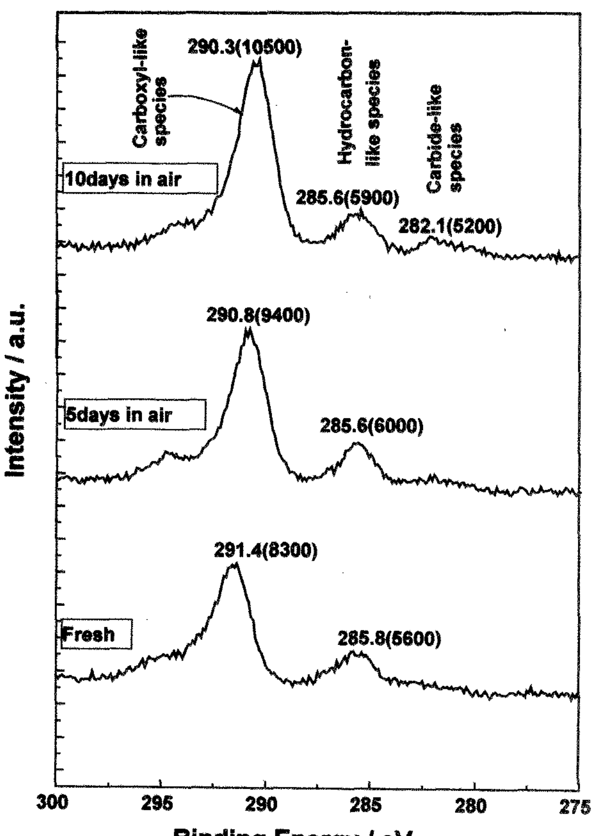

Fig. 8. XPS spectra of carbon 1s from various magnesia samples. The peaks are categorized into three species: carboxyl-like species, hydrocarbon-like species and carbide-like species. Each peak is indicated by the binding energy and the intensity in the parentheses.

From above results, it could be predicted that the presence of the contaminants at the MgO surface are involved during aging in air. The shifts of binding energy and the changes of peak intensity implies that there are compositional changes by chemical reaction at the surface. Therefore, the changes influenced on the flashover treeing appearance at the MgO surface under the electron beam bombardment.

4. CONCLUSIONS

This paper reported about flashover treeing phenomena of polycrystalline magnesia under electron beam bombardment of 25 keV in vacuum. The phenomena included the initiation, propagation and the effect of aging in air on treeing appearance. A simple model of electric field distribution was used to explain the treeing initiation. Although the electric field distribution model was only qualitative, it could contribute to explain the development of avalanche electrons on the surface which lead the surface flashover under electron beam bombardment. The time to flashover of aged samples was shorter than that of the fresh one. The existence of contaminants was a possibility to accelerate the treeing appearance.

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