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Behavior of MgO Based Ceramics under Electron Irradiation

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Abstract

MgO is one of environment friendly ceramic material under harsh environment such as electron irradiation. Different response of MgO ceramics under the irradiation was reviewed. The response was in the appearance of surface breakdown/flashover on the surface of MgO after a certain time under irradiation exposure. The different response was shown for small addition of CaO, ZrO_2 and SiO_2 into high purity of MgO. Addition of CaO resulted in the appearance of flashover could be 30% earlier of that of pure MgO. Moreover, addition of ZrO_2 resulted in 70% earlier. On the other hand, small addition of SiO_2 revealed that the MgO based ceramics could withstand the appearance of flashover under the same energetic electron irradiation.

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Keywords: MgO; surface breakdown; flashover; scanning electron microscope; electron irradiation

1. Introduction

Material behavior under stress up to failure conditions is an important issue due to the development of new and high performance materials to meet a wide variety of industrial needs. In harsh environment such as high lightning strike density [1] the reliable insulator for overhead high voltage transmission line is needed. In space technology, it was reported [2] that spacecraft charging under space irradiation generates problem on electronic devices and may cause accident. In an electrostatic separator, it was [3] shown that materials under electron beam bombardment to prevent breakdown are needed. These entire situations are considered to be very severe to the insulation since they may cause a material failure when the field stress exceeds a critical value.

Magnesia (MgO) is one of environment friendly ceramic materials. It was for long time that MgO is the best ceramic for producing more secondary electrons under electron avalanche in protecting layer of plasma display panel [4]. It is also considered that MgO has many applications in other harsh environment. The MgO is a very good contender as a neutron reflector in fast neutron reactors [5], it is envisaged as a matrix of the ceramic-ceramic composite nuclear fuel for minor actinide transmutation [6], and it could be also used as an electrical insulator for diagnostic components in the ITER fusion reactor [7], making it a potential candidate for applications in the nuclear energy field. Under such operating conditions, the MgO will definitely be subjected to various types of irradiation.

In particular electron irradiation, it was introduced [8] the use of a scanning electron microscope (SEM) as a source of energetic electrons to irradiate uncoated dielectric ceramic to study the capability of the material to withstand the appearance of surface breakdown (flashover). A flashover is a process of charging and discharging on the surface. The charging process will then create an increasing of surface electric which may cause breakdown when the limit of electric field is achieved. MgO was chosen since the materials was very stable under electron irradiation. On the other hand, MgO is very sensitive ceramic to produce secondary electrons when it is exposed under electron irradiation. There are two possibility behaviors of MgO when energetic electrons are directed into it whether suppresses or doubles the production of secondary electrons. The earlier will

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cause the material to withstand surface breakdown and the later one is to allow flashover to occur easily. This paper is to evaluate both behaviors of MgO based ceramics under electron irradiation. First it will be introduced the use of SEM with a certain operating voltage to drive energetic electrons to direct into uncoated dielectric ceramic that may create a surface electric field. Then under the same energy of irradiation, MgO with different addition will be evaluated.

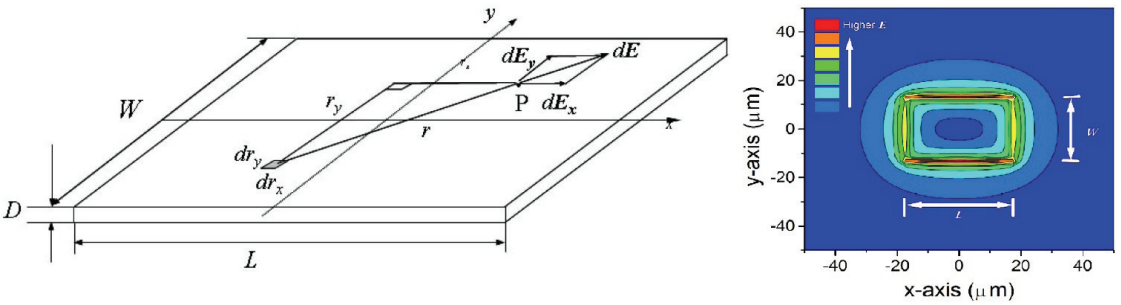
2. Methods

When energetic electrons of a SEM irradiates an insulating material, it causes the emission of secondary electrons. Secondary electrons are electrons which are ejected from a sample during electron beam irradiation. The total secondary electron emission yield δ is given by n_{SE}/n_I where n_{SE} is the number of secondary electrons emitted from a sample irradiated by the number of incident electrons, n_I. When δ is greater than one, the sample surface becomes positively charged. Secondary electrons are generated from a shallow escape depth of D. The electrons are produced along the entirely of the beam electron trajectories within the specimen. The thickness, D, of this region is not well known, but is less than about 50 nm for insulator [9].

Figure 1 (*a*) shows a model of rectangular charged area. The electric field at any point due to a group of point charges is found by an integral of the equation (1) as

$$E(x,y,z) = \int dE = \frac{1}{4\pi\varepsilon}\int \frac{dq}{r^2} \tag{1}$$

where dq is a differential element of the charge distribution, r is its distance from the point P, and dE is the electric field it establishes at that point.



(*a*) (*b*)

Fig. 1. Distribution of electric field: rectangular model (*a*) and plot of the field (*b*) where the highest field (red) is at the edge of scanned area.

The mechanism of a surface flashover (optically-visible flashover treeing) is as follows. Under the period of charging, by using equation 1 above, the highest electric field is created at the edge of the scanned area (shown in figure 1(*b*)). Consequently, the highest field may cause more electrons which are emitted from the subsurface region around the edge. The increase of period of charging may increase the number of electrons above the surface (containing secondary electrons and field-emitted electrons) and cause a potential difference between the edge (more negative charges) and the center (less negative charges or zero) of the scanned area. When the potential difference reaches a critical value, some of the electrons from the edge region may be accelerated and attracted towards the center. The electrons impact upon the surface producing additional electrons by tertiary emission. Some of these tertiary electrons will again strike the surface producing second-tertiary electrons. Continuation of this process results in a cascade along the surface that develops into a tertiary electron emission avalanche. This avalanche, in turn, can lead to a complete breakdown. Some of these electrons will be detected by the Everhart-Thornley (E-T) detector as an optically-visible flashover treeing. Once a flashover treeing is completed, the potential difference between the scanned area edge and the center becomes zero. In this stage, the surface becomes positively charged. Figure 2 shows the process of a flashover appearance on MgO under electrons irradiation.

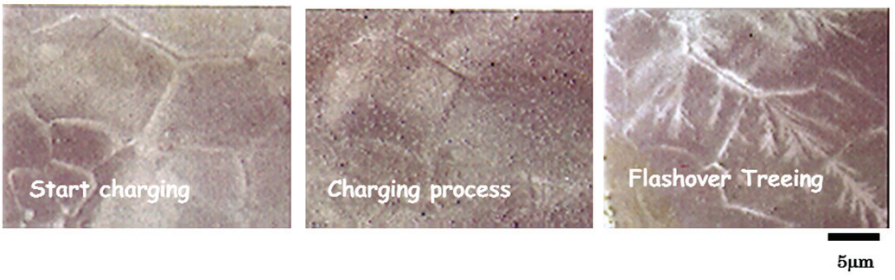


Fig. 2. Flashover on the surface of high purity MgO occurred after 7.5 min (start charging to flashover treeing) under 25keV electrons irradiation energy.

2.1. MgO based ceramics

Parameter which is used to indicate whether suppression or doubling the production of secondary electrons is time to flashover (*ttf*) i.e. exposure time of a sample under electron irradiation to the appearance of a visible flashover on the SEM monitor. One candidate of material addition is CaO. The addition of CaO into MgO is ranged from 0 (pure) to 12.5%. Figure 3 *(left)* shows the results of *ttf* obtained for CaO added MgO surfaces under the SEM conditions of 25 keV and charged area of 27x36 μm^2. It was found that the 2.5 wt% CaO addition reduced the time from 7.5 min to 4 min. It is thought that the small addition of CaO to MgO lowered the *ttf* probably due to the increasing of secondary electron emission yield of the material. The further CaO addition of 5 and 10 wt% suppressed the production of secondary electron emission and increased *ttf*. 12.5 wt% did not show any significant change on *ttf*. Therefore, small addition of CaO at 2.5 wt% might increase secondary electron emission yield compared with the pure MgO. The consistency indicative of CaO addition into pure MgO was also shown from fracture surfaces given in figure 3 *(right)*. Plate like crystallites exist in all samples except figure 3(b), and well developed layered structures showing cleavage planes between the layers are clearly observed on the crystallites in figure 3(a), (c), (d), and (e), contrasting to smooth and structure less surface seen in figure 3(b). These results demonstrate for the sample with 0, 5, 10, and 12.5 wt% a considerable development in the two-dimensionality within the crystallites, which would lead to a significant decrease in the crystal defects and disorders.

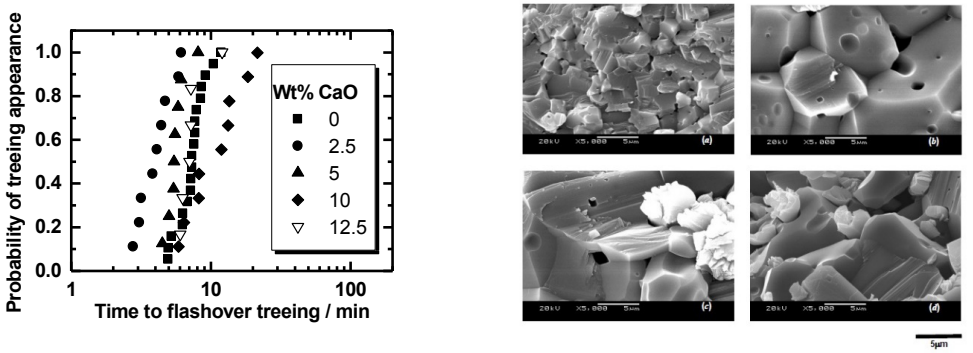


Fig. 3. Various CaO added MgO with electron energy of 25keV for a charged area of 27x36 μm^2: (*left*) *ttf* and (*right*) fracture surface. It was shown the cleavage for 2.5 wt% addition (figure *b*) and platelike crystalike for the rest CaO addition (figure *a*, *c* and *d*).

Figure 4 shows the XRD pattern for all compositions CaO added MgO. There was no noticeable preferential crystal orientations for all investigated samples [10]. The intensity of crystal plane (311) of rock salt MgO was slightly lower at 2.5 wt% CaO addition.

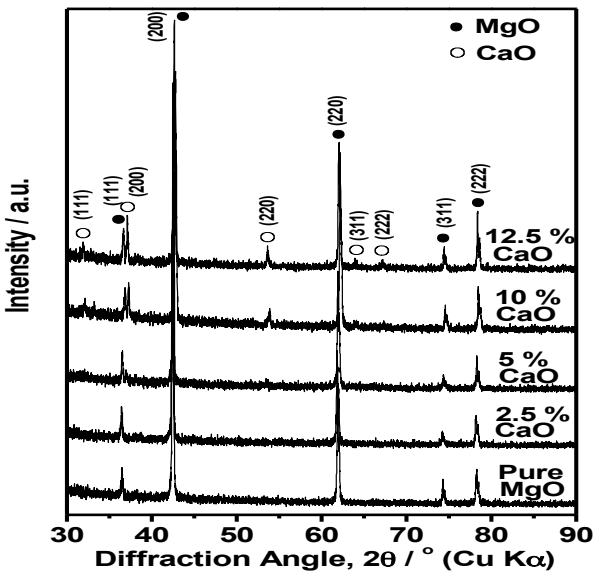


Fig. 4. XRD pattern of CaO added MgO

Another candidate of material addition was SiO_2. Weight % of SiO_2 addition was ranged from 0 to 15 wt%. It was found that *ttf* changed with increasing amount of SiO_2 addition. The time was a little increased for 2 wt% SiO_2 addition and got the maximum value when the 6 wt% addition was applied. Further SiO_2 additions were found to decrease the time. The decreasing of the time might be attributed to the further formation olivine in the crystal [11]. Therefore, when the 6 wt% SiO_2 added MgO was chosen, the time to flashover treeing was delayed from 7.5 min for pure MgO to 41 min, meaning that the breakdown voltage of the surface was increased 5.5 times. The behavior was consistence with it surface conductivity shown in Fig. 4 (*right*). At 6 wt% addition the surface conductivity fall to the lowest one ($2.5x10^{-16}$ Ω^{-1}) compared with the other wt% addition.

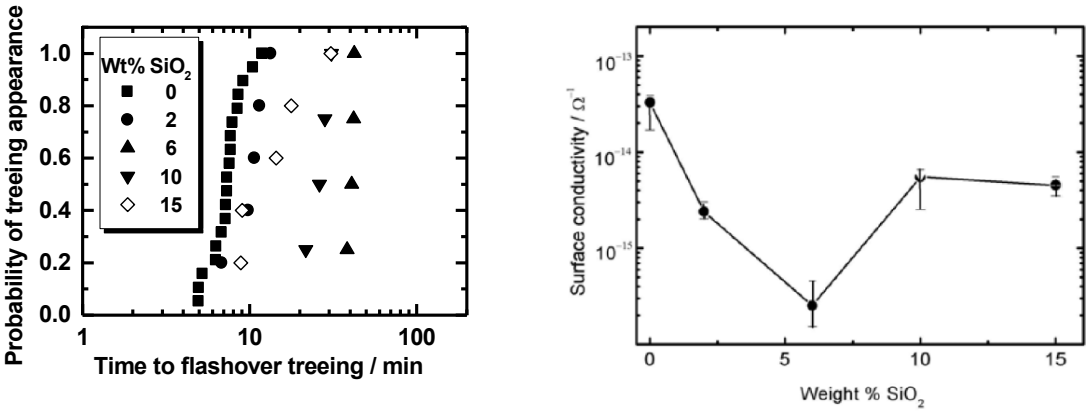


Fig. 5. Various SiO_2 added MgO with electron energy of 25keV for a charged area of 27x36 μm^2: (*left*) *ttf* and (*right*) surface conductivity.

From the compositional maps of SiO_2 added MgO (figure 6), it can be seen that the compositional of Si was spread all over the surface. This condition might give the effective improvement to the insulation property up to 6 wt% SiO_2 addition. Above 6 wt% addition, the formation of olivine will occur and reduces the insulation of MgO [11].

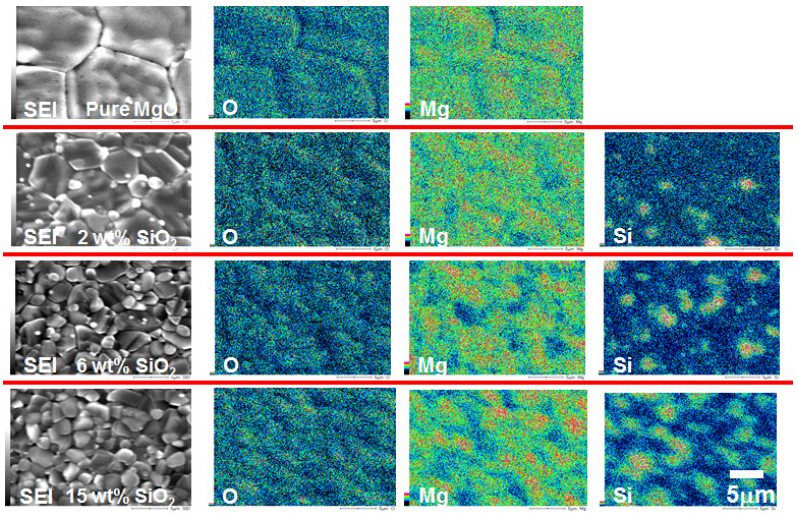


Fig. 6. Compositional maps of SiO_2 added MgO.

Jufriadi, et al. [12] investigated that addition of 5 wt% of ZrO_2 reduced *ttf* from 22 minutes (pure MgO) to only 5 minutes. The ttf was the lowest values among other additions. Secondary electron emission yield of 5 wt% ZrO_2 added MgO could be

predicted to be bigger than those of 2.5 wt% CaO added MgO as well as pure MgO. The appearance of flashover could occur earlier by increasing the secondary electron emission yield. On the other hand, addition of 6 wt% SiO_2 added MgO would suppress the production of secondary electron emission on the material surface and caused material could withstand longer the breakdown.

2.2. Applications

Harsh environment of irradiation in this study was shown by utilizing energetic electrons from an SEM. MgO based ceramics revealed different behavior under electron irradiation. Although MgO is a friendly material and could adapt in such electron irradiation, however MgO was sensitive to any small addition of other materials. Three kind of material additions have been investigated and gave different behavior how to suppress or how to withstand surface breakdown. MgO based ceramic with high secondary electron emission yield is very useful for protecting layer of plasma display panel where energy consumption of firing voltage could be reduced up to 70%. On the other hand the capability of MgO based ceramic on withstanding the surface breakdown is applicable for space materials (spacecraft or space station) where space irradiation might cause space charging that is undesirable. The capability of withstanding the appearance of surface breakdown, therefore, could postpone the life time of spacecraft/space station.

3. Conclusion

MgO responses different behavior by small additive of CaO, SiO_2 and ZrO_2 when the materials were exposed under energetic electrons irradiations. Two kind behavior were investigated: suppression or support the appearance of surface breakdown/flashover which was indicated by visually visible tree like structure on the SEM monitor. Addition of 2.5% CaO or 5wt% ZrO_2 into MgO might accelerate the surface breakdown. It could be predicted that this occurrence was related with the development of secondary electron emission. On the other hand, addition of 6 wt% SiO_2 would withstand the surface breakdown until 5.5 times longer than that of the pure MgO. The behavior, therefore, is very useful for considering the application of MgO in any other harsh environment such as in nuclear irradiation.

Acknowledgements

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References

[1] K.T. Sirait, 18th International Conference on Lightning, Munchen, pp. 16-20, Sept.1985.
[2] N. Feng, D. Li, S. Yang, Y. Chen, D. Tang, C. Zhao, Study of Secondary Electron Emission Depended on Surface Charge of Space Insulator Materials, Applied Mechanics and Materials Vols 716-717 (2015) pp. 137-141
[3] G.R. Mettam, L.B. Adams, How to prepare an electronic version of your article, in: B.S. Jones, R.Z. Smith (Eds.), Introduction to the Electronic Age, E-Publishing Inc., New York, 1999, pp. 281–304.
[4] H. Idzuchi, Y. Fukuma, L. Wang, and Y. Otani1, Spin relaxation mechanism in silver nanowires covered with MgO protection layer, Applied Physics Letters 101 (2012) pp. 022415-(1-4).
[5] R.R. Macdonald, M.J. Driscoll, Magnesium oxide: an improved reflector for blanket-free fast reactors. Trans Am Nucl Soc 102 (2010) pp.488–489.
[6] S. Somiya, Handbook of advance ceramics: materials, applications, processing and properties, Academic Press, Waltham (2013).
[7] T. Shikama, T. Nishitani, T. Kakuta, S. Yamamoto, S. Kasai, M. Narui, E. Hodgson, R. Reichle, B. Brichard, A. Krassilinikov, R. Snider, G. Vayakis, A. Costley, S. Nagata, B. Tsuchiya, K. Toh, Irradiation test of diagnostic components for ITER application in the Japan materials testing reactor, Nucl Fusion 43(7) (2003) pp. 517–521.
[8] A. G. E. Sutjipto, M. Takata, The use of SEM to investigate the effect of an electron beam on the optically-visible flashover treeing of MgO ceramic, J Mater Sci 42 (2007) pp. 6036–6040.
[9] H. Niedrig, Electron backscattering from thin films, J. Appl. Phys. vol. 53R15 (1982).
[10] A.G.E. Sutjipto, The Effect of CaO Addition on the Microstructural, Mechanical and Dielectric Properties of Pure MgO Ceramic, Key Engineering Materials Vols. 345-346 (2007) pp. 1609-1612.
[11] A.G.E. Sutjipto, Surface Breakdown Property of SiO2 added MgO under Electron Bombardment in Vacuum: A promising Materials for Spacecraft and Space Station, Canadian Journal of Pure and Applied Sciences 1(1) (2007) pp. 73-77.
[12] Jufriadi, A.G.E. Sutjipto, R. Othman and R. Muhida, Discharge, microstructural and mechanical properties of ZrO2 addition on MgO for plasma display panel, Materials Research Innovations 13 (3) (2009) pp. 149-152.