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Section 7. Dielectrics, insulators, windows and optics

Role of environment on the surface degradation of Wesgo AL995

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Abstract

Wesgo AL995 alumina samples have been irradiated with 1.8 MeV electrons in high vacuum, dry air, and helium. The irradiations have been carried out at 450°C and approximately 10^{-10} dpa/s, 700 Gy/s with applied electric fields of between 100 kV and 1 MV/m. Severe electrical surface degradation has been observed for this specific alumina grade during irradiation in vacuum, but not in air or helium. The irradiation of the surface is an important factor in the degradation. One possibility is that radiation enhanced vacuum reduction takes place, which may be related to the large grain size and low density of this alumina grade.

1. Introduction

It has recently been shown that, unlike sapphire and Vitox alumina, Wesgo AL995 alumina is highly susceptible to surface electrical degradation when irradiated in high vacuum [1]. In this initial work it was observed that the degradation could be partly removed by heating in air. It was therefore suggested that a radiation-enhanced vacuum reduction takes place in this material. This was supported by SEM X-ray analysis of a degraded surface in which an oxygen deficiency was observed. SEM results also indicated that the conduction layer extends to between 20 and 100 μm below the surface.

The processes involved in the surface degradation of this particular alumina grade have not yet been identified, although they may be related to its high impurity content, large grain size, and/or low density. However three external parameters which play important roles in this radiation enhanced surface degradation have now been identified, these are the electric field strength, irradiation, and environment. The work presented here is concerned with two of these factors; the irradiation environment and the radiation field. It has been shown that irradiation in air or in helium does not produce a surface degradation, and that in vacuum the radiation field itself plays a key role in the process.

The importance of the irradiation environment (vacuum, air, or He) in electrical degradation experiments has not so far been sufficiently considered. This is particularly the case for in-reactor experiments which in general for technical reasons are performed in a helium environment [2,3]. It is essential that one takes into account that fusion applications require the insulating material to perform with at least one face in high vacuum, and that for susceptible materials such a surface degradation could have serious consequences.

2. Experimental procedure

The work reported here has been performed in a chamber mounted in the beam line of a 2 MeV Van de Graaff accelerator, in which alumina samples have been irradiated with 1.8 MeV electrons. The samples may be heated from 15°C to 650°C and maintained at any temperature within 1°C. The experimental set up allows samples to be irradiated with an electric field applied in vacuum, air, or other gases. In the case of vacuum irradiations, the chamber wall is water cooled and liquid nitrogen traps are placed in the beam line at the entrance and exit of the chamber. This provides a clean high vacuum ($\approx 10^{-6}$ mbar) in the sample chamber. No indication of sample surface contamination and related

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electrical degradation resulting from poor vacuum, has been observed in the system for the numerous electrical conductivity experiments already carried out using standard guard ring techniques, even for samples left for several weeks in the chamber. In the case of irradiation in air or other gases, an insert sample chamber has been used with a 0.05 mm aluminium window to allow the accelerator electron beam to enter the sample chamber, while maintaining the sample in air or gas slightly above atmospheric pressure (+20 mm Hg).

In this way samples of Wesgo AL995 alumina, approximately $5 \times 5 \times 1$ mm^3 in size, with sputtered gold electrodes covering both 5×5 mm^2 faces, have been irradiated with a 6 mm diameter collimated beam perpendicular to one large face, in vacuum, dry air, and helium at 450°C, with an electron flux of 6×10^{16} m^{-2} s^{-1} corresponding approximately to 10^{-10} dpa s^{-1}, 700 Gy s^{-1}. Voltages between 100 and 1000 V (corresponding to electric fields of 100 kV m^{-1}–1 MV m^{-1}) were applied in order to study the effect of the radiation and environment on the surface degradation. Measurements were made of total current which included both volume and surface conductivity. In these experiments no attempt was made to measure volume RIC and RIED, the sole purpose being to measure the surface leakage current. The effect of the radiation itself on the surface degradation was examined using samples of $25 \times 25 \times 1$ mm^3, again with electrodes covering both large faces. In this case the irradiated region from the 6 mm diameter beam, was well separated from the edges of the sample. As a standard procedure following electrode sputtering, all the samples are heated in air to 500°C for up to 8 h. At various times during the experiments the irradiation was stopped and the samples were cooled to 20°C and then heated again to 450°C to measure the sample current as a function of temperature. The impurity content of the helium used in ppm, was as follows; H_2O: 2, O_2: 0.5, CO: 0.2, CO_2: 0.2, H_2: 0.2.

3. Results

Irradiation of the whole sample in high vacuum conditions and with an electric field applied at 450°C induced the already observed surface degradation [1]. This is shown in Fig. 1 for 100 and 500 kV m^{-1}, where the total measured current during irradiation is given. Severe degradation is observed which depends on the applied electric field strength. This degradation could be removed by persistent polishing of the sample edges with

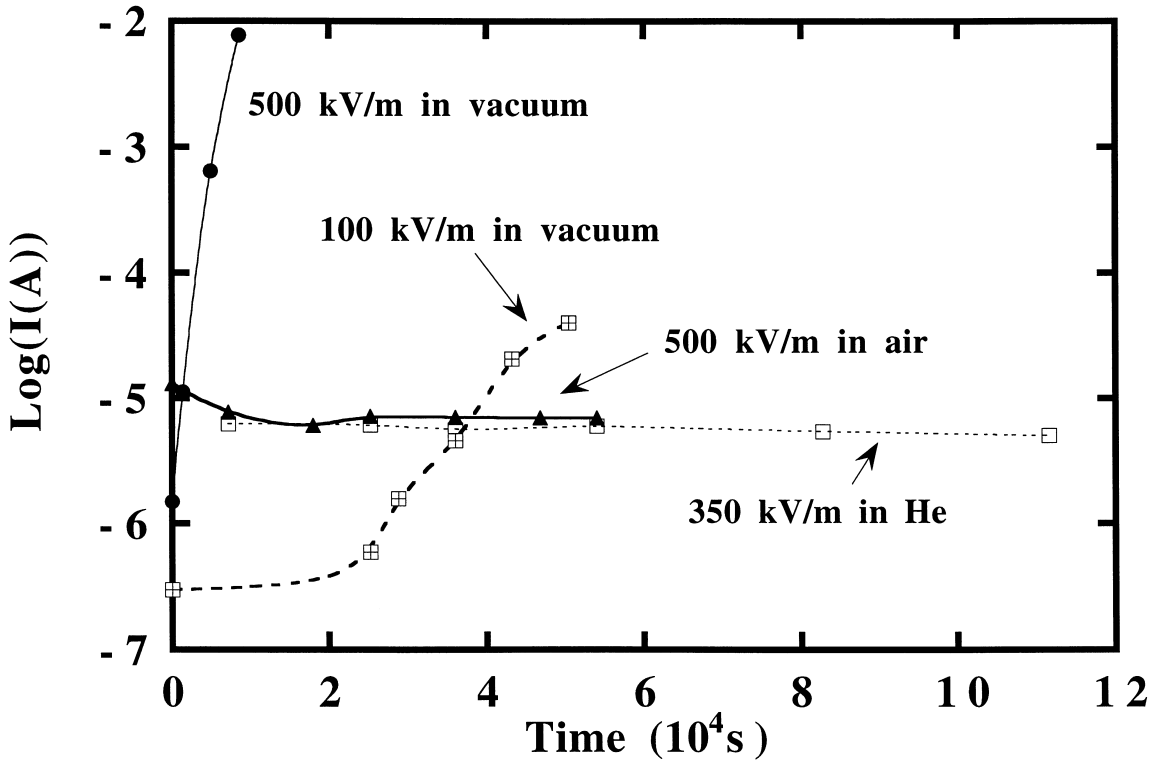

Fig. 1. Total current measurements during irradiation for samples irradiated in vacuum, air, and helium with different applied electric fields.

diamond paste, indicating that the measured current increase was due to leakage through the surface region of the edges. Initially it could also be partly removed by heating the sample in air, strongly suggesting that a reduction process had taken place. However with irradiation time the degraded region extended into the samples and could only be removed by long polishing. This is consistent with the SEM observations which indicated that the degraded region extends to between 20 and 100 μm below the surface [1]. In order to study the role of the vacuum environment on the degradation, two further samples were irradiated in air and in helium at the same temperature with an electric field applied. In these cases the samples did not show any measurable increase in surface conductivity during irradiation (Fig. 1). The measured total current, due to sample and gas radiation induced conductivity (RIC), remained constant during the whole irradiation. It is interesting to note that in the case of helium only 350 V could be applied to the sample due to radiation enhanced breakdown in this gas [4], and that from comparison of the initial currents measured in vacuum, air, and He, approximately 90% of the measured current with helium and air appears to be due to the gas RIC.

The state of the samples was observed at various stages without radiation by heating from 20°C to 450°C and measuring the current. This is shown in Fig. 2, where the apparent conductivity which includes both volume and surface currents, is plotted against T^{-1}. The two samples irradiated in air and helium show activation energy curves almost identical to those observed for the volume conductivity measured in a guarded system [1], indicating that no measurable surface degradation had taken place even after 15 h of irradiation. The sample irradiated in vacuum with an electric field of 500 kV m^{-1} however shows severe degradation corresponding to the surface leakage after only 40 min of irradiation.

In order to study the role of the radiation field itself on the surface degradation, further larger samples were irradiated in vacuum in such a way that the sample edges were not in the electron beam. In this case the samples could be irradiated even with up to 1 MV m^{-1} for more than 50 h before degradation was observable (Fig. 3). Following 10 h of irradiation the sample conductivity was even observed to be reduced, as may be seen in Fig. 2.

In addition to the enhanced surface degradation, a radiation enhanced electrolysis, resulting in a concentration of impurities at the negative electrode, had been

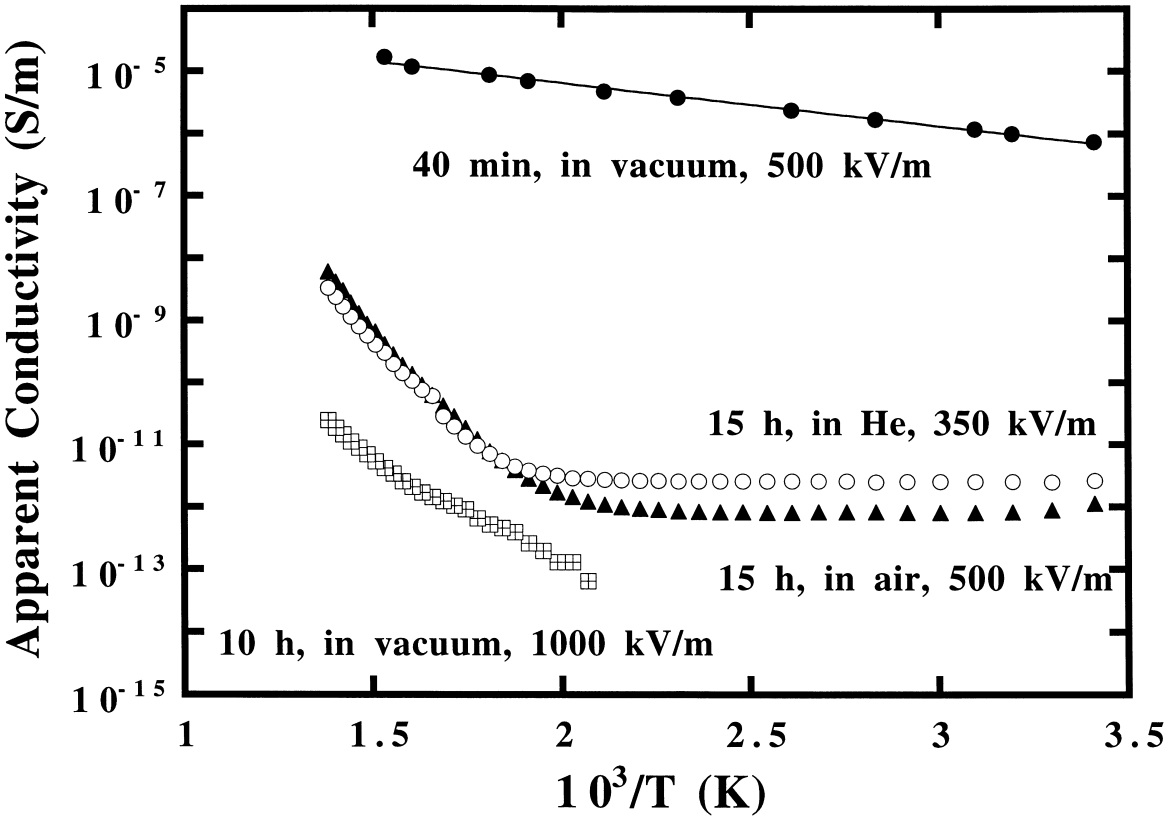

Fig. 2. Apparent electrical conductivities as a function of temperature measured without irradiation for samples irradiated under different conditions.

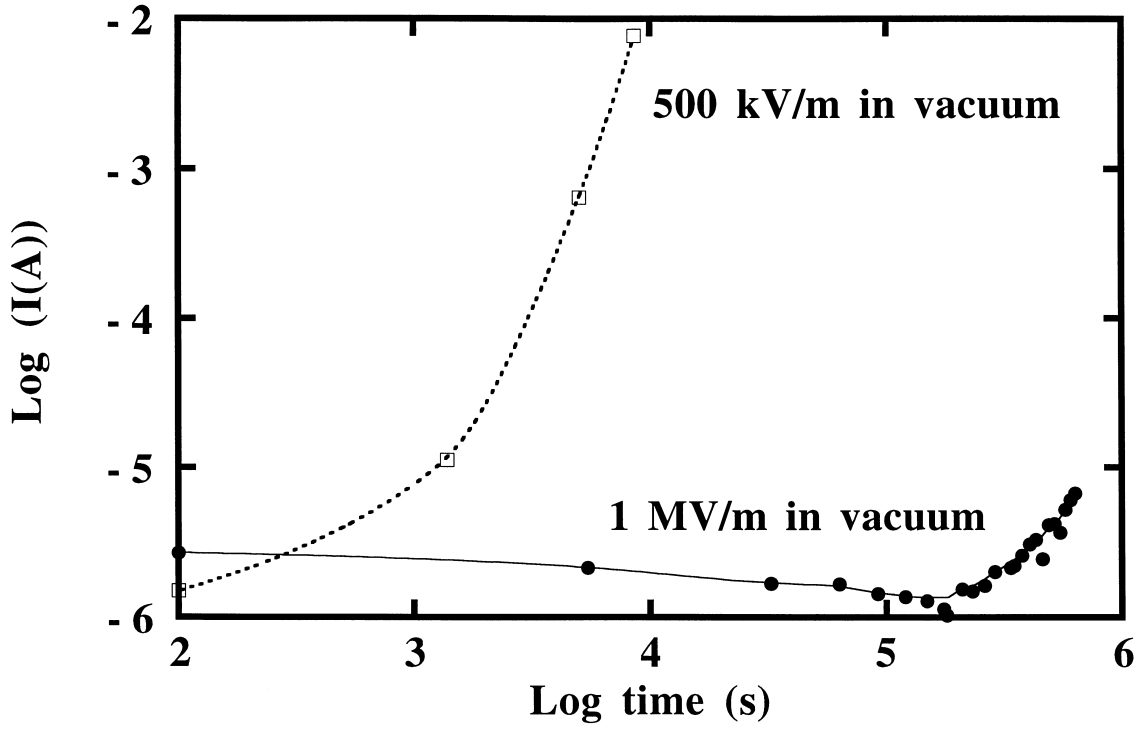

Fig. 3. Total current measurements during irradiation for two samples irradiated in vacuum, for 500 kV m^{-1} with the edge surfaces irradiated, and for 1 MV m^{-1} without irradiating the edge surfaces.

observed [1]. It was at first thought that this high mobility of the impurities could also possibly be related to the surface degradation, however in the experiments reported here a very similar radiation enhanced electrolysis has been observed in the samples irradiated in air and helium, but without surface degradation.

4. Discussion

The results given in Figs. 1 and 2 show quite clearly that surface degradation occurs in vacuum but not in air or helium. The fact that degradation does not occur in helium suggests that the degradation is not due to a lack of oxygen, but rather the vacuum itself, i.e. vacuum reduction. This is in agreement with the observation that partial recovery occurs when heating in air. However it may be possible that the few ppm of oxygen present in the He is still sufficient to prevent reduction. It is also clear that the degradation is a strong function of the electric field, but at this stage it is not possible to know if the increased field or simply the increased leakage current cause the more rapid degradation. The fact that the surface degradation occurs in Wesgo AL995, but not in Vitox nor sapphire under identical conditions would suggest a process related to this particular grade of alumina, rather than radiation enhanced surface contamination [5]. Further experiments in Deranox alumina and Goodfellow pc BeO which have also shown no surface degradation, support this view.

The importance of the radiation field may be seen in Fig. 3 which shows that when the surface region between the electrodes is directly in the radiation rapid degradation occurs, but when this surface region is out of the radiation field degradation is markedly reduced even for a higher electric field. This observation is relevant to earlier conflicting results for Wesgo AL995 alumina where in one case surface degradation was observed, and in another no degradation occurred [6]. In the first case all the sample including the inter-electrode surface was irradiated, whereas in the second case only the inner central electrode area was irradiated. The first case was put down to beam induced breakdown of hydrocarbons [5], however our results show that the surface degradation is related to the specific grade of alumina rather than surface contamination effects.

It is not clear why this particular grade of alumina is susceptible to surface degradation under certain conditions. It was at first suggested that the high impurity content, large grain size, and/or low density could be the cause [1]. The observation here that radiation enhanced impurity electrolysis occurs also in air and helium, but

without surface degradation, would therefore suggest that the impurity content is not an important parameter. However further work is required to clarify this point.

5. Conclusions

Wesgo AL995 alumina has been found to be highly susceptible to surface electrical degradation when irradiated with an electric field applied at high temperature in vacuum. In contrast irradiation in air or helium produces no degradation. The cause of the degradation may be related to the large grain size and/or the low density of this particular alumina grade. The role of the environment in electrical conductivity and degradation radiation experiments has not been sufficiently considered. This is particularly the case for in-reactor experiments, which have been generally performed in a helium environment. It is essential that one takes into account that fusion applications require the insulating material to perform with at least one face in high vacuum. Further work is clearly required to assess the reliability of the different alumina grades under vacuum irradiation.

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