Role of specimen thickness on the electrical conductivity of single crystalline alumina under electron irradiation

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The electrical conductivity of 125-, 332-, and 545-μm-thick single crystalline Kyocera alpha alumina has been measured before, during, and after irradiation with 1 MeV electrons in an applied electric field of 300 kV/m at temperatures up to 723 K. Simultaneous measurements of the bulk and surface conductivity to a total fluence of $8.0\times10^{22}\,e/\mathrm{m}^2$ (9.4×10^{-5} dpa and 5.0×10^{9} Gy) at 723 K show no bulk and no surface degradation in the specimen, rather than only a sort of decrease of the conductivity with total dpa. Strong thickness dependence of radiation induced conductivity (RIC) is found and is believed to be due to the effect of electron charge deposition and the production of charged point defects during irradiation. Finally it is suggested that the thickness dependent RIC of the insulating materials must be considered carefully before designing the coating and window materials of fusion reactors.
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I. INTRODUCTION

Technological applications of alumina in high temperature devices, as well as in proposed fusion reactors as insulators at moderate temperature under irradiations, require an investigation of the electrical performance of the insulators during irradiation.[1] The electrical conductivity of an insulator exposed to ionizing radiation increases to a higher value that is dependent on dose rate but not on accumulated dose and is called radiation induced conductivity (RIC).[2] The increase in conductivity may limit the applications of insulating materials in fusion reactors. Hodgson[3] first reported a phenomenon, called radiation induced electrical degradation (RIED), in which a Union Carbide sapphire insulating material was degraded permanently under 1.8 MeV electron irradiation in the presence of an electric field of 130 kV/m at a temperature of 723 K. The electrical conductivity of the degraded sample did not revert to the unirradiated conductivity when the irradiation was stopped. Later, he and his colleagues confirmed RIED in subsequent experiments.[4–6] In addition, several other groups found RIED in sapphire irradiated with 18 MeV protons, with fast neutrons, with 1.8 MeV electrons, and with ions.[7–11] Pells and Hodgson[10] mentioned that RIED was a bulk effect rather than a surface effect through observing microstructural changes in alumina under 1.8 MeV electron irradiation. On the other hand, RIED became controversial when other groups failed to find RIED in alumina with 1–2 MeV electrons, with 28 MeV He^+ ions and with fast neutrons.[12–19] Jung *et al.*,[20] Kesternich *et al.*,[17,21] and Kinoshita *et al.*[13,15] found RIED-like behavior in alumina that was not a bulk effect, could be explained by surface contamination or internal fracture of the insulator. Hodgson[10] has found the production of point defects during irradiation by optical spectroscopy which influences the observed microstructural changes. In addition, surface breakdown was observed by Morono and Hodgson[22] in Wesgo AL 995 but not in Vitox 999 under 1.8 MeV electron irradiation. In fact, no single study has conclusively demonstrated the cause responsible for RIED. Therefore, further experiments are needed to resolve this controversial phenomenon.

Electrons passing through an insulator undergo frequent elastic collisions which cause range straggling of the electrons.[23] Seltzer and Berger[24] have calculated the transmission and distribution of electrons in many materials using Monte Carlo simulation in order to understand the energy spectrum of straggling electrons. Zong and co-workers[11] have investigated the mechanism of RIED in alumina with 1.8 MeV electrons at 773 K and concluded that the charge of electrons and holes that causes defect clusters during irradiation is responsible for RIED. Therefore, the behavior of the transmitted and deposited electron charge in the insulator may have a great influence on RIC and this influence may correlate with the thicknesses of the samples during irradiation. Due to the growing conflict about RIED with electrons, Zinkle[25] recently proposed the necessity of determing the implanted charge profile in electron irradiated specimens. The purpose of this article is to clarify the effect of charge deposition of electrons and its contributions to the specimen current during measurements of current–voltage ($I-V$) in alumina of various thicknesses under electron irradiation.

II. EXPERIMENTAL PROCEDURE

In this study single crystalline α-Al_2O_3 (alumina) (Kyocera SA 100 with 99.99% purity and $\langle 1\underline{1}02\rangle$ orientation) specimens of 5.5 mm diameter and 125-, 332-, and 545-μm-

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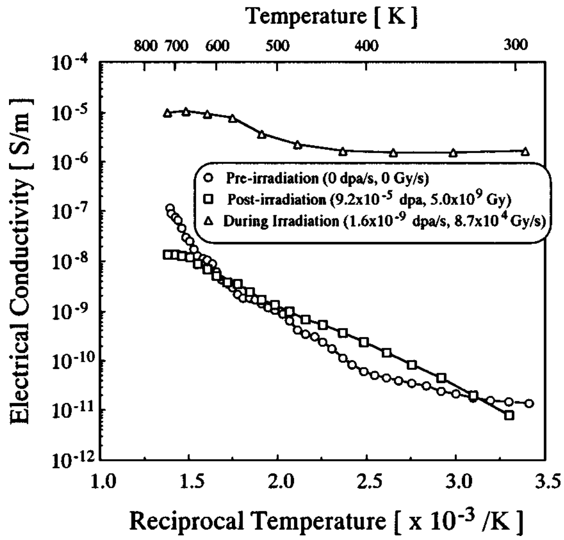

FIG. 1. Semilog electrical conductivity as a function of reciprocal temperature for a 332-μm-thick Kyocera alumina specimen before and during irradiation with a 1 MeV electron flux of $1.4\times10^{18}\,e/\mathrm{m}^2\,\mathrm{s}$ in an electric field of 300 kV/m and after irradiation of total fluence of $7.9\times10^{22}\,e/\mathrm{m}^2$ (9.2×10^{-5} dpa, 5.0×10^{9} Gy).

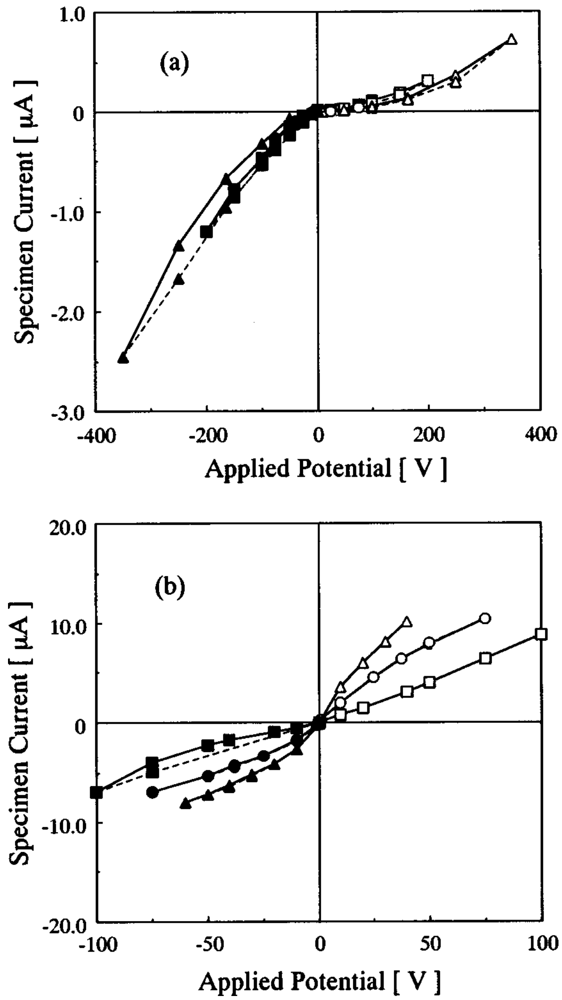

FIG. 2. $I-V$ relationships of 125 (○), 332 (□), and 545 (△) μm-thick alumina with Ti electrodes (a) before irradiation, (b) during irradiation with a 1 MeV electron flux of $1.4\times10^{18}\,e/\mathrm{m}^2\,\mathrm{s}$ at 723 K. The open and filled symbols correspond to the voltage applied to the base and center electrodes, respectively. Solid and dotted lines correspond to the increasing and decreasing applied voltage, respectively.

thicknesses were used and titanium was deposited in vacuum to make the three-electrode system. Prior to irradiation, the $I-V$ and the temperature dependence of electrical conductivity were measured in a bell jar at 10^{-4} Pa, from room temperature (RT) to 723 K while increasing and decreasing the temperature at ~3 K/min. The irradiation experiments were carried out in a high voltage electron microscope (HVEM) under 1 MeV electron beam-on and -off conditions at temperatures ranging from RT to 723 K. The pressure of the HVEM was $\sim10^{-5}$ Pa. Irradiation was done only to the center electrode of the specimen. Both bulk and surface conductivity were measured under irradiation with an electric field of 300 kV/m. More details of the experimental procedure and a specimen holder used are illustrated elsewhere.[15]

III. RESULTS AND DISCUSSION

The mechanism of electrical conductivity of alumina before irradiation is realized to be essential in order to get more insights into the irradiation behavior. Figure 1 shows the electrical conductivity of a 332-μm-thick Kyocera alumina specimen versus reciprocal temperature ranging from room temperature to 723 K before, during, and after irradiation with an electric field of 300 kV/m. The irradiation flux is $1.4\times10^{18}\,e/\mathrm{m}^2\,\mathrm{s}$ (1.6×10^{-9} dpa/s and 8.7×10^{4} Gy/s). The conduction behavior of the specimen can be criticized in terms of the activation energy which can be calculated from the Nernest–Einstein equation $\sigma=A\exp(-E/kT)$, where σ is the electrical conductivity (S/m), T the temperature (K), A the constant, E the activation energy (eV), and k the Boltzmann constant (eV/K). The electrical conductivity increases with increasing temperature with different activation processes. The estimated activation energy before irradiation up to 723 K is 0.40 ± 0.02 eV, represents thermal excitation of electrons from the shallow trapping centers to the conduction band. Depending on the transition of the slope of the curve, the activation energy has been separately estimated, that from RT to 400 K being 0.14 eV and that above 530 K being 0.83 eV. Again, if the conductivity of the specimen is partially thermal process limited during irradiation, then the estimated activation energy during irradiation is to be 0.09 ± 0.02 eV. This reduction of the activation energy during irradiation seems to be due to the dominance of irradiation beam on the thermal behavior. Similar reduction (~0.1 eV) of the activation energy was found in undoped alumina by Klaffky *et al.*[26] In fact, the conductivity slowly changes up to irradiation temperature of about 500 K and then abruptly raises at the higher temperatures. The preirradiation resistivity at RT ($7.3\times10^{10}\,\Omega$ m) of this work is comparable to that of ($10^{10}\,\Omega$ m) Terai *et al.*[27] with the applied electric field of 200 kV/m. Our study also has strong agreement with that of the available data of similar grade of single crystal alumina with respect to amplitude and overall activation behavior of bulk conductivity.

Figure 2(a) shows the electric current in 125-, 332-, and

545-μm-thick alumina as a function of applied voltage before irradiation at 723 K. Measurements were done after 30 s (not given) and after 5 min of applying each voltage to the specimen. No substantial difference between the specimen current measured after 30 s and 5 min was found. Non-ohmic behavior is apparently clear. Nonsymmetric behavior of $I-V$ curves with respect to the polarity of applied voltage indicates electrode-limited blocking effect. This usually happens due to a difference in the work function between the electrode material and the specimen, called the Schottky effect.[26] A sample-thickness dependence of the specimen current is not apparent before irradiation. The $I-V$ curves of 125-, 332-, and 545-μm-thick alumina under 1 MeV electron irradiation with a flux of $1.4\times 10^{18}\, e/\mathrm{m}^2\,\mathrm{s}$ (1.6×10^{-9} dpa/s and 8.7×10^4 Gy/s) at 723 K are shown in Fig. 2(b). Unlike unirradiation $I-V$ measurements, the specimen current measured here required $\sim$1 min at each step to reach a stable reading after the application of electric field. Even though asymmetry exists between the positive and negative quadrant values, the differences between them reduces with irradiation. In the positive quadrant, the specimen current for 545-μm-thick alumina is supralinear whereas for other specimens are sublinear. On the other hand, the specimen current in the negative quadrant for all specimens are sublinear. Comparing these two figures indicates that irradiation either changes or masks the blocking effect between the titanium electrode and specimen. Thickness dependent specimen current is apparent here. The inconsistency of the 332-μm-thick specimen current (less than the 125-μm-thick specimen current) here is due to the different irradiation history (time dependence at every 50 °C stair stepped temperatures was done only in 332-μm-thick specimen). The degree of non-ohmic behavior of $I-V$ curves for alumina irradiated with neutrons at reactor full power is quite different to that of this study.[18] Analogous $I-V$ behavior in a 545-μm-thick Kyocera alumina with and without irradiation was previously found.[13] In addition, the $I-V$ behavior in alumina during and after irradiation with 1.8 MeV electrons by Zong *et al.*[11] is compatible with these results. However, the $I-V$ behavior in this study apparently indicate Schottky effect which behave differently with and without irradiation.

After the first reporting of RIED by Hodgson, numerous studies have been done at quite similar conditions to those of Hodgson. In this study we irradiated alumina specimens with an applied electric field for a long time to observe RIED, and followed IAEA recommendations.[3,14] Figure 3 shows a comparison of the fluence dependence of the bulk RIC for 125-, 332-, and 545-μm-thick specimens with a 1 MeV electron flux of $1.4\times 10^{18}\, e/\mathrm{m}^2\,\mathrm{s}$ (1.6×10^{-9} dpa/s and 8.7×10^4 Gy/s in a dc electric field of 300 kV/m at 723 K. The corresponding potentials for 125-, 332-, and 545-μm-thick specimens are 37.5, 100, and 163.5 V, respectively. Simultaneous measurements of the bulk (between base and center electrodes) and surface (between center and guard electrodes, not shown here) conductivity were done. The RIC promptly increases to a higher value than that without irradiation by about three orders of magnitude due to electronic excitation.[15] Even though the experimental conditions were similar to that of Hodgson, no bulk and surface degradation were observed in the specimens, other than only a decrease of both the conductivity with total dpa of 9.4×10^{-5} (5.0×10^9 Gy), in contrast to the abrupt increase in the surface conductivity at 3.3×10^{-5} dpa in our previous study where the RIED experiment was 7.7×10^{-5} dpa.[3–6,15] Increases of RIC with increasing specimen thickness are found here. We also checked for errors in the calculated conductivity which might have been caused by surface conduction using Kesternich *et al.*'s method[17] and found no error in the bulk conductivity. The postirradiation conductivity after this total damage is less than the unirradiated conductivity. Farnum *et al.*[18] have measured the fluence dependence of specimen and leakage current of sapphire and MI cables, respectively, with reactor neutrons. No permanent degradation was measured. The electrical conductivity returned to the unirradiated value with the reactor off after a fluence of $3.0\times 10^{24}\,\mathrm{n/m^2}$ (0.3 dpa). They have also failed to observe the RIED in alumina with spallation neutrons irradiation in a dc electric field of 50 kV/m at 673–873 K to fluences of 0.02 dpa but observed decrease in the RIC during the irradiation in a course similar to these results.[19] Hodgson[3] concluded that concurrent ionization and displacements are necessary for RIED based on measurements of conductivity in UC alumina irradiated with 1.8 and 0.3 MeV electrons. The drawback of Ref. 3 is that the 1-mm-thick alumina used with 0.3 MeV electrons is far beyond the projected range of the electrons at 0.3 MeV energy (range is about 0.2 mm). Zong and co-workers[28] have suggested that both ionization and displacements are not required for RIED because of two independent mechanisms, of which the former is electronic and the latter is nucleus related. In addition, due to the lack of information in Hodgson's paper it is not clear whether his measurements were showing intrinsic or extrinsic behavior. On the other hand, the observed RIED-like abrupt increase of the surface conductivity of a 270-μm-thick alumina specimen irradiated with 1 MeV electrons with an applied electric field of 93 kV/m was compatible with the results of Kesternich *et al.*,[17,21] which showed a severe surface leakage current. Thus, we conclude that the RIED observed by Hodgson is

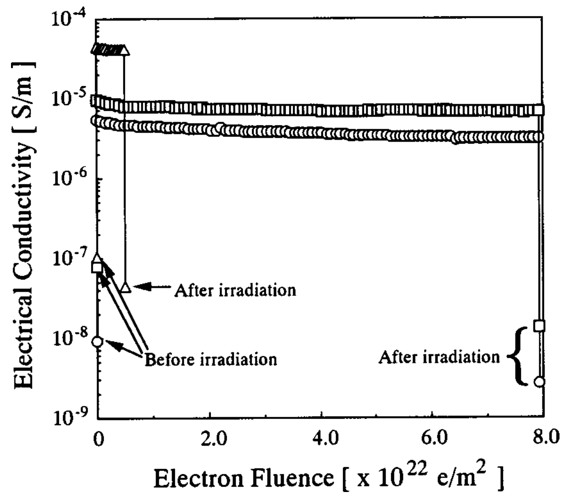

FIG. 3. Electrical conductivity of 125 (○), 332 (□), and 545 (△) μm-thick alumina having Ti electrodes under irradiation with a 1 MeV electron flux of $1.4\times 10^{18}\, e/\mathrm{m}^2\,\mathrm{s}$ in a dc electric field of 300 kV/m at 723 K.

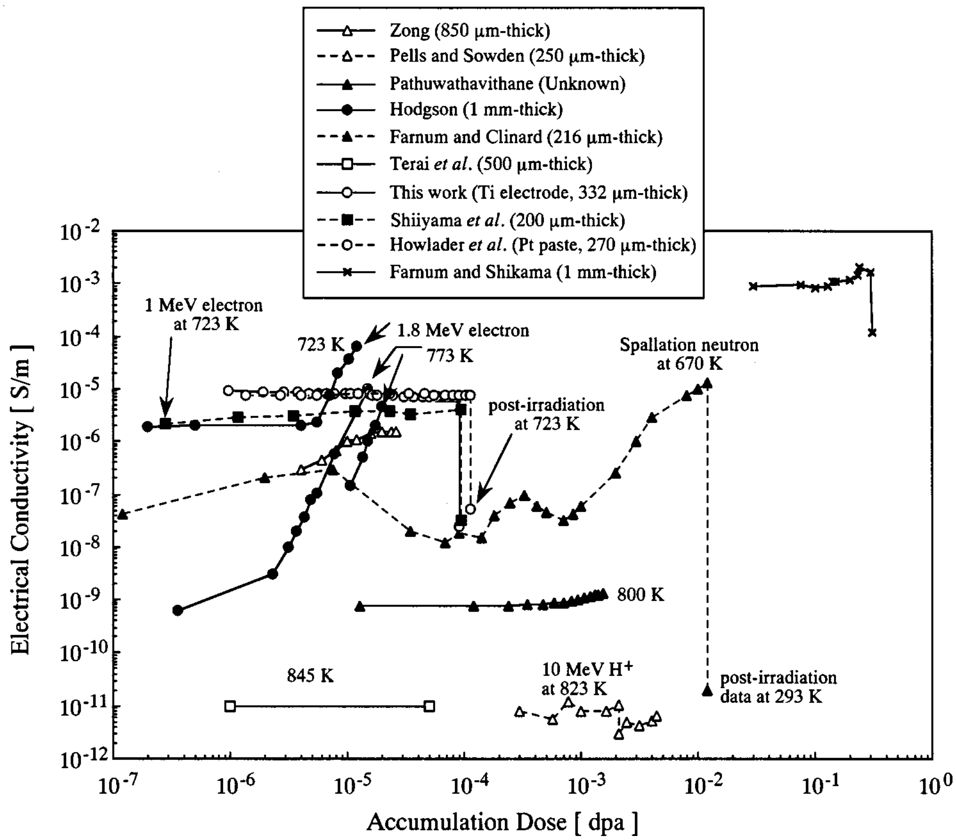

FIG. 4. Compilation of RIED measurements on single crystal alumina specimens irradiated at temperatures between 670 and 845 K. Dose rate of our work is 1.6×10^{-5} dpa/s. No threshold level of RIED is found up to 0.3 dpa except in UV grade sapphire with 1.8 MeV electrons irradiation.

either due to surface contamination or a charge deposition effect. No contamination in this study influenced surface RIC. The value of RIC in our previous study of 270-μm-thick alumina after a few seconds reach stability of the beam current was 7.2×10^{-6} S/m whereas the respective values for 125-, 332-, and 545-μm-thick alumina are 5.3×10^{-6}, 9.7×10^{-6}, and 4.5×10^{-5} S/m, respectively, so the value of RIC of 270-μm-thick alumina is between the 125- and 332-μm-thick specimens.[15] It indicates that RIC is strongly dependent on the specimen thickness rather than either the applied electric field or the type of electrode materials provided (the ionizing dose rate is the same). This dependence of RIC will be discussed below.

Figure 4 shows the compilation of RIED measurements on single crystalline alumina irradiated with various irradiation sources at temperatures ranging from 670 to 800 K. Few studies have failed to observe RIED on sapphire irradiated with electrons, protons, and neutrons.[8,13,15,29,30] In addition we have performed a series of RIED experiments at 723 K in sapphire using Pt paste, Ti and Ti–Au electrodes and have never observed the significant level of RIED in sapphire.[13,15,16] However, an apparent level of surface degradation is found in Kyocera alumina under 1 MeV electron with an applied electric field of 93 kV/m at 725 K. In fact, the irradiation temperature and the applied electric field are in the range of those of Hodgson where he found RIED in UV-grade sapphire irradiated with 1.8 MeV electrons. Definitive levels of RIED have not been observed in the neutron irradiated RIED studies on sapphire.[8,18,31] A RIED-like degradation behavior was observed in sapphire irradiated with spallation neutrons at 670 K, but postirradiation measurements demonstrated that the increase in apparent conductivity was due to surface leakage currents and/or gas ionization effects.[31] In fact, the postirradiation conductivity at room temperature in sapphire irradiated with spallation neutrons to a dose of $\sim10^{-2}$ dpa at 670 K was reduced to $<10^{-10}$ S/m.[31] Application of an electric field of 50 kV/m for 240 h of a total of ~0.03 dpa to a sapphire sample being irradiated at ~620 K did not produce measurable RIED above the existing RIC value of about 1.5×10^{-5} S/m.[24] RIED was not detected in a fission reactor experiment on sapphire performed at ~520 K to a damage level of ~0.3 dpa.[18] Apparent discrepancy in the amplitude of the conductivity and in the total threshold dpa for RIED of sapphire suggests that there may be a factor affecting the RIED quantitively which is not taken into account. This parameter is likely to be the specimen thickness as previously shown (see Fig. 3).

Figure 5 shows a comparison of the RIC in alumina having thicknesses of 125, 332, and 545 μm at 296, 470, and 723 K at various electron fluxes. The RIC proportionately increases with increasing irradiation flux and temperature. At 723 K, the RIC for 545-μm-thick alumina is most sensitive to sample thickness than that at other temperatures. The electrical conductivity (σ) of ceramic insulators during irradiation is expressed by the equation $\sigma=\sigma_0+kR^{\delta}$, where σ_0 is the conductivity in the absence of radiation, k a material dependent constant, R the irradiation flux, and δ the irradiation flux exponent.[32] The temperature dependence of the δ values for all specimens during increasing beam intensity

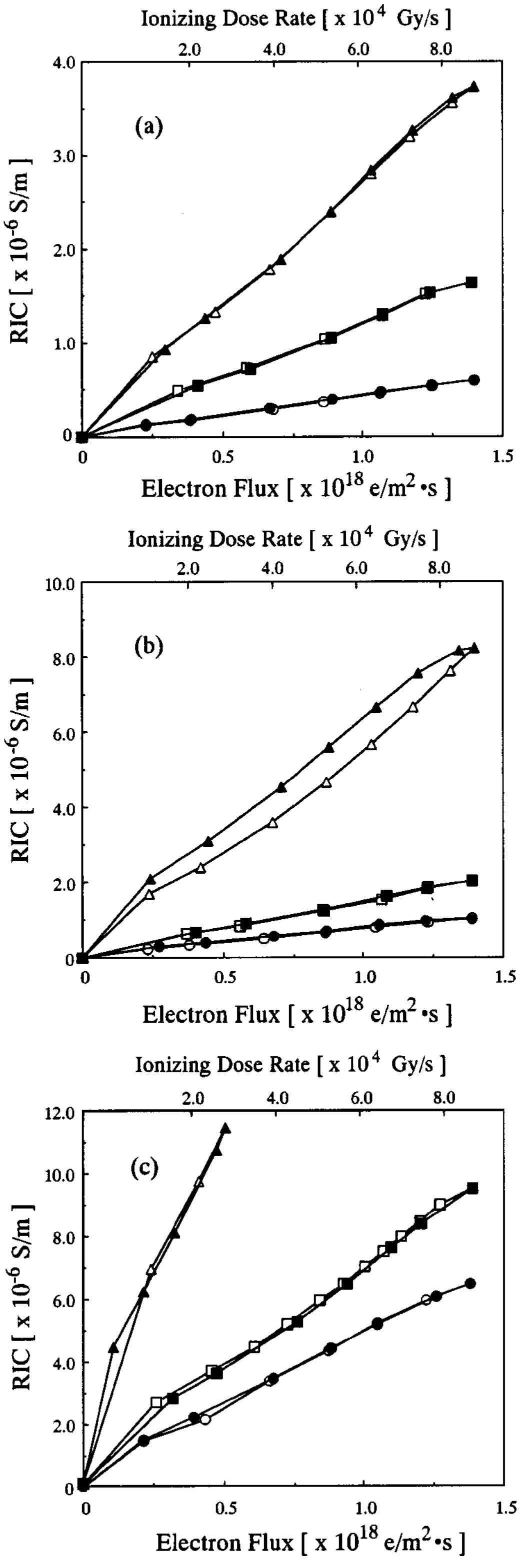

FIG. 5. RIC of 125 (○), 332 (□), and 545 (△) μm-thick alumina having Ti electrodes under 1 MeV electron irradiation with increasing and decreasing beam intensity at (a) 296, (b) 475, and (c) 723 K in a dc electric field of 300 kV/m at the indicated temperatures. The open and filled symbols correspond to increasing and decreasing beam intensity, respectively.

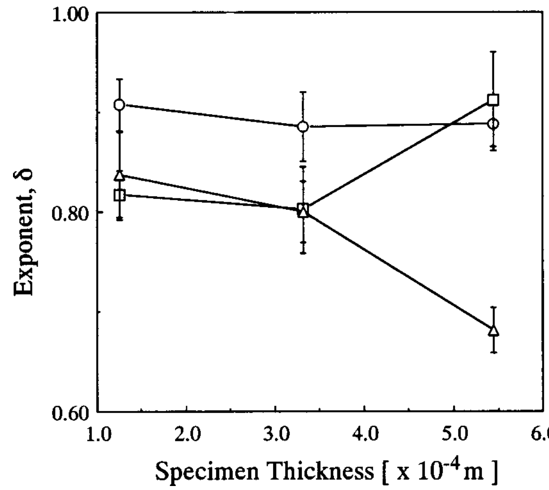

FIG. 6. Thickness dependence of the irradiation flux exponent (δ) of the electrical conductivity (σ) at (○) 296 K, (□) 476 K, and (△) 723 K based on $\sigma=\sigma_0+kR^\delta$ with the experimental data shown in Fig. 3, where σ_0 is the conductivity in the absence of irradiation, k a material dependent constant, and R the irradiation flux.

was obtained from Fig. 5 and it is plotted in Fig. 6. The exponent of irradiation flux decreases with increasing specimen thickness and irradiation temperature within experimental error. The unexplained and exceptional value of δ is found for 545-μm-thick alumina at 476 K. As previously mentioned, there are many reports on the measurements of RIC in alumina of which some report RIED and the rest do not. A few studies have argued that the beam deposition current (that is, the current absorbed in the specimen) can affect the conduction current even though no systematic understanding has developed concerning the relationship between beam deposition current, conduction current, and thickness.[11,23,28] To obtain more information about how the thickness of the specimens affects the conduction current, the thickness dependence of RIC of alumina was calculated for a 1 MeV electron flux of $1.4\times10^{18}\,e/\mathrm{m}^2\,\mathrm{s}$ (1.6×10^{-9} dpa/s and 8.7×10^4 Gy/s) in a dc electric field of 300 kV/m at 296, 470, and 723 K which is shown in Fig. 7. Increasing the sample thickness, increases the RIC nearly linearly at RT whereas it becomes more nonlinear at high temperature. These results showing strong thickness dependence of the

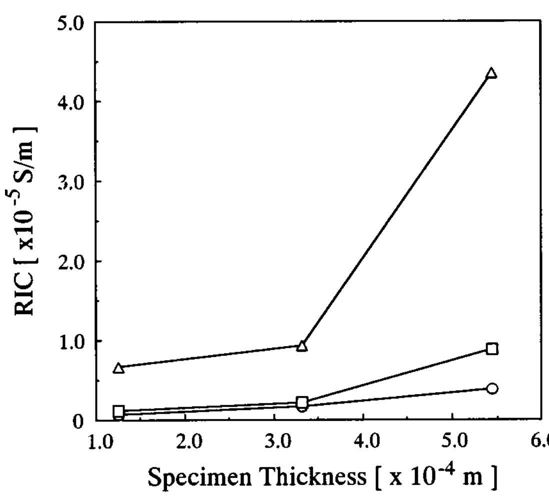

FIG. 7. Thickness dependence of RIC of alumina with a 1 MeV electron flux of $1.4\times10^{18}\,e/\mathrm{m}^2\,\mathrm{s}$ in an electric field of 300 kV/m at (○) 296 K, (□) 476 K, and (△) 723 K.

electrical conductivity under electron irradiation indicate that a part of the electron beam current deposits in the samples producing the electronic excitation and charged point defects. The deposited current increases with increasing specimen thickness and affects the value of the specimen current. The strong increase of RIC at high temperature (723 K) is probably determined not only by electrons but mostly by the formation of charged point defects under electron irradiation. The highest RIC in 545-μm-thick specimen seems to be due to the deposition of most electrons charge and the production of charged point defects in the specimen. Seltzer and Berger[24] calculated the deposition of electron charge in the specimen and found that about 50% of the total electron charge is deposited in a 1-mm-thick specimen with 1 MeV electrons using aluminum foils. Even though the projected range of 1 MeV electrons in aluminum is about 1.1 mm, about 50% of the incident electrons are stopped in a 1-mm-thick foil. We compared Refs. 24 and 33, and found that ~5 to ~20% of the 1 MeV electron beam current may deposit in the 125–545 μm-thick specimens, respectively. The estimated value of beam deposition current for 125-, 332-, and 545-μm-thick specimens will be 2.5×10^{-7}, 1.1×10^{-6}, and 2.0×10^{-6} A, respectively, at an incident flux of $1.4\times10^{18}\,e/\mathrm{m}^2\,\mathrm{s}$. The charge deposition of electrons and holes production during irradiation, which may be responsible for RIED, is argued by Zong *et al.*[11,28]

As already indicated, Pells and Hodgson[10] observed RIED in sapphire and examined the cause of RIED by optical and transmission electron microscopy. They found the mixed formation of alpha and gamma alumina in the bulk of RIED alumina. The formation of small aluminum particles acts as a precursor to the formation of gamma alumina. For this reason, the formation of small aluminum precipitates removes aluminum from the aluminum lattice leaving the necessary vacant aluminum sites required to allow alpha alumina to transform to gamma alumina. Recently Hodgson and Morono[34] have found the presence of aluminum colloids in irradiated samples by optical spectroscopy. Charge deposition including the formation of charged point defects in the samples under electron irradiation may aid the formation of these aluminum colloids and this result is compatible to that of the polycrystalline Wesgo AL995 alumina under electron irradiation.[35] This may possibly explain why Hodgson and his group have consistently found RIED in alumina.

IV. CONCLUSIONS

Nonlinear $I-V$ behaviors may indicate Schottky effect and behave differently with and without irradiation. No RIED was observed up to a total of 9.4×10^{-5} dpa. Strong specimen-thickness dependence of the RIC was found. The RIC dependence on thicknesses is believed to result from the variation of the electron charge deposition and the production of charged point defects under electron irradiation. The very strong increase of RIC at high temperature (723 K) is probably determined by the effect of charged point defects produced under electron irradiation. Charge deposition assisted formation of aluminum colloids may cause the bulk RIED-like degradation of alumina. It can finally be suggested that the thickness dependent RIC of the insulating materials must be considered carefully before designing the coating and window materials of fusion reactors.

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