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In Situ Measurement of Radiation Induced Conductivity in Oxide Insulators During Neutron Irradiation

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In situ measurement of radiation induced conductivity in oxide insulators during neutron irradiation

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An experimental investigation of the *in situ* electrical conductivity of Wesgo Al995 polycrystalline alumina at approximately 450 °C has been performed at the high flux beam reactor at Brookhaven National Laboratory. The measured radiation induced conductivity (RIC) was about 10^{-8} S/m at an ionizing dose rate of 6000 Gy/s. No evidence for permanent radiation induced electrical degradation was observed for an applied electric field of 147 V/mm up to a dose level of ≈ 1.8 displacements per atom. The effect of neutron irradiation on the electrical properties of two mineral insulated cables was also investigated. The RIC in the MgO insulation of a coaxial and a triaxial cable was measured to be in the range of $6\text{–}20 \times 10^{-8}$ S/m at an ionizing dose rate of $\approx$ 6000 Gy/s. [S0021-8979(98)04404-1]

I. INTRODUCTION

Ceramic materials are required in several areas of the International Thermonuclear Experimental Reactor (ITER) where design requirements call for highly resistive materials. There are three general areas of application for ceramic insulators in ITER: microwave windows, insulating current breaks, and diagnostic probes. The electrical properties of ceramic insulators are known to change significantly under irradiation.[1] Radiation induced conductivity (RIC) in oxide ceramics has been shown to increase electrical conductivity by many orders of magnitude at fusion relevant fluxes. Figure 1 gives a compilation of data from several studies[1–5] of RIC in single crystal Al_2O_4 (sapphire) and polycrystalline alumina. As an example, it is seen that the room temperature electrical conductivity of sapphire increases by more than six orders of magnitude to a value of approximately 10^{-6} (S/m) at a representative first wall dose rate of $\approx$ 2000 Gy/s. While this increase in conductivity is dramatic, it is still two orders of magnitude less than the assumed upper limit due to joule heating concerns of 10^{-4} (S/m) for fusion insulators.[6] The conduction lifetime for electrons necessary to produce these levels of conductivity in alumina have been measured[7] and is found to be 0.1×10^{-12} s. This compares with measured conduction lifetimes of 0.15×10^{-12} s and 0.05×10^{-12} s for the ceramic insulators silica and MgO respectively.[7] It is interesting to note that the electron lifetime in several insulating polymers has been estimated[8] to be on the order of 10^{-15} s, several orders of magnitude smaller than the lifetimes measured in these ceramics. In addition to its impact on fusion reactor design RIC is also of interest for other applications, including nuclear thermionics.

In the past several years there has been a considerable amount of research investigating the phenomenon of radiation induced electrical degradation (RIED) in insulating ceramics. The hallmark of this phenomenon is a large, permanent increase in the electrical conductivity of ceramic insulators which have been irradiated while they have had an electric field applied to them. This phenomenon differs from the well studied phenomenon of RIC[1–3,9] in insulators in that the increase in the conductivity persists after the radiation field is removed. RIED has been reported to occur in polycrystalline alumina,[10–12] single crystal alumina (sapphire),[13–15] amorphous alumina,[16] magnesia,[13] spinel[17] and anodized aluminum,[18] with most of the research concentrating on polycrystalline and single crystal alumina. These experiments have helped to define the conditions under which RIED is expected to occur. Some of the physical parameters which have been identified as important for the development of RIED in a particular material are the irradiating species, the magnitude of the applied electric field, and the irradiation temperature. Details of the experimental conditions under which RIED has been observed to occur in different materials have recently been reviewed.[19] Although the parameters under which RIED is expected to develop have become increasingly more well defined in the past several years, the physical basis for RIED has yet to be determined. This fact along with several recent experiments in which RIED did not develop under conditions where it was expected[11,20–24] have made the phenomenon of RIED a somewhat controversial topic.

Briefly, the conditions under which RIED has been observed to occur in polycrystalline alumina (the material used in this experiment) are as follows. RIED in polycrystalline alumina has been reported for several different types of irradiating species including fission neutrons[10] (in Kyocera A 479ss grade alumina), 18 MeV protons,[17] 104 MeV He ions,[11] and electrons[12] (all three in Vitox alumina). This phenomenon has not been observed in alumina for irradiation

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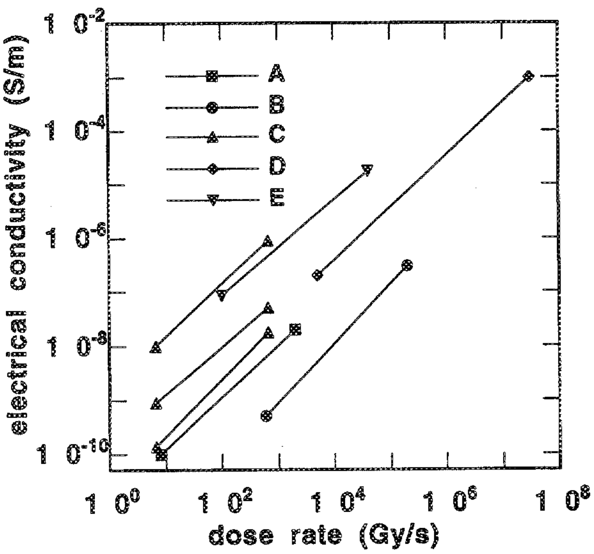

FIG. 1. Radiation induced conductivity in Al_2O_3. Curve A is from Pells (see Ref. 3) for polycrystalline alumina (Vitox) irradiated with 20 MeV protons at 450 °C. Curve B is from Farnum *et al.* (see Ref. 5) for polycrystalline alumina irradiated with 3 MeV protons at 25 °C. The curves labeled C are from Klaffky *et al.* (see Ref. 2) for single crystal Al_2O_3 (Linde Czochralski) irradiated with 1.5 MeV electrons at 450 °C. The lowest and next to lowest C curves are for 0.03 wt % and 0.004 wt % Cr_2O_3 doped samples respectively while the upper C curve is for an undoped sample. Curve D is from van Lint *et al.* (see Ref. 1) for single crystal Al_2O_3 irradiated with 33 MeV electrons at ~20 °C. Finally the E curve is from Goulding *et al.* (see Ref. 4) for polycrystalline alumina (GTE Wesgo AL998) irradiated with fission neutrons in a TRIGA reactor at ~20 °C.

temperatures less than ≈250 °C or greater than 550 °C. The range of applied electric fields in polycrystalline samples which have experienced RIED is from 100 V/mm to 500 V/mm. RIED has been seen to occur for doses as low as $\approx 1 \times 10^{-5}$ displacements per atom (dpa) in electron irradiated specimens[12] and the maximum value of the final bulk conductivity has been as great as 0.1 S/m for He ion irradiated Vitox alumina.[11] It is interesting to note that RIED has not been observed to occur in Wesgo AL995 grade alumina (the material used in this study) under a range of conditions where it may have been expected to occur according to RIED data on other grades of alumina.[11,21,23,24]

RIED may have an impact on the design of components and the application of ceramics in the next generation of fusion reactors, and it is important to understand the conditions under which it does and does not occur and what the underlying physical phenomena are.

This article reports the results of an experiment designed to study RIED in Wesgo AL995 alumina and which was performed at the high flux beam reactor (HFBR) at Brookhaven National Laboratory. This experiment is the third in a series of experiments performed by the authors at the HFBR. The results of the first two experiments (performed at temperatures of ≈80 and 350 °C respectively) were reported previously.[24,25] Neither of the previous two experiments observed the phenomenon of RIED; however the irradiation temperature in the first experiment is below the lower limit for which RIED has been reported to occur and the second experiment is very near to the lower temperature limit.

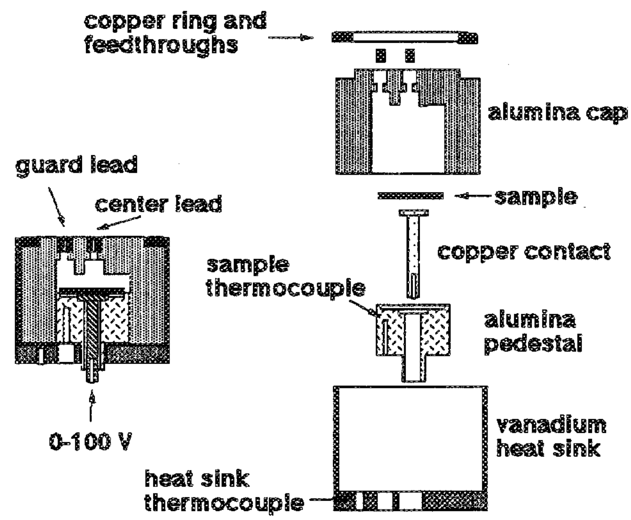

FIG. 2. Schematic of ISEC-3 subcapsule.

II. EXPERIMENTAL DETAILS

The data acquisition system and capsule design for the third *In Situ* Electrical Conductivity (ISEC-3) experiment are very similar to that used in the previous two experiments[24,25] and were described in detail there. The main concern with the data acquisition in this experiment is that an accurate measurement of the electrical conductivity of the sample material is performed. In order to insure this it is necessary that the sample electrodes have a guard ring configuration so that the center electrode current is not diminished by leakage currents. Briefly, the instrument control and data acquisition is accomplished using a computer fitted with an IEEE-488 interface card. This instrument bus controls and acquires data from a Hewlett-Packard power supply (used to supply power to the sample via a mineral insulated coaxial power lead), a Keithley 237 electrometer (used to measure the current through the sample), a digital multimeter (used to monitor thermocouple voltages), and a Keithley 9001 switching unit (to switch between samples and thermocouples). To minimize leakage currents in the data lead the lead to the electrometer is mineral insulated triaxial cable, the center conductor of which is attached to the center electrode of the sample and the inner sheath of which is attached to the guard ring of the sample (the outer conductor is ground).

The design of the subcapsule used in this experiment differs slightly from that used in the previous experiments and is shown in Fig. 2. The primary design difference is that the contacts to the samples were not spring loaded onto the sample electrodes. Instead, a small pad of TiCuSil braze material was vacuum brazed at 870 °C onto the sample surface in the center and guard electrode regions, and then nickel lead wires were laser welded to the pads. Following this, platinum center and guard electrodes were sputter deposited onto the sample. As in the previous experiments this subcapsule was placed inside of an aluminum capsule which was filled with ultra-high purity helium.

Another difference between the ISEC-3 capsule and the previous two is that the termination of the power lead was modified. In the previous capsules the mineral insulated co-

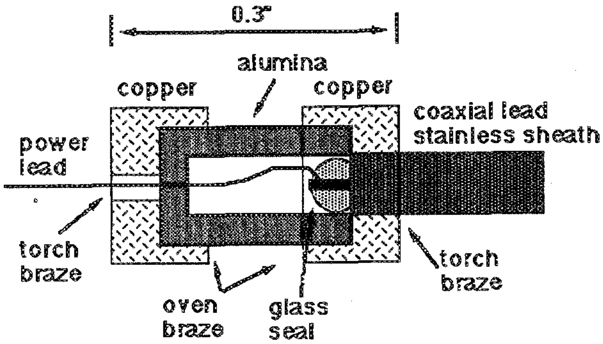

FIG. 3. Schematic of coaxial power lead termination.

axial power leads were either left unsealed in the case of the first experiment[25] or were sealed using a zinc borosilicate glass seal (Corning EG3606) as in the second HFBR experiment.[24] In these previous experiments many of the power leads experienced failure by electrical shorting of the center conductor of the coaxial power lead to the stainless steel outer conductor of the power lead. It was felt that the cause of these failures was that the terminations on the power leads were unsatisfactory, leading to contamination of the cable ends and subsequent shorting and failure. In an attempt to remedy this problem the power lead was terminated as shown in Fig. 3. Corning EG3606 glass was used to seal the end of the cable as before; however, in this case an alumina sleeve with copper endcaps was used to completely seal the power lead from the capsule environment. This power lead has a center conductor of 0.25 mm diameter an outer conductor with a 1.2 mm inner diameter and the insulation is MgO. Excellent results were obtained using this method of termination. In addition to using this new termination on the coaxial power lead, an identically terminated coaxial cable was monitored throughout the irradiation (with a continuously applied voltage) to determine what changes occur in the cable itself.

As in the previous experiments the primary thermal conduction path for this capsule design was radial to the walls of the capsule assembly, which were in contact with the reactor coolant water. The atmosphere inside the capsule was ultra-high purity helium and the design temperature was achieved by selection of the helium gas gap thickness between the sample mount and the capsule wall. The sample temperature was monitored during irradiation by two mineral insulated, type K thermocouples in contact with the alumina pedestal and the vanadium heat sink.

Considerable care was taken to have the highest purity capsule environment as could be reasonably achieved. The specimen was housed in a sealed subcapsule (see Fig. 2) to minimize possible deposition of electrically conductive surface contaminants. The material facing the exposed surfaces of the specimen in the subcapsule interior was alumina. The atmosphere in both the sealed subcapsule and capsule was helium. As in the previous experiments the capsule underwent three cycles of evacuation and back filling using a turbomolecular pump and ultra-high purity helium prior to insertion of the capsule into the reactor. The helium in the capsule was held at a gauge pressure of $\approx 1\times 10^5$ Pa during the irradiation.

The material for this study was polycrystalline Wesgo alumina AL-995.[26] The sample was a disk 0.75 mm thick with an 8.5 mm diameter, the center electrode diameter was 4 mm, and the gap between the center and guard electrode was 1 mm. A dc electric potential of 110 V was continuously applied to the sample during the irradiation using the Hewlett Packard power supply. This corresponds to an applied field strength of 147 V/mm. Also as mentioned above a stainless steel sheathed MgO insulated coaxial cable terminated identically to the sample power lead was placed in the irradiation capsule. A constant potential of 110 V (which corresponds to a minimum and maximum electric field strength in the insulation of 120 and 600 V/mm, respectively) was also applied to this cable during irradiation and the leakage current was monitored throughout the experiment to determine if a breakdown in the insulating properties of the cable occurred during the irradiation. In addition, an MgO insulated triaxial cable identical to that used in the data lead from the alumina sample was placed in the capsule and irradiated without an applied voltage. The outer diameter of this cable was 1.06 mm with a guard inside diameter of $\approx$0.6 mm and a center conductor diameter of 0.15 mm. The center conductor of this cable was periodically sourced with a voltage of from -1 to 1 V with the guard and shield held at ground. The leakage current was measured to determine if the insulation in this cable underwent breakdown.

The capsule was placed into the V-16 thimble of the HFBR during reactor operation. The capsule was located midcore which has an approximately 6000 Gy/s (6 W/g) ionizing dose rate and an associated fast neutron flux of 4×10^{18} n/m^2-s ($E \geqslant 0.1$ MeV). The temperature of the sample was continuously recorded as the capsule was inserted into the reactor and the initial specimen current measurements were taken within 10 minutes of capsule insertion. The capsule was irradiated for 49 full power days, which produced a damage level of $\sim$1.8 dpa in the alumina specimen.

III. RESULTS

The temperature history of the sample is shown in Fig. 4. This plot shows the temperatures measured by the sample thermocouple, which was located $\approx$1 mm below the sample in the alumina pedestal (see Fig. 2), and by a back-up thermocouple which was located in the vanadium heat sink of the subcapsule. The large drops in the temperature which occurred in the intervals between day 11 and day 22 of the irradiation and again between day 53 and day 61 are due to the shutdown of the HFBR reactor for scheduled refueling. The sample thermocouple registered a temperature of $\approx$440 °C for the first 24 days of the irradiation at which point it failed. However the second thermocouple continued to operate for the entire irradiation. The temperature measured by the back-up thermocouple can be seen in Fig. 4 to closely track the temperature measured by the sample thermocouple (although at a lower temperature due to its loca-

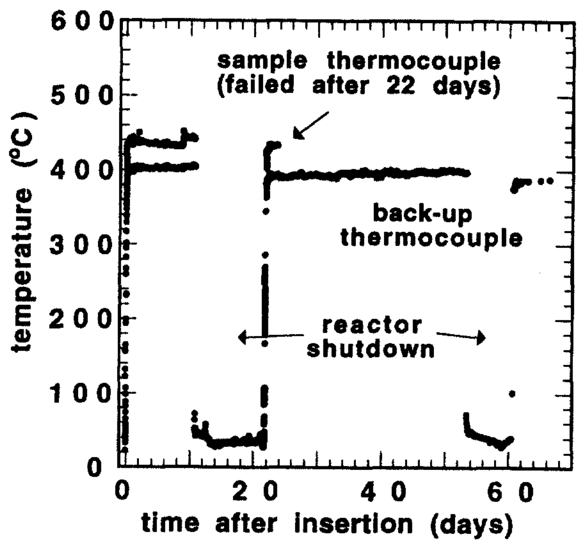

FIG. 4. Temperature history of the ISEC-3 subcapsule. The back-up thermocouple was located in the vanadium heat sink.

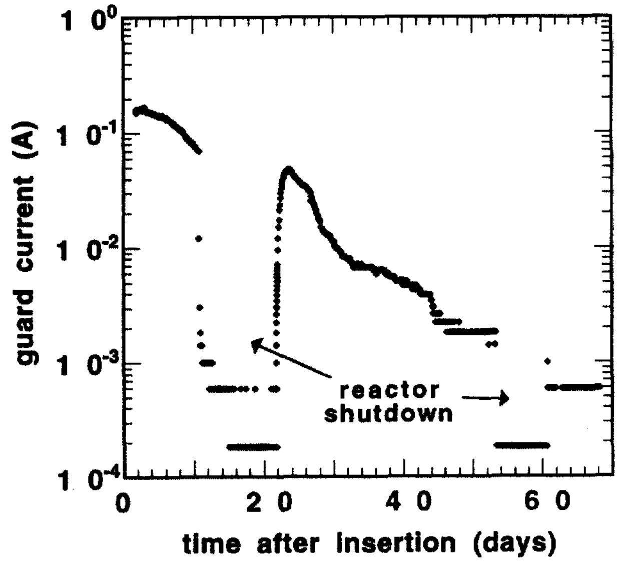

FIG. 6. Guard ring leakage current for the ISEC-3 subcapsule.

tion in the vanadium heat sink). Thus it may be concluded that the temperature of the sample remained at ≈440 °C for the entire irradiation.

Figure 5 is a plot of the time-dependent low side (triax) alumina sample current for an applied potential of 110 V. Note that during the first 2 days of the irradiation the current increased by a factor of 2 and then slowly decreased during the course of the irradiation. It is possible that this decrease is due to a self cleaning of the sample surface at the irradiation temperature (e.g., oxidation of conductive contaminants), thereby reducing spurious surface leakage currents. Note that during both reactor shutdowns the sample current decreased dramatically and that it immediately increased upon restart of the reactor.

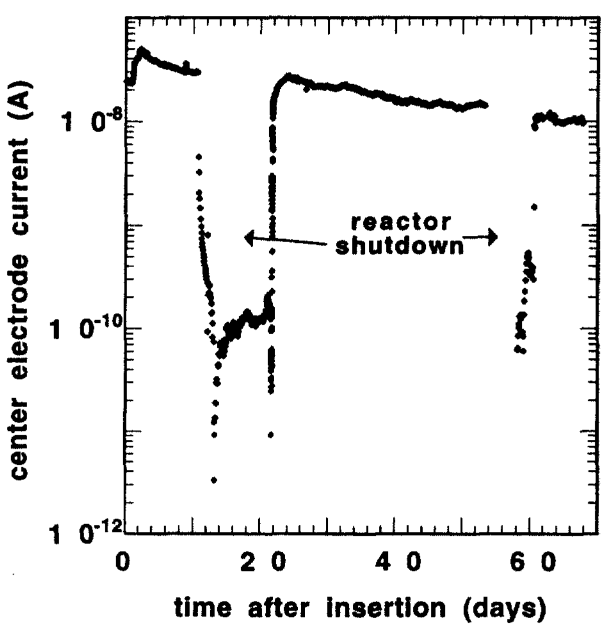

FIG. 5. Center-electrode current for the ISEC-3 subcapsule (110 V applied potential).

Assuming that Ohm's law is observed, the conductivity of the sample is given by

$$\sigma_v = \frac{t}{A}\frac{1}{R_v}, \tag{1}$$

where A is the effective electrode area, t is the thickness of the disk sample, and R_v is the measured volume resistance. The effective area A is given by[27]

$$A = \pi\left(\frac{D}{2} + \frac{g}{2} - \delta\right)^2, \tag{2}$$

where D is the actual center electrode diameter, g is the gap between the center and outer electrode, and δ is a parameter given by

$$\delta = t\left(\frac{2}{\pi}\ln\left(\cosh\left[\frac{\pi}{4}\frac{g}{t}\right]\right)\right). \tag{3}$$

Equations (2) and (3) give an effective area of $A = 16.3$ mm^2 in this case. Taking the data as plotted in Fig. 5 and using Eq. (1), the calculated RIC for most of the irradiation at 450 °C is $\approx 10^{-8}$ S/m, which is at the low end of the scatter band for RIC data on polycrystalline alumina at this dose rate (6000 Gy/s). It is seen from Fig. 5 that during the irradiation the current decreased by about a factor of 3. Also note that during shutdowns the value of the conductivity dropped off rapidly and approached the resolution limit of our equipment ($\approx 10^{-11}$ A) due to the greatly diminished ionizing dose rate and lower specimen temperature when the reactor was shut down. Following the restart of the reactor, the conductivity immediately increased to the level measured immediately prior to shutdown. The damage dose at the end of the irradiation was about 1.8 dpa (1.8×10^{25} n/m^2, $E \geqslant 0.1$ MeV), taking into account the reactor shutdowns. Thus in this sample RIED has not occurred (at least not above the level of RIC) at damage doses up to 1.8 dpa.

The guard ring leakage current from the sample versus time from insertion is shown in Fig. 6. Note the leakage

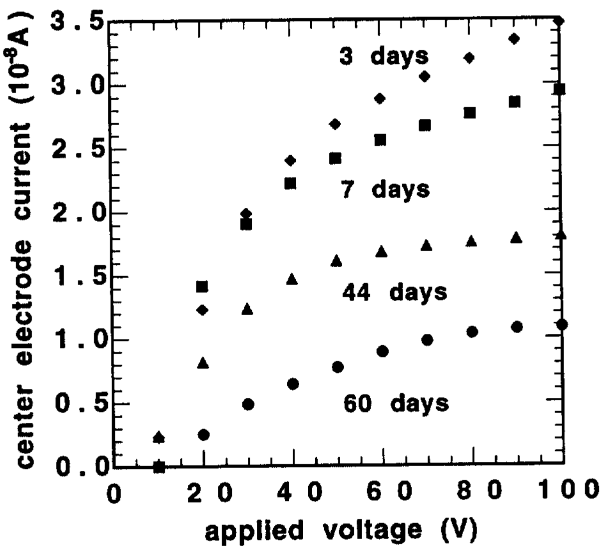

FIG. 7. Ohmic checks for the Wesgo alumina sample at different stages of the irradiation.

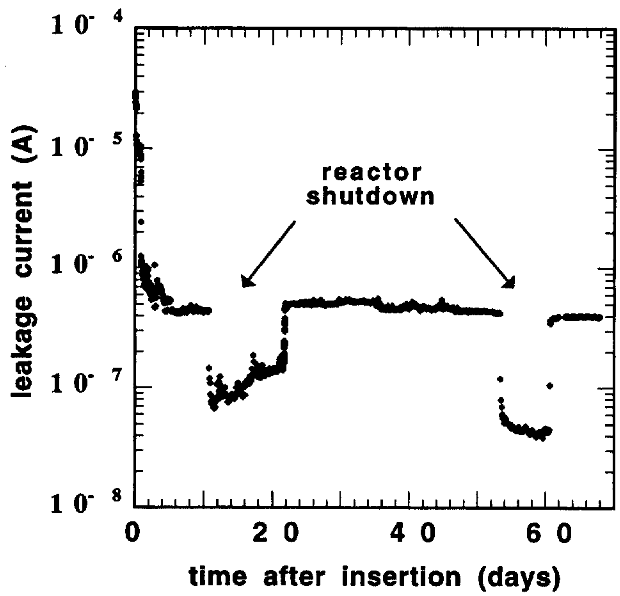

FIG. 8. Leakage current from the terminated coaxial cable sample.

current decreases by more than an order of magnitude during the irradiation. As mentioned, this may be due to self cleaning of the sample during the irradiation, or alternatively to a flaking off of the electrode material under irradiation conditions. However a postirradiation examination of the specimen is needed to confirm either of these possibilities. Again note the decrease in leakage current during the reactor shutdowns. The leakage current at the start of the irradiation was measured to be ~ 0.1 A. This suggests that the surface resistance from the high side electrode to the guard ring at the start of the irradiation was ~1 kΩ. Such a low surface resistance is likely to make the apparent sample conductivity, σ_a, calculated with Eq. (1) differ from the actual sample conductivity, σ_i. It has been estimated[28] that the error in a measurement of this type is given by

$$\Delta\sigma=\sigma_a-\sigma_i=\frac{t}{A}\frac{R_l}{R_s R_{c-g}}, \quad (4)$$

where R_l is the lead resistance from the instruments to the guard electrode (measured in pre-irradiation tests to be ~6 Ω), R_s is the surface resistance from the high side electrode to the guard ring, and R_{c-g} is the surface resistance from the center electrode to the guard electrode and which was measured in pre-irradiation tests to have a value of ≥30 MΩ. Using this equation the uncertainty in the calculated conductivity may be on the order of the calculated conductivity at the start of the irradiation. Once the leakage current drops below 0.01 A, the uncertainty will be less than 10% of the calculated value. However it is reasonable to assume that R_{c-g} may decrease from its pre-irradiation value and that the center-electrode current could come from surface leakage. Thus, due to the large guard-electrode currents which occurred particularly near the beginning of the irradiation, it should be noted that the center-electrode measurement represents an upper limit to the ionization-induced conductivity.

Figure 7 gives an example of the results of ramping the voltage from zero to a maximum of 110 V at varied times during the irradiation. This was done primarily to determine the ohmic nature of the contacts as well as to provide the conductivity of the sample from the slope of the curve (assuming linearity). The plots show that the sample was not ohmic (approximately a factor of 3 difference between a linear fit from the origin to 100 V and the asymptotic slope measured from $V\geqslant 50$ V) and therefore the conductivity cannot be directly obtained from Eq. (1) and Fig. 5. The measured current at a potential of 110 V decreased by about a factor of 3 between an irradiation time of 3 days and 60 days. However, the normalized shapes of the four current versus voltage curves shown in Fig. 7 were all similar. This suggests that there has not been any significant change in the nature of the electrode for this time period. Calculating the sample conductivity using the asymptotic ($V\geqslant 80$ V) slopes of the curves in Fig. 7 gives a conductivity of 6.4×10^{-9} S/m at 3 days and of 1.6×10^{-9} S/m at 60 days.

The results of the leakage current measurements on the terminated coaxial cable sample are plotted in Fig. 8. From this plot it is seen that this cable, which was sourced at 110 V throughout the irradiation, did not experience a breakdown of its insulation despite the presence of a continuously applied electric field of 120–600 V/mm in the MgO insulation. The approximate electrical conductivity (σ_e) of the MgO insulation was calculated from these data to be $\sim 5\times10^{-9}$ S/m using the well-known equation for coaxial cables, $\sigma_e=(I/V)\ln(r_0/r_i)/2\pi L$ where r_0 and r_i are the outer and inner radii of the MgO insulation respectively. Ohmic behavior and an effective irradiated cable length of $L\approx 0.2$ m were assumed for the coax cable RIC calculation.

As mentioned earlier, a triaxial cable which was not connected to a sample was also placed in the capsule. The current was measured as the center electrode of this cable was periodically sourced from −1 to 1 V with the guard electrode and outer sheath grounded. Figure 9 is a representative plot of the voltage versus current for this cable measured during neutron irradiation. The triaxial cable also did not

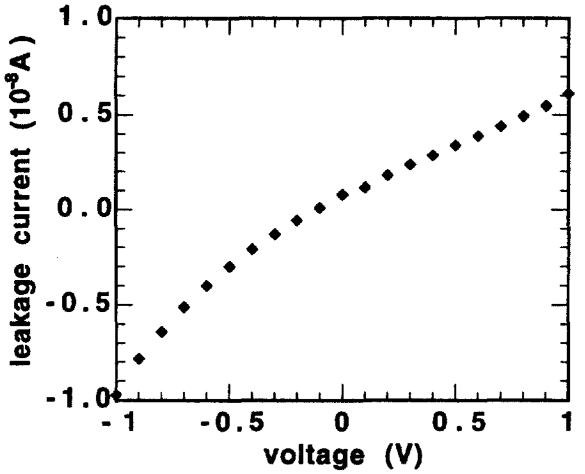

FIG. 9. Ohmic check on the center electrode of the triaxial cable sample after 50 h of irradiation.

experience a breakdown of its insulating properties during the irradiation, although it should be noted that a voltage was not applied to this cable for most of the irradiation so RIED would not be expected to occur. The cable exhibited a slightly non-ohmic behavior, with a positive offset current of $\approx$1 nA when the applied potential was zero. The measured triax cable resistance was ~190 MΩ for positive applied voltages and ≥50 MΩ for negative voltages. This corresponds to an electrical conductivity of 6–20 $\times 10^{-9}$ S/m for the MgO insulation assuming an effective irradiated cable length of 0.2 m, which is in fair agreement with the measured RIC value for the coaxial cable.

IV. DISCUSSION

Polycrystalline Wesgo AL995 alumina has been studied by five different research groups as part of an International Energy Agency (IEA) round robin effort to examine the RIED effect in alumina.[11,19,21–25,29] To this point there has been no bulk RIED observed in the Wesgo material by the five groups. The results presented in this article represent the highest neutron dose level of any RIED study undertaken thus far ($\approx$1.8 dpa) and are at the expected optimum temperature for the onset of RIED (450 °C). For this reason the data presented here further reinforce the position presented in the previous work,[24] that through the selection of the appropriate polycrystalline alumina, e.g., Wesgo AL995, the RIED effect as a bulk phenomenon may not pose a problem for fusion systems such as ITER. Furthermore, it should be noted that RIED was not observed in the coaxial cable which was irradiated with an applied voltage of 110 V (corresponding electric field in the insulation of 120–600 V/mm). The irradiation temperature of the coaxial cable is uncertain since it was not measured directly. The temperature was most likely $\approx$100 °C since it was in contact with the aluminum capsule wall. Therefore, the absence of RIED in the coaxial cable insulation may be due to an irradiation temperature below the regime where RIED has generally been observed, 250–550 °C.

The decrease observed in the RIC in these experiments agrees with previous studies on alumina.[2,3,5] This effect has been explained by taking into account the increased concentration of electron-hole traps produced by the irradiation. RIC occurs when a material is exposed to ionizing radiation of sufficient energy to impart kinetic energy to the valence electrons greater than the band gap energy of the material. Many of the ionized electrons and holes undergo geminate recombination and do not contribute to any current flow. However electrons and holes which escape geminate recombination can diffuse under an applied electric field and contribute to RIC. The RIC can be modified with the introduction of trapping centers in the material which trap the charge carriers and either lead to recombination (recombination centers) of the electrons and holes or the re-excitation of the electrons back into the conduction band (trapping centers). An increase in recombination traps leads to a decrease in the RIC.

In a previous experiment,[24] the alumina sample exhibited more nearly ohmic behavior than the sample in this study. In the ISEC-2 experiment the center and guard electrodes were sputter deposited platinum. In this study the center and guard electrodes were formed by first brazing a small pad of TiCuSil braze material onto the sample and then sputter depositing platinum over these small braze pads. It is possible that the addition of the braze material has lead to the non-ohmic behavior in this case. It is worth noting that another RIED study has also reported non-ohmic behavior of Wesgo AL995 following ion irradiation.[21] In that study the electrodes were formed by a thin (0.3 μm) sputter deposited layer of Ti followed by a 3 μm layer of Pt or Au.

Even with the non-ohmic behavior in this study it is apparent that there was good electrical contact to the sample surface, as demonstrated by the low-side response to applied voltage shown in Fig. 7. Therefore, it may be concluded that an applied field of at least 147 V/mm was applied to the Wesgo AL995 sample to a cumulative dose of approximately 1.8 dpa without catastrophic RIED occurring.

V. CONCLUSIONS

The results of this experiment demonstrate that no RIED occurred in a polycrystalline alumina (Wesgo AL995) sample irradiated with an applied dc potential of $\approx$150 V/mm to a damage dose of 1.8 dpa at a temperature of $\approx$440 °C. This temperature is well within the range of temperatures where it has been reported that RIED will occur. The fact that it was not seen in this case suggests that at least some grades of ceramic insulators may perform satisfactorily in ITER and other fusion reactors.

The termination used for the mineral insulated power lead cable worked well and allowed the power lead to operate for the 68 day reactor irradiation without failure. The fact that the separate coaxial cable sample which was also sourced with 110 V (corresponding electric fields of 120–600 V/mm) for the duration of the irradiation also did not experience a breakdown of its insulating properties is further evidence that this new method of cable termination helps in increasing the life expectancy of the power cables under irradiation.

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