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Dynamic Radiation Effects on Electrical Properties of Ceramic Insulators

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In-reactor measurements of the spontaneous and long-term change in electrical conductivity of α -alumina were carried out in the JMTR fission reactor. The so-called radiation-induced conductivity, RIC, was observed and evaluated qualitatively as well as quantitatively. A long-term increase in electrical conductivity was observed, which is thought to be due to the so-called radiation-induced electrical degradation, RIED.

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I. Introduction

Since the dawn of the irradiation study on materials, it has been well recognized that some radiation-related phenomena would be observable only during radiation. Short-life and transient radiation-induced defects will affect some of the material properties dynamically, especially when the properties are governed by the low-concentration carriers. The typical example is the electrical properties of highly-insulating ceramic materials. The recent development of a nuclear fusion reactor and the advance of the fundamental study on irradiation effects highlights the importance of *in-situ* observations of irradiation effects again.

It has been well known that ceramic insulators increase their electrical conductivity under exposure to ionizing irradiations⁽¹⁾. This phenomenon was named as the radiation induced conductivity, RIC, and has been studied theoretically and experimentally for these three decades⁽²⁾⁽³⁾. This reduction of electrical resistivity of ceramic insulators under ionizing irradiations would inevitably arouse serious technological problems in nuclear fusion reactors⁽⁴⁾⁻⁽⁶⁾. The ceramic insulators are anticipated to play far more important roles under radiation environments in the fusion reactors⁽⁴⁾⁻⁽⁷⁾.

Recently, elegant experimental results of RIC under the X-ray irradiation and accelerated particle irradiations have been accumulated. These results have revealed that RIC depended on the irradiation parameters such as a dose rate, a total dose and a irradiation temperature in a complicated way⁽³⁾⁽⁵⁾⁽⁸⁾⁻⁽¹⁰⁾. Also, the ratio of displacement damage rate vs electronic excitation rate (ionizing dose rate) is found to be an important factor⁽¹¹⁾. However, the total doses and their rates in these experiments were far

smaller than those expected in nuclear fusion reactors⁽⁴⁾. The effects of neutron irradiation have been scarcely reported⁽¹⁰⁾, because of many difficulties such as interferences by parasitic electrical currents caused by various reasons in performing the in-pile experiments⁽¹⁰⁾. When we think of the importance of the ratio of the dpa (displacement per atom) rate vs ionizing dose rate as well as the importance of the data in the higher ionizing dose ranges, we clearly realize the importance of the reactor irradiation with neutrons for the RIC study.

Recently, Hodgson⁽¹²⁾ demonstrated the phenomenon, the so-called radiation-induced electrical degradation (RIED) on α -alumina. This phenomenon means far more deleterious and permanent degradation of electrical-insulating ability of ceramic insulators than that caused by RIC. Hodgson claims that synergistic effects of ionizing irradiation and displacement damage under an electrical field would cause this phenomenon⁽¹²⁾. Concerning its mechanism, he suggested that some precipitates were formed by irradiation and the precipitates would increase the electrical conductivity substantially. He suggested that the precipitate would be aluminum colloids⁽⁵⁾⁽¹²⁾.

This implies that the co-existence of the displacement damage and the electric field would convert the electronic defects into the displacement damage. Namely, the so-called radiolysis might take place even in α -alumina. Up to now, it has been well established that the radiolysis, which is very, popular in the typical insulators such as alkali-halides, would not occur in α -alumina.

In electron irradiation, we can control the radiation variables and conditions very well. So, in general, electron irradiation is an appropriate tool for a fundamental study of the radiation effects. However, the electron irradiation sometimes causes unique phenomena which might not take place in other irradiation environments.

One example is colloid formation which might be the key issue for the RIED. Pells and Shikama⁽¹³⁾⁽¹⁴⁾ demonstrated the formation of aluminum colloids in α -alumina by 1 MeV electron irradiation in a high voltage electron microscope (HVEM). However, in subsequent irradiation studies with neutrons and ion beams, the aluminum colloids formation has not been observed. This suggests that the electron irradiation conditions, especially in a HVEM, would cause the colloid formation uniquely. Here, it would be pointed out that the electron irradiation gives a very high ratio of ionizing dose rate/dpa rate. So, it is strongly demanded to demonstrate RIED in other radiation sources, especially under neutron radiation.

Here, we tried to carry out in-pile measurements of electrical conductivity on α -alumina, which is the strongest candidate for electrical insulators in fusion reactors. We developed small subcapsules containing a specimen in a vacuum⁽¹⁵⁾. The specimen should be electrically insulated from the environments well enough in the capsule. The fission reactor used in the experiment is the Japan Materials Test Reactor (JMTR)⁽¹⁶⁾ of the Oarai Research Establishment of Japan Atomic Energy Research Institute.

II. Experimental Procedures

Figure 1(a) shows a schematic cross section of the first subcapsule design, the subcapsule I. The subcapsule was composed of highly pure and highly dense sintered alumina and 316-type stainless steel. Two electrical cables go through each end of the subcapsule. The electrical insulation between two electrode was planned to be guaranteed by the high resistant alumina cylinder and by the magnesia insulating electrical cable (MI cable) with a diameter of 1.5 mm. The alumina cylinder and the stainless endcaps were brazed to make vacuum tight. Before sealing, the subcapsule was evacuated to below 1×10^{-4} Pa at room temperature.

The electrical insulation between the two electrodes were measured to be greater than $1 \times 10^{14} \Omega$ at room temperature before irradiation. However, it was found that the brazing process short-circuited the alumina cylinder. So the observed insulation was thought to be maintained mainly by the magnesia insulating cable.

Figure 1(b) shows the cross sectional drawing of the second subcapsule design, the subcapsule II. In this subcapsule, we tried to improve the electrical insulation between the two electrodes. The brazing between the insulating

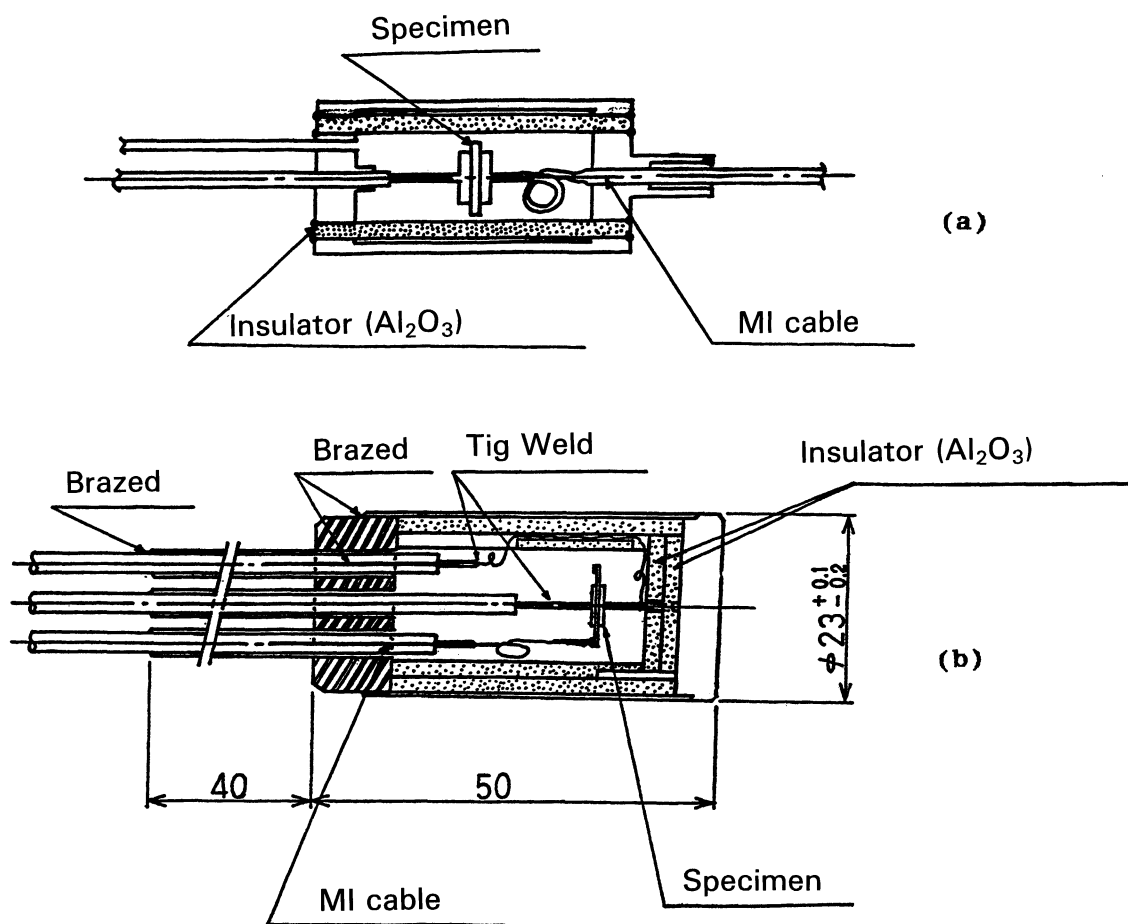


Fig. 1 Schematic cross sections of subcapsules: (a) the first design, (b) the second design.

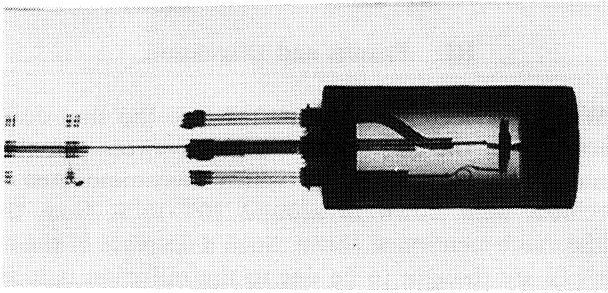


Fig. 2 X-ray transmission photograph of subcapsule II.

alumina components and the stainless-steel components was carefully carried out. As a result the insulation between the two electrodes were improved to greater than $1 \times 10^{15} \Omega$ at room temperature. However, we had many troubles with air leakage through the brazed sections. These troubles mainly resulted from the complicated configuration of the subcapsule as can be seen in Fig. 1 in comparison with the initial subcapsule design. The feed-through sections of the MI cables were the most troublesome. These problems were successfully overcome, and the success of the brazing is demonstrated in Fig. 2, where the completed subcapsule was examined by

the transmission X-ray photography. The gas leakage rate was determined to be less than $2 \times 10^{-10} \text{ atm cm}^3/\text{s}$ with a helium leak detector at room temperature.

The subcapsule and the attached MI cables were degassed at about 800 K, just above the planned irradiation temperature, for a few hours to obtain a vacuum better than $1 \times 10^{-4} \text{ Pa}$ and sealed. The subcapsules were installed in the instrumented irradiation rig as shown in Fig. 3. The MI cables for electrical measurements go up from the reactor core through the reactor pressure boundary to the measuring instruments directly. The length of the MI cable is more than 20 m⁽⁵⁾⁽¹⁵⁾.

The first experiment was carried out at 600–630 K, using the subcapsule I. The AC-voltage of 0.5 V was applied to a 99.9% pure single crystal. α -alumina, Kyocera SA 100, of 1 mm thick in the course of irradiation. The fluxes were 3.4×10^{17} and $1.8 \times 10^{18} \text{ n/m}^2\text{s}$ for fast ($E > 1.0 \text{ MeV}$) and thermal ($E < 0.6286 \text{ eV}$) neutrons, respectively. The ionizing dose rate was estimated to be $2.7 \times 10^3 \text{ Gy/s}$. The irradiation was carried out for 96 reactor full power days (rfpd) with three reactor-shutdown periods of about a few months and four short intermissions of a few days. The electrical resistivity was measured by the conventional AC (Alternating Current)-

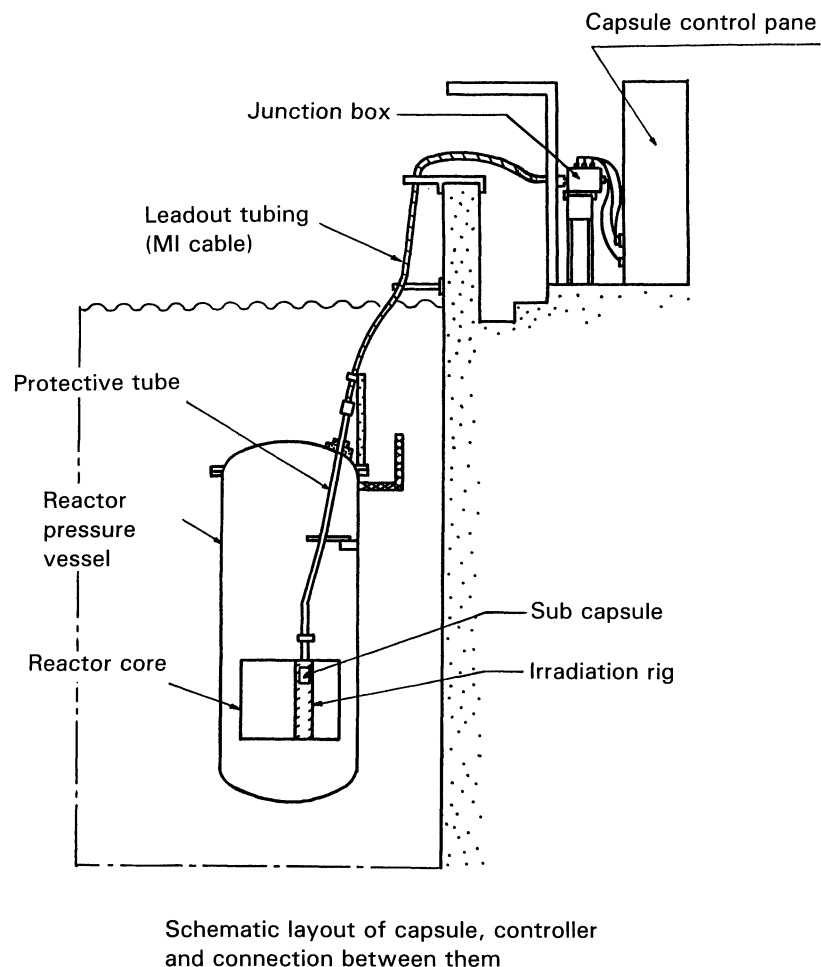


Fig. 3 Schematic of accommodation of subcapsules in the irradiation rig and their insertion into the JMTR reactor. Also, this shows the schematic experimental configuration.

Table 1 Irradiation conditions the first and second experiments.

| | The first | The second |
|--|--------------------|----------------|
| Period | | |
| Start | 19, March, 1990 | 8, March, 1991 |
| End | 17, December, 1990 | 4, June, 1991 |
| No. of Cycles | 4 | 2 |
| Temperature (K) | 600–630 | 770–800 |
| Neutron Flux (10^{17} n/m ² s) | | |
| Fast ($E > 1$ MeV) | 3.4 | 15.1 |
| Thermal ($E > 0.6826$ eV) | 18.0 | 23.9 |
| dpa rate (10^{-8} dpa/s) | 3.4 | 15.1 |
| γ -ray dose rate (10^3 Gy/s) | 2.8 | 5.3 |
| Specimens | Single Crystal | Polycrystals |
| Electric field | 500 V/m | 0 and 500 kV/m |

technique in the frequency range of 100 Hz–10 MHz. The instrument used was a HP model 4194A impedance analyzer. The DC resistivity at zero frequency, R_{DC} was estimated from the Cole-Cole plot and through the following relationships:

$$R_0 = Z / \cos \theta \quad (1)$$

$$X^2 + (R - 1/2R_{DC})^2 = R_{DC}^2, \quad (2)$$

where

R_0 = AC resistivity, namely, inverse of AC conductivity,

Z = absolute value of the measured complex impedance,

η = measured phase difference,

R = real part of the measured complex impedance,

X = imaginary part of the measured complex impedance.

R_{DC} = DC resistivity.

In the first experiment, we observed a long-term degradation of electrical resistivity of α -alumina, which was similar to the phenomenon reported as the radiation-induced electrical degradation, RIED, by Hodgson⁽¹²⁾. He irradiated α -alumina with electrons. So, in the second experiment, we tried to observe RIED in a fission-reactor irradiation conditions. The second experiment was carried out at a higher neutron flux and ionizing-dose-rate region in the reactor at the higher temperatures of 770–800 K. We used the subcapsule II. The irradiation duration was 48 reactor full power days. The details of the irradiation condition as well as those for the first experiment are tabulated in Table 1⁽¹⁶⁾. In the second experiment, two identical polycrystal α -alumina, Kyocera A 479 SS, were irradiated in the same irradiation rig at the same time. The specimen configuration was 8 mm in diameter and 0.5 mm thick. To one specimen, the DC-voltage of 250 V was applied during the irradiation, and no DC-voltage was applied to the other specimen. The DC-resistivity of the specimens was measured using the high resistant meter of HP 4329A in the voltage range of 10 V–1 kV and using a high DC-voltage supplier and a picoammeter. The AC-resistivity was measured also using the same method as in the first experiment.

III. Results and Discussion

When a subcapsule was inserted into the shut-down reactor core, the electrical resistivity between the electrodes of the subcapsule which accommodated a specimen went down to around 10^{10} – $10^9 \Omega$ from the initial value mentioned above. Such a decrease of the resistivity are thought to be due to the radiation induced conductivity, RIC, of the alumina specimen in the subcapsule. Here, we confirmed that the α -alumina specimen was electrically insulated from its surroundings with electrical resistance of more than one order higher than the resistance of the specimen, itself. The magnitude of the observed decrease of resistivity can be explained by RIC of α -alumina under a ionizing dose rate of around 10^6 R/h, which is the estimated dose rate in the shut-down reactor core.

Figure 4 shows a typical example of measured resistance when the reactor power goes up from 500 kW to 10 MW. Here, the resistance change was clearly observed, which followed details of the reactor power change. Figure 5 shows that RIC of the present α -alumina is about $3 \times 10^{-8} (\Omega m)^{-1}$ at the reactor-shut-down, namely, at the γ -ray dose rate of 0.5 – 1.5×10^1 Gy/s and is about 6 – $15 \times 10^{-6} (\Omega m)^{-1}$ at the reactor-full-power operation, where the ionizing dose rate was estimated to be 2.8×10^3 Gy/s. The RIC measured in the present experiment changed with the reactor power as shown in Fig. 5, where the measured electrical conductivity was logarithmically plotted as a function of the reactor-

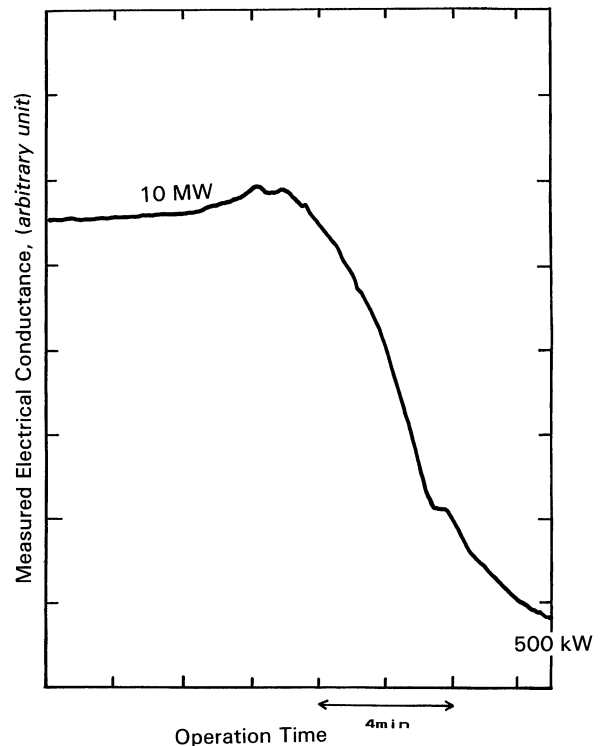


Fig. 4 Observed change of electrical conductance as the reactor power goes up.

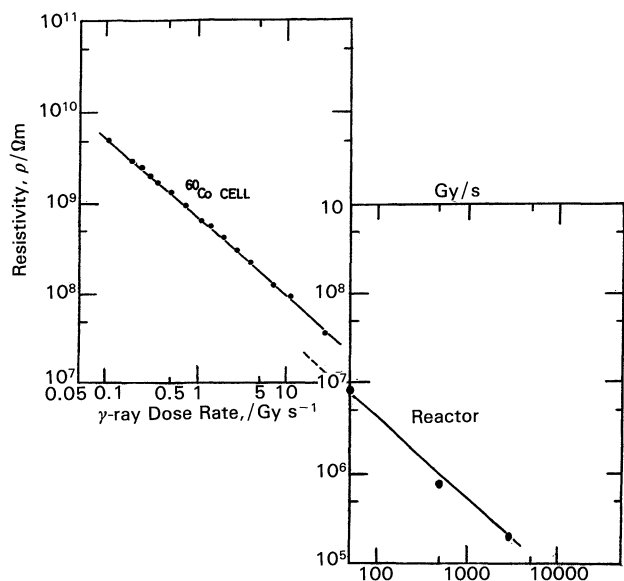


Fig. 5 Measured electrical resistivity of the α -alumina as a function of the ionizing dose rate of JMTR irradiation. Results are compared with data obtained under ^{60}Co γ -ray irradiation.

power, in comparison with data obtained under ^{60}Co γ -ray irradiation⁽¹⁷⁾. In the present experiments, RIC of α -alumina is described as below:

$$\text{RIC} = K \times P^{1.05-1.2}, \quad (3)$$

where

RIC = Radiation induced conductivity,
 K = Constant of proportionality,
 P = Reactor power.

The ionizing-dose-rate dependence of the observed RIC and also the magnitude of the RIC agreed relatively well with previously reported data⁽³⁾⁽⁸⁾ obtained at far lower dose rates with X- and γ -rays, and light ion irradiation. In our experiments, which covered the far more extended dose range which included the expected dose rate in a fusion environment, RIC did not saturate distinctly.

However, a detailed examination suggested that there was some trend for RIC to saturate at the highest dose rate region in the polycrystal α -alumina. This may suggest that the lifetime of excited electrons from the valence band may be affected by the density of holes in the valence in the higher dose rate region in materials containing high structural defects.

Figure 6 shows a long-term change of electrical conductivity of the single crystal α -alumina under full power irradiation in the first experiment. At the initial stage of the irradiation, the measured conductivity was nearly constant at $6 \times 10^{-6} (\Omega\text{m})^{-1}$. After about 10 reactor-full-power-day (rfpd) irradiation, the electrical conductivity began to increase. The increment, C, depends on the irradiation duration, t, as follows:

$$Ct^n \quad (\text{Here, } n=0.7). \quad (4)$$

This increase of the electrical conductivity under the irradiation resembled the phenomenon of RIED. In this ex-

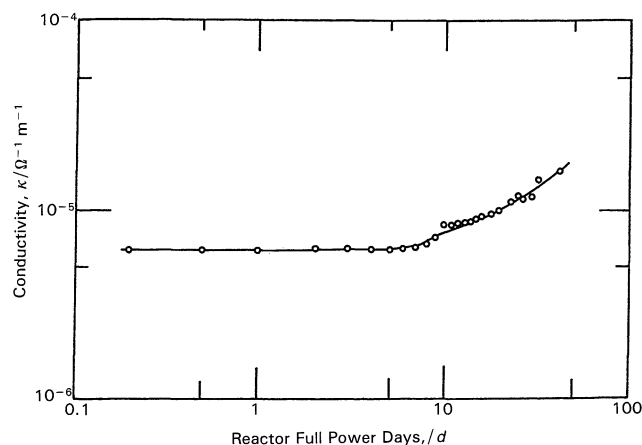


Fig. 6 Long-term change of the electrical conductivity of the α -alumina under the full-power reactor irradiation.

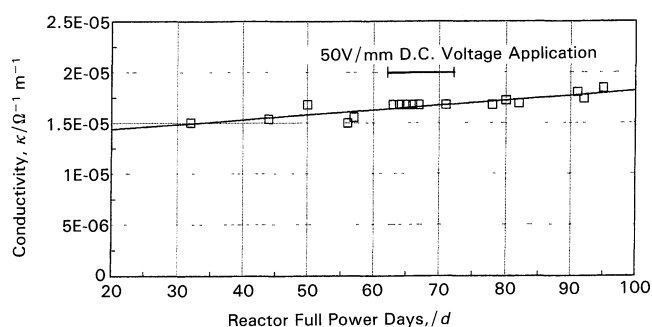


Fig. 7 Effect of high voltage application on the long-term degradation of electrical insulation during the first experiment.

periment, we measured the AC-resistivity continuously during the irradiation. As a result, 0.5 V AC-voltage was kept to be applied to the specimen. The magnitude of the electric-field was, however, quite low compared to that applied in the Hodgson's experiment⁽¹²⁾. So, we applied a DC-voltage of 100 V for 10 rfpd, after the specimen was irradiated for 50 rfpd. Figure 7 shows the effect of this voltage-application. We did not observe any enhancement of the conductivity increase by the high DC-voltage application.

The conductivity-increase in the first experiment has many dissimilarities to the phenomenon observed by Hodgson⁽¹²⁾. Hodgson reported that RIED took place at about 10^{-5} dpa and about 10^{5-6} Gy ionization dose⁽¹²⁾. In the present experiment, the conductivity-increase took place after about 10 rfpd. The dpa was evaluated to be about in the range of 10^{-2} and the ionization dose exceeded 10^9 Gy. Also, the time dependence of the conductivity-increase, namely, n in Relation (4), in the present experiment, was very small of about 0.5 compared with the values of 4–6 reported by Hodgson⁽¹²⁾.

These differences may be explained by the differences in the experimental conditions. Especially the temperature difference may play an important role. If the mechanism of RIED was due to the aluminum colloid formation in the alumina matrix, the temperature is a very important factor, as extensively studied in alkali-

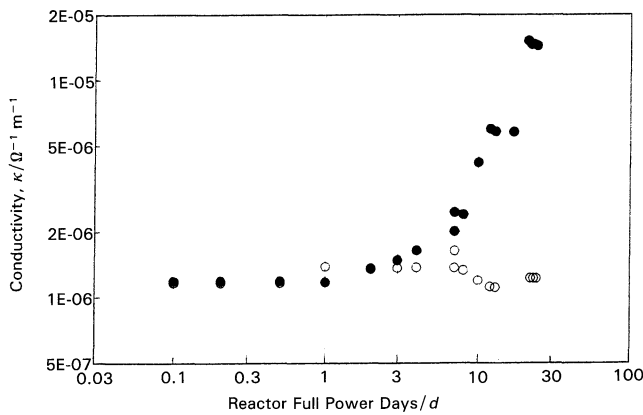


Fig. 8 Electrical conductivity of α -alumina under reactor irradiation, with 0 (●) and 500 kV/m (○) DC applied electric fields.

halides⁽¹⁸⁾⁽¹⁹⁾. Lower temperature irradiation means lower formation rates of colloids. Then, the low irradiation temperature in the present experiment would necessitate more displacement damage and more ionization dose. Also, the differences in the ionizing dose rate and in the dpa rate between two experiment would affect the behavior of RIED. The ratio of the rates of ionizing dose and of the displacement damage also would play a significant role as suggested by Zinkle *et al.*⁽¹¹⁾ Electron irradiation gives quite large ratios of ionization dose rate to displacement damage rate compared with fission reactor irradiation.

There are other possible causes for the electrical conductivity increase observed in the first experiment. As opposed to experiments with electron irradiation, the in-reactor measurements have many experimental difficulties. The hostile reactor irradiation environment causes parasitic electrical signals which could contribute to the observed long-term electrical conductivity increase. One example is the degradation of vacuum around the specimen. In the second experiment, the vacuum and electrical insulation of the specimen were improved. Two identical specimens were irradiated to compare especially the effect of voltage application. The irradiation temperature was selected to be higher, namely, 770–800 K.

Figure 8 shows the results obtained. The specimen with an applied DC voltage of 250 V (500 kV/m) showed a distinct increase in electrical conductivity. The increase was similar to that observed in the first experiment and was proportional to t^n as described in Relation (4). However, in this case n was estimated to be 0.75–0.8, larger than that of the first experiment. The increase began just after about one rfpd irradiation. In the meantime, the specimen, which had no voltage applied did not show any increase in conductivity during 48 rfpds. The conductivity may have even decreased a little in the late stage of irradiation as shown in Fig. 8.

The comparison between these results obtained with and without an applied voltage strongly suggests that the cause of the observed increase in electrical conductivity is intrinsic and is not caused by parasitic effects that would

occur in both cases. Also, the results indicate that the application of a voltage is essential for the increase of electrical conductivity as was claimed by Hodgson⁽¹²⁾.

In the second experiment, the increase of electrical conductivity took place after about 1 rfpd, which was about 5–7 times earlier than the first experiment. In the second experiment, the dpa rate is about 4–5 times larger and the ionizing dose rate is about 2–3 times larger than those of the first experiment. So, the increase took place at about the same dpa and about one-fifth to one-third to one-fifth of the ionization dose. Also, n in Relation (4) in the second experiment is larger than that in the first experiment.

Since the second irradiation was carried out at a higher temperature than the first, it would be expected that the increase would take place sooner and at a higher rate in the second experiment. If alumina is similar to alkali halides⁽¹⁸⁾⁽¹⁹⁾, aluminum colloid formation would take place in the temperature range of 700–1000 K. The irradiation temperature is well within this range in the second experiment. The higher dpa rate and the higher ionizing dose rate as well as the higher voltage-application would play a role in the earlier take-off and the higher rate of the increase in electrical conductivity in the second experiment. We should also point out that we used a single crystal sample in the first experiment but polycrystalline sample in the second experiment. As a whole, these results suggest that a long-term increase in electrical conductivity of α -alumina will take place in the fission reactor irradiation environment, if a voltage is present. The behavior of the observed increase in electrical conductivity is similar to the RIED reported by Hodgson⁽¹²⁾.

However, there are quantitative disagreements in the total doses at which the phenomenon took place and in the dependence of the conductivity-increase rate upon the irradiation period, t , namely n in Relation (4), among the experiments. Some of these disagreements could be explained by the differences in ionizing dose rates, dpa rates and the irradiation temperature. Also, the difference in damage cascade structure between electron and neutron irradiation might cause the disagreements.

However, we must report the important qualitative disagreement between the present results and those of Hodgson's⁽¹²⁾. Hodgson reported clearly that the RIED is the permanent effect. Even after the irradiation stops, the increase of the electrical conductivity due to RIED would stay. In the meantime, the present results indicated distinctly that, at least, substantial part of the increment in electrical conductivity due to RIED recovered after the reactor power went down. Also, Hodgson reported a superlinear dependence of the conductivity increase on irradiation time, namely a value of 4–5 for n in Relation (4) and claimed that this superlinear dependence is quite similar to that of colloid growth in the alkali halides⁽¹⁷⁾⁽¹⁸⁾. In the meantime, we observed a sublinear dependence, namely, a value of 0.5–0.8. This disagrees with the growth rate of colloids in alkali halides, but agrees with the 0.5–0.6 power dependence of colloid

growth observed in the HVEM irradiation on α -alumina⁽¹³⁾⁽¹⁴⁾. Recently, Chen *et al.* carried out the RIED study using electron beams⁽²⁰⁾. Their results showed some disagreements with those of Hodgson's. Their results might suggest that the cause of RIED might be the agglomerate of F^+ centers⁽²⁰⁾. Further and extensive study is strongly needed to clarify RIED.

IV. Conclusion

The electrical conductivity of highly electrically-insulating ceramics of α -alumina was measured *in-situ* in a fission reactor, JMTR. We observed radiation induced conductivity, RIC of alumina, which is quite similar to those observed in different irradiation environments. Namely, its electrical conductivity increases dynamically and linearly with the ionizing dose rate under the reactor irradiation. The magnitude of the increase also agreed well. However, some of the results suggested the trend of saturation of RIC at the highest dose rate in the present experiment, although the data were scarce and inconclusive as yet.

The long-term change of electrical conductivity of the α -alumina was studied under the irradiation in the fission reactor, JMTR. The obtained results indicated that the increase of the electrical conductivity would occur after a certain period of irradiation when the specimen was irradiated with the applied voltage. This phenomenon was thought to be the same as that was observed and reported as the radiation induced electrical degradation, RIED, by Hodgson, although we observed some qualitative disagreement.

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Here, one of the authors would like to emphasize the importance of the collaboration among people of a wide variety of research and technical fields, for the research studies like the present one. He is quite confident that many new-types of reactor experiments will be needed for the development of the fusion reactors. And, some new-types of reactor experiments are essential for the materials science to contribute to the fusion develop-

ments. Also, the new-types of reactor experiments yield a kind of strong interactive field among different research fields. Sometimes, this interactive field will open very fresh and growing new research field. However, these promising new-types of reactor experiments can be realized only through the close collaboration among the different research activities.

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