

# Electrical Degradation of Ceramic Insulators Due to Dynamic Irradiation Effects

著者	Shikama Tatsuo
journal or publication title	Materials Transactions, JIM
volume	36
number	8
page range	997-1003
year	1995
URL	<a href="http://hdl.handle.net/10097/52236">http://hdl.handle.net/10097/52236</a>

## OVERVIEW

# Electrical Degradation of Ceramic Insulators due to Dynamic Irradiation Effects<sup>†</sup>

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Degradation of electrical insulating ability of ceramic insulators is attracting keen attention especially in fusion reactor developments. A permanent increase of the electrical conductivity of the ceramic insulators has been observed recently in certain irradiation conditions, which is called the radiation induced electrical degradation (RIED). Its nature and mechanism are still controversial, but recent experimental results are confirmative to support the existence of the phenomenon causing the degradation of the bulk electrical conductivity of some ceramic insulators under certain irradiation conditions. The paper will review the recent experimental results and discuss the nature and the mechanism of the degradation.

(Received January 20, 1995)

*Keywords: ceramic insulators, radiation effects, electrical conductivity, radiation induced electrical degradation*

### I. Introduction

A terminology of 'Radiation Effects' is not clearly defined especially when it is translated into Japanese 'Syosha koka' or 'Houshasen koka'. It is mainly because the word is used interdisciplinarily in different meanings in different research fields. Also, it can be partly ascribed to an ambiguous definition of the word 'radiation' in Japanese.

Initially, the 'radiation' was used to describe unknown radiations from materials and from some physical processes, which had an ability to ionize surroundings. However, the 'radiation' has interactions not only with electrons but also with lattice atoms. As a general concept, the 'radiation' can be defined as energetic radiations having an activity of interacting energetically with electrons and nuclei in materials. The radiation effects, then, can be described as phenomena of property changes of materials by the irradiation; interactions with the radiation.

Up to now, researchers in radiation chemistry and radiation biology have long been focusing their interest on effects of the ionization or the electronic excitation (electronic effects). In the meantime, the interactions of radiation with lattice atoms have been much important in the nuclear materials science up to now. Especially a process of displacement of lattice atoms by the irradiation attracted considerable attention of researchers concerned with

metallic materials. This effect can be called displacive effects.

However, both the electronic and the displacive effects are important to understand the radiation effects in materials. Furthermore, recent studies are empathizing importance of interactions between the electronic and displacive effects<sup>(1)(2)</sup>. Interesting phenomena have been recognized especially in ceramics, which can happen through the interactions between the electronic and the displacive effects<sup>(1)</sup>.

Peculiar degradation of an electric insulating ability of ceramics has been observed by some researchers<sup>(2)-(6)</sup>. The phenomenon itself is still controversial and further experimental evidence will be needed to establish its reality as a physical phenomenon. Some researchers<sup>(7)</sup> demonstrate that the phenomenon is an artifact due to surface contamination. Some of the experimental results<sup>(8)</sup> can be explained by the surface contamination. However, it can be said that the results of experiments by different researchers<sup>(9)(10)</sup> show that there exist some phenomena causing an increase of bulk electrical conductivity.

In this paper, the present experimental results of this phenomenon will be reviewed in view of the interaction of the electronic and the displacive effects.

### II. Electrical Conductivity of Ceramic Insulators under Irradiation

The displacive effects are primarily important for changes of structural properties such as dimensions and mechanical properties. The radiation induced lattice defects will interact with other defects and resultantly

<sup>†</sup> A part of this paper was originally published in Japanese in *Materials Transactions, JIM*, 33 (1994), 1363.

radiation induced microstructures will be evolved. This evolution of microstructures are responsible for many radiation induced changes of properties. Here, the changes of properties depend mainly on an accumulated dose of the irradiation and can be evaluated after the irradiation has finished (PIEs: post irradiation examinations).

In the meantime, processes of the interactions of the radiations with materials themselves will change some properties of materials dynamically. It is emphasized that the dynamic changes of properties can be studied only by *in-situ* type experiments and never can be observed by the conventional PIEs<sup>(11)</sup>. Figure 1 demonstrates the dynamic change of optical transmissivity of fused silica ( $\text{SiO}_2$ ) under irradiation in a pulse-operated fission reactor, TRIGA of University of Illinois<sup>(12)</sup>. The optical transmissivity decreases substantially under the irradiation but it recovers promptly after the irradiation ceases. Each abrupt decrease and recover corresponds to each pulse operation of the TRIGA.

An electrical conductivity of insulators is another typical example. In ceramic insulators, density of electrons in a conduction band is very low at moderate temperatures. Once they are exposed to ionizing radiations, however, many electrons are excited from valence bands to a conduction band and they contribute to electrical conduction. Thus, the electrical conductivity of ceramic insulators would increase in many orders of magnitude under the exposure to the ionizing radiation, that is called a radiation induced conductivity (RIC). Figure 2 shows the RIC of alumina ( $\text{Al}_2\text{O}_3$ ), the strongest candidate for the ceramic insulators of nuclear systems, under X-ray irradiations<sup>(13)</sup>.

The RIC is independent of the accumulated dose of irradiation, and will be determined by the electron densities in valence bands, the electronic excitation rate, and the band gap energy of the material<sup>(14)(15)</sup>. However, changes of the RIC can be considered theoretically as ir-

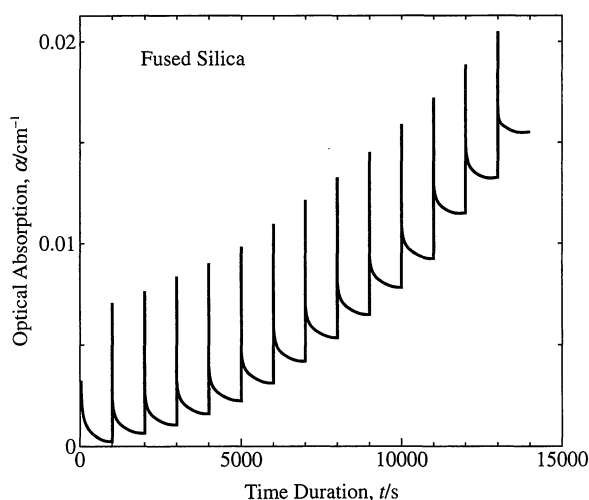


Fig. 1 Optical absorption of fused silica under irradiation in the pulse-operation TRIGA reactor<sup>(12)</sup>.

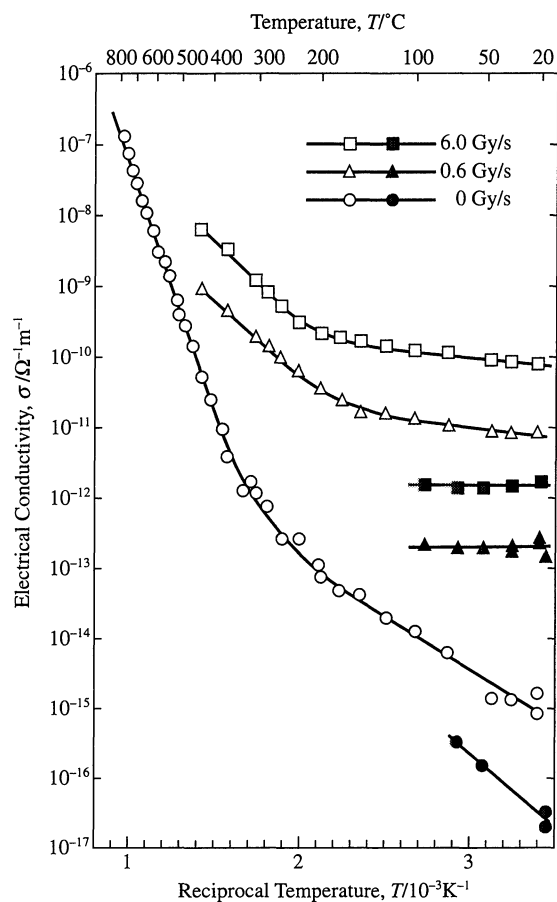


Fig. 2 Electrical conductivity of alumina under ionizing irradiation<sup>(13)</sup>. Open: without pre-irradiation, closed: after irradiation in a fission reactor.

radiation is continued, as the irradiation effects will modify donor-levels and acceptor-levels of electron bands as well as a mean life time and a mobility of excited electrons<sup>(16)</sup>. Experimentally, changes of the RIC have been observed<sup>(17)(18)</sup>. A typically gradual decrease of RIC with increasing accumulated dose is observed and is interpreted as a depletion of electrons in the donor levels or decrease of the mobility of the excited electrons. The decrease of the RIC of the alumina by accumulated neutron irradiation is shown in Fig. 2<sup>(13)</sup>.

The increase of the RIC has been also observed especially in materials having high impurity contents. Figure 3 shows the increase of the RIC of impure (95%) silicon nitride ( $\text{Si}_3\text{N}_4$ ) in an initial stage of irradiation in a fission reactor, JMTR (Japan Materials Testing Reactor in Oarai Research Establishment of Japan Atomic Energy Research Institute). The increase may be interpreted by the depletion of holes in the acceptor-level.

The RIC and its change in the course of irradiation, for example, the increase of RIC shown in Fig. 3 are temporary phenomena and once the irradiation is ceased, it will disappear and the electrical conductivity should return back to the initial value. However, Hodgson<sup>(3)</sup> observed peculiar behavior of the electrical conductivity of alumina under electron irradiations. The results are

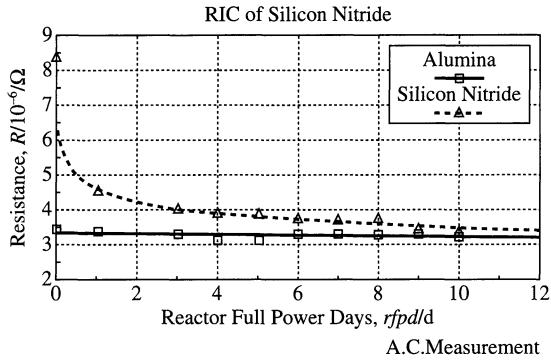


Fig. 3 Electrical resistance of silicon nitride under irradiation in JMTR, in comparison with that of alumina.

shown in Fig. 4. Drastic and permanent increase of the electrical conductivity was observed in addition to the RIC. The electrical conductivity,  $\sigma_t$ , increases with the duration time  $t$  of irradiation, in the following relationship, after a certain incubation period,  $i$ .

$$\sigma_t = \sigma_0 + \sigma_1(t - i)^n + \sigma_2 D^q. \quad (1)$$

Here,  $\sigma_1$  and  $\sigma_2$  are constants and  $D$  is the ionizing dose rate.  $n$  and  $q$  are constants which depend on experimental conditions. The first term,  $\sigma_0$ , is the electrical conductivity without irradiation, and the third term describes

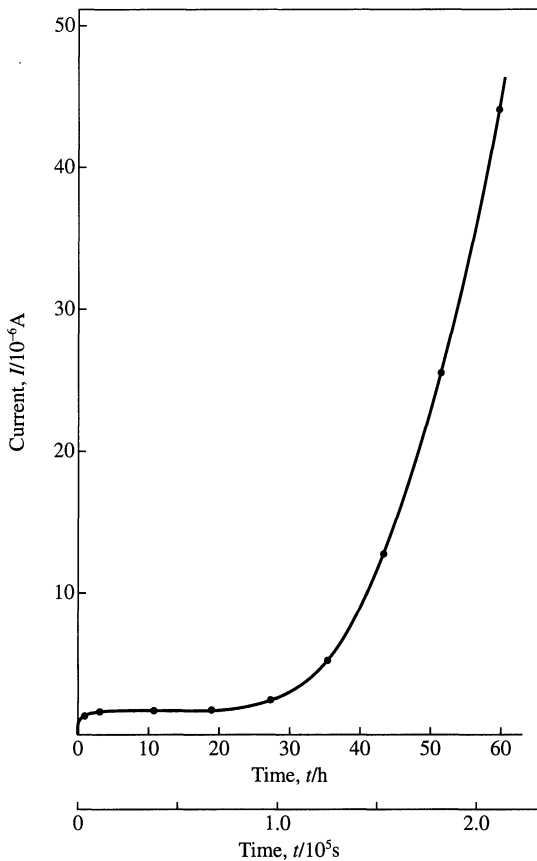


Fig. 4 Drastic and permanent increases in electrical conductivity of alumina under electron irradiation with external electric field<sup>(3)</sup>.

the RIC, which is temporarily observed only during irradiation. The second term describes the permanent increase of the electrical conductivity due to the irradiation. Hodgson named this increase the radiation induced electrical degradation (RIED).

Subsequently, the same kind of increase of the electrical conductivity in the long-period irradiation was observed in proton irradiation<sup>(4)</sup>, in fission reactor irradiation<sup>(5)(6)</sup>, and also in electron irradiation<sup>(19)</sup>. However, Keseternich *et al.*<sup>(7)(20)</sup> could not observe the RIED in well defined experimental conditions in proton irradiation. They carried out circuit analysis extensively and claimed that the observed increase of the electrical conductivity could be explained by the increase of a surface electrical conductivity caused by surface contamination in the course of long-period irradiation in not-well-controlled environments. Farnum *et al.*<sup>(8)</sup> could explain their observation of RIED-like behavior in a spallation neutron irradiation (LMPF/LASREF; Los Alamos Meson Production Facility/Los Alamos Spallation Neutron Radiation Effects Facility) by the increase of surface conductivity proposed by Kesternich *et al.*<sup>(7)(20)</sup>

Now, it is clear that detailed methods of measuring the electrical conductivity are key and crucial issues to demonstrate real nature of the RIED.

### III. Methods for Measuring Electrical Conductivity of Ceramic Insulators

Measurements of the very low electrical conductivity is not easy. The typical electrical conductivity of the ceramic insulators are in the range of  $10^{-10}$ – $10^{-17}$   $\text{Sm}^{-1}$  at room temperature without irradiation. Even under the ionizing irradiation of  $10^4$   $\text{Gys}^{-1}$ , the electrical conductivity will be in the range of  $10^{-4}$ – $10^{-6}$   $\text{Sm}^{-1}$ . Thus, we have to measure electrical currents in the range of pico to micron amperes even with a thin specimen of a large cross section. Also, we have to separate a bulk conductivity from a surface conductivity. The ceramic insulators tend to have a higher surface conductivity than a bulk conductivity. The standard method for measuring electrical conductivity of insulators are established for non-irradiation environments at a room temperature<sup>(21)</sup>.

Figure 5 shows the scheme of measuring the electrical conductivity of insulators<sup>(22)</sup>. Two electrodes and one

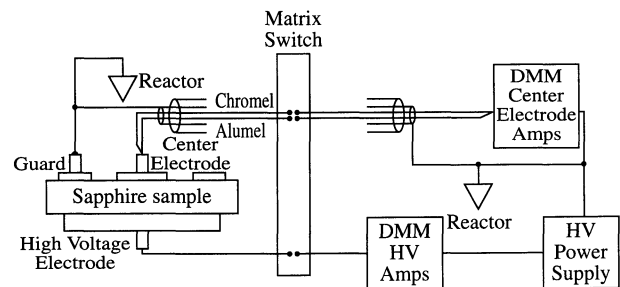


Fig. 5 Scheme for measurement of electrical conductivity of ceramic insulators<sup>(22)</sup>.

guard ring are attached to a specimen and a current from a high-voltage side electrode through the specimen to a low-voltage side one is measured by an ampere meter which is placed between the low-voltage side electrode and a common ground. The ampere meter should have a low internal impedance and an electrical resistance of wires between the low-voltage side electrode and the ampere meter should be as low as possible to make the electrical potential of the low-voltage side electrode as close as to the grounded potential. The surface leak current can be excluded by the guard ring. The electrical potential of the guard ring should be as close as to the grounded potential, too, being the same electrical potential as the low-voltage side electrode.

The above conditions can be attained in conventional measurements without radiation near room temperatures. Surroundings of a specimen can be controlled in principle to avoid unexpected increase of surface leak currents. However, several precautions should be made for the control of the surroundings under the irradiation, especially in a high-power-density fission reactor. After extensive discussions in successive international specialist meetings, the standard procedure of *in-situ* measurements of the electrical conductivity of ceramic insulators has been recommended in the IEA meeting<sup>(23)</sup> at Stresa. The points are:

(1) The temperature of a specimen should be measured directly. An unsheathed thermocouple is recommended to attach to the specimen directly.

(2) The specimen should be floated from the ground to make a current measurement at a low-voltage side.

(3) The guard ring configuration is essential for a reliable measurement.

(4) Degree of a surface contamination should be monitored through an experiment by measuring the electrical conductivity between the guard ring and the low-voltage side electrode.

(5) Electrical resistance of wires connecting a guard ring to the ground and a low-voltage side electrode to an ampere meter should be measured through the experiment.

(6) A double sheathed MI-cable (Triax cable) should be used for the electrical wire connecting the low-voltage side electrode to the ampere meter.

Figure 6 shows a specimen configuration developed for the *in-situ* measurement in the JMTR under the US/Japan collaboration. A prototype assembly was irradiated in JMTR and some data were obtained<sup>(24)(25)</sup>. An improved assembly of specimens will be irradiated in HFIR (High Flux Isotope Reactor in Oak Ridge National Laboratory in US) and in JMTR under the collaboration.

#### IV. Radiation Induced Electrical Degradation (RIED)

As described in the Section II, the phenomenon of the RIED is controversial. Zinkle and Hodgson<sup>(26)</sup> and Zinkle<sup>(27)</sup> summarized results of the RIED on alumina up

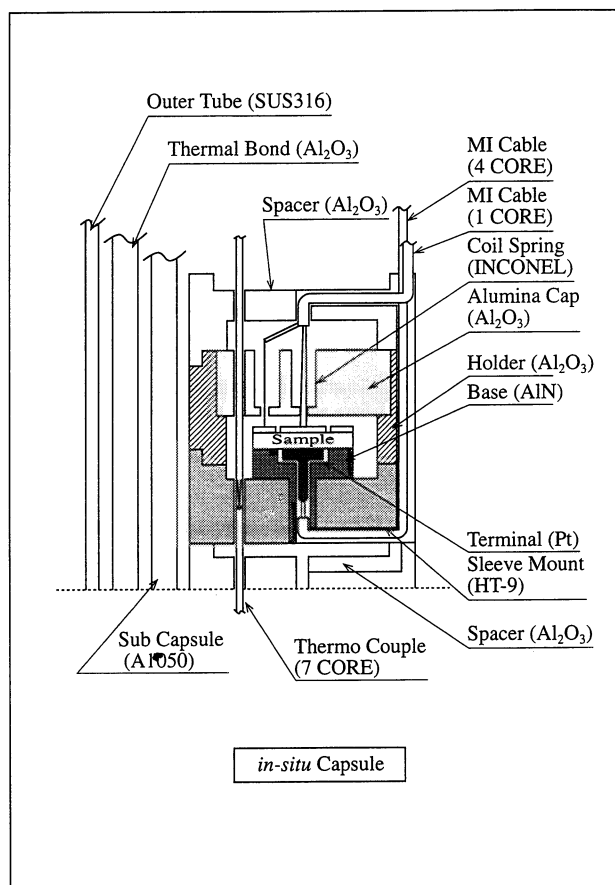


Fig. 6 Cross sectional view of specimen for RIED study in a fission reactor.

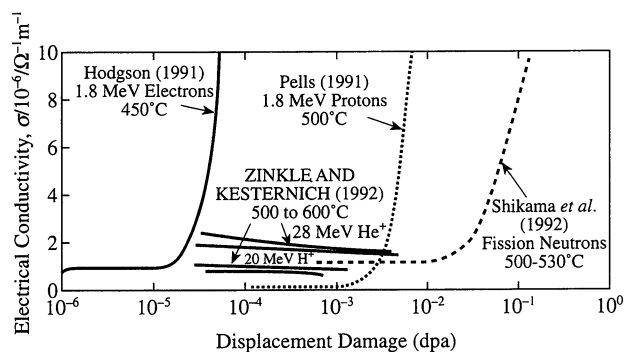


Fig. 7 Results on RIED data compiled by Zinkle<sup>(25)</sup>.

to 1992 as shown in Fig. 7. At that time, the most reliable data was that by Kesternich and Zinkle<sup>(7)</sup> in Fig. 7. Kesternich *et al.*<sup>(20)</sup> carried out further extensive experiments especially on 99.5% pure alumina, WESGO 995 in proton irradiations. They claimed that the RIED is an artifact due to the surface contamination during the experiments. Debates have been evoked but they were barren in general up to the IEA meeting<sup>(23)</sup> at Stresa, where a very constructive recommendation has been made for the experimental procedure as described in the previous section.

Results are being obtained from experiments which

satisfy the recommendations mentioned above. Chen *et al.*<sup>(19)</sup> have carried out such experiment under electron irradiations after the first international debates<sup>(28)</sup> concerning the RIED in Vail in 1991. They observed the RIED-like behavior, but they could not observe the same microstructural change observed by Hodgson<sup>(3)</sup>, namely the formation of some precipitates in the irradiated alumina matrix. Instead, they observed formation of dense dislocation networks under the irradiation conditions where the RIED-like phenomenon happened<sup>(19)</sup>.

Moslang *et al.*<sup>(9)</sup> carried out the same experiment satisfying the criteria mentioned above on WESGO 995 and could not observe the RIED. Snead *et al.*<sup>(29)</sup> did the experiment in a HFBR (High Flux Beam Reactor in Brookhaven National Laboratory) on WESGO 995 and could not observe the RIED, however, their irradiation temperature was very low for the RIED phenomenon.

Moslang *et al.*<sup>(9)</sup> carried out also the same experiment on Vitox alumina whose purity is 99.9% and observed the RIED-like phenomenon just the same as that reported by Hodgson<sup>(3)</sup>. Hunn *et al.*<sup>(10)</sup> carried out studies of the RIC of ceramic coatings under proton irradiations. They observed a permanent increase of the bulk electrical conductivity of the alumina, which is similar to the RIED.

So, the situation is still controversial but the recent experimental results are confirmative for the existence of the phenomenon of deleterious increase of the bulk electrical conductivity of some ceramic insulators under the irradiation in certain conditions. The conditions, where the RIED-like behavior would take place, (the RIED-conditions) can be summarized as follows:

- (1) Both the displacement damage and the electronic excitation are needed.
- (2) The application of electric field is needed.
- (3) The phenomenon takes place in a certain temperature range. In the case of electron irradiation, the temperature range was reported to be 420–900 K.
- (4) Some specimens show the RIED-like behavior but some do not. The results on the alumina suggest that impurities may have some crucial role in the RIED-like phenomenon.
- (5) The radiation induced conductivity (RIC) is thought to play an important role in the RIED.

Specimens which showed the RIED-like behavior have been found to become fragile and the PIEs of the specimens are difficult in general<sup>(2)</sup>. However, some PIEs showed far higher density of radiation induced defects in the specimens under the RIED-conditions, when compared with the density of relevant defects in the specimens which did not show the RIED-like behavior (It means that the specimens irradiated in the same conditions but without an electric field) at the same displacement dose.

The accumulated results suggest that the RIED-like behavior takes place earlier under the irradiation having lower displacement dose rate, when results are compared as a function of the displacement dose. Hodgson<sup>(30)</sup> analyzed the results and claimed that the take-off duration of the RIED will be inversely proportional to the square root of

the displacement dose rate. However, these results may be interpreted that the electronic excitation is playing an important role in the RIED. The data may be analyzed better as a function of the electronic excitation dose.

They may imply that some electronic defects have been converted into lattice defects (radiolysis) with a help of an applied electric field. The radiolysis is the phenomenon well studied in alkali halides<sup>(31)(32)</sup>, where ionizing irradiation having no ability of displacing atoms can produce lattice defects such as metal colloids, halogen bubbles and dislocations. It is proposed that the electronic defects at anion sites can be converted into Frenkel-type defects pair (vacancy and interstitial). The same phenomenon is thought to happen in some ceramic insulators such as silica (SiO<sub>2</sub>)<sup>(1)</sup>, but is thought not to happen in the alumina in usual irradiation conditions.

Recent studies are revealing that there are strong interactions between the electronic excitation and the radiation induced lattice defects even in metals which have strong electron-phonon interactions<sup>(33)</sup>. The electronic excitation can anneal the lattice defects dynamically and sometimes it will be converted into lattice defects even in metals at low temperatures. Up to now, quantitative comparison of damage efficiency in the ceramic insulators have not been done in different irradiation sources having different ratios of the electronic excitation rates and the displacement rates. Zinkle<sup>(34)(35)</sup> recently showed that the stability of radiation induced lattice defects is strongly dependent on the ratio of the electronic excitation rate to the displacement rate.

Morono and Hodgson<sup>(36)</sup> observed that the density of the  $F^+$  center increased in the RIED-conditions. So, it is plausible that the radiolysis may take place in alumina even in usual irradiation conditions. The experimental results on the RIED will indicate that the application of the electric field will enhance the radiolysis drastically.

The external electric field is very small compared with the internal electric field in ceramic insulators and some mechanism is needed to amplify the external electric field locally in the lattice. Here, the RIC is thought to play a role. As described in the Section II, the electrical conductivity increased substantially under the ionizing irradiation. Thus, under the strong ionizing irradiation, the ceramic insulators become a good electrical conductor. If there are some electrical barriers in ceramic insulators, the electrical charge-up will take place and the strong electric field will be generated there.

Stoneham<sup>(37)</sup> and Zinkle<sup>(38)</sup> suggested independently that the electrical charge-up (electrets) will take place and mechanical separation will result at some boundaries. They suggested that this mechanical breakdown will be responsible for the RIED. The electrets mechanism will explain the result that the RIED could be observed most apparently under the electron irradiation which has the highest potential for causing the electrical charge-up. Also, even a small amount of impurities will affect the RIED, when they are segregated at grain boundaries in this mechanism.

In the meantime, Hodgson suggested that the metallic

colloid formation will be responsible for the RIED<sup>(3)(30)</sup>. However, up to now aluminum colloids were not detected in the specimen which showed the RIED. Pells reported that the gamma alumina precipitated in the alumina in the RIED-conditions<sup>(2)</sup>. The formation of the aluminum colloids was demonstrated in electron irradiations by Pells and Shikama<sup>(39)</sup>. Hasegawa *et al.*<sup>(40)</sup> reported that the metallic aluminum precipitated on surfaces of voids formed in neutron irradiated sapphire. In the meantime, Chen *et al.* ascribed the RIED to the far denser dislocation networks formed under the RIED conditions as described above<sup>(19)</sup>.

At present, the conclusive mechanisms for the RIED can not be established. Furthermore, the phenomenon of the RIED itself can not be well understood. It is plausible that there are multiple mechanisms which cause the RIED-like behavior. In our experiments in the JMTR, we observed the RIED-like behavior in the alumina and magnesia, but major part of the observed increase of the electrical conductivity disappeared when the irradiation stopped and it reappeared when the irradiation restarted<sup>(1)(22)(24)(25)(41)(42)</sup>. The observed behavior can be explained by the increase of ionic conduction under the irradiation. Ivanov *et al.*<sup>(5)</sup> observed a similar phenomenon in alumina and silica base ceramic composites in fission reactor irradiations. They proposed the increase of the ionic conduction as a responsible mechanism<sup>(5)</sup>. However, experiments in fission reactors up to now could reveal only the initial stage of the degradation. If the degradation was caused by the increase of ionic conduction, further application of the electric field will cause an electrolysis which will result in the similar end products observed by Hodgson<sup>(3)</sup> and others<sup>(4)(9)(10)</sup>.

Recent results by Moslang *et al.*<sup>(9)</sup> are strongly suggesting that some uncontrollable properties of alumina is a determinant factor for the occurrence of the RIED. At present, the impurities will be the most probable player but other possibility such as grain sizes, preferred orientation may be also important. Further and detailed experiments are needed and the detailed PIEs are strongly anticipated.

The RIED-like phenomenon as well as the RIC is important for applications of the ceramic insulators to irradiation environments such as fusion reactors, fission reactors, and space facilities. International collaborative efforts are demanded to study the RIED and several scientific meetings have been held as described above. Under the recommendations from these meetings, the collaborative experiments will be carried out in the world largest experimental fission reactor, HFIR under the Japan/US collaboration on fusion reactor materials (so-called JUPITER (Japan Us Program on Irradiation TESts for fusion Research) project) from 1995. It is expected that the conclusive results can be obtained from the experiments and that the experiments will yield the quantitative as well as qualitative data suitable for the design of fusion reactors in near futures.

## V. Summary

Recent experimental results are confirming a phenomenon which causes deleterious degradation of the electrical insulating ability of ceramics under irradiations in certain conditions. The phenomenon named the radiation induced electrical degradation (RIED) will evoke serious technological problems for applications of ceramic insulators in irradiation environments, which are inevitable for fusion reactors, advanced fission reactor systems, and space facilities. The experimental results indicate that the RIED will take place at the displacement damage level from down to far less than  $10^{-3}$  dpa (displacement per atom) in highly ionizing irradiation<sup>(30)</sup>. In the case of fission reactor irradiations, which will be the most relevant irradiation environments for actual irradiation environments, the RIED would start at about  $10^{-1}$ – $10^{-2}$  dpa<sup>(3)-(6)(9)(10)</sup>.

The nature and the mechanism of the RIED are still controversial, and further and extensive experiments are needed especially in a high-power-density reactor. However, it is currently considered that the electronic effects as well as the displacive effects and their mutual interactions play important roles in the RIED with the assistance of the external electric field.

It should be mentioned that there is some possibility that multiple mechanisms exist which will cause the electrical degradations. In the fusion reactor irradiation environments, the solid transmutants such as sodium in alumina and magnesia will cause the electrical degradation<sup>(6)</sup>. Also, the hydrogen-isotope environments are thought to play deleterious roles. Thorough the understanding of deleterious mechanisms and their countermeasures, developments of ceramic insulators highly resistant to the degradations are essential for the successful developments of the fusion reactors and other concerned systems.

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