

Journal of Nuclear Materials 233-237 (1996) 1289-1293



Section 16. Dielectrics, insulators, windows, optics

## Electrical resistivity of ceramic insulators under irradiation using 14 MeV neutrons

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#### Abstract

Ceramic insulator materials used for various components such as diagnostic systems, in-vessel components, RF windows, etc. in fusion reactors are exposed to severe irradiation environments which are characterized by high energy neutrons with energies up to 14 MeV. The electrical resistivity of ceramic materials decreases due to radiation-induced conductivity (RIC) during irradiation. The RIC due to 14 MeV neutrons for  $Al_2O_3$  in the temperature range 300–570 K was measured in the neutron dose rate range  $10^{-2}$  to  $10^0$  Gy s<sup>-1</sup> using the fusion neutronics source (FNS) at the Japan Atomic Energy Research Institute. The RIC of  $Al_2O_3$  was estimated for the more severe irradiation environment in ITER and a prototype fusion reactor (SSTR) by extrapolating the data due to 14 MeV neutrons in the present study and those due to gamma-ray in another study. The estimated electrical degradation due to the RIC is considered to be accommodated with appropriate fusion reactor designs.

#### 1. Introduction

Many ceramic materials will be used as electrical insulator materials for various diagnostics components, the first wall current break, reduction of electromagnetic force to various in-vessel components, the RF windows, etc. in D-T fusion reactors. During operation of the fusion reactors, radiation-induced conductivity (RIC) takes place in the ceramic insulators by exciting electrons in the valence band to the conduction band with high energy neutrons and gamma-ray. An energy released for electronic excitation in the materials by a 14 MeV neutron is about ten times as large as that by a fission neutron of which energies are lower than several MeV. Thus, evaluation of degradation of electrical resistivity due to the RIC induced by 14 MeV neutrons is one of the research and development issues for various ceramic insulator materials which are expected to be used in the fusion reactors.

The RIC due to X-ray, gamma-ray, electron, proton, and fission neutron irradiation has been measured for ceramic insulator materials such as  $Al_2O_3$ , MgO, etc. [1–6]. However, the data of RIC due to 14 MeV neutrons

are very limited. Only one data set is preliminary data of RIC at 300 K for  $Al_2O_3$ , which was measured using the fusion neutronics source (FNS) at the Japan Atomic Energy Research Institute (JAERI) by our group [7].

In this study, data of electrical resistivity measured for  $Al_2O_3$  in the temperature range 300-570 K during 14 MeV neutron irradiation using FNS are presented as a function of the neutron dose rate.

#### 2. Experimental

### 2.1. Specimens

Specimens used are high purity  $Al_2O_3$  single crystal disks of about 10 mm in diameter and about 0.3 mm in thickness which were obtained by cutting the single crystal purchased from Rare Metallic Co. Ltd. Impurities in the specimens were as follows: Mg = 22 ppm, Ca = 3 ppm, Ti = 54 ppm, Mn = 10 ppm, Fe = 20 ppm, W < 1 ppm, C < 1 ppm, Ni < 1 ppm, V < 1 ppm, Mo < 1 ppm, Zn < 1 ppm. An electrode and a guard ring to avoid influence of surface leak current on the resistivity measurements were made on one side of the specimens by painting platinum paste and heating at about 1270 K for several hours. Then,

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another electrode on the opposite side of the specimen was made by gluing to a platinum plate with the platinum paste and heating in the above-mentioned procedure.

# 2.2. Electrical resistivity measurements under 14 MeV neutron irradiation

FNS is a pure 14 MeV neutron source which generates D-T fusion neutrons by injecting 330 kV deuterons into tritium targets. The characteristics of the neutron irradiation field of the FNS have been described elsewhere [7].

An irradiation chamber for in-situ electrical resistivity measurements at various temperatures was developed on the basis of the irradiation chamber by which the electrical resistivity of  $Al_2O_3$  under 14 MeV neutron irradiation at 300 K was measured [7]. A schematic illustration of the chamber is shown together with electrical diagram of the electric resistivity measurements in Fig. 1. The features of the chamber are as follows:

(1) A specimen is set to the target of FNS as closely as possible in the irradiation field with a large flux gradient, while insulators  $(Al_2O_3)$  between electrodes are located at a distance of about 30 cm from the specimen. Such a configuration results in the RIC due to the neutrons for the insulators which is smaller than that for the specimens by about two orders of magnitude. This enables proper measurements of the resistivity of specimens under irradiation.

(2) High vacuum can be attained using a turbo molecular pump system so as to avoid influence of leak current due to the ionization of surrounding gas which leads to overestimation of the RIC of the specimen.

(3) Temperature of the specimen can be varied in the range 300-870 K by a heater attached.

The resistivity measurement was carried out with a three-terminal DC method with the guard ring using a HP4339A high resistance meter. In the measurement system, only the current of the circuit through the platinum plate-the bulk specimen-one electrode was measured. The in-situ measurement of electrical resistivity of the  $Al_2O_3$  specimen was carried out in the temperature range 300–570 K at various 14 MeV neutron flux levels up to  $10^{15}$  n m<sup>-2</sup> s<sup>-1</sup>. Neutron flux during experiments was measured by a <sup>232</sup> Th fission counter.

#### 3. Results and discussion

14 MeV neutron irradiation experiments for each irradiation period of a few minutes were carried out at 300, 490 and 570 K in the neutron flux range  $10^{12}-10^{15}$  n m<sup>-2</sup> s<sup>-1</sup> (corresponding dose rates:  $10^{-3}-10^{0}$  Gy s<sup>-1</sup>) for Al<sub>2</sub>O<sub>3</sub>. The resistivity of an Al<sub>2</sub>O<sub>3</sub> single crystal specimen was measured before, during and after each irradiation. Fig. 2 shows a typical example of electrical resistivity behavior of the specimen in the in-situ measurements at 300 K. The resistivity decreased at once just after the start of the neutron irradiation and a constant value of the resistivity was immediately attained. The constant resistivity was kept with a constant neutron flux. When the neutron irradiation was stopped, the resistivity was immediately increased into the level which was slightly smaller than that before irradiation. And then, the resistivity increased



Fig. 1. Schematic illustrations of irradiation chamber for in-situ measurements of electrical resistivity of ceramic insulator materials and electric diagram of the measurement system.



Fig. 2. Typical electrical resistivity behavior of  $Al_2O_3$  specimen at 300 K during in-situ measurements.

gradually to the level before the irradiation. Such gradual increase of the resistivity can be attributed to RIC due to gamma-ray generated from the activated rotating target of FNS [7]. The gamma-ray from the target area activated during neutron generation decayed gradually after the neutron irradiation stop, resulting in the gradual decrease of RIC due to the gamma-ray, i.e. the gradual increase of the resistivity. Similar resistivity behavior was observed for 490 and 570 K.

Fig. 3 shows the electrical conductivity of the  $Al_2O_3$  single crystal specimen under irradiation at 300, 490 and 570 K versus 14 MeV neutron dose rate in a log-log plot. The conductivity of ceramic insulator materials under irradiation,  $\sigma$ , can be expressed by

$$\sigma = \sigma_0 + KR^d, \tag{1}$$

where  $\sigma_0$  is the conductivity in the absence of irradiation and  $KR^d$  is the contribution due to RIC. *R* is the ionization dose rate, and *K* and *d* are constants strongly depending on materials (*d* is generally 0.5–1.0) [1].

Several recent studies indicate that prominent permanent increases in the electrical conductivity of ceramic insulators, i.e. radiation-induced electrical degradation (RIED), may occur during irradiation in the temperature range 470–870 K even for small damage level of  $10^{-5}$ –0.1 dpa, if an electrical field is applied during the irradiation [1,2,8–10]. In the present study, RIED, i.e. a change of  $\sigma_0$ , was not virtually found, because of the negligibly small damage levels due to the very low neutron flux level and the short irradiation periods.

The dose rate exponent factors, d, for Al<sub>2</sub>O<sub>3</sub> under 14 MeV neutron irradiation in the neutron flux range  $10^{12}$ – $10^{15}$  n m<sup>-2</sup> s<sup>-1</sup> (i.e.  $10^{-3}$ – $10^{0}$  Gy s<sup>-1</sup>) were evaluated to be 0.96, 0.90 and 0.91 at 300, 490 and 570 K, respectively. The values of d were almost the same as the dose rate exponent for 0.004 wt% Cr<sub>2</sub>O<sub>3</sub> doped Linde Al<sub>2</sub>O<sub>3</sub> in the temperature range 300–570 K under electron irradiation

(d = 0.95) [3], in which impurity levels were considered to be similar to those in this study.

RIC of Al<sub>2</sub>O<sub>3</sub> has been measured by in-situ measurements using X-ray, gamma-ray, electron, proton and fission neutron irradiation [1-6]. Fig. 4 shows a comparison of the relationships between the electrical conductivity of Al<sub>2</sub>O<sub>3</sub> under irradiation in the range 300-570 K and ionization dose rates in the present study with those in the similar temperature range in some of the other studies mentioned above. The values which are obtained by extrapolating the present results in the range 300-570 K to higher dose rate region are in good agreement with those of 0.004 wt% Cr<sub>2</sub>O<sub>3</sub> doped Linde Al<sub>2</sub>O<sub>3</sub> single crystal specimens irradiated with electrons and the Vitox Al<sub>2</sub>O<sub>3</sub> polycrystal specimen irradiated with protons [3,4]. On the other hand, the extrapolated values are smaller than those of undoped Linde single crystal specimens, and larger than 0.03 wt% Cr<sub>2</sub>O<sub>3</sub> doped Linde single crystal specimens [3]. The RIC at dose rates around  $10^{\circ}$  Gy s<sup>-1</sup> in the present study are larger than that in the Vitox Al<sub>2</sub>O<sub>3</sub> polycrystal specimens irradiated at 300 K with X-ray [4].

RIC depends on impurity levels and radiation damage levels; RIC decreases with concentration of impurities and radiation damage in general [1,2]. The impurity levels of 0.004 wt%  $Cr_2O_3$  doped Linde  $Al_2O_3$  single crystal specimens are considered to be similar to those in the present study. The impurity levels of the specimens in the present study is higher than those of the undoped Linde single crystals, while lower than those in 0.03 wt%  $Cr_2O_3$  doped Linde single crystals. In the temperature range 300–570 K, and when RIED does not occur, the electrical conductivity of  $Al_2O_3$  under irradiation is predominantly contributed by RIC, except at very low dose rate levels. From this standpoint, the experimental results on RIC in the present study in which the damage levels are negligibly small are considered to be quite reasonable.



Fig. 3. Electrical conductivity of  $Al_2O_3$  specimen under irradiation at 300, 490 and 570 K as a function of 14 MeV neutron dose rate.



Fig. 4. Comparison of relationship between electrical conductivity of  $Al_2O_3$  single crystals under 14 MeV neutron irradiation and ionization dose rate in the present study with those for various  $Al_2O_3$  specimens under X-ray, gamma-ray, electron, proton, fission neutron irradiation in other studies.

A typical dose rate due to neutrons and gamma-ray for ceramic insulator materials at the first wall in ITER is expected to be about  $2 \times 10^3$  Gy s<sup>-1</sup> [11]. Operation temperatures for most of the ceramic insulators are supposed to be 570 K or below in ITER, although the temperature also depends strongly on the design. The RIC of Al<sub>2</sub>O<sub>3</sub> at the first wall of ITER, where the irradiation condition was more severe than the other regions, was evaluated by extrapolating data of RIC due to 14 MeV neutrons at 570 K in the present study and of RIC due to gamma-ray (Kyocera SA 100) in the previous work [6]. The values of RIC extrapolated to the dose rate levels of  $10^{3}$ -10<sup>4</sup> Gy s<sup>-1</sup> using the above-mentioned two data sets are almost identical, and the RIC at the first wall of ITER (dose rate level:  $2 \times 10^3$  Gy s<sup>-1</sup>) is evaluated to be about  $8 \times 10^{-7} \ \Omega^{-1} \ m^{-1}$  if such extrapolation is appropriate for the evaluation. The neutron irradiation produces irradiation damage which decreases the RIC in general [1], and therefore, the RIC at high fluence levels may be smaller than  $8 \times 10^{-7} \ \Omega^{-1} \ m^{-1}$ .

For prototype or demonstration fusion reactors, the irradiation environments will be more severe than that of ITER. The highest dose rate of about 10<sup>4</sup> Gy s<sup>-1</sup> (total of dose rates due to neutrons and gamma-ray) will be attained for Al<sub>2</sub>O<sub>3</sub> at the first wall in SSTR which was designed as a prototype fusion reactor at JAERI [12]. The RIC level at the region near the first wall in the SSTR is evaluated to be about  $4 \times 10^{-6} \ \Omega^{-1} \ m^{-1}$  in the same manner as the evaluation for ITER.

For most ceramic insulator applications, the electrical conductivity during irradiation should be less than  $10^{-4}$   $\Omega^{-1}$  m<sup>-1</sup> in order to prevent dielectric breakdown due to excessive Joule heating. However, even for low power

applications typical for many diagnostic components, it is desirable to keep the conductivity  $\sigma < 10^{-4} \ \Omega^{-1} \ m^{-1}$  to prevent instrument decalibration due to spurious currents [1]. From this standpont, the electrical conductivity in Al<sub>2</sub>O<sub>3</sub> under operation condition of the fusion reactors is not considered to provide any significant problems for ceramic insulator components, if only RIC is taken into account for electrical degradation in the irradiation field. It is, however, possible that very low conductivity levels ( $\sigma < 10^{-6} \ \Omega^{-1} \ m^{-1}$ ) will be needed for the better accuracy of measurements in some diagnostic components. In such cases, the RIC level can be decreased to the desired level by moving the position of the diagnostic components from the region near the first wall to the outer region of the blanket.

RIED was observed to occur during irradiation with an electrical field in the temperature range 470–870 K even for small damage level of  $10^{-5}$ –0.1 dpa in several studies [1,2,8–10], while RIED as a bulk effect not in some other studies [2,11,13]. The observed RIED phenomena were very complicated and depended on the applied electric field, temperatures, dose rates, fluence levels, impurity levels, etc. [2]. The RIED observed in some studies exceeded the conductivity of  $10^{-4} \Omega^{-1} m^{-1}$ , and therefore RIED could severely limit the usefulness of ceramic insulators in fusion reactor environments, when it occurs [2]. Further extensive studies are required to clarify the RIED. In-situ experiments to investigate RIED due to 14 MeV neutrons will be carried out in near future using FNS.

In the present study, the RIC due to 14 MeV neutrons for  $Al_2O_3$  single crystals was evaluated in the range 300–570 K. Various ceramic insulators other than  $Al_2O_3$ will be used in fusion reactors, and use of ceramic insulators at the higher temperatures is also expected. Thus, further studies to evaluate RIC due to 14 MeV neutrons are required to obtain material data of various ceramic materials in the wider temperature range for fusion reactor designs.

#### Acknowledgements

The authors wish to express their thanks to Drs. S. Funahashi, H. Katsuta, H. Maekawa for their interest in this work. The authors are also indebted to the FNS operation group.

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