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Radiation-induced changes in the physical properties of ceramic materials

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The results from recent studies on radiation-induced changes in the electrical conductivity, loss tangent, and thermal conductivity of ceramics are reviewed, with particular emphasis on in-situ data obtained during irradiation. The in-situ measurements have found that the physical property degradation during irradiation is generally much more severe than indicated by post-irradiation measurements. In particular, permanent degradation in the electrical resistivity may occur after irradiation to damage levels of $< 10^{-3}$ displacements per atom (dpa) for temperatures near 450°C. This radiation-induced electrical degradation (RIED) is observed only if an electric field is applied during the irradiation.

1. Introduction

It has become increasingly apparent in recent years that radiation-induced changes in the physical properties of ceramics will have a significant impact on the design of fusion reactors. Although ceramic components constitute only a small volume fraction of a fusion reactor plant (< 1%), virtually every aspect of the heating, control, and diagnostic measurement of the fusion plasma is dependent on the satisfactory performance of a ceramic material. The physical properties of importance for fusion ceramics include thermal conductivity, electrical conductivity and dielectric breakdown strength under dc or ac conditions, and the loss tangent at high frequencies. Table 1 summarizes the anticipated operating environments for insulating ceramics in a fusion reactor. The operating conditions span a wide range of flux, fluence, and electric fields. Of particular importance is the use of ceramics in various diagnostic components, where there is concern about possible decalibration of instruments due to gradual degradation in the electrical resistivity.

The topic of radiation effects in ceramics has been the subject of several previous reviews [1-11], with recent emphasis given to fusion reactor applications [3-10]. Most of the studies on ceramics have concentrated on post-irradiation analyses of the microstructure, density, and strength changes, in a manner analagous to radiation effects studies on metals. It has generally been observed that irradiation causes a modest degradation in the structural and physical properties, although spectacular reductions were occaisonally observed. In some cases, improvements in properties (e.g. higher electrical resistivity) were reported.

Table 1

Typical operating conditions for ceramics in the proposed ITER fusion reactor. Operating temperatures in a commercial fusion reactor will generally be higher

Component	Electric field [V/mm]	Irradiation flux		Temper-
		Displacive [dpa/s]	Ionizing [Gy/s]	ature [°C]
Diagnostics (magnetic coils, coaxial		<u> </u>		
cables, insulators)	10 - 10000	$\sim 10^{-8}$	~ 1000	100-350 °
Active coil	~ 500	5×10^{-8}	125	~ 200
Divertor coil	~ 500	< 10 ⁻⁷	~ 1000	< 200
First wall current break	~ 20	$\sim 10^{-7}$	~ 3000	> 300
Vacuum vessel current break	0.3	< 10 ⁸	~ 200	100
Neutral beam insulators	~ 1000	$\sim 10^{-10}$	≤ 10	~ 50
rf windows (ICRH)	< 1000	< 10 ⁻⁹	< 100	< 300
Ceramic breeder insulators	~ 500	< 10 ⁻⁷	~ 1000	~ 400

^a Up to 1500°C for carbon tile diagnostics.

2. Mechanical properties and structural changes

A rather extensive data base has been established on mechanical property and volumetric changes in ceramics following irradiation with fast neutron fluences up to about 3×10^{26} n/m² (30 dpa) [1-10,13-15]. These bulk measurements have been complemented by numerous transmission electron microscopy investigations (e.g., see reviews in refs. [8,16,17]). The mechanical strength and Weibull modulus of ceramics are generally degraded by irradiation, with significant (> 20%) decreases typically occurring after doses > 1 dpa [10,14]. Substantial volumetric swelling also develops at comparable doses for elevated temperature irradiations, and anisotropic swelling in materials such as Al₂O₃ and BeO can produce microcracking and dramatic strength decreases [1,2,8]. Several studies have found that spectral differences between fusion and fission reactors may be significant [18-20]. In particular, the available evidence indicates that the increased concentration of transmutation products such as H, He, and C in a fusion environment will greatly enhance the amount of void swelling in swelling resistant ceramics such as $MgAl_2O_4$ [19,20].

3. Irradiation effects on physical properties

Whereas radiation-induced volumetric changes and reduction in mechanical strength are significant in ceramics for damage levels > 1 dpa, substantial degradation in the physical properties may occur at damage levels $< 10^{-3}$ dpa. In the following sections, the influence of irradiation on the electrical conductivity, loss tangent, and thermal conductivity is reviewed, with emphasis on the available in-situ data. Since the physical properties are sensitive to the concentrations of point defects and conduction electrons, it is natural to expect that these properties may be different during irradiation compared to the post-irradiation value.

3.1. Electrical conductivity

For most insulator applications, the electrical conductivity during irradiation should be less than 10^{-4} S/m 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 $\sigma < 10^{-4}$ S/m to prevent instrument decalibration due to spurious leakage currents.

There is an extensive data base documenting the instantaneous increase in the electrical conductivity of insulating ceramics induced by exposure to ionizing radiation [1,9,12,21-41]. This radiation-induced conductivity (RIC) is due to the excitation of valence electrons into the conduction band, and has been observed during X-ray, gamma ray, electron, proton, and



800600

10⁻⁶

10-7

10⁻⁸

Fig. 1. Electrical conductivity of polycrystalline alumina versus reciprocal temperature, with and without ionizing irradiation [30].

Reciprocal Temperature (K⁻¹) x 10⁻³

Temperature (°C)

100 50 20

Al₂O₃

400 300 200

fission neutron irradiations. The electrical conductivity during irradiation is given by

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

where σ_0 is the conductivity in the absence of irradiation, and R is the ionizing dose rate. The value of the proportionality constant K has a strong material dependence, with typical values at room temperature ranging from 10^{-15} to 10^{-9} s/(Gy Ω m) for polymers and ceramics [27,31,41,48]. The value of the radiation dose rate exponent, d, is generally between 0.5 and 1.0, although supralinear values as high as 1.6 have been reported [9,12,26,27,31,37,48]. The value of the dose rate exponent is determined by the balance between production of conduction electrons by ionization, and trapping and recombination of electron-hole pairs. The general aspects of the observed RIC behavior can be explained by multitrap photoconductivity theory [9,12,21,26,27,37].

Fig. 1 shows a typical example of the temperaturedependent RIC of a ceramic insulator during exposure to ionizing radiation [30]. The electrical conductivity is weakly dependent on temperature at low temperatures (it may either decrease [26] or increase [23,26,30] with increasing temperature). From a technological perspective, it may be concluded that the RIC increases are significant ($\sigma \sim 10^{-7}$ S/m for a fusion-relevant irradiation flux) but can be accomodated with appropriate reactor designs.

Several studies have investigated the electrical conductivity of oxide ceramics during extended irradiation, with and without an applied electric field [1,12,24-26,28,32-41]. It is generally observed that the RIC decreases in specimens irradiated without an applied field [1,26,33,40,41], which may be attributable to a buildup of electron traps produced by the displacement damage. On the other hand, recent work has shown that large, irreversible changes in the electrical conductivity may develop in oxide ceramics if an electric field is applied during irradiation [12,28,32-36,39]. This radiation-induced electrical degradation (RIED) develops at moderate temperatures (300-550°C) after irradiation to damage levels of only 10^{-5} to 10^{-2} dpa, and occurs only if displacement damage and an electric field are simultaneously present. Fig. 2 shows the typical fluence-dependent behavior of the electrical conductivity of Al₂O₃ [12]. The base conductivity σ_0 measured in the absence of radiation steadily increases with increasing dose and eventually becomes much larger than the initial RIC.

Fig. 3 shows the base conductivity σ_0 in Al₂O₃ after electron irradiation to a damage level of 4×10^{-5} dpa [12,33]. The electrical conductivity of the specimen irradiated without an applied electric field was comparable to the nonirradiated condition, with an activation energy of ~ 1.5 eV. The specimen irradiated with an applied electric field showed a dramatic increase in the base conductivity and the activation energy was reduced to 0.18 eV, which suggests a change in the



Fig. 2. Fluence-dependent behavior of the electrical conductivity measured in-situ during 1.8 MeV electron irradiation at 450°C with an applied dc electric field of 130 V/mm and an ionizing dose rate of 2800 Gy/s [12].



Fig. 3. Comparison of the postirradiation electrical conductivity of Al_2O_3 irradiated with 1.8 MeV electrons with and without an applied electric field ([12,33] and unpublished data).

conduction mechanism. Pells [36] has made similar studies of RIED in Al_2O_3 and $MgAl_2O_4$ after proton irradiation with an applied field. The conductivity activation energy decreased in some cases to < 0.1 eV, which indicates the presence of electronic conductivity.

Significant degradation in the electrical resistivity of Al_2O_3 has been observed following electron irradiation even for electric fields as low as ~ 10 V/mm [34,35]. However, an accelerated rate of degradation was observed if the electric field applied during the irradiation was above ~ 60 V/mm. In contrast to the well-known phenomenon of thermal-induced dielectric breakdown in nonirradiated ceramics [42], RIED has been observed to be essentially identical for dc and ac fields for frequencies as high as 126 MHz [35]. A comparable degradation rate has been observed for both single and polycrystals [32].

The available data indicate that the severity of RIED may be strongly dependent on irradiation temperature and dose rate. Little or no degradation occurred in Al_2O_3 or Y_2O_3 for neutron doses of 0.01 to 0.1 dpa at temperatures of 700 to 1100°C with an applied field of ~ 40 V/mm [25,45] (the applied electric field strength was not given in ref. [25]). No electrical degradation was observed in Al_2O_3 and $MgAl_2O_4$ during proton irradiation with an electric field of 500



Fig. 4. Temperature dependence of radiation induced electrical degradation in Al_2O_3 after irradiation for 30 h with 1.8 MeV electrons with an applied electric field of 130 V/mm and a dose rate of 2800 Gy/s [32,33]. The dashed curve shows the general theoretical temperature dependence for colloid formation (not a fit to the data).

V/mm to a damage level of $\sim 10^{-3}$ dpa for irradiation temperatures less than 400 and 300°C, respectively, whereas substantial degradation occurred in both materials at 500°C [36]. Fig. 4 shows the temperature-dependent base conductivity measured in Al₂O₃ following electron irradiation to a damage level of 6×10^{-5} dpa with an applied electric field of 130 V/mm [32,33]. The dashed curve shows the general temperature dependence predicted for the growth of colloids (metallic precipitates) in irradiated ionic crystals [4,43,44]. Colloid formation occurs in irradiated ionic materials as the result of clustering of anion vacancies (F centers), which produces microscopic regions containing only the complementary metal cations. The basic equations describing colloid aggregation are directly analogous to the chemical rate theory equations used to model void swelling in metals. Although it has not yet been directly shown that colloid formation is responsible for the observed RIED in oxide ceramics, the change in the activation energy for electrical conduction [12,33,36] and the flux dependence of RIED [32,33] support this mechanism.

A comparison of the RIED data obtained from different irradiation sources indicates that the irradiation spectrum may have some influence, although systematic trends are not apparent [12,24,25,28,32–39,45]. There are some indications that radiation fields that have high ratios of ionizing to displacive radiation (e.g., electrons and protons) produce an acceleration of the RIED compared to neutron irradiation. However, all of the available results indicate that a large, irreversible degradation of the electrical resistivity occurs in oxide ceramics at very low damage levels $(10^{-5} \text{ to } 10^{-2} \text{ dpa})$ at irradiation temperatures between 350 and

 500° C when an electric field is applied during the irradiation.

3.2. Dielectric properties at high frequencies

The loss tangent (tan δ) is a measure of the amount of power absorbed in a dielectric material from an incident electromagnetic wave, which is of particular importance for the ceramic windows and feedthroughs associated with resonant frequency heating components for fusion reactors. The loss angle, δ , is related to the phase difference between the applied ac field and the resulting current. For an ideal loss-free dielectric, the current leads the voltage by 90° and the loss angle $\delta = 0$ [46]. Integration of the product of the current and voltage yields the power absorbed in lowloss dielectrics:

$$P = \omega \epsilon_0 \epsilon' \tan \delta E^2, \tag{2}$$

where ω is the angular frequency of the ac electric field with rms amplitude *E*, and ϵ_0 and ϵ' are the free space and dielectric permittivities. According to fusion design analyses, the dielectric loss tangent for ion cyclotron (~100 MHz) and electron cyclotron (~100 GHz) resonance heating applications should be less than 10^{-3} and 10^{-6} , respectively.

The value of tan δ for low-loss dielectrics is dependent on temperature and frequency [9,10,46–49]; a typical value for a high-quality ceramic at room temperature is tan $\delta \sim 10^{-4}$. The low value of tan δ required for electron cyclotron heating systems can only be-achieved in present dielectrics by cooling to cryogenic temperatures. There are two major mechanisms responsible for energy losses in dielectrics [46,48]:

$$\tan \delta = \frac{\sigma}{\epsilon' \epsilon_0 \omega} + \frac{\chi''}{\epsilon'}.$$
 (3)

The first term represents electrical conductivity losses, which can be significant in ceramics during irradiation (cf. section 3.1) but becomes negligible for frequencies above ~ 100 MHz due to the inverse dependence on frequency [40,46,48]. The second term in eq. (3) represents polarization losses, where χ'' is the imaginary part of the susceptibility [48]. Resonances in the polarization losses occur at frequencies corresponding to electron, ion, and defect jump polarizations [9,46]. In practice, the widths of these resonances are very large due to a distribution of relaxation times, and tan δ varies slowly with frequency at frequencies relevant for plasma heating [10].

As noted in previous reviews [9,10], there have been relatively few studies of radiation-induced changes in the dielectric properties of ceramics. Most of these studies involved postirradiation measurements on specimens that were neutron irradiated at elevated temperatures. The post-irradiation data indicate that radiation-induced changes in the permittivity are generally vcry small (~ 1-5%), with either slight increases [13,48,50,52] or decreases [51] reported. The loss tangent was observed to increase in all irradiated materials studied, but the magnitude of the increase depended on the material, irradiation conditions, and measurement frequency [9,10,13,41,48,50-53]. The amount of increase was generally larger for the lower irradiation temperatures. Neutron irradiation to damage levels < 0.1 dpa at 70 to 200°C increased the loss tangent of Al₂O₃ by about a factor of 3 to ~ 10⁻³ for frequencies of 30 MHz to 35 GHz [48,52].

Several in-situ loss tangent measurements have been performed on ceramics during proton [40,41,54] or neutron [55,56] irradiation near room temperature at frequencies ranging from near dc to 10 GHz. Cryogenic in-situ tests at a frequency of 1 kHz have also been performed during neutron irradiation on organic dielectrics with relatively high loss factors [57]. These in-situ studies have found that large transient increases in the loss tangent may occur under certain conditions. Fig. 5 shows the loss tangent of two ceramics measured in-situ at 100 MHz during pulsed neutron irradiation [56]. Most of the increase and subsequent decay of the loss tangent during the pulsed irradiation may be attributed to radiation-induced conductivity (cf. eq. (3)).



Fig. 5. Loss tangent of Al_2O_3 and AlN at a frequency of 100 MHz measured in-situ during pulsed fission reactor irradiation at room temperature [56]. The reactor power pulse is shown on the same time scale. The peak ionizing and displacive radiation fields were 6×10^4 Gy/s and 1.8×10^{-7} dpa/s, respectively.



Fig. 6. Dielectric loss factor of polycrystalline Al₂O₃ at a frequency of 60 MHz measured in-situ during 3 MeV proton irradiation near room temperature [54].

At higher frequencies (1–10 GHz), where the electrical conductivity contribution to tan δ is reduced (eq. (3)), the loss tangent of a wide range of ceramics increased only slightly during pulsed neutron irradiation at a damage rate of ~ 10⁻⁵ dpa/s [55].

Several studies have determined that significant annealing of the induced damage in ceramics may occur at room tempcrature [40,41,53]. For example, postirradiation measurements performed on a range of ceramics irradiated with protons to damage levels of $\sim 2 \times 10^{-2}$ dpa found that a substantial part of the loss tangent increase at 100 MHz was recovered within a matter of hours [41,53]. This indicates that postirradiation measurements of the loss tangent may be an underestimate of the in-situ value even at high frequencies where the in-situ electrical conductivity contribution is small.

Buckley and Agnew [54] have recently studied the effect of accumulated damage on the in-situ loss tangent of Al₂O₃. As shown in fig. 6, the loss tangent showed a steady increase during proton irradiation at 300 K for damage levels greater than $\sim 10^{-4}$ dpa. The value of tan δ as measured in-situ became unacceptably high $(> 10^{-3})$ at damage levels on the order of 10^{-3} dpa. This degradation rate in the loss tangent is higher than postirradiation measurements would indicate, and may be due to the absence of postirradiation annealing effects [41,53]. Alternatively, the rapid degradation shown in fig. 6 may also be associated with the ~ 10 V/mm electric field that was applied during the irradiation. The results of Hodgson [35] indicate that the application of even this small electrical field could cause significant degradation of the electrical properties over a wide frequency range. Additional in-situ measurements performed over a wide temperature range and in different irradiation spectra are needed to further examine this issue.

3.3. Thermal conductivity

A high thermal conductivity is desirable to minimize the influence of thermal stresses in ceramic components during fusion reactor operation. The thermal conductivity of ceramic insulators is controlled by phonon scattering [58,59]. In contrast to metals, heat conduction in ceramics by electrons is negligible compared to the lattice heat conduction even during highly ionizing irradiation conditions which produce cnhanced levels of conduction electrons (RIC). Numerous postirradiation measurements performed on a wide range of ceramics have demonstrated that the thermal conductivity may be significantly degraded by neutron irradiation [1,2,5,8,10,60-70]. The thermal conductivity measured at room temperature is generally observed to saturate at 10 to 50% of the preirradiation value for damage levels greater than about 0.1 dpa. Irradiation and/or measurement at elevated temperatures produces proportionately less degradation in the thermal conductivity.

The radiation-induced decrease in the thermal conductivity is much more severe at low temperatures than at room temperature [67,69,70]. As shown in fig. 7, the thermal conductivity of single crystal Al₂O₃ at cryogenic temperatures is initially very high (up to 15 times the room temperature copper value). However, neutron irradiation near 50°C to a damage level of $\sim 5 \times$ 10^{-3} dpa reduced the peak conductivity to ~ 3% of its nonirradiated value [67]. The low-temperature in-situ data by McDonald [69] on irradiated BeO suggests that the rate of degradation shown in fig. 7 would have been significantly faster if the irradiation was conducted at cryogenic temperature: Irradiation of BeO at 350 K [68] required 16 times higher fluence to produce the same thermal conductivity degradation observed after irradiation at 90 K [69]. McDonald also found that 25% of the thermal conductivity degradation in



Fig. 7. Low-temperature thermal conductivity of single crystal Al_2O_3 measured after fission reactor irradiation [67]. The irradiation temperature was not specified, but was presumably near 50°C (in-pile irradiation).

BeO was recovered by isochronal annealing for 15 min at room temperature.

Although point defects are generally more important than extended defects in degrading the thermal conductivity of ceramics [58,59], a recent analysis [73] indicates that the typical point defect supersaturation (beam on versus beam off) during irradiation is not sufficient to produce significant instantaneous degradation. This analysis is supported by several low-flux $(\sim 10^{-10} \text{ dpa/s})$ in-situ neutron irradiation studies which did not detect any instantaneous changes in thermal conductivity as the irradiation source was turned on and off [24,69]. On the other hand, there is clear evidence from in-situ [71] and postirradiation isochronal annealing [69] thermal conductivity measurements that postirradiation measurements may underestimate the degradation present during irradiation, due to point defect (and defect cluster) annealing effects that occur over an extended period of time. Keilholtz et al. [71] have also shown that the rate of thermal conductivity degradation in BeO measured in-situ during neutron irradiation at 920°C was flux-dependent. The rate of degradation was markedly slower for the lower flux irradiation, due to defect annealing that occurred during the irradiation.

4. Discussion

The available data suggest that there can be large in-situ physical property degradations in ceramics not observed in postirradiation measurements. Prompt effects associated with RIC can cause significant, albeit manageable, changes in σ and tan δ . Further differences between extended in-situ irradiation and postirradiation properties can be attributed to two general effects. First, despite ceramics refractory nature, a substantial amount of the defects produced during irradiation may be annealed during postirradiation storage at room temperature prior to making measurements [40,41,53,69]. This thermal instability of radiation-induced defects also causes room and elevated temperature irradiations to yield smaller amounts of physical property degradation than what occurs during lower temperature irradiations [9,10,52,61,64-72]. Second, new radiation-induced processes such as RIED may be activated if the ceramic is irradiated under certain design-relevant conditions, e.g., with an applied electric field [12,28,32-36,39].

These in-situ observations underscore the importance of performing irradiations and measuring property changes under conditions that closely resemble the anticipated operating environment. The combined effect of ionization, displacement damage, and an applied electric field (dc or ac) produces serious electrical degradation (RIED) in oxide ceramics, whereas this degradation does not occur if the electric field is absent. One particularly disconcerting aspect associated with RIED is that the degradation cannot be removed by high temperature annealing. Instead, the work by Pells [36] indicates that the degradation process may be further enhanced by postirradiation annealing. Most of the studies concerning the newly discovered phenomenon of RIED have utilized electrical conductivity measurements alone. It seems apparent that the other physical properties such as loss tangent and possibly thermal conductivity may also be further impaired by a similar process if an electric field is imposed during the irradiation.

The investigations of RIED have so far been limited to oxide ceramics. Due to their predominantly ionic bonding, oxides are more susceptible to colloid formation compared to ceramics with other types of bonding. It would certainly be worthwhile to extend the RIED investigations to insulating ceramics with predominantly covalent bonding, such as nitrides, to determine if a similar degradation occurs.

There are some indications that ceramics may be more sensitive than metals to irradiation spectrum differences, particularly with regard to the ionizing radiation component. For example, it has been demonstrated that the amount of volumetric swelling is reduced [74] and extended defect clusters do not form [75] in oxide ceramics if the irradiation tield is highly ionizing. Similarly, the reduction in thermal conductivity in Al_2O_3 was apparently less for an irradiation with very high energy neutrons (LAMPF) compared to fission neutron irradiation [64,66]. Further work is needed to understand these spectrum effects before presentday irradiation results can be applied to fusion reactor designs.

5. Design guidelines

In general, postirradiation physical property data should not be considered appropriate for design purposes since it often is an undermeasure of the in-situ property degradation. Recent in-situ experiments have demonstrated that ceramics cannot be satisfactorily operated under certain experimental conditions. There is some urgency to solve these emerging challenges, in that a fusion reactor may become inoperative within a few weeks after initiation of D-T experiments due to electrical degradation of key ceramic components. Unacceptable degradation apparently develops at extremely small doses $- \sim 10^{-3}$ dpa depending on the temperature and dose rate, which corresponds in the first wall region to less than 1 h of full power operation. This may be contrasted with structural degradation in metals and ceramics, which typically becomes of concern only after damage levels greater than 1-10 dpa. There does not appear to be a strong incentive for operating ceramics at cryogenic temperatures for damage levels $> 10^{-3}$ dpa unless in-situ anneals can be performed, due to the higher degradation rates in the thermal conductivity and loss tangent at low temperatures.

There are hopeful indications that catastrophic degradation of the physical properties of ceramics may be avoided by judicious choice of the operating temperature. The data presently available suggest that for first wall irradiation conditions the temperature range of 300 to 550°C should be avoided. Operation at lower or higher (up to 800 to 1000°C) temperatures may give acceptable performance. For ceramic applications in lower dose rate regimes such as near the vacuum vessel, colloid growth theory [43] would predict that the temperature range for serious degradation would shift to lower temperatures. Obviously, considerable work is needed to confirm and theoretically comprehend the present results before firm guidelines can be established.

6. Conclusions

Recent in-situ radiation effects studies have uncovered the existence of operational conditions in which unacceptable degradation of the physical properties of ceramics may occur at exceptionally low doses ($< 10^{-3}$ dpa), due to a synergistic relation between ionization, displacement damage, and applied electric field. Postirradiation physical property data are often not indicative of the property values during irradiation, and generally should not be used for design purposes.

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