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Journal of Nuclear Materials 283–287 (2000) 903–906

journal of nuclear materials

www.elsevier.nl/locate/jnucmat

In-beam dielectric properties of alumina at low frequencies

R. Vila *, E.R. Hodgson

Euratom/CIEMAT Fusion Association, Institute of Investigacion Basica, Avenida Complutense 22, 28040 Madrid, Spain

Abstract

Loss tangent data for three commercial alumina grades measured in the range 0.1–300 kHz together with the effect of radiation are presented. Very different dielectric behaviour has been found for the three grades irradiated at 150°C and 250°C with 1.8 MeV electrons at dose rates of up to 3.5 kGy/s. In the case of Ceraten C9999, an extremely pure alumina, the increase in the loss tangent with dose rate and its dependence on frequency is due to the dc radiation induced conductivity (RIC). For Wesgo AL995 and Morgan Matroc Vitox 999 the behaviour is more complex. The influence of an applied electric field has also been examined. The importance of material selection is noted, as is the dose-dependent degradation for Wesgo AL995.

1. Introduction

Alumina is considered to be a good candidate as a general purpose insulator for many different fusion reactor diagnostics. Requirements (voltages, temperatures, etc.) and working frequencies (dc to ac/rf) vary greatly. Systems such as bolometers, pick-up coils, and pressure gauges will need insulating materials to act as feedthroughs, stand-offs, cable insulation, and coil formers for low-voltage signals in the frequency range from about 1 kHz to 1 MHz [1]. For such applications dielectric loss (loss tangent) and permittivity, for different alumina grades over a wide frequency range, together with the effect of radiation are of vital importance for the design of the systems. Although data does exist for the radiation effects on dielectric properties of aluminas for the plasma heating frequency ranges (100 MHz to 200 GHz range) [2,3], this is not the case at low frequencies (<1 MHz). With the aim of providing this information and assessing the problem, the dielectric loss behaviour of three high-purity alumina grades has been studied, in which the loss as a function of dose rate, dose, temperature and applied electric field has been examined.

* Corresponding author. Tel.: +34-1 346 6580; fax: +34-1 346 6068.

E-mail address: vila@ciemat.es (R. Vila).

2. Experimental procedure

To provide representative loss tangent and permittivity data, samples of Ceraten C9999, Morgan Matroc 'Vitox' 999, and Wesgo AL995 high-purity alumina grades have been measured with a commercial LCR meter (HP 4284A) which has a frequency range from 20 Hz to 1 MHz. Data on these three grades, to be referred to as C9999, MM999 and W995 respectively, are given in [4]. The C9999 and MM999 are high-density small grain size aluminas, whereas the W995 has low density and large grain size. Available total impurity analysis indicates C9999 $<$ 100 ppm, MM999 $\approx$ 1000 ppm, and W995 $\approx$ 5000 ppm. To enable the effect of radiation to be determined, a special sample chamber has been built and installed in the beam line of a 2 MeV Van de Graaff electron accelerator. The chamber contains a heating system and thermocouples to control the sample temperature. Alumina samples (30 mm diameter, 1-mm thick) with evaporated common, guard and central aluminium electrodes have been irradiated in vacuum (10^{-4} mbar) with 1.8 MeV electrons at temperatures up to 250°C and dose rates of up to 7 kGy/s (10^{-9} dpa/s), and the loss tangent and permittivity determined. For the complete experimental set-up, the frequency range for reliable data, imposed by system noise and sample impedance, is between about 0.1 and 300 kHz, and the lower limit for loss about 10^{-4}. Both dose rate and dose, as well as temperature effects have been recorded. In addition, to check the effect of an electric field on the

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PII: S0022-3115(00)00091-X

dielectric degradation, a dc voltage of 40 V was superimposed on the 1 V LCR meter measuring signal for the MM999 and W995 materials.

3. Results and discussion

For the three alumina samples very different dielectric behaviour has been found. All three materials show large increases in loss during irradiation. Furthermore, both W995 and MM999 show permanent degradation in the loss with dose. For simplicity the results for each alumina grade will be discussed separately.

C9999: Before irradiation, this extremely high-purity alumina (<100 ppm) presents very low losses at 20°C ($\leqslant 10^{-4}$) [4], and at 150°C is still below the measuring limit. At the onset of irradiation at 150°C for dose rates up to 400 Gy/s, a very fast (<1 s) increase in the loss ($\tan\delta$) occurs which exhibits an f^{-1} dependence (Fig. 1). Dielectric loss may be expressed as

$$\tan\delta \approx \sigma/f\epsilon\epsilon_0 + \chi/\epsilon,$$

where σ is the dc conductivity, f the frequency, ϵ the relative permittivity, ϵ_0 the permittivity of free space, and χ is the imaginary part of the electric susceptibility. The first term represents the dc contribution, and the second term that due to defect related polarization mechanisms [5]. Hence the 1/*f* behaviour together with the fact that the permittivity remains constant indicates that the radiation induced dc conductivity (RIC) dominates in the loss. The rapid increase at the onset of irradiation, indicating that the RIC is due mainly to electron–hole pair production with little defect charge trapping, also supports the above interpretation [5]. The losses increase linearly with dose rate, but for dose rates above 400 Gy/s, this linearity and the 1/*f*-dependence are only valid for higher frequencies (Figs. 1 and 2). The derived dc radiation induced conductivity obtained from the measurements at 10 kHz for 3.5 kGy/s is 10^{-6} S m^{-1}, in good agreement with direct RIC measurements for very pure alumina and sapphire [6]. In order to study possible dose effects, this material was irradiated at 150°C and 3.5 kGy/s. Up to a total dose of 330 MGy (5×10^{-5} dpa) no permanent degradation was observed in the post irradiation loss, nor in the loss measured during irradiation. Under all conditions the permittivity remained almost unchanged.

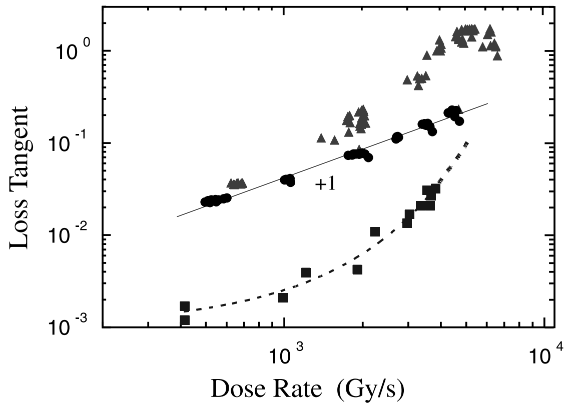

Fig. 2. Loss tangent at 10 kHz and 150°C for (●) C9999, (■) W995, and (▲) MM999 as a function of dose rate. The dotted line is an exponential curve fit.

W995: Before irradiation this material has a loss of about 2×10^{-4} over the measured frequency range at 20°C [4]. This increases to about 10^{-3} at 150°C and 2×10^{-3} at 250°C (Figs. 3 and 4). On irradiation at 150°C and 250°C, the behaviour is very different from that observed in C9999. At the onset of irradiation a delay of several minutes occurs before the dielectric loss approaches saturation, and the loss with frequency varies approximately as $f^{-0.6}$ under irradiation (Fig. 3).

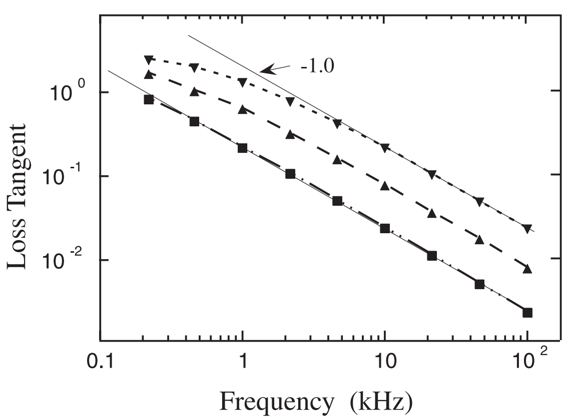

Fig. 1. Loss tangent at 150°C as a function of frequency for C9999 at dose rates of (■) 0.35, (▲) 1.4 and (▼) 3.5 kGy/s.

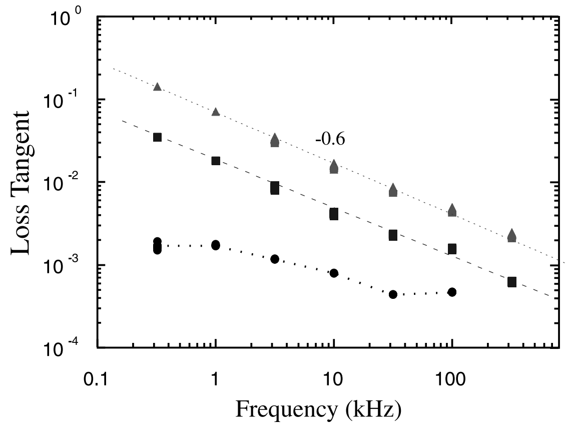

Fig. 3. Loss tangent at 150°C as a function of frequency for W995 at (●) 0, (■) 2.1 and (▲) 3.5 kGy/s.

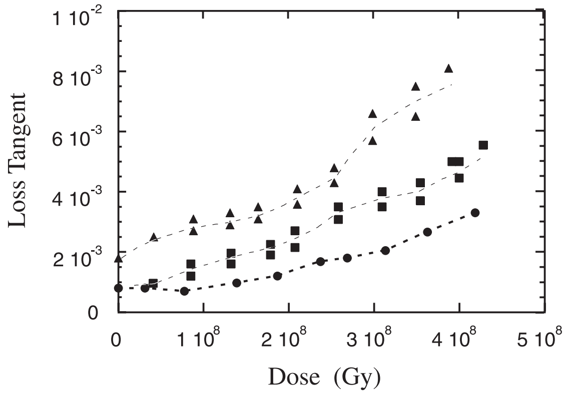

Fig. 4. Loss tangent for W995 at 10 kHz without radiation, measured after several electron irradiations up to the indicated accumulated doses. (●) Irradiated at 150°C, (■) irradiated at 150°C and 40 kV/m, and (▲) irradiated at 250°C and 40 kV/m.

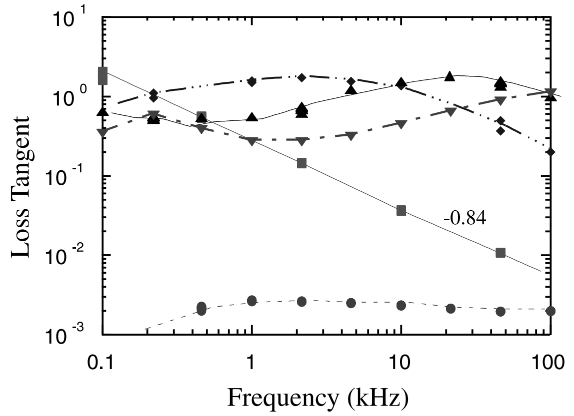

Fig. 5. Loss tangent at 150°C as a function of frequency for MM999 at dose rates of (●) 0, (■) 0.35, (◆) 2.6, (▲) 3.5 and (▼) 4.4 kGy/s.

These two observations indicate the important role of charge trapping in this lower purity material. The dielectric loss at a fixed frequency increases exponentially with dose rate (Fig. 2). The effects of dose for irradiations at 150°C, and at 150°C and 250°C with an applied dc field of 40 kV/m, have been observed for doses up to 420 MGy. With dose a continuous increase in the post irradiation loss occurs for all three cases (Fig. 4). This permanent dose-dependent damage is high for irradiation at 150°C, higher when irradiating with an applied dc field at the same temperature and even higher when irradiating at 250°C with field. Clearly both temperature and field have an effect. The applied field is well below that expected for RIED in this material (1 MV/m), so the observed degradation may be related to the reported surface degradation observed in W995, for similar temperatures and fields [7,8]. The data for permittivity indicate that the maximum changes will occur at the lower frequencies, where increases of the order of 20% have been recorded at 320 Hz for the highest dose rate and dose. By 10 kHz this increase reduces to 4%.

MM999: Before irradiation, the loss is low at 20°C ($\leqslant 10^{-4}$) [4], increasing to about 10^{-3} at 150°C (Figs. 5 and 6). Irradiation has been carried out at 150°C with 40 kV/m applied. This sample exhibits the most complicated behaviour. As for W995, at the onset of irradiation the material shows a delay of several minutes before the loss approaches saturation. At low dose rates, the loss shows a similar behaviour to that observed for C9999 and W995, i.e. a straight line in the log–log plot with a slope of −0.8 in this case. At higher dose rates a peak structure appears (Fig. 5). The frequency of the peak maximum is dose-rate dependent and moves to higher frequency on increasing dose rate. This results in a complex dependence of the loss with dose rate when measuring at a fixed frequency (Fig. 2). On increasing the dose rate as the peak maximum approaches the measuring frequency the loss increases in a similar way to that observed for W995, but then the loss reaches saturation and begins to decrease as the peak moves to higher frequency. The results for dose dependence show that the post irradiation loss increases quite rapidly by 200 MGy, but then saturates with very little change up to 460 MGy (Fig. 6). The permittivity shows a complex behaviour with frequency and dose rate; however the largest increases again are observed in the low-frequency region, where extreme changes of up to 20 times have been recorded.

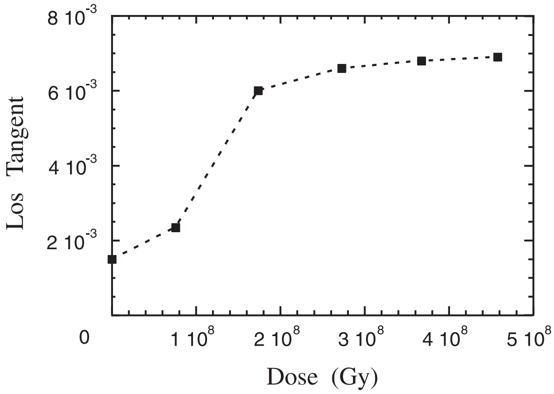

Fig. 6. Loss tangent at 10 kHz for MM999 without radiation after different accumulated electron doses at 150°C.

4. Conclusions

These results emphasise the importance of measuring the exact alumina grade to be employed for low-frequency diagnostic components. Not only are large differences observed in both the dielectric loss and permittivity for the three materials, but also in the general behaviour under radiation. Only the very pure alumina grade (C9999) exhibits a predictable behaviour, with a 1/*f*-frequency dependence for the loss. For the other materials a lower exponent is observed, and in the case of MM999 an absorption band in the loss spectrum induces an extremely complex behaviour. At the highest common dose rate of 3.5 kGy/s, the measured loss at 10 kHz is highest in the MM999 (2.0) and lowest in W995 (3×10^{-2}). One must also take into account the possibility of dose-dependent degradation. In the case of the C9999 material no degradation was observed in the post irradiation measurements, and in the case of MM999 a saturating degradation of about 7×10^{-3} in the loss was recorded. However, in the case of W995 serious degradation was observed with the post irradiation loss reaching about 10^{-2} by 400 MGy, with no indication of saturation. This value is comparable with the maximum value measured during irradiation (3×10^{-2}), so for this material both dose rate and dose effects must be considered.

Acknowledgements

The authors are indebted to Mr J. Montesinos and Mr E. Sánchez-Cabezudo for their valuable help in these experiments. The Wesgo material was kindly provided by Dr R. Stoller, ORNL, USA, as part of the ITER 'round robin' study. The work forms part of the European Fusion Technology Programme.

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