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BA materials activities: Radiation induced electrical degradation of HP SiC

A. Moroño*, R. Vila, T. Hernandez, J. Manzano, E.R. Hodgson

Euratom/CIEMAT Fusion Association, Avenida Complutense 22, 28040 Madrid, Spain

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ABSTRACT

Within the EU-Japan Broader Approach (BA) materials activities, the Euratom/CIEMAT fusion association will carry out work to characterize basic properties of silicon carbide materials. Work has begun on hot pressed (HP) SiC. The material has been irradiated with 1.8 MeV electrons and implanted with 50 keV He in order to study radiation induced electrical degradation in the bulk and surface of the material. For both 1.8 MeV electron irradiation and 50 keV He implantation amorphization of HP SiC occurs together with notable changes in the electrical behaviour. Microstructure has been examined using X-ray diffraction and SEM.

1. Introduction

Within the EU-Japan Broader Approach (BA) materials activities, the Euratom/CIEMAT fusion association will carry out work to characterize basic properties of silicon carbide materials (SiC and SiC_f/SiC), considered as potential long term reduced activation structural materials, as well as possible flow channel inserts in Li–Pb tritium breeding blanket modules. Measurements planned include volume and surface electrical conductivity, and H diffusion, as well as the effects of H and He on microstructure and electrical conductivity.

Studies of silicon carbide based ceramic composites for applications in fusion have been carried out for more than 10 years, and although understanding of the basic radiation damage processes as well as microstructural evolution has shown significant advances, considerable further work is required to fully understand the physical mechanisms and varied irradiation effects for the different forms of SiC and SiC_f/SiC [1]. Clearly in order to improve the radiation behaviour of SiC_f/SiC, one must fully understand the basic radiation response of the SiC matrix material.

Work has begun on hot pressed (HP) SiC. Results for radiation induced degradation of the electrical conductivity, show almost an order of magnitude decrease in the volume conductivity for electron irradiation in high vacuum to 420 MGy at 450 °C. In contrast surface conductivity markedly increases after both electron irradiation and 50 keV He bombardment (implantation). Following He bombardment surface amorphization is observed, but of more concern is the unexpected amorphization of SiC within the volume after electron irradiation to low dose, and at moderate temperature.

2. Experimental procedure

Hot press monolithic commercial SiC samples were cut to approximately 10 mm × 10 mm × 2 mm, and then irradiated with either 1.8 MeV electrons to examine the effects of ionizing radiation on the bulk material, or 50 keV He^+ ions to study surface effects. The electron irradiations were made in a special chamber mounted in the CIEMAT HVEC Van de Graaff accelerator beam line, where samples mounted on an oven, may be irradiated in high vacuum (≈10^{-6} mbar), at controlled temperatures between 15 and 650 °C. Gold central and guard electrodes sputtered onto one 10 mm × 10 mm sample face, and a single earth electrode onto the opposite face allow an electric field to be applied to measure volume and surface electrical conductivity. The voltage applied to the sample was 100 mV (50 V/m), and the current sensitivity 10^{-5} A. In this way the sample was heated at 0.2 °C/s from 20 to 450 °C to measure the volume and surface electrical conductivities as a function of temperature, and then irradiated at 450 °C, 7 kGy/s (≈10^{-9} dpa/s on the C sublattice), to 140 MGy. This was repeated up to 420 MGy. Volume and surface resistances (inverse of conductivity) could be measured to about 1 Ω. The central and guard contact resistances were measured before and after irradiation and in all cases found to be less than 0.5 Ω. To check for purely temperature effects a further sample was maintained under the same conditions at 450 °C for 16.5 h (the equivalent time to reach 420 MGy).

The He implantation was performed in the beam line of the CIEMAT Danfysik 60 kV ion implanter, using a chamber identical to that described above. In this case a 5 mm diameter stainless

* Corresponding author. Tel.: +34 91 346 7913; fax: +34 91 346 6124.
E-mail address: morono@ciemat.es (A. Moroño).

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steel collimator was used in order to define the implanted area. To measure the surface conductivity two gold electrodes separated by 1.5 mm were sputtered onto the 10 mm × 10 mm sample face to be implanted. The sample was implanted at 450 °C with 50 keV He^+ ions, 0.5 μA/cm^2, in steps doubling the dose up to 2×10^{18} ions/cm^2. Following each step the sample was cooled and conductivity measured from 20 to 450 °C. The ion range for 50 keV He^+ in SiC is about 0.3 μm [2].

The crystalline phases for the as-received material were investigated by X-ray diffraction (XRD) using a Philips diffractometer (X-Pert-MPD with Cu Kα radiation and Si monochromator). Scanning electron microscopy (SEM) using a JEOL JSM 6400 at 20 kV and ADX LINK INCA was applied to visualize the microstructure. The crystalline evolution after irradiation was examined by XRD. In the case of the He implantation, the surface was analyzed by XRD at a glancing angle of 0.5°, by means an X'pert PRO MRD equipped with a high horizontal resolution goniometer, using the Kα wavelength 1.54056 Å.

3. Results

The volume resistance of the unirradiated SiC at 20 °C was 788 Ω, corresponding to a volume conductivity of about 0.25 S/m. On heating to 450 °C the volume conductivity increased to 13 S/m. At the onset of irradiation at 450 °C with 1.8 MeV electrons the volume conductivity further increased to about 16 S/m due to beam heating. At this high level of conductivity no radiation induced conductivity (RIC) was detected. The volume conductivity then slowly decreased with irradiation time (dose). Fig. 1 shows Arrhenius plots of electrical conductivity as a function of inverse temperature for the SiC unirradiated and following irradiation at 450 °C with 1.8 MeV electrons to 140 and 280 MGy. A clear decrease in volume conductivity with dose is observed, more pronounced at 20 °C. Fig. 2 shows this decrease in volume electrical conductivity measured at 20 °C as a function of dose up to 420 MGy, with no indication of saturation. Volume conductivity has been reduced by about a factor 6 (volume resistance at 20 °C increasing from 788 to 4925 Ω). In contrast the surface conductivity at 20 °C showed a very different behaviour (Fig. 3). Up to 120 MGy the surface conductivity remained constant, and then increased by about three times to a higher constant value.

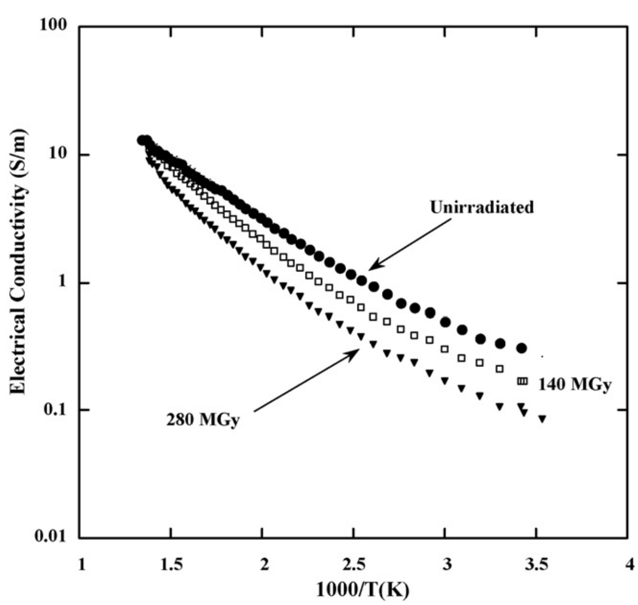

Fig. 1. Arrhenius plot of volume electrical conductivity for HP SiC, unirradiated and 1.8 MeV electron irradiated at 450 °C to 140 and 280 MGy.

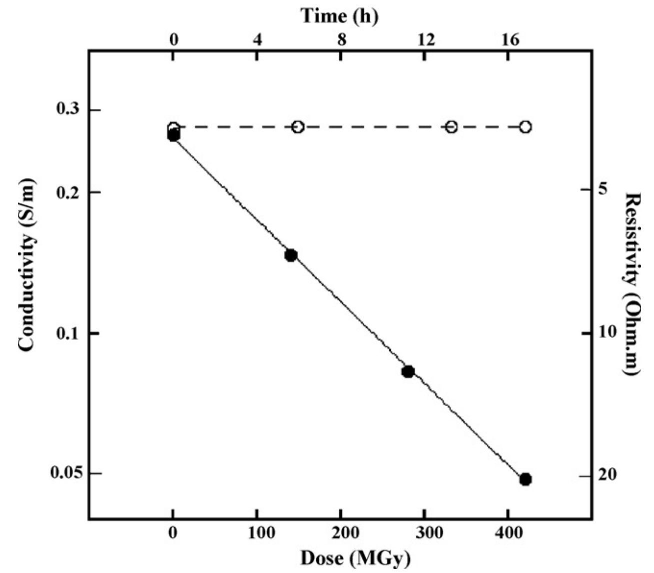

Fig. 2. Electrical volume conductivity at 20 °C; 1.8 MeV electron irradiation at 450 °C vs. dose (closed circles), and unirradiated at 450 °C vs. time (open circles).

The results for the effects of temperature alone (Fig. 2) indicated no change in volume or surface conductivity, i.e. the observed effects are due to the radiation.

Fig. 4 shows the Arrhenius plots for the surface conductance in Siemens ($S = 1/\Omega$) for unimplanted SiC and implanted with 50 keV He^+ at 450 °C to 2×10^{18} ions/cm^2. Although the surface conductivity changes by less than an order of magnitude from 20 to 450 °C compared with two orders of magnitude for the volume conductivity (Fig. 1), the form is more complex, showing little increase at the lower temperatures. After implantation the change in conductivity is more noticeable at the lower temperatures. As with the electron irradiations, for implantation the surface conductivity increases with dose, and shows a very similar behaviour. The surface electrical conductance measured at 20 °C for the SiC implanted at 450 °C with 50 keV He as a function of dose is shown in Fig. 5. No change occurs up to a dose of 10^{17} ions/cm^2. The conductivity then starts to increase, but by $<2 \times 10^{18}$ ions/cm^2 the degradation begins to saturate after an increase in conductivity of about 50%.

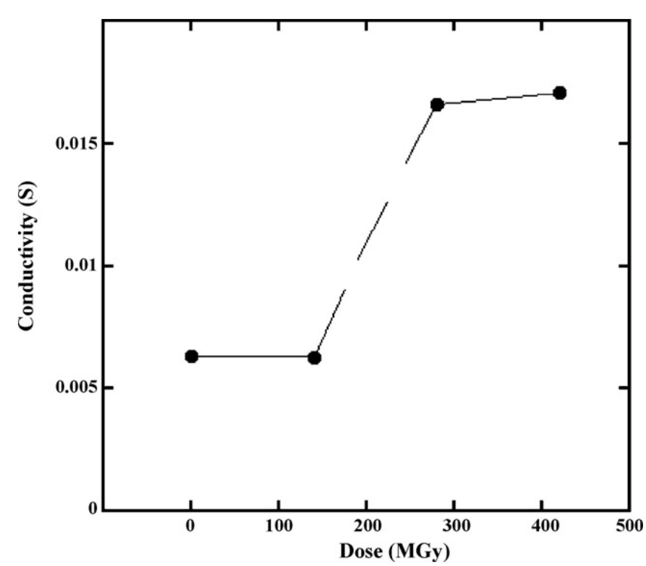

Fig. 3. Electrical surface conductivity at 20 °C vs. dose for 1.8 MeV electron irradiation at 450 °C.