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Trapping and thermal diffusion for energetic deuterium implanted into SiC

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ABSTRACT

During ITER and DEMO reactor operation Li–Pb blanket flow channel inserts made from SiC will be exposed to both radiation and tritium. Absorption, desorption, and tritium diffusion are expected to occur and will strongly depend on the irradiation conditions; temperature, and neutron and gamma fluxes. Reaction bonded (RB) SiC samples were deuterium implanted at both room temperature and 450 °C at different implantation energies and the corresponding TSD spectrum was obtained for each implantation energy. After implantation the samples were subjected to SIMS analysis. The TSD spectra obtained for all the samples implanted at different energies are very similar and characterized by a prominent deuterium desorption occurring at temperatures between 450 and 1000 °C with a maximum that exhibits a clear trend to shift toward higher temperature as either implantation energy or implantation temperature increase. SIMS analysis before heating the deuterium implanted samples indicates that the implanted deuterium has a tendency to become bonded to Si rather than to C. The SIMS analysis shows that once heated up to 1000 °C only part of the implanted deuterium was thermally released. The temperature shift observed when increasing the deuterium implantation energy and, hence, penetration, implies a deuterium diffusivity value at 700 °C of about 8.5×10^{-17} m^2/s.

Introduction

SiC materials are primary candidates for flow channel inserts in blankets due to their excellent thermo-mechanical and corrosion properties [1,2]. For this application hydrogen isotope absorption is of fundamental importance. During reactor operation the SiC material will be exposed to tritium in a hostile radiation environment. Absorption, diffusion, and desorption are expected to occur, depending strongly on the radiation conditions, neutron flux, and ionizing radiation. The operation temperature for this material in a future fusion reactor could be from about 500 up to 1000 °C depending on the type of blanket concept [3]. The main aim of this work is to study the behaviour of energetic deuterium implanted into SiC in terms of trapping, detrapping, and diffusion within the temperature range of between 400 and 1000 °C.

Reaction bonded (RB) SiC samples were deuterium implanted at both room temperature and 450 °C at different implantation energies. The implantation energies used for this work ranged from 20 keV up to 50 keV. After deuterium implantation, the samples were heated up to 1000 °C at a rate of 0.16 C/s and the deuterium released was measured as a function of temperature obtaining in this way the corresponding Thermo Stimulated Desorption (TSD) spectrum for each implantation energy. After implantation the samples were subjected to secondary ion mass spectrometry (SIMS) analysis both before and after being heated up to 1000 °C.

The TSD spectra obtained for all the samples implanted at different energies are very similar and characterized by a prominent deuterium desorption occurring at temperatures between 450 and 1000 °C with a maximum that exhibits a clear trend to shift toward higher temperature as either implantation energy or implantation temperature increase. The temperature shift observed when increasing both the deuterium implantation energy and, hence, penetration, implies a deuterium diffusivity value near 700 °C of about 8.5×10^{-17} m^2/s within the range of the value expected from the diffusivity parameters found in the literature [4].

Experimental procedure

1 mm thick RB SiC samples ($\rho = 3.10$ g/cm^3), were cut and optically polished from the same 15 mm diameter bar (Goodfellow) to ensure uniformity. This commercially available RB material has low apparent porosity (0%), fine grained, and highly crystalline (90%

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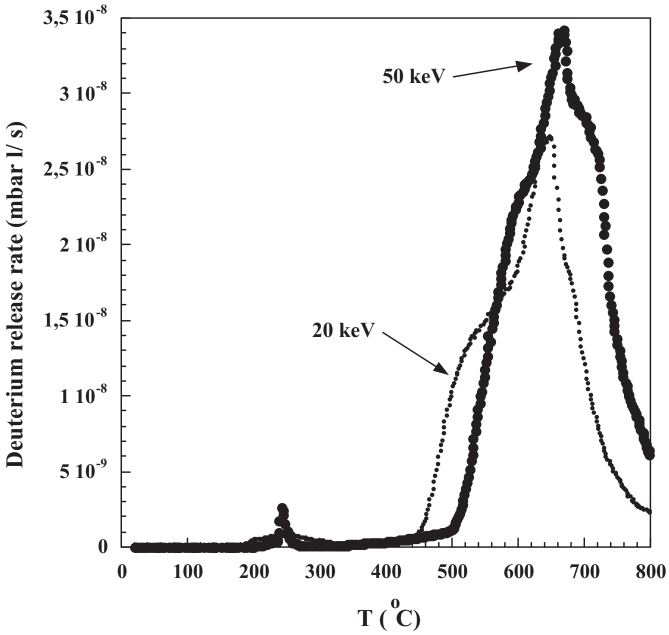

Fig. 1. TSD spectra for D^+ implanted RB–SiC samples at 25 °C at different energies up to 1×10^{16} D/cm^2.

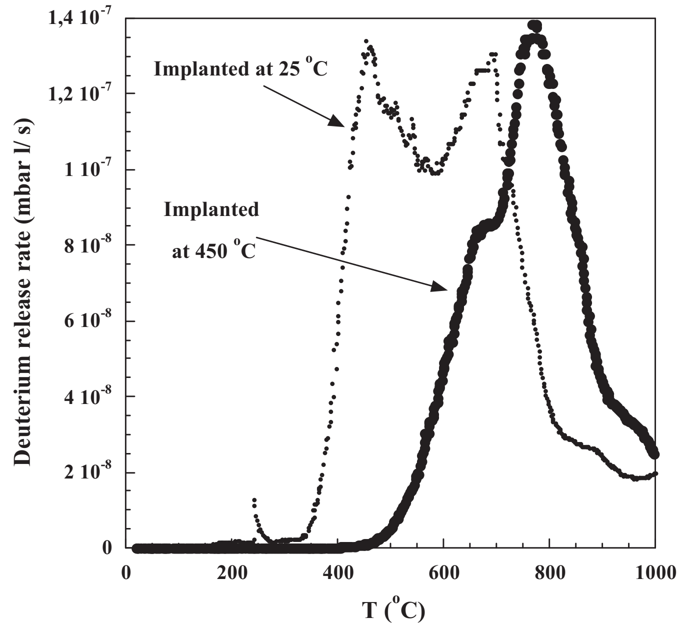

Fig. 2. TSD spectra for two RB–SiC samples implanted with 20 keV D^+ one at 25 °C and the other at 450 °C up to a dose of 1×10^{17} D/cm^2.

hexagonal phase and 10% free crystalline silicon, typical impurities; B, Al, N, Fe, Mo). Further microstructural characterization is given in a previous work [5]. Before being deuterium implanted all the samples were heated up to 1000 °C in vacuum (2×10^{-6} mbar) for 2 h in order to clean the sample of vapours, gases, and humidity.

Two of the samples were deuterium implanted at 25 °C with 20 and 50 keV respectively with deuterium ions (D^+), at 0.5 microamp/cm^2, up to a dose of 1×10^{16} ions/cm^2, at the CIEMAT Danfysik 60 kV ion implanter. In order to study both the dose and the temperature effect another two samples were implanted up to a higher dose of 1×10^{17} ions/cm^2 with 20 keV D^+ at 0.5 microamp/cm^2, one of them was implanted at 25 °C and the other at 450 °C.

Once deuterium implanted, each of the samples was consecutively mounted in a special cell provided with an oven which permitted to heat the samples from 25 up to 1000 °C. A helium leak detector was connected to the cell in order to measure the D_2 implanted into the samples. In this way TSD measurements were carried out. The sensitivity for this TSD measuring system was equivalent to $\geq 5 \times 10^{-12}$ (mbar l)/s or 0.13×10^8 D_2/s. For all the samples the heating rate during TSD measurements was 0.16 °C /s and the background pressure was 2×10^{-6} mbar.

Before and after the TSD measurements, the deuterium implanted samples were analysed using SIMS (Hiden Analytical, 5 keV O, 150 nA) to compare the amount of deuterium remaining as a function of depth, determined using a profilometer to measure the final sputter crater depth. For the higher implantation dose (1×10^{17} ions/cm^2) deuterium was clearly detected by SIMS meanwhile for the samples implanted at the lower dose (1×10^{16} ions/cm^2) it was not possible to detect the implanted deuterium as it was below the detection limit.

Results

In Fig. 1 one can see the corresponding TSD spectra for two D^+ implanted RB–SiC samples at 25 °C up to 1×10^{16} D/cm^2 one implanted with 20 keV D^+ and the other with 50 keV D^+. The spectra are very similar being dominated by a structure formed by three bands between 450 and 800 °C. One can see that this group of bands shift to higher temperature as the implantation energy is increased. It is expected that there is not an important influence of HD molecules as the background pressure was better than 2×10^{-6} mbar.

The TSD spectra for two RB–SiC samples implanted with 20 keV D^+ one at 25 °C and the other at 450 °C up to a dose of 1×10^{17} D/cm^2 is shown in Fig. 2. In the case of the implantation at 25 °C the spectrum shows a prominent desorption around 450 °C together with another band around 600 °C similar to the band observed for the lower implantation dose shown in Fig. 1 In the case of the implantation at 450 °C the spectrum is similar for those shown in Fig. 1 for the samples implanted at lower dose but in this case the maximum for desorption is shifted from about 600 to 800 °C.

In Fig. 3 one can see the SIMS D profile for two 20 keV D^+ implanted RB–SiC samples up to a dose of 1×10^{17} D/cm^2, one at 25 and the other at 450 °C. Both implantations agree with the expected implantation range obtained from SRIM [6]. The quantity of deuterium measured as D is higher for the lower implantation temperature.

In Fig. 4 the SIMS SiD profile for two 20 keV D^+ implanted RB–SiC samples up to a dose of 1×10^{17} D/cm^2, one at 25 and the other at 450 °C are shown. For the implantation at 25 °C the SiD profile is similar to those shown in Fig. 3 however one can clearly see that for the implantation at 450 °C SiD is observed for depths higher than the expected penetration range.

SIMS D profile for a RB–SiC sample implanted with 20 keV D^+ up to a dose of 1×10^{17} D/cm^2 at 25 °C both before and after being heated up to 1000 °C are shown in Fig. 5. One can see in Fig. 5 that the total integrated D quantity measured by SIMS after heating up to 1000 °C is about 30% lower than the total integrated D quantity measured by SIMS before heating.

Fig. 6 shows the SIMS D profile for a RB–SiC sample implanted with 20 keV D^+ at 450 °C up to a dose of 1×10^{17} D/cm^2 both before and after being heated up to 1000 °C. Nearly no change is observed after heating the sample up to 1000 °C.

In Fig. 7 it is shown the SIMS SiD profile for a RB–SiC sample implanted with 20 keV D^+ at 25 °C up to a dose of 1×10^{17} D/cm^2

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