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Influence of radiation damage on the thermal properties of silicon carbide implanted with heavy noble gas ions

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ABSTRACT

Diffusion of heavy noble gas atoms in irradiation damaged single crystalline silicon carbide and the thermal etching of it is investigated at temperatures of 1300 °C and 1400 °C. For this purpose 360 keV krypton and xenon ions were implanted in commercial 6H-SiC wafers at 600 °C, which is far above the critical amorphization temperature of the target material. Width broadening of the implantation profiles and the retention of krypton and xenon during isothermal annealing was determined by RBS-analysis, whilst damage profiles were simultaneously obtained by α-particle channelling. No diffusion and no loss of the implanted species is detected in the implanted samples after isothermal annealing for 40 h at 1400 °C. However, thermal etching of the target material is observed at both annealing temperatures and leads at 1400 °C to a significant shift of the implantation profile towards the surface due to sublimation. RBS analysis shows that this occurs mainly during the initial stage of isothermal annealing, while surface loss during prolonged annealing is minimal. The resulting topographical modification of the surface during annealing was studied by scanning electron and atomic force microscopy. It indicates that the observed phenomenon is due to a relatively strong dependence of thermal etching on the defect density in the surface region, while the evolving surface roughness seems not to play a decisive role.

1. Introduction

Development of modern high-temperature gas-cooled nuclear reactors (HTGR's) is taking place world-wide within the framework of the *International Generation IV Reactor Program*. These reactors normally employ fuel elements containing triple isotropic (TRISO) fuel particles, consisting of fuel kernels encapsulated by four successive layers of low-density pyrolitic carbon, high-density pyrolitic carbon, silicon carbide and high-density pyrolitic carbon. In this configuration the silicon carbide cladding plays a key-role concerning the retention of fission products inside the fuel particle. Up to 1000 °C, which is the typical operating temperature of HTGR's currently in use, most of the important fission products are retained [1]. However, in order to enhance the efficiency, some designs propose operating temperatures significantly above 1000 °C.

As very little information on transport properties above this temperature were available, our group started a systematic investigation on diffusion behaviour in silicon carbide at temperatures up to 1500 °C, by studying implantation profile broadening of various atomic species during annealing. At the upper end of this range, not only broadening, but a shift of the whole profile towards the surface was observed. Such a shift can be the result of either thermal etching [2] or a Markov-type diffusion process [3] in a non-uniform medium. Due to irradiation induced damage the target material is indeed non-uniform with defect densities extending far beyond the ion ranges. Experimental evidence shows enhanced diffusion in radiation damaged silicon carbide. Hence, a shift deeper into the target would be expected. The observed forward shift can therefore only be explained by thermal etching. This could eventually destroy the silicon carbide layer of the fuel particles, exposing the fission products accumulated in the kernel. To get more insight in this matter, we investigate in this work the behaviour of krypton and xenon implants in 6H-SiC during isothermal annealing at temperatures of 1300 °C and 1400 °C. The heavy noble gas implants were chosen, because previous work showed that no Gaussian diffusion is observable at these temperatures [4,5].

2. Experiment and analysis

Hexagonal 6H-SiC wafers from *Intrinsic Semiconductors®* were used in this study. Krypton and xenon ions were implanted at 600 °C with an energy of 360 keV and fluences of 2×10^{16} cm^2 and 1×10^{16} cm^2, respectively. The high implantation

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temperature, which is far above silicon carbide's critical amorphization temperature of 350 °C [6], was chosen to ensure that the basic crystal structure is retained. Ion flux was kept below 10^{13} cm^{-2} s^{-1} to prevent target heating. RBS-analysis before and after isothermal vacuum annealing at 1300 °C and 1400 °C was employed to determine widths and projected ranges of the implantation profiles. These measurements were combined with the results of α-particle channeling spectroscopy along the *c*-direction to obtain defect density profiles as a function of implantation and annealing temperatures. A detailed description of the experimental techniques is given elsewhere [7].

Diffusion coefficients were extracted from the broadening of implantation profiles during isothermal annealing. Assuming a Gaussian depth distribution the following relationship between the final and original widths holds [8]:

$$[W(t)]^2 = 4Dt\ \ln(2) + [W(0)]^2$$

$W(t)$ is the full width at half-maximum after annealing for time t. Hence, the diffusion coefficient D at temperature T_a is directly obtained from the slope of a plot of $[W(t)]^2$ versus annealing time t.

For the determination of defect density profiles, Rutherford backscattering spectra were measured for random and aligned incidence along the (0001)-direction, using 1.44 MeV and 1.65 MeV α-particles. Irradiation damage in the 6H-SiC samples consists of uncorrelated and correlated displaced atoms due to point defect clusters/amorphous regions and extended defects respectively. The computer code DICADA1 [9], can treat a mixture of uncorrelated point defects and dislocation loops, but is applicable only to mono-elementary targets. However, by introducing certain approximations it can be successfully applied to a binary target as shown and discussed in Ref. [4]. A similar approach is also used in this work, assuming a Gaussian density distribution of dislocation loops with z_{max} = 200 nm and σ = 40 nm.

Structural information on the samples before and after annealing was obtained by scanning electron microscopy (SEM), employing a Zeiss Ultra 55 field emission scanning electron microscope with an in-lens detector as discussed in Ref. [10]. Topographical surface profiles were determined by atomic force microscopy (AFM) employing a *Bruker's Dimension Icon* scanning probe. Surface roughness was measured along a number of different lines across the sample to obtain average values and their statistical uncertainties.

3. Results and discussion

The extracted defect density profiles before and after isothermal annealing at 1300 °C and 1400 °C are shown for the krypton and xenon implants in Figs. 1 and 2, respectively. Also shown are implantation profiles obtained by TRIM simulations [11]. In contrast to room temperature implantations into silicon carbide, which form an amorphous surface region with a thickness in which the implanted species are completed embedded [4,5], the basic

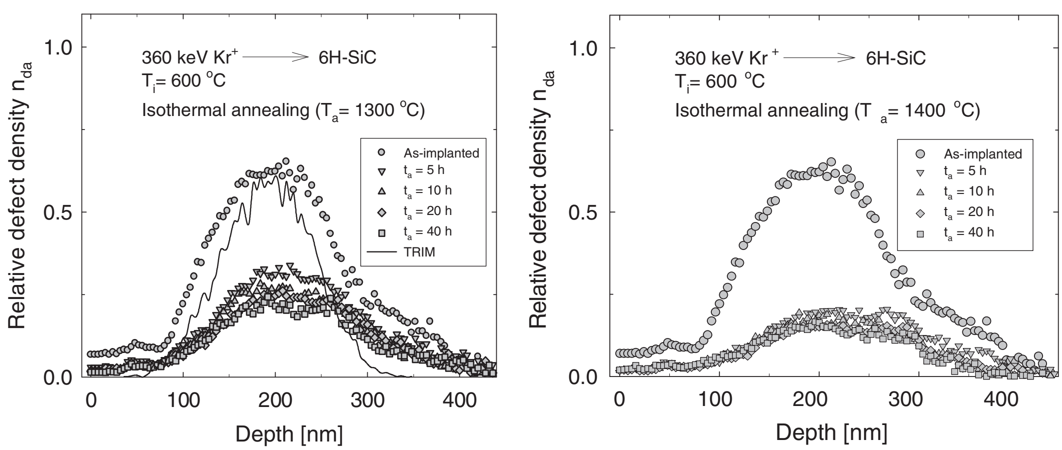

Fig. 1. Uncorrelated defect density distributions of krypton implanted 6H-SiC before and after isothermal annealing at 1300 °C and 1400 °C. The solid line in the left graph shows the implantation profile obtained by TRIM simulation [11].

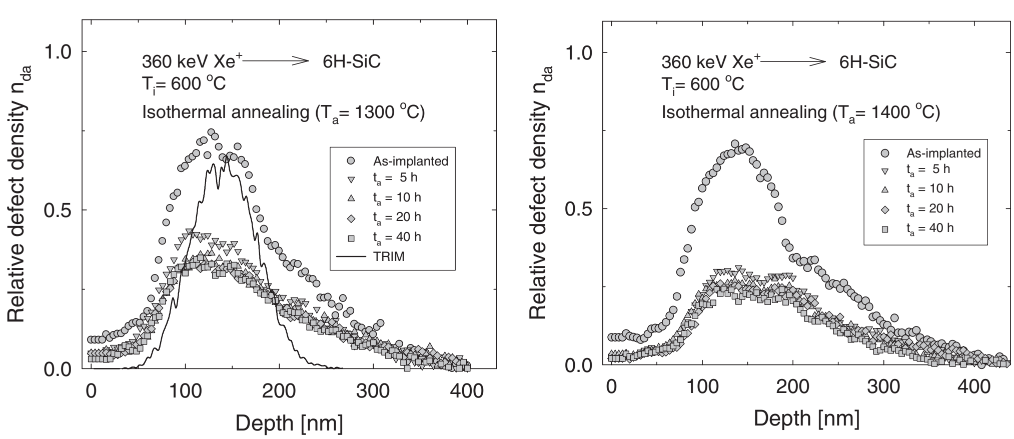

Fig. 2. Uncorrelated defect density distributions of xenon implanted 6H-SiC before and after isothermal annealing at 1300 °C and 1400 °C. The solid line in the left graph shows the implantation profile obtained by TRIM simulation [11].