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Dose dependence of helium bubble formation in nano-engineered SiC at 700 °C

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ABSTRACT

Knowledge of radiation-induced helium bubble nucleation and growth in SiC is essential for applications in fusion and fission environments. Here we report the evolution of microstructure in nano-engineered (NE) 3C SiC, pre-implanted with helium, under heavy ion irradiation at 700 °C up to doses of 30 displacements per atom (dpa). Elastic recoil detection analysis confirms that the as-implanted helium depth profile does not change under irradiation to 30 dpa at 700 °C. While the helium bubble size distribution becomes narrower with increasing dose, the average size of bubbles remains unchanged and the density of bubbles increases somewhat with dose. These results are consistent with a long helium bubble incubation process under continued irradiation at 700 °C up to 30 dpa, similar to that reported under dual and triple beam irradiation at much higher temperatures. The formation of bubbles at this low temperature is enhanced by the nano-layered stacking fault structure in the NE SiC, which enhances point defect mobility parallel to the stacking faults. This stacking fault structure is stable at 700 °C up to 30 dpa and suppresses the formation of dislocation loops normally observed under these irradiation conditions.

1. Introduction

Because of its low neutron capture cross-section, outstanding chemical and thermal stability, silicon carbide (SiC) is considered as a promising candidate for structural component and cladding material in the next generation fusion and fission nuclear reactors [1–3]. For advanced nuclear applications, SiC will be exposed to high temperatures, intense fluxes of fast neutrons, and the production of helium and hydrogen gas atoms from nuclear reactions. In advanced fission reactors, the helium generation rate is reported to be 2.5 appm He/dpa [4]. In a fusion reactor, SiC will be exposed to not only a high irradiation dose (up to 200 dpa), but also high helium generation rates (70 appm/dpa in the reactor, 130 appm/dpa for the first wall) [5–7]. The damage accumulation from high temperature irradiation may lead to different types of defects that will induce hardening effects, microstructure evolution and volume swelling [8]. In order to enhance the high temperature radiation tolerance, the response of different types of SiC to radiation damage has been investigated both computationally [9–14] and experimentally [7,8,15–17]. Ion irradiation is widely employed in experimental studies since it provides an effective approach [18–20] to investigate the irradiation response of SiC to high irradiation dose and high helium accumulation at controlled temperatures. Some promising paths for improving radiation resistance, such as grain refinement [21], fiber-bonded reinforced composites [22,23], and increase volume fraction of grain boundaries [24,25], have been reported. Composites made with the advanced fibers of Nicalon Type S and the UBE Tyranno SA retain dimensional stability and have negligible change in strength up to 10 dpa at 800 °C [5]. SiC composites also show great irradiation resistance under neutron irradiation to doses of 30–40 dpa at 300–800 °C [26]. However, the

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irradiation performance of the composite is strongly affected by the degradation of interfacial shear properties [27]. Recent investigations have shown that nano-engineered (NE) SiC, which contains high-densities of stacking faults (SFs) with nm spacing formed within a nano-sized grain structure, can confine three-dimensional random point defect migration to a two-dimensional like movement parallel to the SFs [24,28]. As illustrated in Fig. 1, the irradiation-induced defects encounter higher migration barriers across the SFs; thus point defects can either migrate two-dimensionally parallel to the SFs or recombine within a short distance. As a result, this nano-engineered structure enhances the rate of point defect annihilation, leading to improved radiation tolerance of SiC.

Helium pre-implanted NE SiC is employed in this study to investigate subsequent heavy ion irradiation on bubble formation. Although two-dimensional diffusion is supported by density functional theory calculations [25], the nucleation and growth of helium bubbles in NE SiC, as well as the irradiation response and stability of this novel structure at high temperature, are not well understood. Also, the high-density of SFs with average spacing of 1–2 nm could result in significant defect migration and bubble coarsening along grain boundaries, especially under high dose heavy ion irradiation. Such bubble growth at grain boundaries may lead to degradation of mechanical properties. In our previous study [29], helium bubble formation was not observed in NE SiC following helium implantation at 277 °C nor after thermal annealing at 700 °C for 1 h. Helium bubbles of measurable size (up to 5 nm) were only observed after Au ion irradiation to 10 dpa at 700 °C. Consequently, we in the present work investigate the evolution of helium bubbles in NE SiC at much higher doses under prolonged irradiation (up to 30 dpa) at 700 °C in order to evaluate helium bubble growth and microstructural evolution, and to gain insight on the performance of NE SiC under extreme irradiation environments.

2. Experimental methods

NE SiC with a high density of <111>-type SFs (spacing of 1–2 nm) within columnar grains of 100–300 nm size were grown along the <111> direction on (1 0 0) silicon substrates using low-pressure chemical vapor deposition. Processing techniques and fabrication of these NE SiC thin films are described elsewhere [29]. The average thickness of the NE SiC films is 530 nm. In this study, the corresponding implanted helium concentration and dose in displacements per atom (dpa) were calculated using Stopping and Range of Ions in Matter (SRIM) full-cascade simulations (version 2012) [30,31]. The parameters used in the simulations are threshold displacement energies of 20 and 35 eV for the C and Si atoms, respectively [32], and a sample density of 3.21 g cm^{-3}.

As the first step, helium ion implantations were performed using the 200 kV ion-implanter at Los Alamos National Laboratory (LANL) [33]. The SiC thin films were implanted with 65 keV helium ions to fluences from 1 × 10^{15} to 1 × 10^{16} ions cm^{-2} at a temperature of 277 °C, which is above the critical temperature for amorphization [34]. The helium concentration peak was located at 320 nm from the surface, as illustrated in Fig. 2. After the helium ions were implanted, subsequent heavy ion irradiations were carried out in a multi-purpose target chamber using the 3.0 MV tandem accelerator facilities in the Ion Beam Materials Laboratory at the University of Tennessee [35].

The ion irradiations were conducted using 9 MeV Au^{3+} ions at 700 °C to ion fluences of 1.5 × 10^{16} (average dose of 20 dpa) and 2.3 × 10^{16} ions cm^{-2} (30 dpa) to produce a relatively flat-damage profile at the helium peak region (depth of 270–390 nm) under

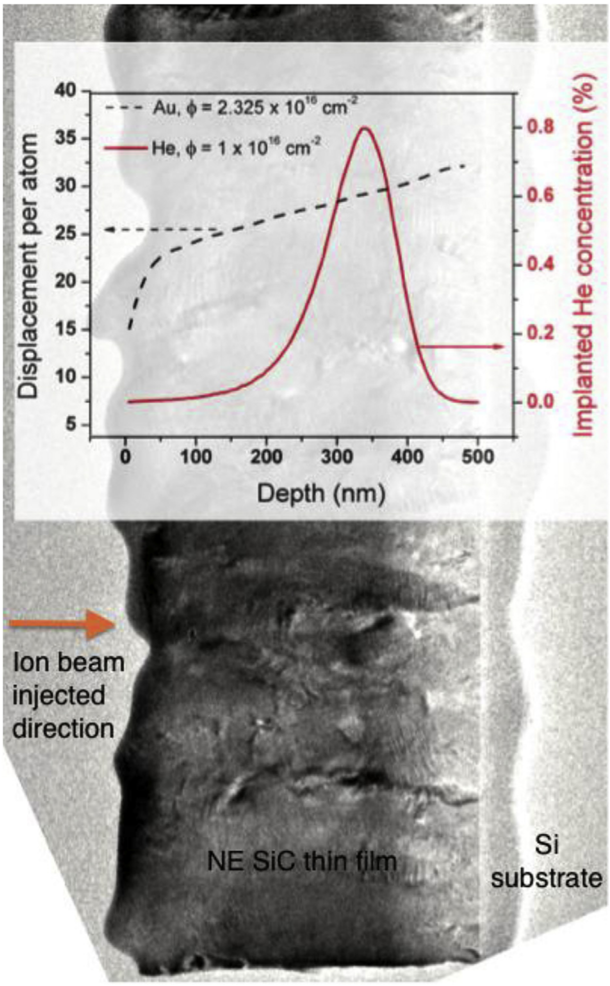

Fig. 2. SRIM 2012 simulation of the irradiation damage prediction and implanted helium concentration in NE SiC. The depth profile of SiC film on Si substrate is shown in background TEM image.

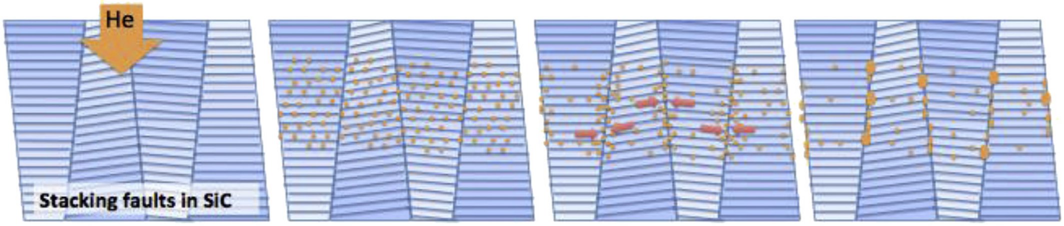

Fig. 1. Defect diffusion schematics. Migration occurs mainly between SFs, following a two-dimensional process in NE SiC. Since SFs prevent defect migrating across layer parallel to surface, helium atoms may tend to diffuse to grain boundaries.