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# Defect Microstructure in Irradiated Silicon Carbide

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Recent interest in silicon carbide (SiC) and its composites (SiC/SiC composites) has been motivated historically by its possible utilization as a structural and functional material in fusion blankets in 1970s [1]. They are now attracting attention as alternative materials for fuel cladding for operating fission reactors (LWR) because of the conceivable better chemical stability and strength under LOCA or beyond design basis conditions in addition to the fusion and gas cooled reactors [2]. The advantages include perceived radiation stability at temperatures <1000 °C that comes from retention of radiation produced nano-structured defects up to high fluences. The knowledge regarding the microstructural change in SiC during irradiation has thus far been mainly limited to the low-to-intermediate temperature regime, and has hampered the determination of upper temperature and fluence limits for the severe use conditions now being considered. Important rapid property changes are anticipated at very high temperatures, where irradiation induced microstructural defects undergo unstable growth. This paper will review our recent results on the evolutions of dislocation and void microstructures in ion- or neutron-irradiated β-SiC, especially for the high temperatures beyond the upper service temperature limits for metallic heat resistant structural alloys. Some of advanced techniques on the defect analysis will also be touched on.

The literature offers only limited microstructural information on β-SiC subjected to high temperature neutron irradiation until late 1990. Price (1973) first observed irradiation induced void formation in SiC deposited on graphite discs, the irradiation condition of void formation was neutron fluence of $4.3 \times 10^{25}$ n/m$^2$ (E>0.18 MeV) and an irradiation temperature of 1250 °C or higher [3]. Price (1969) and Blackstone (1971) et al. reported macroscopic volumetric expansion in neutron irradiated SiC at high temperatures (~1500˚C) [5]. No information on the relation between microstructure and macroscopic length change in irradiated β-SiC, however, has been available until not long ago. Several self-ion irradiation experiments also reported cavity formation in SiC, where cavities were observed at >1000 °C [4]. Recently, Snead et al. reported the temperature and fluence dependent swelling in very high purity SiC (produced through chemical vapor deposition (CVD)) in the high temperature regime [6], and formation of voids was considered the probable cause of the swelling. Our work clarified that cavities were predominantly spherical in shape below 1300 °C. Additional voids faceted with {111} planes were dominating at 1460 °C, which were basically tetrahedral truncated at the corners with {111}. The tetrahedral shape was unexpected as the surface-to-volume ratio is larger than the alternative {111} octahedral void common in both metals and ceramics. From a geometric viewpoint, all faces of the observed voids are either Si- or C-terminated surfaces. By comparing the surface area with the octahedral void (composed of the both Si- and C-surfaces) of the same volume, the considerable difference in surface energy between the Si(111) and C(-1-1-1) was implicated [7]. One can conclude now that those are responsible for the high temperature swelling in SiC.

Other microstructural features in neutron-irradiated SiC are black spot defects and/or small dislocation loops (r~3nm) after irradiation at relatively low fluences between 300 and 1100 °C. The small dislocation loops were identified as lying on {111} lattice planes, and have been tentatively identified as Frank loops without Burgers vector analysis. Recently, Katoh et al. (2006) suggested that these were a mixture of Frank loops and other type defect clusters, for example having Burgers vector of Shockley partial, by means of Burgers vector analysis for CVD SiC irradiated to $7.7 \times 10^{25}$ n/m$^2$ (E>0.1 MeV) at 800°C. No evidence was reported because of their small size, but these defects were believed to be interstitial type based on difference in the mobilities of interstitials and vacancies in SiC. A high-resolution electron microscope image of a relatively larger planner defect on (111) in heavy-neutron irradiated SiC has been estimated to be an interstitial-type Frank loop by image simulation [8]. In our work, large dislocation loops were identified as interstitial-type, $a_0/3$<111> Frank loops using the inside/outside contrast method. It would appear that small loops at 1130 ˚C were also Frank loops: at least we confirmed the habit plane was {111} and their Burgers vector was parallel to <111>.

In order to obtain the information about the interstitial motion in SiC, the temperature-dependent width of the interstitial loop denuded zone (DZ) formed along the random grain boundaries was evaluated [10]. The quantitative analysis showed a positive temperature dependence of the DZ width, where the smallest DZ width of 8.9 nm was observed in SiC irradiated at 1010 °C and the largest of 57 nm was observed at 1380 °C. Significant populations of small TEM invisible voids ($r_v$ = 0.2–0.7 nm) were theoretically found to be formed in specimens irradiated below 1130 °C based on a simple reaction-diffusion equation, which were supposed to be limiting the interstitial motion at lower temperatures. The temperature-dependent diffusion coefficient estimated from the loop-denuded width showed an activation energy of interstitial migration of $1.5 \pm 0.1$ eV in SiC, which is likely associated with the slower-moving species of Si interstitials. Finally, we concluded that the excellent irradiation stability of SiC is attributed to its stable microstructural defects due to the very high sink strength of vacancies even at high temperature and fluence. Advanced analytical technique in an electron microscopy may be best for understanding the ceramic specific features of the irradiation defects in a covalent bonding SiC, such as the stoichiometry constraint of the loops and the being of charged defects.

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