## Recovery Behavior of High-Purity Cubic SiC for First-Wall Applications in Fusion Reactors by Post-Irradiation Annealing After Low-Temperature Neutron Irradiation

Abstract

Two types of high-purity cubic ( $\beta$ ) SiC polycrystals, PureBeta-SiC and CVD-SiC, were irradiated in the BR2 reactor (Belgium) to a fluence of  $2.0-2.5 \times 10^{24}$  (E > 0.1 MeV) at temperatures ranging from 333 to 363 K. Changes in macroscopic length were analyzed through post-irradiation thermal annealing using a precision dilatometer, employing a step-heating method up to 1673 K. Each specimen was held at a given temperature for 6 hours, and length variations were recorded during isothermal annealing from 373 K to 1673 K in 50 K increments. The recovery curves were evaluated using a first-order model, and rate constants were determined for each annealing step.

Defect recovery in neutron-irradiated high-purity  $\beta$ -SiC occurred in four distinct stages, each associated with different activation energies. In the range of 373–573 K, the activation energy for PureBeta-SiC was 0.17–0.24 eV, while for CVD-SiC, it was 0.12–0.14 eV. At 723–923 K, the values were 0.002–0.04 eV and 0.006–0.04 eV, respectively. In the 923–1223 K range, activation energies were 0.20–0.27 eV for PureBeta-SiC and 0.26–0.31 eV for CVD-SiC. At higher temperatures (1323–1523 K), the values increased to 1.37–1.38 eV and 1.26–1.29 eV, respectively.

Below approximately 1223 K, defect recombination likely occurred between closely spaced C and Si Frenkel pairs, without the need for long-range migration. This mechanism accounted for nearly three-fourths of the total neutron irradiation-induced recovery. Additionally, at 1323–1523 K, recombination of slightly separated C Frenkel pairs and increased long-range migration of Si interstitials may have taken place in both PureBeta-SiC and CVD-SiC. The migration of both vacancies appeared to be restricted up to ~1523 K. Compared to hexagonal  $\alpha$ -SiC, high-purity  $\beta$ -SiC exhibited faster recovery, particularly at lower annealing temperatures below 873 K, with the most significant recovery occurring below 573 K.

Numerous reports of silicon carbide (SiC) show outstanding features in thermal and mechanical properties, such as thermal stability at temperatures above 1273 K, and excellent mechanical strength (>600 MPa). Due to its excellent properties, it had been used as an important material in tristructural-isotropic (TRISO) fuel of high temperature gas-cooled reactors. This type of a ceramic exhibits tremendous stability in a harsh radiation environment, induced by fission products and neutrons in reactors. Moreover, SiC is used in internal combustion engines, magneto hydrodynamic (MHD) generators, power electronic semiconductors, transport system components, and first wall in fusion reactor. Nevertheless, the main flaw of monolithic SiC is its intrinsic brittle nature, leading to a high risk of failure under excess load.

Hence, to ensure enhanced reliability with increasing damage tolerance, the proposed materials for future structural components were SiC fiber-reinforced SiC composites (SiC<sub>f</sub>/SiC) which composed of infiltrated SiC matrix into SiC fiber preform where it can be woven into 2D or preferably 3D fabric. SiC<sub>f</sub>/SiC composites are fabricated for improving radiation resistance, and are being considered as an attractive and promising material for light water reactor (LWR) fuel cladding and channel box, gascooled fast reactors components, and future blanket material for fusion energy system. However, a fusion reactor environment near the first wall is considerably severe for materials, due to a wide energy range of ions, an extreme heat and electromagnetic flux, and a crucial dose of penetrating neutrons. In addition, the most significant threat to structural materials is fusion neutrons, which are able to trigger the atomic displacement and the production of gaseous and solid transmutation products.

These irradiation effects are potentially liable for degradation of material performance of SiC and SiC<sub>f</sub>/SiC. Thus, in order to understand the fundamental mechanical properties of SiC and SiC<sub>f</sub>/SiC composites after neutron irradiation, the dimensional change, lattice parameter and microstructure of these materials should be measured and examined in detail. Furthermore, physical property changes of SiC during nuclear operation, particularly with elevated temperatures, can be determined through observation of recovery behavior by postirradiation annealing of lower temperature irradiated specimens. Seven of monolithic SiC or SiC<sub>f</sub>/SiC composite specimens (PureBeta, Chemical Vapor Deposition (CVD)-SiC, Liquid Phase Sintering (LPS)-SiC, Nano-powder Infiltration and Transient Eutectoid (NITE) A and B, SiC<sub>f</sub>/SiC of Chemical Vapor Infiltration (CVI), NITE and Electrophoretic Deposition (EPD) methods) with different fabrication processes were irradiated in the BR2 reactor (Belgium) up to a fluence of 2.0-2.5 x  $10^{24}$  (E > 0.1 MeV) at 333-363 K. Subsequently, changes in macroscopic length and lattice parameter were measured, and observation of microstructure before and after the neutron irradiation were conducted for clarifying basic irradiation effects on each material.

Additionally, recovery behavior assessment by post-irradiation annealing was conducted to clarify recovery stages of high purity monolithic b-SiC (PureBeta and CVD-SiC) polycrystals. Post-irradiated isochronal annealing up to 1673 K and in-situ length change measurement at 6 h isothermal annealing with increment of 50 K from room temperature was performed using a precision dilatometer to confirm recovery stages in high purity b-SiC.