

5th International Symposium on Innovative Nuclear Energy Systems, INES-5, 31 October – 2 November, 2016, Ookayama Campus, Tokyo Institute of Technology, JAPAN

Recovery behavior of SiC_f/SiC composites by post-irradiation annealing up to of 1673 K

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Abstract

Silicon carbide fiber-reinforced silicon carbide composite (SiC_f/SiC) has been proposed as a satisfied material to accommodate an extreme and corrosive irradiation ambience such as first wall of fusion reactors. Chemical Vapor Infiltration (CVI) and Electrophoretic Deposition (EPD) are the processes those have been conducted in order to fabricate the composites. In this study, CVI-SiC and EPD-SiC composites were neutron-irradiated in the BR2 reactor up to a fluence of $2.0\text{--}2.5 \times 10^{24}$ n/m² ($E > 0.1$ MeV) at 333–363 K. Heat treatments were carried out with step-heating method in a precision dilatometer in order to understand their recovery behavior. The specimens were held each temperature step for 6 h. Length change was recorded during each isothermal annealing step from room temperature up to 1673 K with 50 K increment. Between 1473 K and 1523 K, the EPD composite recovered abruptly and completed. However, the CVI specimen did not recover completely even though it was heated up to 1673 K. The recovery curves were analyzed with a first order model, and rate constants at each annealing step were obtained. Activation energies for both specimens were generally increased with increasing the annealing temperature, except for stage 2 in the CVI and stages 2 and 3 in the EPD specimens. Compared to the activation energies of monolithic SiC, nearly similar values were observed except for the activation energy at stage 4. So, the recovery processes of stage 4 for both specimens and stage 3 in the EPD specimen were different from those of high-purity monolithic SiC.

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Peer-review under responsibility of the organizing committee of the 5th International Symposium on Innovative Nuclear Energy Systems.

Keywords: SiC/SiC composite; neutron irradiation; swelling; recovery behavior; activation energy

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1. Introduction

Silicon carbide (SiC) has excellent properties such as low activation and short half-life by neutron irradiation, good thermal conductivity, resistance in high temperature condition and physical stability in severe neutron environment. Thus, it has been used as one of the layers in tristructural-isotropic (TRISO) fuel of high temperature gas-cooled reactors to retain fission products at elevated temperatures [1,2]. However, silicon carbide fiber-reinforced silicon carbide composites (SiC_f/SiC) have been proposed for future nuclear applications because of avoiding brittle nature of monolithic SiC. SiC_f/SiC composites are stable in an extreme and corrosive irradiation ambience and have become a candidate material for the first wall of fusion reactors. Newsome et al. [3] reported that SiC_f/SiC composites were less swelled (1.4%) compared to CVD SiC (2.0%) after exposure to neutron irradiation up to 1 dpa at 573 K. In addition, our previous report stated that SiC_f/SiC composites fabricated by chemical vapor infiltration (CVI), nano-infiltration transient eutectoid (NITE) and electrophoretic deposition (EPD) were swelled approximately 1.0%, compared with that of the monolithic SiC of 1.3% after the irradiation by intermediate neutron fluence (0.25 dpa) at 363 K [4].

It is learned that crystalline defects introduced into SiC and SiC_f/SiC composites by neutron irradiation particularly in nuclear reactor caused swelling and would degrade their mechanical and thermal properties, hence, lifetime of these ceramics would be less. Moreover, it is understood that the swelling of SiC and SiC_f/SiC composites decreases by increasing the irradiation temperatures [5]. It is believed that recombination of interstitial atoms and vacancies occurred more frequently in irradiated SiC during neutron irradiation at elevated temperatures. Therefore, the swelling is smaller and accumulation of defects in SiC could be lesser during high temperature irradiation lower than 1273 K. Furthermore, study on recovery behavior of neutron-irradiated high-purity SiC and SiC with sintering additives showed the recombination occurred for closely positioned C and Si Frenkel pairs below 1223 K. In addition, at 1323–1523 K, recombination of slightly separated C Frenkel pairs and more long-range migration of Si interstitials may have occurred. By determining the activation energy, both monolithic SiC specimens have similar recovery mechanism. However, the irradiated SiC ceramics containing 12 wt.% and 18 wt.% of sintering additives were shrunk by thermal annealing whereas they did not recover completely owing to the crystallization of secondary phases by annealing up to 1673 K [7,8].

Recovery behavior of neutron-irradiated monolithic SiC that constitutes the matrix of SiC_f/SiC composites is relatively well understood by post-irradiation annealing method. Similar technique had been used in this study on SiC_f/SiC composites to elucidate the recovery behavior at elevated temperatures. In this study, the neutron-irradiated SiC_f/SiC composites fabricated by CVI and EPD were annealed from room temperature (RT) up to 1673 K in a precision dilatometer. The stability of defects in the SiC matrix and SiC fibers could be estimated from the length change during isothermal annealing. Furthermore, the effects of the sintering additives in the EPD specimen on their recovery behavior were examined and compared with those of the high-purity SiC and SiC with sintering additives [7,8]. In fusion reactors, the neutron dose can reach up to 200 dpa at elevated temperatures (1273 K). However, this study was conducted to understand the fundamental irradiation effects of SiC_f/SiC composites.

2. Experimental procedures

2.1 Materials

Two types of SiC_f/SiC composites fabricated by CVI and EPD processes were evaluated in this study. Commercially available near stoichiometric SiC fibers (Tyranno™ SA3 fiber, Ube Industry, Japan) were used for the CVI composite. This composite was manufactured by Hyper-Therm High-Temperature Composite, Inc. (Huntington Beach, CA, USA). In the CVI process, SiC matrix was deposited from gaseous reactants on a heated SiC fiber preform at temperatures lower than 1473 K (<0.5 atm). The fiber was coated with a layer of pyrolytic carbon (PyC) of nominally 150 nm thickness using an isothermal isobaric CVI process as proper fiber/matrix interface. No sintering additives were used to fabricate the composite. Then, it was cut into bar shape of 25 mm x 2 mm x 2 mm in size.

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