Ion irradiation effects on the matrix phase of SiC_f/SiC composites prepared by the whisker growing assisted CVI process

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1. Introduction

SiC_f/SiC composites are one of promising candidates for structural material of the next generation energy system such as GFR and fusion reactors [1]. A number of fabrication methods have been studied for obtaining an outstanding SiC_f/SiC composite with a high density, high crystallinity and purity. SiC_f/SiC composites consisted of whisker-reinforced matrix have a great potential at the viewpoint both of the fabrication process and the mechanical properties. SiC whiskers formed between SiC fibers improve the densification of SiC matrix during CVI process. In addition, the reinforced whiskers would be likely to enhance the mechanical properties of matrix and SiC_f/SiC composite. While there has been significant developmental work on manufacturing the SiCf/SiC composite by the whisker growing assisted CVI process, detailed understanding of what effects the complex in the operating conditions combined with realistic materials property data is not adequately understood. Especially, its irradiation effects are even less clear and not well understood. A method of charged-particle irradiation is the most important R&D topics for simulating the core conditions of the advanced nuclear systems. Many studies on radiation effects of SiC and SiC_f/SiC composites using a method of ion irradiation have in progress for R&D of the advanced nuclear systems [2, 3].

In this present work, changes of the mechanical property of SiC whisker-reinforced matrix in SiC_f/SiC composite were evaluated by means of the depth-sensing indentation method before and after charged-particle irradiation.

2. Experimental Procedure

The material for ion irradiation was a SiC_f/SiC composite with the SiC whisker-reinforced matrix, which was produced by CVI process. Reinforced SiC whiskers was grown using a gas mixture of methyltrichlorosilane (CH_3SiCl_3, MTS, Aldrich Co., 99%) and purified H_2 (purity: 5 N) where H_2 acted both as a reducing agent and a carrier gas for MTS vapor. To obtain SiC_f/SiC composite with SiC whisker reinforced matrix, the sequential and repeated processes were performed, including pyrocarbon coating on SiC fiber, SiC infiltration, whisker growth and infiltration of SiC matrix. Fig. 1 shows the microstructure of whiskers grew in the spaces between SiC fibers and the whiskers exhibit diameters with submicron scale. The detailed description of this process had reported in elsewhere [4].

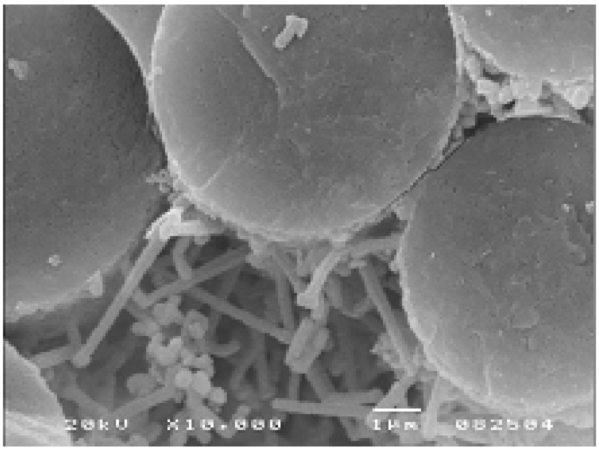

Fig. 1 Microstructures of grown whiskers inter SiC fibers in SiC/SiC composites.

Self-ion irradiation experiments were conducted by accelerator (a NEC model 5SDH-2, a tandem Van de Graaff) with the conditions of 5.1 MeV Si^{2+} ions (1.7 MeV terminal voltages). The irradiation temperature, displacement damage rate and total dose were room temperature, 2.24×10^{-3} dpa/s, and 20 dpa, respectively. Fig. 2 shows the displacement damage level (dpa; displacement per atom) and the Si ion distribution with the depth from the surface of SiC. The profiles were calculated by SRIM2003 code [5] assuming the average displacement threshold energy of 35 eV, stoichiometric chemical composition and a mass density of 3.21 g/cm^3. The total dose of ion-irradiated SiC/SiC composite was determined by selecting the average value of displacement per atom. The 20 dpa indicates at the depth of 1400 nm.

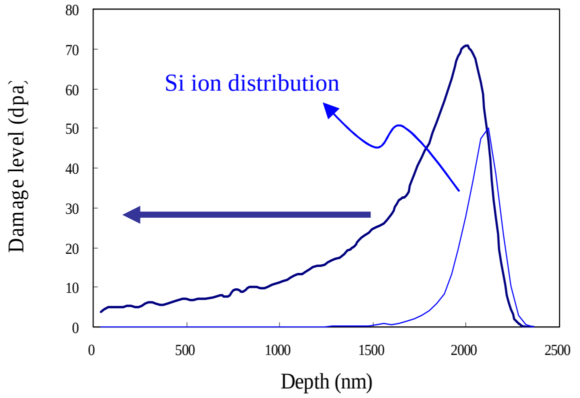

Fig. 2 Profiles of displacement damage (dpa), and distribution of injected silicon ions.

The evaluation of the mechanical property of SiC whisker-reinforced matrix before and after ion irradiation was carried out by the depth-sensing indentation method with the nano-indentation device (NanoTest, Micro Materials Ltd.,) and a Berkovich diamond tip. The contact depth (h_c) was controlled below 250 nm, which would cause the actual indenter-depth of 125 nm because of blunting of the diamond tip. The indentation-induced deformation is formed to 10 times depth compared to that of the penetration depth of actual indenter. The evaluation method under these test conditions provides a reliable data of the ion-irradiated materials. The relation between the deformation range and the penetration depth of actual indenter was explained in elsewhere [6].

3. Results and Discussion

Fig. 3 shows the load-depth curves obtained by one complete indentation process. The load-depth (*P*-*h*) relationship was determined by the nano-indentation test that was capable of measuring the sample hardness and modulus from the initial portion of the unloading curve. In Fig. 3, apparently decreased contact depth (h_c) was exhibited in the ion-irradiated sample under the same applied load of 50 mN. The narrower area surrounded by the loading-unloading curve was identified in the ion-irradiated sample. This behavior exhibits an obvious evidence of hardness increase by the ion irradiation. The slope (stiffness, *S*) of the initial portion of the unloading curve is involved in the indentation modulus. Generally, a steep slope manifests the lower modulus compare to a gentle slope. Therefore, we can carefully suppose the modulus increase of SiC whisker reinforced matrix by ion irradiation. The decreased indenter depth (contact depth, h_c) may be related to the difficulty of dislocation migration, which causes restriction of the growth of deformation zone.

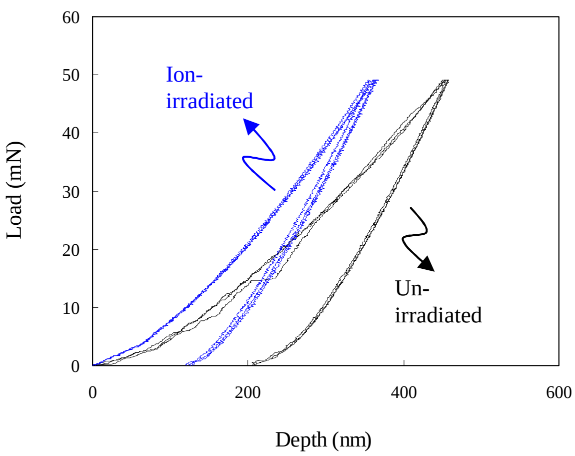

Fig. 3 The load-depth curves of SiC matrix reinforced with SiC whiskers before and after Si ion irradiation. The stiffness of the virgin and the ion-irradiated material is 0.273 and 0.261 mN/nm, respectively.

As can be expected from reference survey [7], amorphization of SiC is occurred by ion irradiation at room temperature. However, no data on radiation effects of SiC whisker reinforced SiC material in connection with amorphization is exist. Also, mechanical property changes of pure SiC after radiation-induced amorphization is not exist. The more studies on radiation effects of SiC and whisker-reinforced SiC/SiC composite are essential.

4. Conclusions

The indentation load-depth curve of SiC whisker-reinforced SiC matrix was clearly different by the ion irradiation. The decrease of indenter contact depth (h_c) and stiffness was exhibited after the ion irradiation. The ion irradiation seemed to create a stress concentration in the target material, and the more analysis is necessary.

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