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Effect of Ion Beam Irradiation on Silicon Carbide with Different Microstructures

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1. Introduction

SiC and SiC/SiC composites are one of promising candidates for structural materials of the next generation energy systems such as the gas-cooled reactors and fusion reactors [1]. This anticipation yields many material issues, and radiation effects of silicon carbide are recognized as an important research subject. Silicon carbide has diverse crystal structures (called polytypes), such as α-SiC (hexagonal structure), β-SiC (cubic structure) and amorphous SiC. Among these polytypes, β-SiC has been studied as matrix material in SiC/SiC composites. Near-stoichiometric β-SiC with high crystallinity and purity is considered as suitable material in the next generation energy system and matrix material in SiC/SiC composites because of its excellent radiation resistance. Highly pure and crystalline β-SiC and SiC/SiC composites could be obtained by the chemical vapor deposition (CVD) and Infiltration (CVI) process using a gas mixture of methyltrichlorosilane (CH_3SiCl_3, MTS) and purified H_2. SiC produced by the CVD method has different grain size and microstructal morphology depended on the process conditions such as temperature, pressure and the input gas ratio [2].

In this work, irradiation effects of silicon carbide were investigated using ion beam irradiation with emphasis on the influence of grain size and grain boundary. MeV ion irradiation at low temperature makes amorphous phase in silicon carbide. The microstructures and mechanical property changes of silicon carbide with different structures were analyzed after ion beam irradiation.

2. Experimental Details

The materials used for this study were single crystal 6-H α-SiC (Cree Systems), polycrystalline 3-C β-SiC (Rohm & Haas Co.), and CVD-SiC fabricated using a gas mixture of methyltrichlorosilane (CH_3SiCl_3, MTS, Aldrich Co., 99%) and purified H_2 (purity: 5 N). Explanation on the materials chosen for the MeV ion irradiation is listed in table 1.

The ion beam irradiation was 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 voltage). The irradiation temperature, displacement damage rate and total dose were room temperature, 2.24×10^{-3} dpa/s, and 20 dpa, respectively. The samples listed in table 1 were irradiated at the same time. The damage level was calculated by SRIM2003 code [3] assuming the average displacement threshold energy of 35 eV, stoichiometric chemical composition and a mass density of 3.21 g/cm^3. The representative total dose of ion irradiated materials was determined by selecting at the depth of 1400 nm. Unfortunately, accurate irradiation temperature could not be measured. The evaluation of the mechanical property change 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 detailed procedure of depth-sensing indentation test was explained in elsewhere [4]. The sample preparation by FIB milling system for TEM observation was described in detail in the previous paper [5]

3. Results and Discussion

Fig. 2 shows the TEM microstructure of CVD-SiC (ID: SiC02, Rohm and Haas, Co.) after 20 dpa ion irradiation. Si ions accelerated with the energy of 5.1 MeV were injected to SiC and stopped at the depth of about 2.3 μm. This damage distribution is well agreed with the result by the SRIM calculation. In the case of single crystal SiC, amorphous phase was clearly observed in TEM microstructure with a electron diffraction pattern. However, CVD SiC (ID: SiC02, Rohm and Haas, Co.) exhibited the mixed phases of amorphous and crystal structure. The selected diffraction pattern in the damaged region showed diffuse rings and diffraction spot at the same time, which indicates typical amorphous and crystal materials, respectively. On the other hand, as-fabricated CVD SiC

Table 1. The materials used in this study.

Sample ID / explanation	SiC 01	SiC 02	SiC 03	SiC 04
Manufacturer	CREE, Inc.	Rohm and Haas, Co.	As-fabricated	As-fabricated
Structure	6H (hexagonal)	3C (cubic)	3C (cubic) Fabrication at 1300 °C	3C (cubic) Fabrication at 1100 °C
Grain Size	Single Crystal	10 ~ 30 μm	~ 10 μm	~ 1 μm

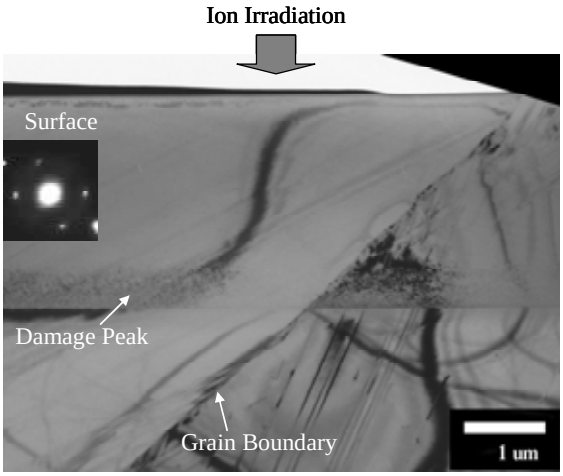

Fig. 1 TEM microstructure of CVD SiC with grain size of 10 ~ 30 μm.

with comparatively smaller grain size (>10 μm) showed the almost crystalline diffraction patterns with clear diffraction spots. The degree of an amorphization formation was strongly depended on the grain size of silicon carbide. Snead [6] reported that the nanometer scaled islands of amorphous phase were formed in radiation induced SiC depended on irradiation temperatures and doses.

The amorphization behavior governed by grain size also affected mechanical properties of SiC. Elastic moduli of four SiC samples were shown in Fig. 2 before and after ion beam irradiation. The data were arranged

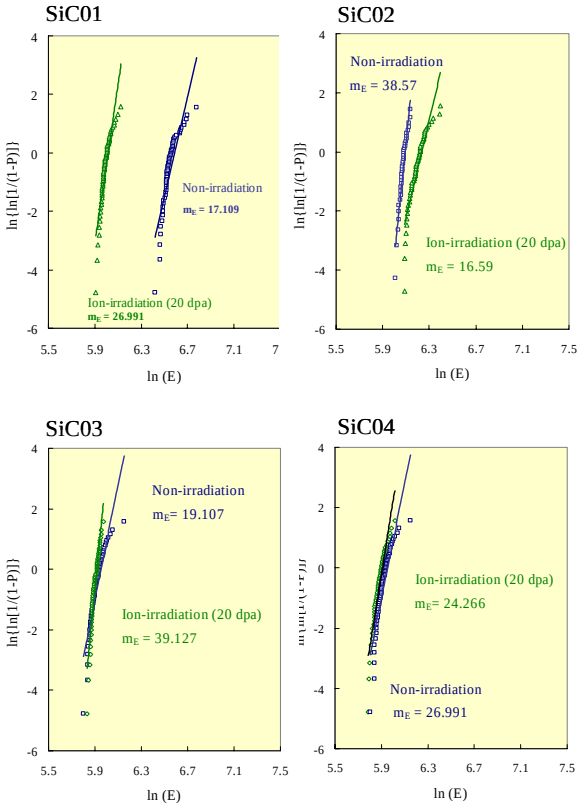

Fig. 2 Weibull distribution on elastic moduli of four types SiC before and after 20 dpa ion irraidation

statically with weibull modulus. The single crystal SiC showed a clear reduction of the elastic moduli by ion beam irradiation. However, CVD SiC (ID: SiC02) exhibited slight increase of the elastic moduli. And comparatively little change of the elastic moduli was shown in SiC with the small grains (SiC03 and SiC04).

It seems that damage evolution like amorphization and point defects is depended on the grain size in SiC. The corresponding mechanical properties of SiC are sensitively affected by this behavior. Further studies on radiation induced amorphization, microstructural evolution and mechanical property changes have to be investigated in various SiC under the carefully controlled irradiation experiments.

4. Conclusions

Four types of SiC with different grain size were irradiated by MeV ion accelerator. SiC with no grain boundary and relatively large grains exhibited the easy formation of amorphous phase. It revealed that the amorphization did not occur in SiC below few micrometers grain size. Large amounts of amorphous phase in SiC cause a severe reduction of the elastic moduli, although the elastic moduli was increased in SiC with small amounts of amorphous phase. Little change of microstructure and mechanical property was shown in SiC with the small grain sizes (below 10 μm).

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