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Influence of irradiation-induced defects on fracture behavior in highly pure SiC

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Abstract

Charged-particle irradiation to highly pure SiC was performed to investigate the influence of irradiation-induced defects on its fracture behavior. To investigate the fracture behavior of SiC, the microstructural analysis was carried out by observing cross-sections of a residual indentation impression. An apparent increase of indentation fracture toughness was shown in the highly pure SiC upon ion irradiation. In the case of ion-irradiated SiC, a number of micro-cracks were created around the indentation-induced deformation region. Indentation-induced slip bands on the (1 1 1) crystallographic plane were observed in both the virgin and the ion-irradiated SiC. The indentation-induced slip bands and the pre-existing defects provided one of the sites for micro-crack creation for the ion-irradiated SiC. The formation of the micro-cracks may dissipate the fracture energy for brittle fracture, which seems to be one of the mechanisms for the toughening behaviors of SiC by an energetic particle irradiation.

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1. Introduction

A highly pure silicon carbide (SiC) has been widely used as a matrix material of SiC/SiC composites for advanced nuclear fusion reactors. Many studies on fabrication of SiC-based materials have been carrying out and those have provided data for many grades of SiC and SiC/SiC composites. Among those materials, SiC based-materials with high purity and crystallinity have been shown to have excellent irradiation tolerances [1]. In the design for a SiC fusion reactor, the SiC/SiC composites are formed to a blanket shape for surrounding the toroidal plasma. Over its lifetime, the SiC composite would be expected to receive dynamic irradiation damage: high displacement damage (as much as 100 dpa), high temperature (∼1000 °C) and transmuted gases.

Silicon carbide produced by chemical vapor deposition (CVD) has been considered as a reference material for study of irradiation effects of SiC/SiC composite, since the CVD process gives

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highly-crystalline, stoichiometric and pure SiC, which is fundamental requirement for good irradiation resistance. While there has been significant developmental work on manufacturing the SiC and SiC/SiC composite, details of the effects that the complex operating conditions have on the properties of SiC are not adequately understood. Silicon carbide and SiC/SiC composites have been extensively studied to explore neutron irradiation-induced changes such as mechanical properties [2,3], amorphization [4], swelling [5], thermal conductivity [6] and microstructural evolution [7], although its irradiation effects are even less clear and not well understood. Especially, fracture behavior of SiC is critical because the brittle nature of SiC-based materials is one of the most limiting factors in design of fusion applications. Little has been done with fracture behavior of irradiated SiC, aimed at better understanding the effect of irradiation and basic comprehensions of fracture behavior of SiC-based materials. Cracks associated with Vickers indentation are widely used as artificial defects of 'known' size for the fracture toughness measurement of ceramics. Fracture toughness of SiC-based materials under irradiation is a critical parameter to obtain and one that reflects their growing importance in the engineering design of advanced fusion reactor devices. Cracks initiate within the SiC matrix of SiC/SiC composites under expected fusion core conditions. In recent reports, SiC and SiC/SiC composite with high purity, crystallinity and good stoichiometry showed enhanced fracture toughness and mechanical properties after ion and neutron irradiations [1,3,8,9]. Charged-particle irradiation is a very powerful tool for the simulation study of neutron irradiation damage of expected operation conditions in fusion reactors.

In this paper, a high voltage accelerator was used to ion irradiate SiC in order to study the effects of irradiation damage on fracture mechanisms. Indentation-induced cracks provide adequate information on the fracture mechanics of SiC, including crack initiation, crack extension pattern and fracture toughness. The initial stage of crack initiation and fracture pattern of highly pure SiC were investigated before and after ion irradiation.

2. Experimental

A highly pure SiC, which was produced by chemical vapor deposition (CVD), Rohm & Haas Co., was used for ion irradiation with sample dimensions of a 3 mm diameter and a 0.25 mm thickness. The crystal grains of CVD–SiC are in a columnar structure which consists of 50 μm columnar grains that have a preferred crystallographic orientation of ⟨1 1 1⟩ direction parallel to the CVD-growth direction but randomly oriented in the normal plane.

Si^{2+} ions irradiated the (1 1 1) oriented crystallographic plane of CVD–SiC as shown in Fig. 1. The texture in CVD–SiC is mostly along the ⟨1 1 1⟩ direction and this texture leads to mainly {1 1 1} facets on the surface [10].

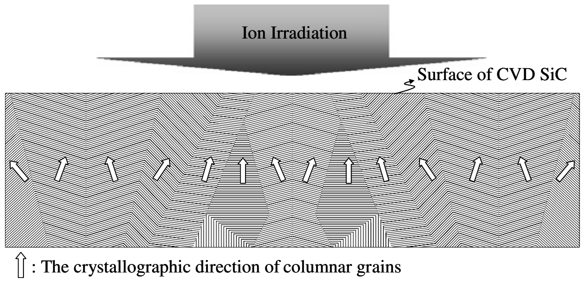

Fig. 1. Expected morphology of CVD–SiC with mainly {1 1 1} facets on the surface.

Fracture behavior of SiC was investigated by observing the crack progress, which was induced by a point indentation. In order to observe the cracks around indentation imprint, a newly contrived FIB (Focused Ion Beam) method was used to prepare the sample for transmission electron microscope (TEM) and scanning electron microscope (SEM) observation. Detailed procedure of the sample preparation was described elsewhere [11].

3. Results and discussion

Fig. 2 shows indentation fracture toughness of CVD–SiC as a function of irradiation temperature.

Toughening behavior by silicon-ion irradiation was obviously exhibited in SiC and depends strongly on the irradiation temperature. It is noted that the mechanical property changes of highly pure SiC are independent of irradiation damage level (dpa). These results indicate that higher fracture toughness was obtained in samples ion-irradiated at lower temperature. However, it is difficult to make a quantitative assessment of the effect of irradiation and to interpret the reason why SiC is toughened by irradiation. It has been shown previously that critical toughening mechanisms were clearly seen in the crack patterns of SiC after ion

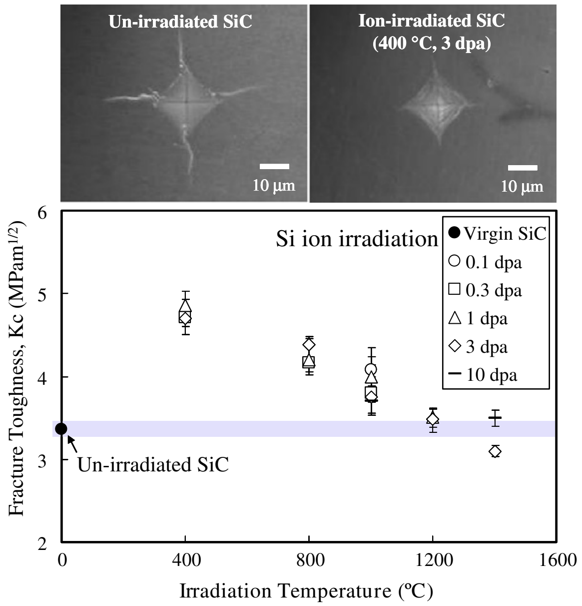

Fig. 2. Indentation fracture toughness of SiC with and without Si-ion bombardment as a function of irradiation temperature.

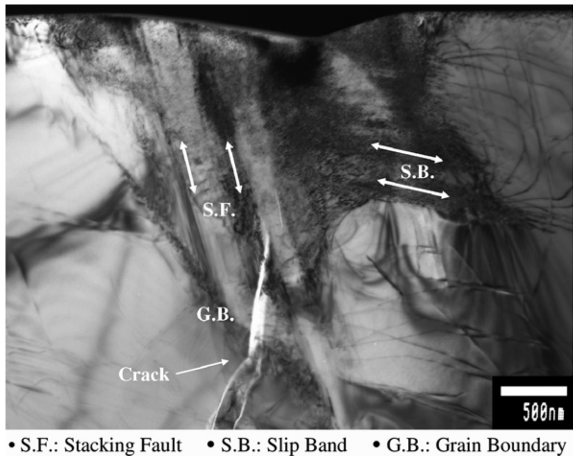

Fig. 3. Cross-sectional TEM image of residual indentation impression in the un-irradiated SiC.

irradiation including crack deflection, tilting, micro-cracking and branching [12]. However, the previous study offers no explanation of why micro-cracking appears dominant in many cases and why crack toughening mechanisms occur in ion-irradiated SiC.

This result will be examined more critically herein by observing the cross-section of the residual impressions of indentations induced by relatively low applied load. The present study is focused on SiC irradiated below 800 °C due to its clearly toughened behaviors.

A cross-sectional TEM image of an indentation residual impression of un-irradiated SiC is given in Fig. 3, which depicts the crack initiation in the deformed region. The cross-sectional size of a residual impression was 250 nm from the surface and the deformed region was roughly 10 times larger than that of the indentation depth. The slip bands were exhibited on the (1 1 1) crystal plane, causing initiation and growth of dislocations. The primary crack was initiated at the root of stress concentration in the deformed region with crack propagation along the pre-existed stacking faults. This, in turn, caused the crack propagation path to change direction parallel to the indentation applying direction. It seems that the median crack branches at the grain boundary. However, no additional cracks were observed around the deformed region.

The cross-sectional image of the ion-irradiated SiC was also observed to compare with the un-irradiated SiC. These SiC samples were irradiated at the temperature range of 400–800 °C, and with dose of 1–3 dpa. Point defect and clusters are anticipated as the main defects in SiC with no irradiation-induced amorphization or voids under the irradiation conditions of present study [5,13]. The indentation-deformed microstructures of SiC after ion irradiations are shown in Fig. 4. No median cracking appeared dominant in the ion-irradiated SiC. Comparatively clear observation of the slip band and the grain boundary can be obtained in ion-irradiated SiC by applying the indentation. The crack configurations of ion-irradiated SiC were found to involve the defects such as the indentation-induced slip bands (SB), the stacking faults (SF) created during fabrication process, the grain boundaries (GB) and those interactions. As can be seen in Fig. 4, the crack has propagated tracing the stacking faults (A, C2) and the slip bands (B, C2). It is interesting to note that the pattern of the crack extension was affected by the interaction among the indentation-induced slip band, the pre-existing stacking faults and grain boundary. The crack on the slip band was clearly deflected at a point of contact with the grain boundary in Fig. 4(B). The crack in Fig. 4(C3) initiated at the intersection of the slip band and the stacking fault. The possible interpretation of preferential crack extension on the site of intrinsic (SF, GB) and extrinsic (SB) defects is that the defects by the ion irradiation seem to interact with the lattice and modify the structure of

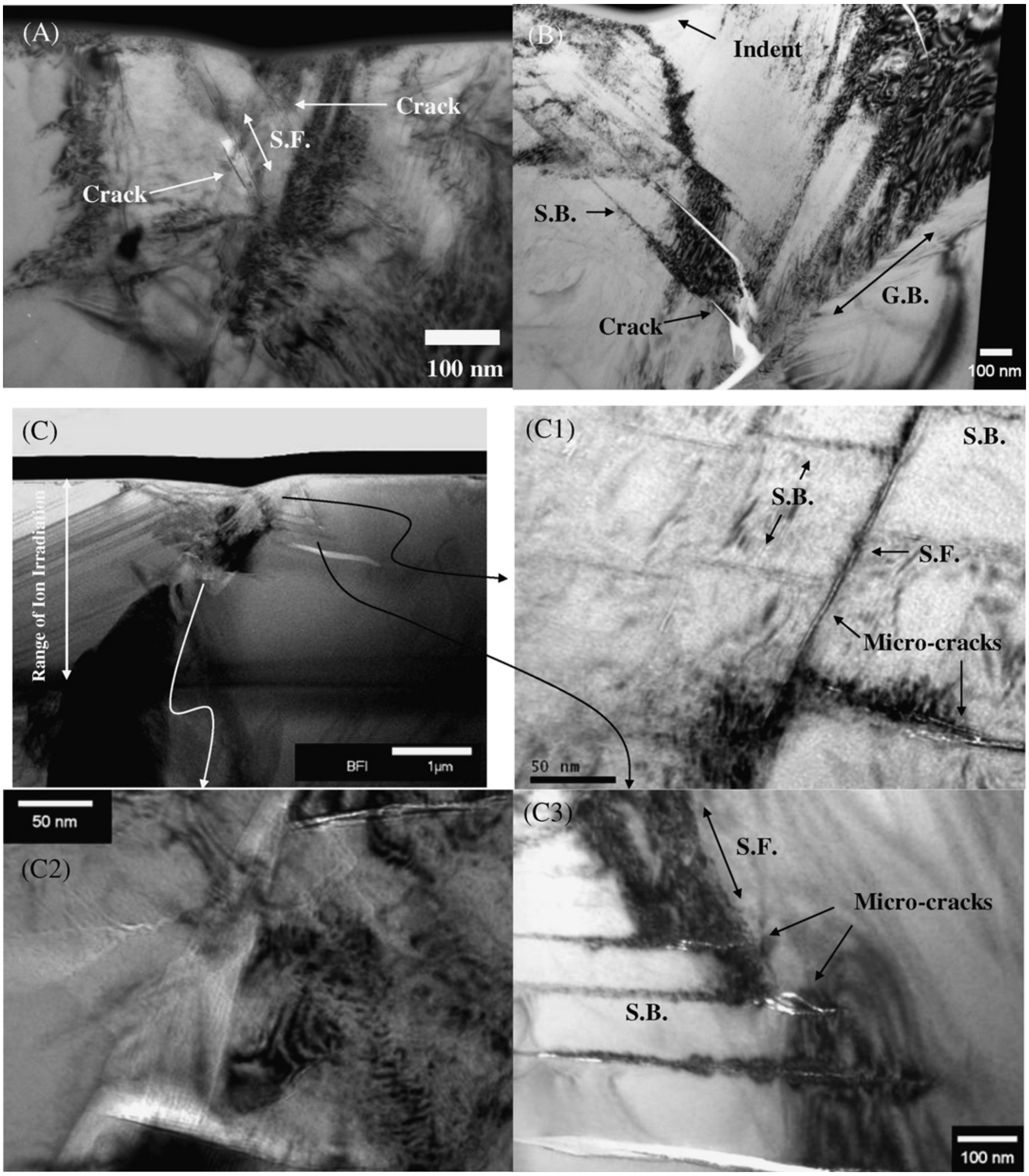

Fig. 4. Cross-sectional TEM images of residual indentation impression in SiC after ion irradiation. The SB, SF, and GB in the image indicate the slip band, stacking fault and grain boundary, respectively. The irradiation temperature and dose are as follows, respectively: (A) 800 °C and 1 dpa, (B) 600 °C and 3 dpa, and (C) 400 °C and 3 dpa. The irradiation range from the specimen surface is a 2.4 μm depth along the normal to the entry face.

pre-existing defects to form an easy site for crack initiation and propagation. Additional micro-cracks were also observed at the point of stress concentration in the indentation-deformed region as shown in Fig. 4(C2). Qualitative descriptions of the un-irradiated structure indicate brittle crack features, while the irradiated SiC can be described as including the toughened crack configurations, such as micro-cracking and branching behaviors. Further study on the relationship between irradiation-induced defect and crack extension is essential.

4. Summary

In this paper, the fracture behavior of highly pure SiC was studied by observing residual indentation impressions using TEM images taken beneath indents before and after ion irradiation.

The irradiation-induced defects seem to cause comparatively easy sites of crack initiation at the pre-existing stacking fault and grain boundary, and indentation-induced slip bands. One of the important mechanisms for irradiation-induced toughening behavior of highly pure SiC is that a number of micro-cracks initiated in ion-irradiated SiC allow for the dissipation of fracture energy for brittle fracture, but also promote crack toughening.

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