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Evaluation of dual-ion irradiated β-SiC by means of indentation methods

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Abstract

Dual-ion irradiation using the high-voltage accelerators and the micro- and/or nano-indentation scheme are useful techniques to evaluate the irradiation-induced mechanical property changes of fusion materials. In this study, polycrystalline β-SiC was irradiated by silicon ions with and without simultaneous injection of helium ions using DuET Multi-beam Facility. β-SiC irradiated with dual-beam was carefully tested by means of micro-indentation technique to establish hardness and fracture toughness for determining micro-mechanics. Simultaneous irradiation of Si and He ions clearly enhances radiation-induced hardening than that by single Si ion irradiation. Indentation fracture toughness of radiation-induced β-SiC increased, however, the enhancement decreased with increasing irradiation temperature and time.

1. Introduction

SiC has been extensively studied to explore the irradiation-induced effects such as mechanical properties, amorphization, density change, swelling, thermal conductivity change and micro-structural evolution, since SiC/SiC composite is recognized as a promising structural material for fusion applications [1]. However, to realize the application of SiC/SiC composite as fusion materials, there still are many issues to be solved or proven, which include the micro-structural stability and mechanical property changes under fusion environment [2].

Dual-beam ion irradiation is particularly an effective method to study the irradiation effects in SiC due to its capability of high-fluence bombardment, high temperature irradiation, helium co-implantation and target versatility [3]. In fusion material study typically the dual-ion irradiation experiment is used to simulate the fusion environments, especially synergistic effects, which include the atomic displacement damage and the helium production through (n,α) reaction. However, the majority of studies relating to SiC following ion irradiation have been limited so far to measure radiation-induced swelling and micro-structural evolution [4,5]. Because the induced damage by ion irradiation occurs within thin surface layers of typically a few micrometer thickness. Meanwhile, micro- and/or nano-indentation have been considered to be a useful technique for probing the mechanical properties on very thin films and ion induced materials.

The purpose of this article is to establish the irradiation-induced property changes of SiC by means of indentation technique, reflecting their growing importance for the realization of fusion reactor devices.

2. Experimental detail

The material used was a polycrystalline β-SiC, 3 mm diameter × 0.25 mm thickness, which was produced using a chemical vapor deposition process (Roam and

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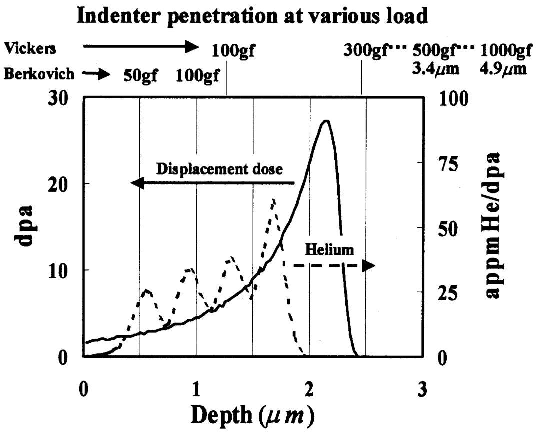

Fig. 1. TRIM calculation related to the depth profile of displacement damage and deposited helium ions in β-SiC (with condition of 60 appm He/dpa).

Hass Co.). The sample is irradiated at Dual-beam for Energy Technologies, University of Kyoto (DuET facility) [3]. Specifically, 5.1 MeV Si^{2+} ions were implanted as single-beam by a tandem accelerator (Model 4117MC+) operating at 1.7 MV and an additional single-ended accelerator was used for simultaneous implantation of 1.0 MV He^{+}. Helium ions were energy-degraded by thin aluminum foils.

The depth profiles of displacement damage and deposited silicon and helium were calculated using TRIM-98 code [6,7], (see Fig. 1) by assuming an average displacement threshold energy of 35 eV, stoichiometric chemical composition and a mass density of 3.21 g/cm^3. The irradiation temperature, displacement damage rate and total dose were 873–1473 K, 1×10^{-3} dpa/s, and 0.1–10 dpa, respectively. The helium-to-dpa ratio in the dual-beam experiment was 60 appm He/dpa. The micro- and/or nano-indentation testing was carried out for evaluating mechanical property changes of ion induced β-SiC using micro-indentation (model: Akashi HM-101) and Akashi MZT-3 instrumented nano-indentation device, with Vickers and Berkovich diamond tips, respectively. Hardness tests were conducted with the nano-indentation device and a Berkovich diamond tip. The indentation fracture toughness was determined at the load of 100 g (Berkovich indentation) and 100, 300, 500 and 1 kg (Vickers indentation) for evaluating the relation between the depth of the displacement damage and the indenter penetration. This indenter penetration is depicted in the upper scale of Fig. 1.

3. Analysis

We conducted the micro- and/or nano-indentation experiments, and proved that it is a useful method for evaluating the micro-mechanics such as hardness and fracture toughness of dual-ion irradiated β-SiC. In the following section, there will be simply introduced the profiling method of micro-hardness and fracture toughness. Details have been reported elsewhere [8–12].

3.1. Hardness

The load–depth (P–h) relationship was determined by nano-indentation tests, which were capable to measure the sample hardness from the initial portion of the unloading curve. The hardness evaluation was conducted by procedures given by Oliver and Pharr [8], where the hardness H is defined as

$$H = \frac{P_{\max}}{A(h_c)}.$$

$P_{\max}$ is the peak load and $A(h_c)$ the projected contact area, which is a function of the contact depth h_c and the shape of indent tip. After ascertaining the threshold load (about 55 g) of radial crack creation by field-emission SEM observation, hardness tests were conducted below 50 g with a constant loading rate of 0.15 g/s.

3.2. Fracture toughness

A number of analytical models relevant to fracture toughness of brittle materials have been conveniently developed by means of indentation technique with Vickers and Berkovich indenters [9–11]. The following simple relationship between the fracture toughness K_c and the length of the radial cracks c is given by Lawn et al. [9]. Lawn took advantage of the potential for measuring the fracture toughness of small specimens and ion-beam induced β-SiC

$$K_c = \alpha\left(\frac{E}{H}\right)^{1/2}\left(\frac{P}{c^{3/2}}\right).$$

E is the Young's modulus and H the hardness, both of them are calculated from low-load nano-indentation (load–depth curve). P is the applied load and α an empirical constant, which is 0.016 for a Vickers indenter and 0.032 for a Berkovich indenter [12]. The length of radial cracks was measured using FE-SEM (JEOL JSM 6700F field emission scanning electron microscope).

4. Results and discussion

The results of hardness, which was determined using low-load scale ($\leqslant 50$ g), are presented in Fig. 2. Open and solid symbols indicate single- and dual-beam irradiation, respectively. The initial letter in legend of figures (Figs. 2–4) indicates the single (S) and the dual irradiation (D) and the numbers indicate the irradiation tem-

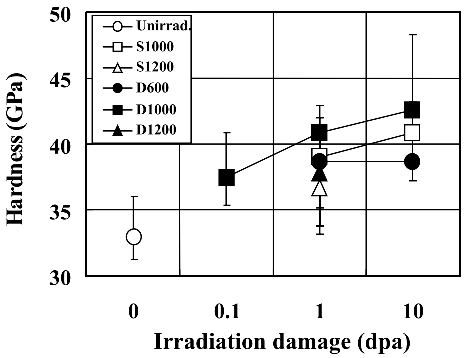

Fig. 2. Results of hardness changes after single and dual-ion beam irradiation.

perature (for example, S1000: single irradiation at temperature of 1000 °C). These results show that unirradiated β-SiC exhibits the lowest hardness of about 33 GPa.

Apparently the hardness is increasing with the radiation damage and simultaneous irradiation of Si and He ions clearly enhances radiation-induced hardening compared to that by single Si ion irradiation. The hardness is dependent on the dislocation movement, since it is related to the yielding phenomenon in crystalline materials. Therefore, it is clearly evident that radiation-induced point-defect clusters and helium production acted as barriers for dislocation movements. A temperature effect cannot be drawn due to an insufficient number of experimental data, but the irradiation at higher temperatures (over 1000 °C) decreases the hardness of the samples. One possible explanation for this decreasing hardness is an easy migration and combination of helium ions and vacancies, which might have occurred due to annealing of dislocation populations or recovery of stress concentration in β-SiC.

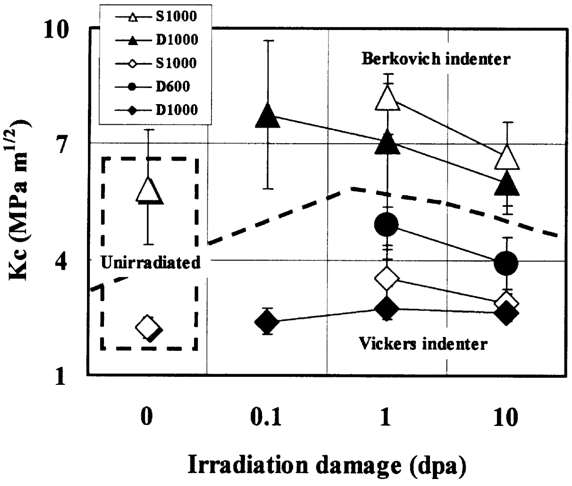

Fig. 3. Indentation fracture toughness as a function of irradiation temperature and time.

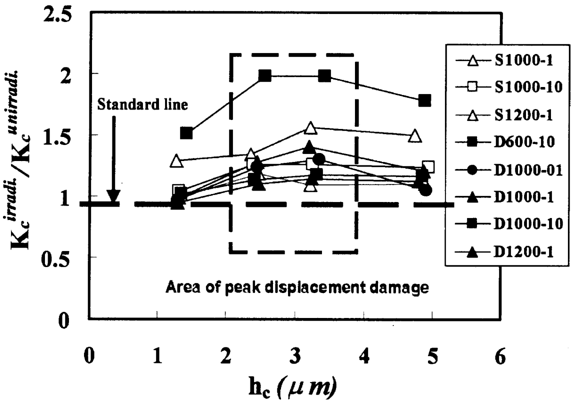

Fig. 4. Normalized fracture toughness ($K_c^{\text{irradi.}}/K_c^{\text{unirradi.}}$) for evaluating the relation between depth of displacement damage and indenter penetration.

When arranging the indentation fracture toughness of β-SiC, this value is governed by the irradiation temperature and time. The results of the correlation between fracture toughness and irradiation temperature and dose, which is related to the irradiation time, are plotted in Fig. 3.

Although fracture toughness is enhanced by single- and dual-ion irradiation, the increment decreased with increasing irradiation temperature and time. The fracture toughness is related to the crack propagation; therefore, the fracture toughness is dependent on the easiness to create cracks around the indent. When β-SiC is irradiated, vacancies and point-defect populations are produced in the sample. The reason for the increasing fracture toughness is obstacles, which interrupts crack propagations. However, sample annealing removes the obstacles of crack propagation.

Finally, we evaluated the relation between the range of displacement damage and the indenter penetration. The results are shown in Fig. 4.

At the area of the peak displacement damage (dot-square area, range 2–4 μm), which is calculated by TRIM code (see Fig. 2), we can see the largest difference between the standard line and the normalized fracture toughness. This means, that an optimum indenter depth have to be determined within the limits of the area of the peak displacement damage in order to establish an effective evaluation for the fracture toughness of ion irradiated β-SiC.

5. Conclusion

Dual-beam ion irradiation with conditions up to 10 dpa at the maximum temperature of 1473 K and 60 appm He/dpa was carried out to polycrystalline β-SiC by irradiating Si and/or He ions using DuET facility. The results may be summarized as follows:

The indentation method was proved to be an excellent technique for evaluating the micro-property changes of ion irradiated β-SiC. The single or dual-ion irradiation increases the indentation hardness. Especially, simultaneous irradiation with Si and He ions clearly enhances the radiation-induced hardening compared to single Si ion irradiation. The fracture toughness is increased after ion irradiation, however, this enhancement decreased at higher temperatures and longer duration of irradiation. We could carefully expect that an optimum indentation load for evaluating ion-irradiated β-SiC have to be determined within the limits of the range of the peak displacement damage.

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