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Anisotropic evolution of Frank loops in ion-irradiated silicon carbide

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Abstract

Frank loop evolution in highly damaged polycrystalline cubic silicon carbide irradiated with 5.1 MeV Si^{2+} ions at 1673 K was studied by transmission electron microscopy (TEM). Individual TEM images of Frank loops formed on each {1 1 1} plane revealed that their population strongly depended upon their orientation with respect to the incident beam direction. However, no significant difference in loop growth rates was observed between each habit plane. The anisotropic loop evolution has not been reported for neutron-irradiated SiC. Our examination shows that a grain, containing {1 1 1} planes nearly parallel to the irradiated surface, was largely strained toward the free surface by the preferential formation of Frank loops on the plane. Compressive stress following the anisotropic swelling in ion-irradiated specimen may affect the loop evolution. The possible mechanism of the anisotropic loop evolution observed here is accounted for by the stress induced preferential nucleation of Frank loops.

1. Introduction

Silicon carbide (SiC) and its composites may to be used as structural materials for fusion power reactors due to their superior high-temperature properties, thermo-chemical stability, irradiation tolerance, inherent low activation, and low after-heat properties [1]. Before SiC and/or SiC/SiC composites may be considered for use in nuclear environments, several technical issues must be examined, including dimensional instability, modification of thermal and electrical transport properties, and mechanical property changes [2].

Several microstructural examinations of SiC have been carried out after neutron irradiation. Price [3] observed the formation of small Frank dislocation loops in β-SiC irradiated with fast-neutrons at temperatures below 1273 K. Observations by high-resolution electron microscopy revealed interstitial dislocation loops primarily formed on {1 1 1} habit plane in β-SiC irradiated at 4.8×10^{26} and 1×10^{27} n/m [4,5]. The fluence and temperature dependence of microstructural changes, studied by an ion-irradiation method, indicated that black spot defects were dominant for irradiation temperature below 1073 K, while larger Frank loops were not observed until irradiation temperatures exceeded 1473 K [6].

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In general, ion-irradiation data include the potential ion-irradiation-specific phenomenon resulting from higher damage rate, limited ion range, surface effects, etc. A careful analysis of microstructural defects in ion-irradiated specimen may lead to understandings of the underlying physics for irradiated effects which may not be revealed by neutron irradiation experiments. The objective of the present work is to clarify the mechanisms of newly detected anisotropic evolution of the Frank loops in ion-irradiated β-SiC.

2. Experimental procedures

A high-purity (>99.9995%) polycrystalline β-SiC specimen produced through chemical vapor deposition process (by Rohm & Haas Co., Woburn, MA, USA) was irradiated with 5.1 MeV Si^{2+} ions at 1673 K at DuET facility [7], Kyoto University. The deviation from targeted temperature was confirmed to be ±5 K with high-resolution thermography during irradiation. The displacement damage and deposited Si concentration profiles were calculated by SRIM98 [8] and are shown in Fig. 1. A schematic image defining 'θ' as the angle between Frank loop normals and incident beam direction, which corresponds to the angle between the loop planes and the irradiated surface, are depicted in the figure.

Thin foils were produced from the irradiated samples using gallium ions in a focused ion-beam micro-processing device (Micrion JFIB2100). For microstructural observations, the thinned samples were examined in a transmission electron microscope (TEM, JEOL JEM-2010). Frank loop images were taken using streaks of diffraction spots arising from the loops. The streaks are due to presence of the {1 1 1} stacking faults in the loops and can be used to image the Frank loops independent of the surrounding crystalline matrix.

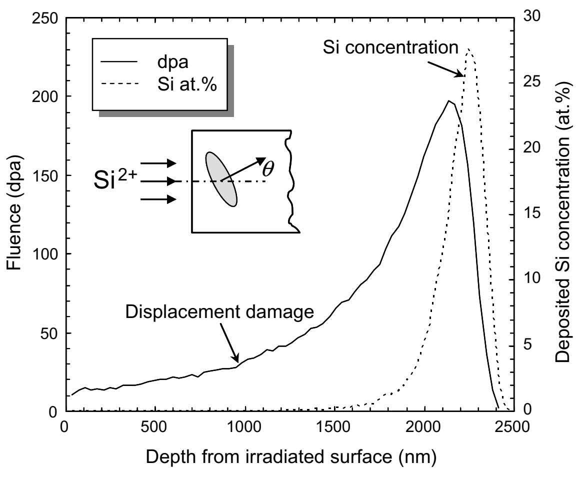

Fig. 1. Damage and Si concentration profiles for Si^{2+}-irradiated SiC calculated by SRIM98. The parameter θ is defined as the angle between {1 1 1} normal and the beam direction.

3. Results

The microstructures of two different grains named 1 and 2 were examined in detail in this study. The typical mean grain size of the sample is about 5–10 μm. In Fig. 2, TEM images of Frank loops formed at a depth of 1050 nm from the irradiated surface (corresponding to a damage level of approximately 34 dpa), and corresponding Thompson's tetrahedron are shown. Frank loops on two specific planes in four {1 1 1} habit planes are clearly viewed edge-on. These Frank loops were identified as interstitial-type by inside–outside contrast analysis. We could not differentiate between loops formed on two other planes viewed non-edge-on because of their small sizes. However, the number density of these non-edge-on loops was measured, and the results are summarized in Table 1.

The Frank loop size distributions for three orientations formed at the damage levels of 20, 34, and 70 dpa are shown in Fig. 3. Total number densities of loops formed on the planes with smaller θ were higher than that in planes with larger θ at all damage levels. In particular, relatively small loop ($d < 10$ nm) populations were largely affected by θ.

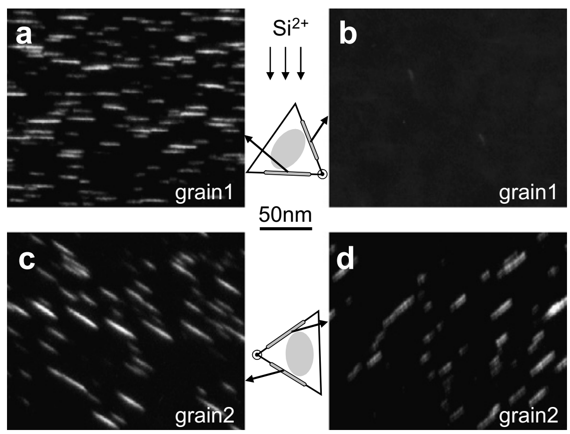

Fig. 2. TEM images of Frank loops in four specific planes of {1 1 1} family and Thompson's tetrahedron for θ: (a) 2.8°, (b) 71.9°, (c) 32.0°, and (d) 38.5°.

Table 1
Frank loop microstructural data at 34 dpa for ion-irradiated SiC

	Parameters	$(\bar{1}\bar{1}1)$	$(\bar{1}1\bar{1})$	$(1\bar{1}\bar{1})$	(111)
Grain 1	θ (°)	2.8	71.9	67.7	31.0
	Mean diameter (nm)	12.1	5.77	–	–
	Number density ($\times 10^{21}$ m^{-3})	12.0	0.2	<0.4	<0.4
	Area per unit volume ($\times 10^{5}$ m^{-1})	18.0	0.05	–	–
Grain 2	θ (°)	87.2	38.5	32.0	87.2
	Mean diameter (nm)	–	17.0	12.4	–
	Number density ($\times 10^{21}$ m^{-3})	<0.3	3.2	6.2	<0.3
	Area per unit volume ($\times 10^{5}$ m^{-1})	–	7.82	8.90	–

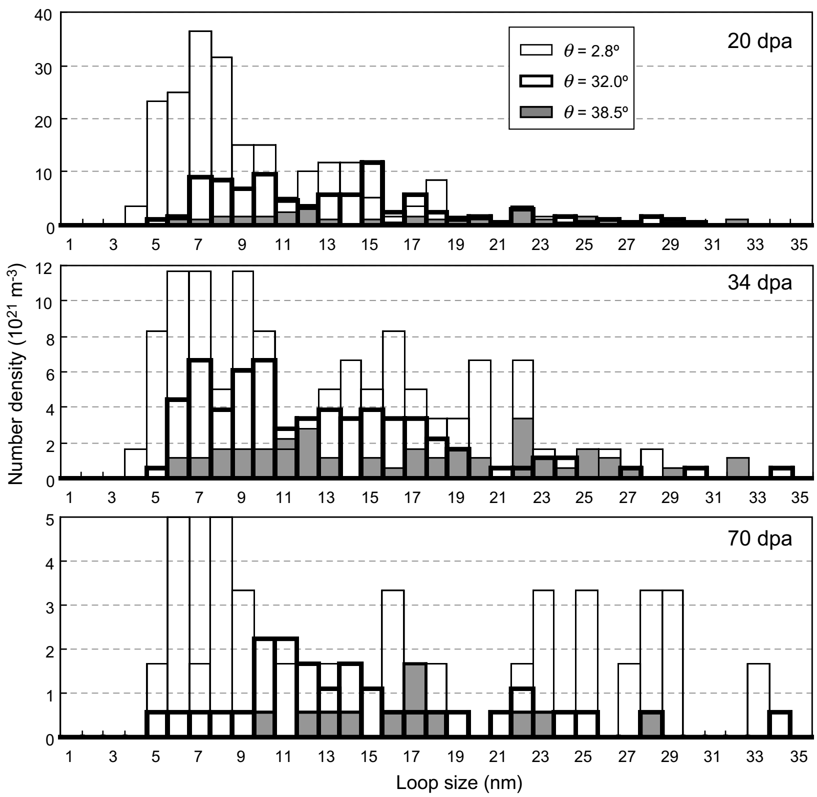

Fig. 3. Frank loop size distribution determined for three θ-orientations.

The number density of the small loops formed on the plane nearly parallel to the irradiated surface were high even at 70 dpa. A few perfect loops with Burgers vector of $a_0/2[1\,1\,0]$ were also observed at 70 dpa.

Fig. 4 shows the specific mean diameter of Frank loops formed on three {1 1 1} planes and their growth trends. The sizes of loops increased with increasing damage level. No significant differences were observed between $\theta = 2.8°$ and 32.0° in loop growth rates with increasing fluence. The reason for the relatively small numerical value for the case of $\theta = 2.8°$ is a large number of smaller loops were formed on the plane. For the case of $\theta = 71.9°$, data exhibited significant scatter because the loop population formed on the plane was small.

The θ dependence of Frank loop area per unit volume is shown in Fig. 5 for three damage levels. The area of Frank loops is roughly proportional to the number of interstitial atoms that constitute the loops. Frank loop area decreased with increasing θ for all damage levels.

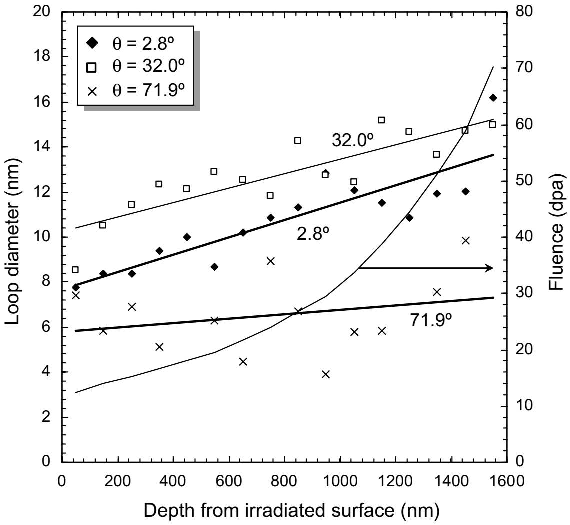

Fig. 4. Frank loop sizes as a function of depth for three θ-orientations.

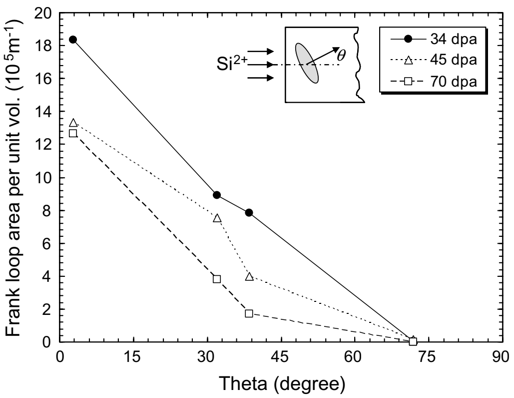

Fig. 5. Frank loop area per unit volume as a function of θ-orientation.

4. Discussion

The present observations provide orientation dependence of Frank loop evolution in ion-irradiated β-SiC. The formation of loops with $\mathbf{b} = a_0/3[1\,1\,1]$ was also observed in neutron-irradiated SiC [9], however, the loop orientations were not so anisotropic. For example, Akiyoshi et al. [10] reported that the Frank loops in neutron-irradiated SiC were not formed only on specific {1 1 1} planes, but appeared to be uniformly distributed, and their structure does not allow intersection in the same family plane. They concluded when Frank loops encounter loops on other planes, their growth, and the nucleation in the space enclosed by the loops are restricted.

In this experiment, the growth of the loops on $(\bar{1}\,1\,\bar{1})$ plane appeared to be stopped at the contact point with loops on $(1\,\bar{1}\,\bar{1})$ plane in grain 2. This phenomenon is consistent with the neutron data described by Akiyoshi. On the other hand, in grain 1, loop contacts were rare because few loops were formed on the three {1 1 1} planes other than the $(\bar{1}\,\bar{1}\,1)$ plane. That may be one of the reason for the larger number density of small loops on $(\bar{1}\,\bar{1}\,1)$ plane in grain 1 than other planes even at 70 dpa (shown in Fig. 3). However, because the growth rates of observed loops were nearly independent of θ, loop contacts were not common, and had almost no effect on growth and nucleation of loops under the irradiation condition studied here.

The strain parallel to the beam direction for the neighboring two grains was estimated from the area swept by the Frank loops and the results are summarized in Table 2. Strain in grain 1, which contained the $(\bar{1}\,\bar{1}\,1)$ plane nearly perpendicular to the beam direction, were larger than that of grain 2 at all damage levels. The excess strain in grain 1 is associated with the large Frank loop area attributed to the preferred nucleation of loops on the $(\bar{1}\,\bar{1}\,1)$ plane. This observation suggests that volume expansion (swelling) toward the free surface was enhanced by the anisotropic loop evolution under high-temperature ion-irradiation conditions.

The possible mechanisms leading to the observed θ dependence of evolution of Frank loops are explored. Effects of electrical field on density of loops in He-ion-irradiated Al_2O_3 have been reported by Yasuda et al. [11]. They showed that the applied electric field of 100 kV m^{-1} decreased the number density of interstitial-type dislocation loops and increased their size. The electric field will enhance the diffusivity of defects such as interstitial atoms. However, in this work, there was no applied electric field and loop growth rates were nearly inde-

Table 2
The calculated strain in the incident beam direction estimated from the area swept by the Frank loops in the two examined grains

Depth (nm)	Fluence (dpa)	Strain in beam direction	
		Grain 1	Grain 2
1050	34	4.6×10^{-4}	2.8×10^{-4}
1250	45	3.3×10^{-4}	2.0×10^{-4}
1550	70	3.2×10^{-4}	9.4×10^{-5}

pendent of loop orientation. Zinkle accounted for observed preferential loop nucleation in planes nearly parallel to beam direction for Al-ion-irradiated $MgAl_2O_4$ by considering the cascade anisotropy [12]. However, the loops observed in this work preferentially nucleated on planes nearly parallel to the irradiated surface, not parallel to the beam direction. These proposed physical mechanisms of anisotropic loop distribution seem to not be correlated with our results.

In contrast, stress induced preferred nucleation (SIPN) [13] of Frank loops was observed in early neutron-irradiation experiments of 316 stainless steel under applied stress [14]. In the present study, the damage range existed only in a thin layer (approximately 2.5 μm in depth) at the irradiated surface. Ion-irradiated bulk specimens are allowed to expand toward the irradiated surface without spatial restraint. Thus, induced compressive stress perpendicular to the beam direction by anisotropic swelling may affect the nucleation of loops. The θ dependence of the size distribution of small loops observed here strongly supports the mechanism of anisotropic evolution of Frank loops by SIPN of these loops.

5. Conclusion

The Frank loop population in ion-irradiated β-SiC was strongly affected by orientation of {1 1 1} planes (expressed by θ; angle between loop planes and the irradiated surface). In particular, the numbers of small loops formed on the plane nearly parallel to the irradiated surface was much higher than that formed on other {1 1 1} planes. However, the loop size was nearly independent of loop orientation. It is noted that this anisotropic loop nucleation does not occur in neutron-irradiated SiC.

In response to these results, the anisotropic swelling mechanism is proposed as the potential cause of the θ dependence of the observed Frank loop formation. Compressive stress following the initial anisotropic swelling likely inhibits the loop nucleation in planes perpendicular to the stress axis. In contrast, preferred nucleation of loops occurs in planes nearly parallel to the free surface. The results suggest that the SIPN mechanism controls the formation, distribution, and orientation of the Frank loops, and it is identified as an irradiation creep mechanisms for SiC at high temperatures.

Acknowledgements

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