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Blister formation and erosion due to blister fracture of SiC or Si

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Abstract

For silicon wafer and silicon carbide, helium ion irradiation followed by annealing was repeated, and the weight loss was measured. The blister formation and its fracture processes were also examined. For Si, many blisters were observed at the surface after the first ion irradiation. After the annealing at 1173 K after the irradiation, small pores formed by helium desorption were observed. With the increase of the cycle number, surface roughness gradually increased. The weight loss due to annealing was about a half of that by ion sputtering. The sputtering yield due to the ion irradiation was approximately twice the reference value in the present experiment, perhaps due to the bubble fracture during the irradiation. For SiC, small blisters were observed on SiC crystals after the ion irradiation. The erosion amount due to only the process of annealing at 1323 K was several times larger than that of ion sputtering, although the sputtering yield due to the ion irradiation was roughly the same as the reference value. The present erosion was significantly large, so that this erosion has to be taken into account regarding the wall lifetime and impurity level of the core plasma.

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1. Introduction

Plasma wall interactions are critical issues for the development of fusion demonstration and commercial reactors [1,2]. In fusion devices, wall surfaces are exposed to ions and charge exchange particles (hydrogen isotopes, helium ash and so on), and then blisters are formed on the surface. The blister is fractured by the increases of inner pressure and thermal stress when the wall temperature rises due to high heat fluxes. Then, the fracture of blisters causes the erosion of plasma facing materials. In addition, the fractured powders enhance the impurity concentration of the core plasma. So, it is necessary to examine the blister fracture process and the erosion for next step fusion devices. Recently, the plasma facing wall was modified by surface coatings such as B and Si [3–6], consequently the plasma confinement was significantly improved. Silicon coating and silicon carbide are attractive as plasma facing materials because of the low chemical erosion and high thermal strength [7–12]. Studies on blister formations and fractures of silicon crystal [13–16] and silicon carbide [17–19] due to helium ion irradiation have been carried out so far. However, the erosion amounts due to the blister fractures for these materials have not been investigated systematically. In the present study, we examined the blister formation and its fracture process of both silicon crystal (Si) and silicon carbide (SiC) due to helium ion irradiation followed by annealing, and measured the erosion by this process.

2. Experimental procedure

For the samples, we used boron doped Si wafer and SiC converted graphite [20]. The thickness of the sample

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was approximately 0.5 mm. The boron concentration in the Si wafer was $1 \times 10^{21}/m^3$. First, the sample was degassed for 1 h before the helium ion irradiation. The degassed temperature was 1173 or 1323 K for Si or SiC, respectively. After that, the sample was irradiated by helium ions with an energy of 5 keV in an ECR ion irradiation apparatus [20] at RT. The fluence per irradiation cycle was 1×10^{22} He/m^2. For Si, the irradiation direction was [1 0 0]. After the irradiation, the sample was linearly heated up with a ramp rate of 0.5 K/s, and held for 1 h at 1173 or 1323 K for Si or SiC, respectively. During the heating, we measured helium desorption rates of the irradiated sample and the amount of retained helium by a technique of thermal desorption spectroscopy. These irradiation/annealing cycles were repeated four times. After the irradiation or annealing, the surface morphology of the sample was observed by a scanning electron microscope, SEM. In addition, the weight loss of the sample was measured by a microvalance for evaluation of the erosion amount. The resolution of the microvalance was 2 μg/cm^2.

3. Results

Surface morphologies of Si after the 1st cycle of irradiation and annealing are shown in Fig. 1(a) and (b), respectively. For Si, many small blisters were observed at the surface after the first irradiation. The diameter and areal density of these blisters were approximately 50 nm and 5×10^{13} m^{-2}, respectively. In addition to these small blisters, craters with a diameter of approximately 400 nm were observed. It was reported that the craters appear due to the exfoliation of the blister by annealing [13,15]. These craters may be caused by blister fracture due to heavy irradiation in this study. Detailed analysis is necessary to know the reason that these craters appeared. After the first annealing at 1173 K, small pores, which were formed by helium desorption, were observed at the surface. There was no clear correlation between the positions of the pores and the craters. The diameter of the pores was approximately 50 nm and the areal density was approximately 1×10^{12} m^{-2}. The surface roughness gradually increased with the increase of the irradiation/annealing cycle number. In particular, a flaked surface appeared after the 4th annealing.

Surface morphologies of SiC after the 1st cycle of irradiation and annealing are shown in Fig. 2(a) and (b), respectively. For SiC, small blisters were observed on the SiC crystal with a size of 100 μm after the first irradiation. The diameter and areal density of the blisters on SiC after the first irradiation were 60 nm and approximately 2×10^{13} m^{-2}, respectively, similar to those of Si. After the first annealing at 1323 K, small pores were observed at large SiC crystals. The size and areal density of the pores were similar to those of the blisters before the annealing. In addition, the pores, which are similar to those at the large SiC crystal, were observed on small SiC crystals with a size of 1 μm. These pores may be formed by fractures of blisters. With the increase of the irradiation/annealing cycles, the degree of uplifting became large. In addition, the number of small particles (shown in a circle in Fig. 2(a)) on large SiC crystals gradually increased. These small particles may be formed by both the sputtering and redeposition of Si, C and SiC during helium ion irradiation. The blisters, which appeared after the 1st irradiation/annealing cycle, were also observed after the 2nd, 3rd and 4th cycles.

Fig. 3 shows the thermal desorption spectra of helium for Si. After the first irradiation, the spectrum had two peaks at 850 and 1100 K. On the other hand, after the 2nd irradiation, the peak at 950 K was observed with a shoulder at around 800 K. The spectrum did not significantly change when the cycle number increased after

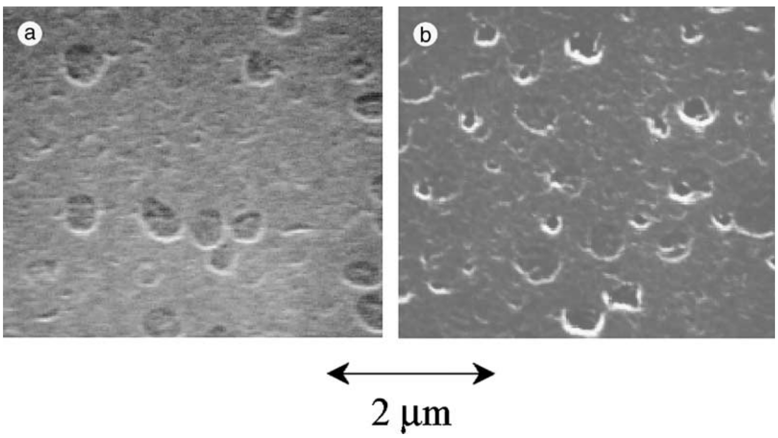

Fig. 1. Surface morphologies of silicon wafer after (a) 1st cycle of helium ion irradiation and (b) 1st cycle of annealing at 1173 K.

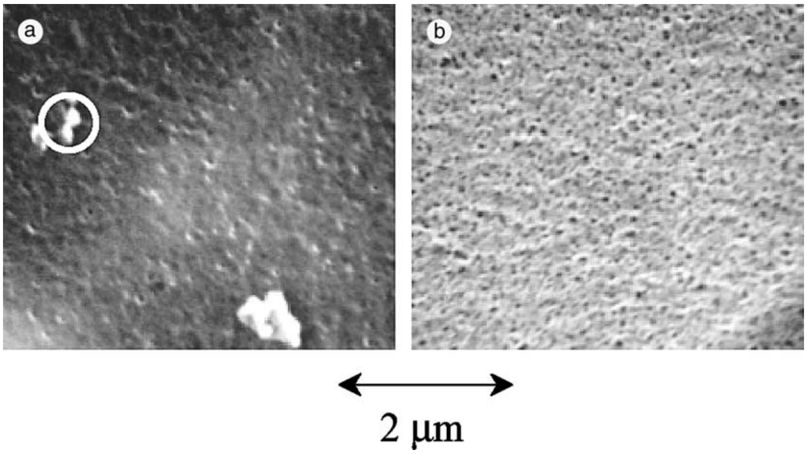

Fig. 2. Surface morphologies of silicon carbide after (a) 1st cycle of helium ion irradiation and (b) 1st cycle of annealing at 1173 K.

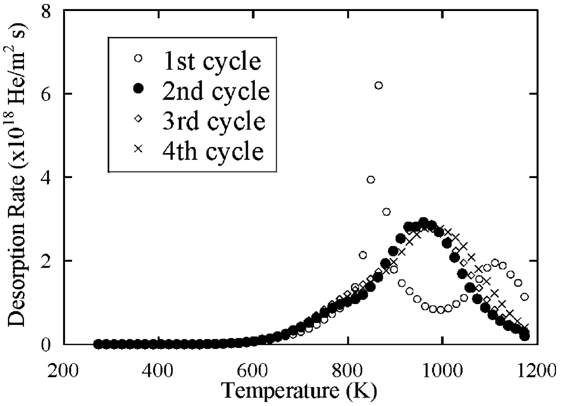

Fig. 3. Thermal desorption spectra of helium from silicon wafer.

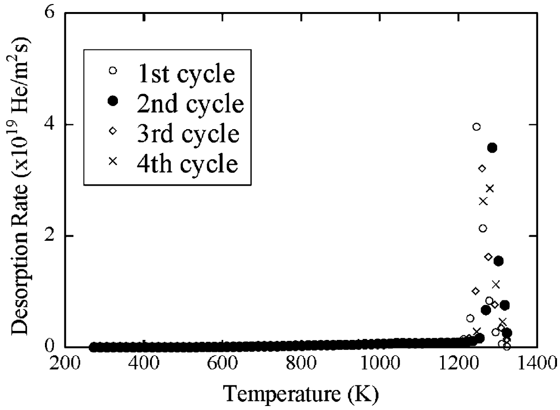

Fig. 4. Thermal desorption spectra of helium from silicon carbide.

the 2nd cycle. The helium desorption rate rapidly decreased while holding at 1173 K, and then desorption was not finally detected during the holding. Therefore, it was concluded that the helium implanted in Si was completely desorbed during the thermal desorption experiment. The integration of the desorption rate with respect to the heating time gives the amount of retained helium. The amount of retained helium was approximately 1.6×10^{21} He/m^2, which was roughly the same in every cycle. Fig. 4 shows thermal desorption spectra of helium for SiC. In the case of SiC, the thermal desorption spectrum of helium was very similar in every cycle. The helium desorption took place at 400 K and the desorption rate gradually increased with the heating temperature, and then a very sharp peak appeared at around 1300 K. The sharp peak may be caused by the sudden fracture of blisters by increases of inner pressure and thermal stress. The helium implanted in SiC was also completely desorbed while holding at 1323 K for 1 h. The amount of retained helium was approximately 3×10^{21} He/m^2, which was roughly the same in every cycle.

Fig. 5 shows the accumulated weight loss after irradiation/annealing cycles for Si. The weight loss after irradiation, i.e., the sputter erosion was 6 μg/cm^2, which corresponds to the sputtering yield of 0.14. This sputtering yield was about two times larger than the calculated value in the [21]. The increase of the yield may be due to flaking and/or effect of thermal annealing before irradiation. After the annealing at 1173 K, the weight loss of Si became a half of that by irradiation. The weight loss by annealing gradually decreased with the increase of the cycle number. However, it is noted that the erosion in the 4th cycle was larger than that in the other cycles. This large erosion may be due to the formation of more weak structures by repeated irradiation/annealing cycle. Fig. 6 shows the accumulated weight loss of SiC after irradiation/annealing cycles. Here, it is

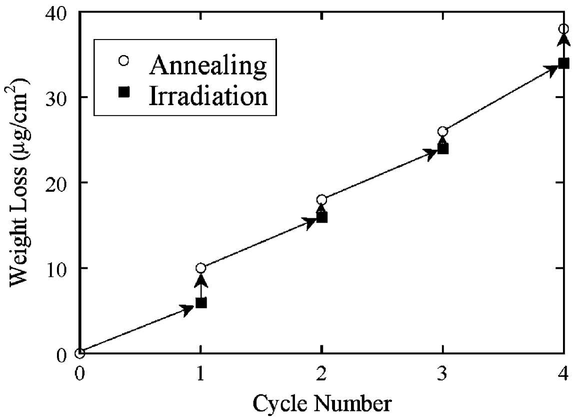

Fig. 5. Accumulated weight loss of silicon wafer after irradiation/annealing processes.

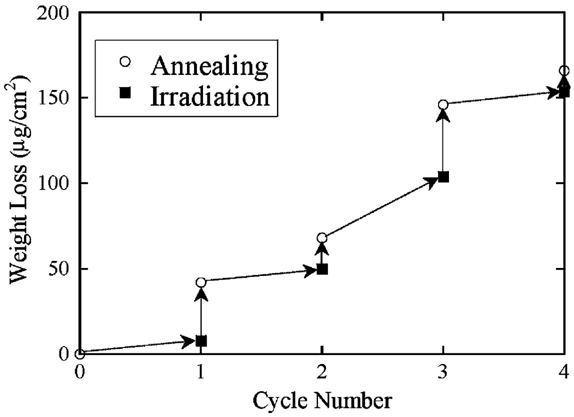

Fig. 6. Accumulated weight loss of silicon carbide after irradiation/annealing processes.

noted that the weight loss of 30 μg/cm^2 sometimes occurred during the handling process, particularly in the 3rd cycle. The erosion amount by ion irradiation was 8 μg/cm^2 (0.24 of sputtering yield), which was similar to the numerically calculated value [21]. The erosion amount by annealing, i.e., fracture of blisters, was 1.5–4 times larger than the value by ion irradiation. The erosion due to the fracture of blisters gradually decreased as the cycle number increased. This tendency was similar to that of Si.

4. Conclusion

In the present study, the blister formations and its fracture processes of Si and SiC due to helium ion irradiation followed by annealing were examined. For Si, many blisters were observed at the surface after the first irradiation. After annealing at 1173 K, small pores formed by helium desorption were observed at the surface. As the cycle number increased, the surface roughness gradually increased. Weight loss by annealing at 1173 K was approximately half of that by ion sputtering. This sputtering yield due to the ion irradiation was approximately twice the reference value. This increase may be due to the fracture of bubbles during ion irradiation. For SiC, small blisters were observed on the SiC crystal after the irradiation. The diameter of the bubble was approximately 60 nm. The fracture of the blister occurred at 1200 K. The erosion amount due to only the process of annealing at 1323 K was several times larger than the value by ion sputtering, although the erosion yield per annealing process gradually decreased with the increase of the cycle number. The sputtering yield due to ion irradiation was roughly the same as the reference value. In the present study, it was found that the erosion by the fracture of blisters was significantly large. Therefore, the present erosion process should affect the impurity level of the core plasma and the lifetime of the plasma facing wall.

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