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Surface morphology changes of silicon carbide by helium plasma irradiation

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ABSTRACT

Silicon carbide (SiC) and its composites are candidate materials for the blanket components and for the first wall in a fusion reactor. If the SiC is used without any armor materials for the first wall, it is exposed by helium (He) plasma as well as hydrogen plasma. Characteristic surface morphology changes are reported for various materials by He plasma exposure. Thus, we exposed SiC specimens to He or simultaneous deuterium (D) and He (D + He) plasma by various conditions and then observed surface morphology changes by SEM. As a result, needle-like structures and whiskers-like structures at the tip were formed in He plasma and D + He irradiation, while only needle-like structures were formed in D plasma. Therefore, it indicated that the effects of He were attributed to form whiskers-like structures. Although the structures are different among He plasma, simultaneous D + He plasma and D plasma irradiations, sputtering is considered to be a dominant process for the formation of the structure formation. However, the effects of He atoms in the structure could also be attributed to form whiskers-like structures.

1. Introduction

Silicon carbide(SiC) has been proposed for the candidates as structural materials in a fusion reactor because of high strength at high temperature and low induced activity [1, 2]. When SiC is used without any armor materials for the first wall, SiC is directly exposed by hydrogen isotopes and helium (He). Several metals (tungsten, palladium, titanium and etc [3-6]) are known to form surface nanostructures of a shape of hole or fiber by helium plasma irradiation. It is known that the shape and dimension of helium induced nanostructure are dependent on fluence and surface temperature [7,8]. Besides metals, silicon surfaces irradiated by helium plasma became needle-rod-like structure [9]. However, morphology changes of SiC by helium plasma irradiation is barely reported [10].

To evaluate surface morphology changes, SiC specimens were exposed to He, deuterium (D), and mixed (He + D) plasmas. In He plasma irradiation, we investigated dependence on He fluence and specimen surface temperature. Formations of nano-scale structures were observed after the irradiations by a field emission scanning electron microscopy (FE-SEM). Classification of these structures are discussed in the beginning of Section 3. Then, dependence of nanostructure size on the total irradiation fluence (Section 3.2) and on the specimen temperature during irradiation (Section 3.3) are discussed for the He irradiation. Comparison among He, D + He and D irradiation is also discussed in Section 3.3.

2. Experiments

SiC specimens were fabricated by a private company using Chemical Vapor Deposition. Very high purity β-SiC was obtained which impurity content is ~100 ppb in total. As received SiC specimens were irradiated by electron cyclotron resonance (ECR) plasmas over the temperature of 593–843 K which was measured by a thermocouple touching to the back surface of the specimen. The ECR plasma exposure was performed using a linear plasma device, named LaPlex, at Osaka University, Suita. Sample temperature was controlled by a rate of coolant flow (water or air) in the sample holder which removes heat load from the plasma. In the beginning of the plasma irradiation, specimen temperatures gradually increased to the desired values. Typically, 20–30 K temperature overshooting for 3–5 min was observed, then stable temperature at the desired value was obtained. Once a steady-state condition was achieved, temperature fluctuations were within 5 K. The temperature range is consistent with the anticipated operating temperatures of the blanket plasma facing surface. Base pressure of the ECR device was $\sim 10^{-7}$ Torr with the gas filling pressure of 5×10^{-2} Torr (He), 2.7×10^{-2} Torr (D), and mixture of 0.3×10^{-2} Torr (He) and 3.0×10^{-2} Torr (D) for plasma operations. The irradiation flux and the irradiation fluence were 1.5×10^{21} He/m^{2}s^1 and (1–10) $\times 10^{25}$ He/m^2, respectively. The incident ion energy was controlled by sample biasing and kept at 80 eV. Surface morphology changes were observed by FE-SEM (Zeiss, ULTRA55) at Kyoto University, Uji. Si/C ratio of

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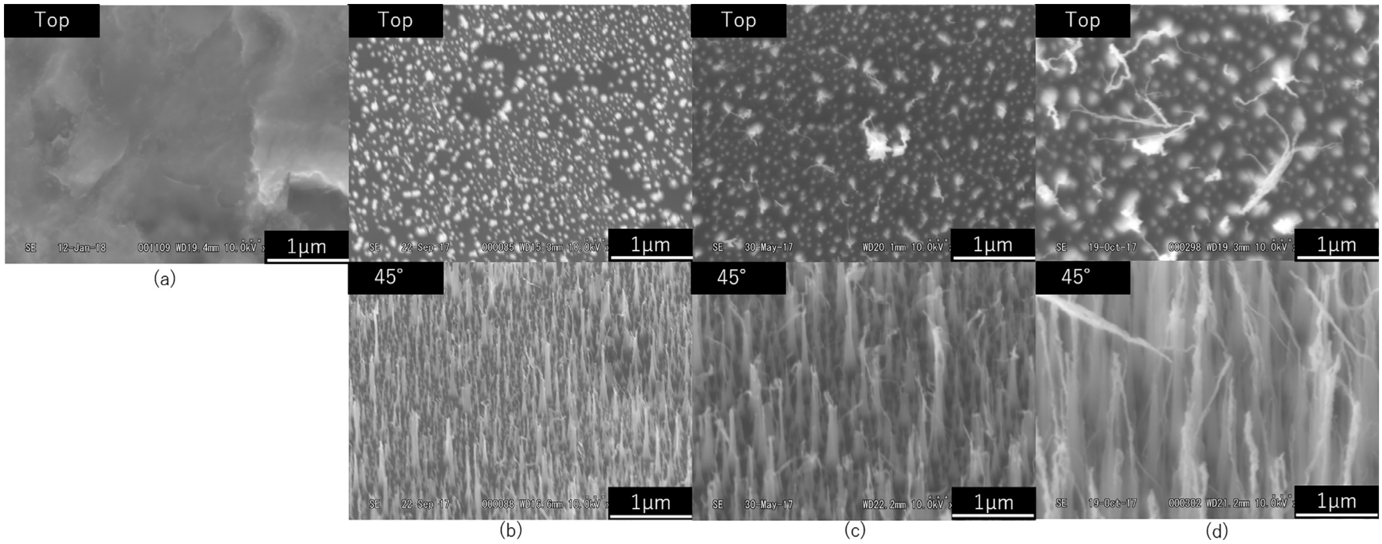

Fig. 1. FE-SEM micrograph of SiC surface before (a) and after irradiation by helium plasma (b) 1×10^{25} He/m^2 (c) 3×10^{25} He/m^2 (d) 1×10^{26} He/m^2 at 783 K.

irradiated surface was analyzed by energy dispersive X-ray analysis (EDX).

3. Result and discussion

Fig. 1 shows FE-SEM images of SiC surface before and after He plasma irradiation. Formation of nanoscale structure was observed. The structure can be classified into two components; a needle-like structure and a whisker-like structure. Needle-like structure is a thick and straight structure grown from the base surface. In contrast, whisker-like structure is a thin and bent structure grown from tip of a needle-like structure. We studied fluence dependence and temperature dependence of the formation of these structures which are important for formation of He induced nanostructure on metals. In addition, surface morphology changes by simultaneous D + He irradiation and D irradiation were also performed to clarify He effects on the structure.

3.1. Size dependence of SiC nanoscale structure on the irradiation fluence

Fig. 1 shows FE-SEM images of SiC surface before (a) and after the He irradiation fluence of (b) 1×10^{25} He/m^2 (c) 3×10^{25} He/m^2 (d) 1×10^{26} He/m^2. Specimen temperature was kept at 783 K for all specimens. It is seen that, as fluence increased, the heights of these structures become larger. It is also seen that the erosion amounts by the irradiation, measured by mass loss, also become larger. Thus, we compared the heights of nanostructure with erosion thickness (see Fig. 2). It is found that the thicknesses of eroded layers are larger than the heights of needle-like structures. This comparison indicates that the needle-like structures are caused by an etching process of the plasma exposure. Unlike the fuzz structures, the nanoscale structures on SiC surface did not grow over the original surface. The exact formation mechanism is unclear, but both sputtering and re-deposition processes should have influences on the formation of the needle-like structures. Influences of surface migration and impurities from the sample holders also need to be considered.

3.2. Size dependence of SiC nanoscale structure on the specimen temperature during irradiation

Fig. 3 shows FE-SEM images of the sample surface after irradiation at (a)593 K (b)783 K (c)843 K. Total fluence was kept at 3×10^{25} He/m^2.

Both needle-like structures and whiskers-like structures at the tip were observed for the entire tested temperature range (593–843 K). As

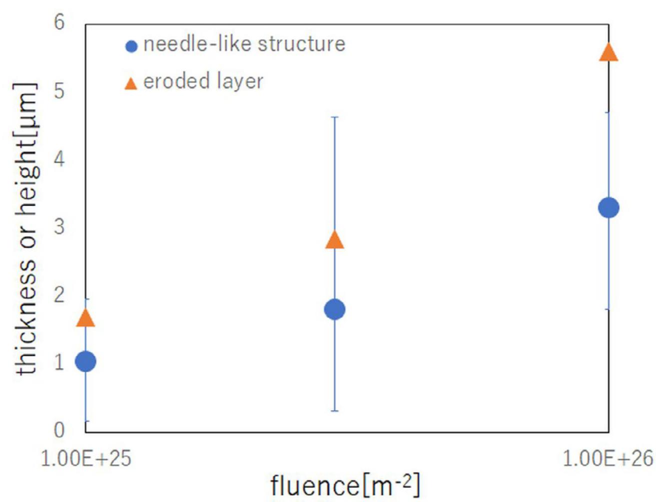

Fig. 2. The relationship by fluence dependence between thickness of eroded layer calculated from mass loss and heights of needle-like structure.

the temperature rises, whiskers-like structures became more apparent and thicker, while needle-like structures showed similar in size as summarized in Fig. 4. Hence, formation of nanoscale structures on SiC is weakly influenced by temperature, which is different from the cases for refractory metals (e.g. tungsten). The formation of He induced fuzz structures on tungsten [3] is strongly influenced by the specimen temperature during irradiation. It is known that sputtering cones, observed not only by He irradiation but by other ion species, can be formed in a wide temperature range. Thus, the formation of needle-like structures could be simply dominated by sputtering, while formation of whisker-like structures was temperature-controlled processes to some extent.

3.3. SiC nanoscale structure formation by simultaneous D + He plasma irradiation or D plasma irradiation

Fig. 5 shows FE-SEM images of surface morphology after the simultaneous D + He (10%) plasma irradiation. Here, 10% of He content is based on the neutral gas pressure. The irradiation was performed with the specimen temperature of 743 K, incident energy of 80 eV and total fluence of 2×10^{25} D + He/m^2.

The needle-like structures were observed mainly in the D + He

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