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Disruption and sputtering erosions on SiC doped CFC

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Abstract

This paper describes the disruption and sputtering erosions on newly developed SiC doped Carbon fiber reinforced composite (CFC). Disruption experiments were performed under heat fluxes of 1000–2000 MW/m^2 with pulse length of 2–4 ms in the temperature range from RT to 1000°C at the JEBIS facility. Sputtering experiments were performed under the deuterium particle flux of 1–2 × 10^{20} m^2/s with energy of 50–100 eV in the temperature range from 200°C to 600°C at the SLEIS facility. As a result, it is clear that the sputtering yield from 200°C to 450°C is about 0.09 and gradually decreases to 0.067 with increasing sample temperature and that the disruption erosion of SiC doped CFC was about double that of the non-doped CFC and the erosion was mainly caused by the loss of the matrix part.

1. Introduction

Carbon fiber reinforced composite (CFC) with high thermal conductivity is selected as a candidate material of the armor tiles for ITER divertor [1]. Since CFC is eroded by chemical sputtering with hydrogen isotopes during normal operation, boron (B) or silicon (Si) doping into the CFC is investigated for the reduction method of the chemical sputtering. In the previous work [2,3], we developed B_4C doped CFC and recognized B_4C doping is effective for the suppression of the sputtering behavior by a scanning electron microscopy (SEM) observation. However, the use of B to the armor tiles is not desirable in view point of the nuclear reaction with neutron. Then, we tried to develop SiC doped CFC and successfully performed the development of the conventional fabrication method. Since erosion data is important for the prediction of the life time of divertor armor tiles, it is necessary to measure the erosion data of the new SiC doped CFC. This paper presents the sputtering and disruption erosions on the newly developed SiC doped CFCs.

2. Newly developed SiC doped CFC and sample preparation

The fabrication method of the newly developed SiC doped CFC is almost the same as that of B_4C doped CFC [2]. SiC was doped into only the matrix part of CFC because of the prevention from the reduction of the thermal conductivity. SiC doping into the matrix part was carried out by sticking SiC on the carbon fibers in the fabrication process of CFC. The content of SiC in the CFC was controlled by the quantity of SiC powder in the solution. The advantage of this doping method is very simple, it does not lead to increase of the fabrication cost and time.

In this experiment, the SiC content in CFC was 10 wt%, which means Si content was 5%. The ratio of the fiber weights to the total weight of this base CFC was around 60% and the diameter of the fibers was ~10 μm. Typical dimensions of the sample were 25 mm in width, 25 mm in length and ~3 mm in height for the sputtering experiment and ~15 mm in height for the disruption

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experiment. All samples were washed in the acetone bath with an ultrasonic cleaner for 10 min to completely remove carbon powder from the samples. After washing, all samples were baked up to 1000°C by an electric furnace in vacuum for 30 min in order to reduce outgassing from the samples.

3. Sputtering erosion

The SLEIS facility was applied to the present experiments. The SLEIS facility can produce high particle flux of $\sim 10^{21}$ m^{-2}/s at very low particle energy of 50–150 eV by means of tungsten wire acceleration grids which were originally developed in JAERI [4]. The beam extraction area of SLEIS is 60 mm × 100 mm and beam uniformity of ± 5% can be achieved over an area of 40 mm × 60 mm at the sample position. The species of the charged particles were analyzed with a magnetic mass-analyzer. Since more than 85% of total charged particles was D_3^+ for deuterium beam, beam energies were indicated as one-third of the acceleration voltages. The particle flux was calculated with ion current, i.e. total particle fluxes were obtained as summation of D^+, twice of D_2^+ and three times of D_3^+ ion fluxes. Light impurity ions such as oxygen were less than 1%. The neutral particle flux at the sample surface was estimated to be less than a few percent.

The test sample was placed about 15 cm downstream from the ion source. The samples were heated up to 600°C by using an electric heater and the temperature was measured by the thermocouples during irradiation. The weight loss of the sample was measured in atmosphere by a microbalance. Sputtering yields of the sample were calculated from the weight loss and the particle fluences. The change in the surface composition was measured with an electron probe micro-analyzer (EPMA). Irradiation conditions for samples in this experiment are summarized in Table 1.

Table 1
Deuterium and sample conditions of sputtering experiments

Ion source species	D_1^+ (~10%), D_2^+ (~5%), D_3^+ (~85%)
Acceleration voltage	200 V
Flux	$1–2 \times 10^{20}$/m^{-2}·s
Fluence	$1–3 \times 10^{24}$/m^{-2}
Target temperature	200–600°C

Temperature dependence of the sputtering yield of SiC doped CFC is shown in Fig. 1. The error of the data is included in the circles. The sputtering yield from 200°C to 450°C is almost constant, 0.09, and gradually decreased to 0.067 with increasing sample temperature. The sputtering yield of pure SiC has no temperature dependence between 400°C and 600°C with 1 keV hydrogen beam as shown in previous work [5]. On the other hand, the sputtering yield of 1D CFC with 67 eV deuterium beam has a broad peak around 400°C, which means the sputtering yield at 350°C and 450°C is larger than that at 200°C and 600°C, as shown in recent data [6]. Therefore, it is considered that the temperature dependence of SiC doped CFC, which was composed with SiC doped matrix part and non-doped fibers, is a combination of pure SiC and 1D CFC in the higher temperature range (350–600°C). However, more investigations in the lower temperature range (200–350°C) are necessary since the sputtering yield at 200°C is not lower than that at 350°C.

The measurement by EPMA was performed at three different points in the matrix part of each sample. The Si

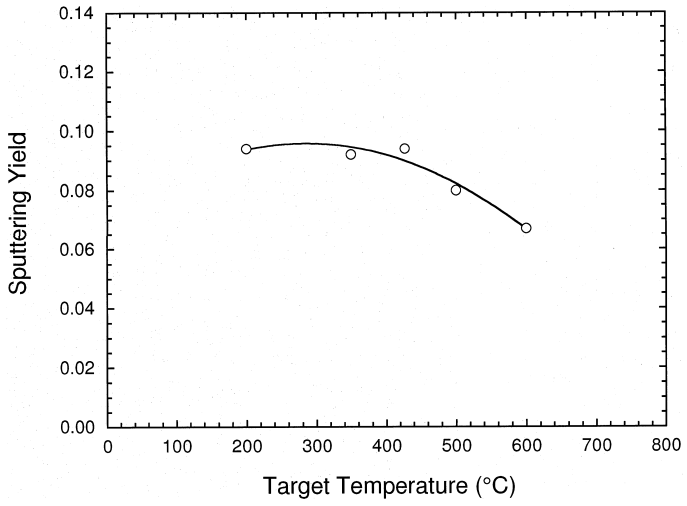

Fig. 1. Temperature dependence of sputtering yield of SiC doped CFC with deuterium beam irradiation.

Table 2
Si concentration in the matrix part before and after deuterium beam irradiation

	Si concentrations (%)		
	Before irradiation	Irradiated at 350°C	Irradiated at 600°C
1	1.24	15.45	4.16
2	5.54	7.37	16.22
3	4.67	9.69	7.15
Average	3.82	10.84	9.28
Change ratio (after/before)		2.84	2.43

concentration of the three points, the average of the three points and the change ratio after to before irradiation are shown in Table 2. In this measurement, Si concentration increased up to 2–3 times after irradiation. This result insists on the existence of the selective sputtering of carbon in the matrix part, which means that the remain of SiC crystals in the matrix part are by irradiation. The change ratio of Si concentration at 350°C was 17% larger than that at 600°C. However, this difference is less than the difference of 37% in the sputtering yield both at 350°C (0.092) and 600°C (0.067).

The surface morphologies of un-irradiated and irradiated samples are shown in Fig. 2. Many crystals were observed on the surface in the matrix part of all irradiated samples. Since the sputtering yield of SiC is generally one order lower than that of carbon [5], the crystals are estimated to be SiC crystals in addition to the result of EPMA measurement. No morphological difference in the matrix part was observed on the irradiated surface at 200°C and 600°C, although the sputtering yields at 200°C were 40% higher than that at 600°C.

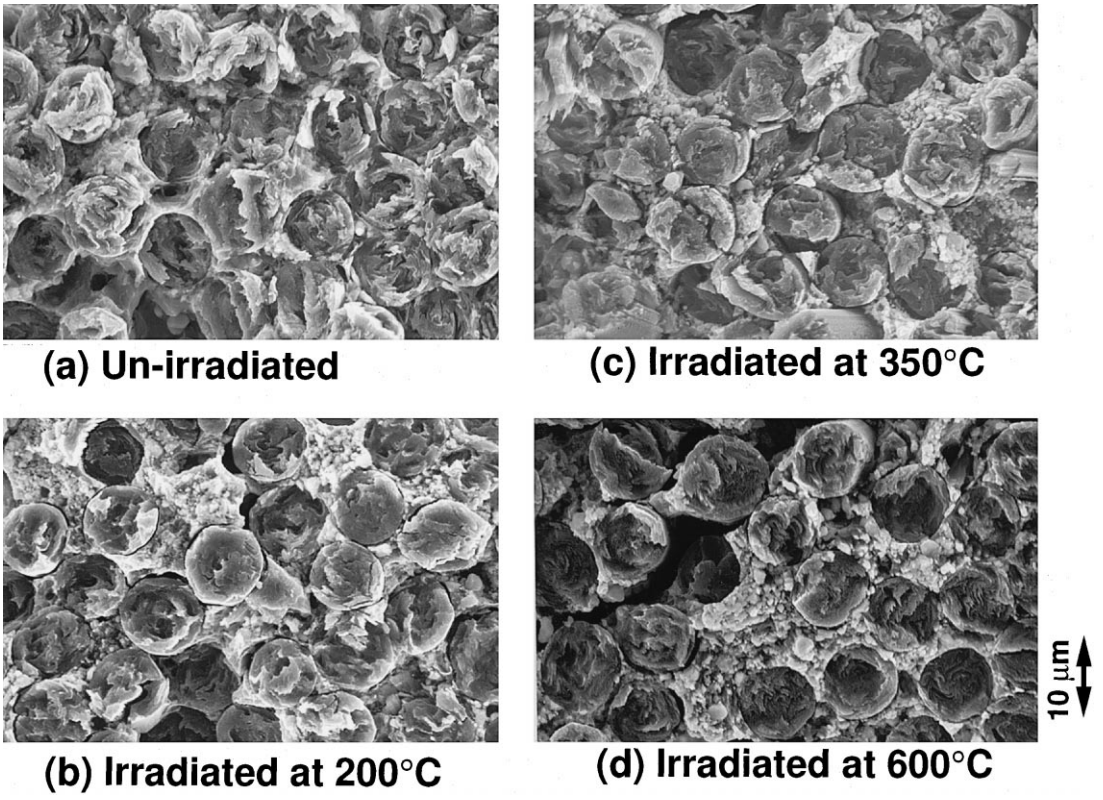

Fig. 2. Surface morphologies of SiC doped CFC before and after deuterium beam irradiation by SEM observation.

Table 3
Electron and sample conditions of disruption experiments

Electron energy	70 keV
Peak heat flux	1000, 2000 MW/m^2
Pulse length	2.0, 4.0 ms
Peak absorbed energy	4 MJ/m^2
Sample temperature	RT∼1000°C.

4. Disruption erosion

Disruption experiment was carried out in the JEBIS facility [7]. The condition of the electron beam irradiation is shown in Table 3. The test was performed under two different heat loads, i.e. high and low heat flux conditions. The high heat flux condition was 2000 MW/m^2 for 2 ms and the low heat flux condition was 1000 MW/m^2 for 4 ms. The absorbed energy density was 4 MJ/m^2 in both cases. Samples were heated by a single beam shot. The samples were preheated with very low heat flux electron beam (0.8 MW/m^2) before irradiation with the disruption heat loads. The temperature of the samples before irradiation was measured with the infrared camera. The weight loss of the samples was measured with a microbalance, and the surface morphology was observed with SEM.

The weight loss of SiC doped and non-doped CFCs is shown in Fig. 3. In the figure, the heat flux and the preheated temperature dependence of the weight loss of SiC doped CFC were observed. Namely the weight loss of SiC doped CFC with a heat flux of 2000 MW/m^2 was about double of that with a heat flux of 1000 MW/m^2 at the same preheated sample temperature. The weight loss of SiC doped CFC at 1000°C was larger than that at RT at the same heat flux. Both dependences are almost the same in the case of 10% B_4C doped CFC. The weight loss of SiC doped CFC was larger than non-doped CFC at similar irradiation conditions. The surface morphology of SiC doped CFC after irradiation is shown in Fig. 4. It is observed that the matrix part was selectively eroded for all irradiation conditions. From these experimental results, the weight loss of SiC doped CFC by the electron beam irradiation mainly came from the loss of the matrix part, which was caused by the lower thermal conductivity compared with fibers.

5. Conclusions

Sputtering and disruption experiments on SiC doped CFC were performed in JEBIS and SLEIS and the results are summarized as follows

Sputtering erosion:

(1) The sputtering yield from 200°C to 450°C is almost 0.09 and gradually decreased to 0.067 with increasing sample temperature.

(2) SiC crystals were observed in the matrix part after deuterium irradiation by SEM observation.

(3) Increase of Si concentration in the matrix part was observed by the deuterium irradiation.

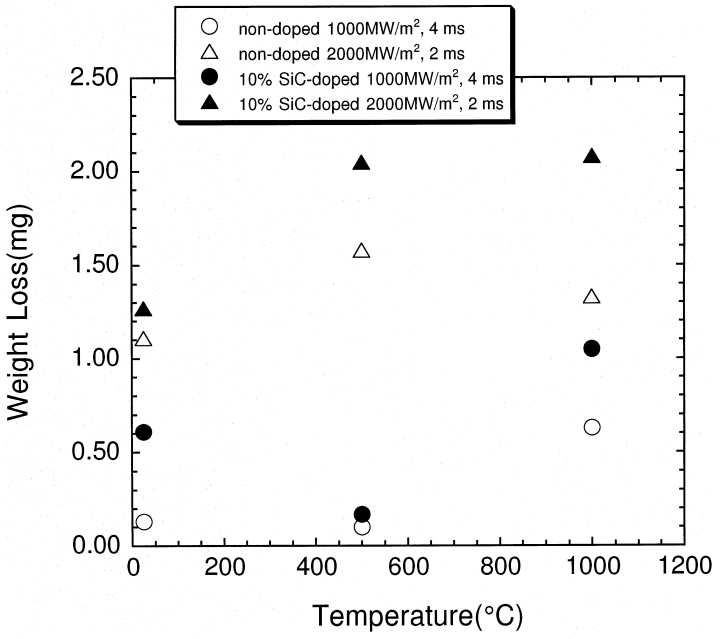

Fig. 3. Temperature dependence of weight loss of SiC doped CFC on electron beam irradiation.

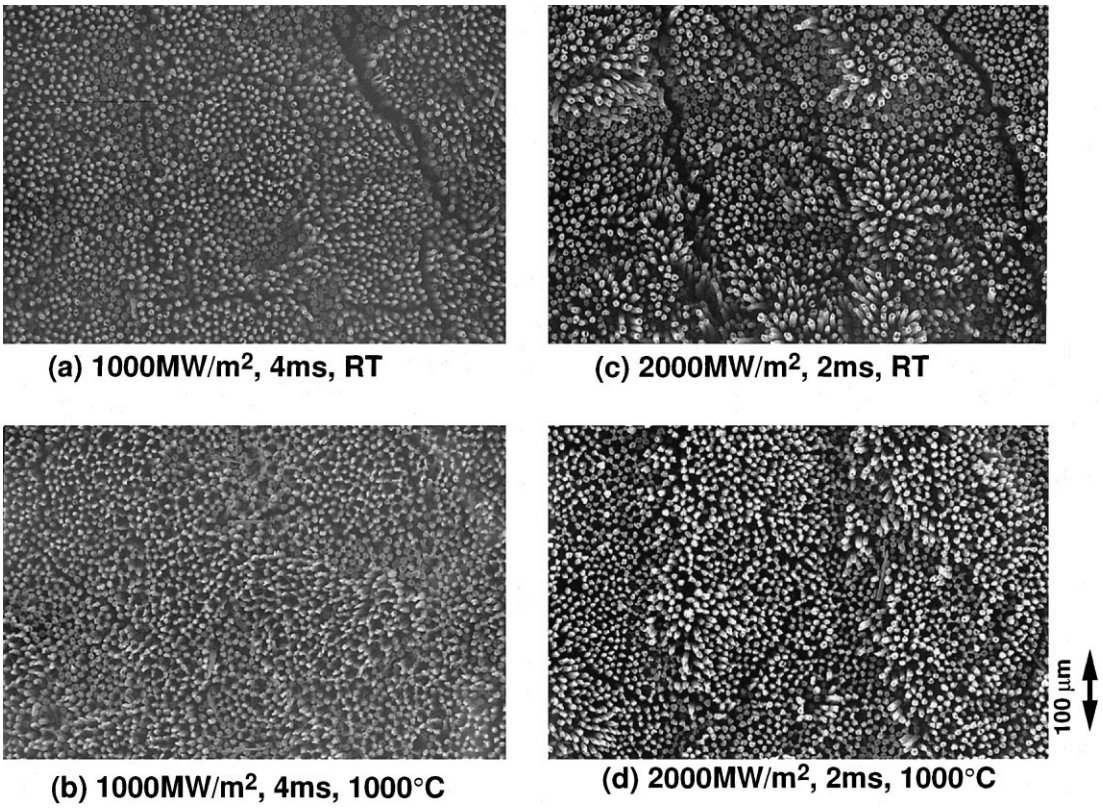

Fig. 4. Surface morphologies of SiC doped CFC after electron beam irradiation by SEM observation.

Disruption erosion:

(4) The disruption erosion with a heat flux of 2000 MW/m^2 was about double of that with a heat flux of 1000 MW/m^2 at the same incident power of 4 MJ/m^2.

(5) The disruption erosion at 1000°C was larger than that at RT under the same heat fluxes.

(6) The disruption erosion of SiC doped CFC was about double that of non-doped CFC and the erosion was mainly caused by the loss of the matrix part.

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