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Sputtering experiments on B_4C doped CFC under high particle flux with low energy

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Abstract

Sputtering property of newly developed 5–10% B_4C doped CFCs with high thermal conductivity, ~ 160 W/m/K, was investigated under high particle flux with low energy by using the super low energy ion source (SLEIS) facility at Japan Atomic Energy Research Institute (JAERI). Hydrogen beam irradiation was performed with particle flux of $2-3 \times 10^{20}/m^2/s$ and particle fluence of $3-9 \times 10^{24}/m^2$ at 50 eV. Reduction of erosion of the matrix of CFCs by B_4C doping was observed with a scanning electron microscope, while the carbon fibers were preferentially eroded. The sputtering yield of the B_4C doped CFCs was 0.025–0.034.

Keywords: Physical erosion; Low *Z* wall material

1. Introduction

Plasma facing components in fusion experimental reactors such as ITER are exposed to high particle flux with low energy from plasma during normal operation, which is predicted up to $\sim 10^{24}/m^2/s$ with ~ 50 eV [1]. Therefore, it is very important to investigate the sputtering yields of the plasma facing materials by the plasma particles, in particular hydrogen isotopes, for evaluation of the life time. Carbon fiber reinforced carbon composites (CFCs) are one of the promising materials for the plasma facing materials because of their high thermal conductivity and high tensile strength. In our previous studies [2], sputtering properties of various types of CFCs i.e. MFC-1, MCI-felt and CX-2002U, were investigated under high particle flux with low energy in the SLEIS facility and it was found that CFCs, which consist of fibers and matrix, were selectively sputtered by the hydrogen isotopes from the results of SEM observations, namely the sputtering erosion of the matrix was larger than that of the fibers. This fact implies that suppressing erosion of the CFCs matrix would be effective to reduce the sputtering erosion of CFCs. Based on this consideration, B_4C doped or overlaid CFCs have been envisaged for the plasma facing materials, because boron carbide (B_4C) is well known as low sputtering material [3–6]. In particular B_4C doped CFCs have been preferred for next fusion devices, because the plasma facing materials will be exposed to severe particle fluence and high heat loads from reactor plasma. Since the thermal conductivity of B_4C itself is as low as about 40 W/m/K, it is critical to develop B_4C doped CFCs without reduction of the thermal conductivity. From this point of view, we proposed a combination with a unidirectional CFC, which has high thermal conductivity and the B_4C doped carbon matrix and successfully developed the B_4C doped CFCs which has the thermal conductivity of as high as around 160 W/m/K [7]. In this report, the sputtering characteristics of newly developed B_4C doped CFCs against the hydrogen beam is described.

2. Experimental conditions and materials

The SLEIS facility [8] 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

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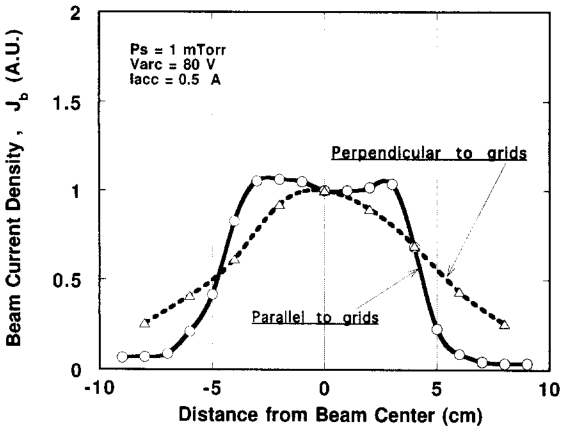

Fig. 1. Distribution of the hydrogen beam, which is extracted from tungsten wire grids. Pressure of the vacuum chamber was 1 mTorr. Arc voltage and current were 80 V and 0.5 A, respectively. Beam current density parallel to the grids is similar to a rectangular profile and that which is perpendicular to the grids is similar to Gaussian.

50–150 eV by means of tungsten wire acceleration grids which were originally developed in JAERI. The beam extraction area of the ion source is 100 × 100 mm with a beam transparency of about 50%. The typical beam profile of the SLEIS facility is shown in Fig. 1. Hydrogen gas pressure in the arc chamber, arc voltage and arc current were 1 mTorr, 80 V and 0.5 A, respectively . The abscissa is the distance from the beam center and the ordinate is the hydrogen beam current density in arbitrary unit. The hydrogen beam uniformity of ±5% can be achieved over an area of 40 × 60 mm at the sample position. The species of the charged particles were analyzed with a magnetic mass-analyzer. Since more than 90% of the total charged particles was H_3^+ for hydrogen beam, beam energies were indicated as one-third of the acceleration voltages. The particle flux was calculated with ion current i.e. total particle flux were obtained as summation of H^+, twice of H_2^+ and three times of H_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 %. The test sample was placed about 15 cm downstream from the ion source. Temperature of the sample

Table 1
Irradiation conditions of sputtering experiments with SLEIS

Irradiation particles	Hydrogen
Acceleration voltage (V)	150 (as E_0)
Particle energy (%)	$E_0/3$ (85–90)
	$E_0/2$ (3–5)
	E_0 (7–10)
Particle flux density (/m²/s)	2–3 × 10^{20}
Particle fluence (/m²)	3–9 × 10^{24}
Temperature (°C)	200–300

Table 2
Characterization of B_4C doped CFCs for sputtering experiments

Doping of B_4C	
Diameter of B_4C particles (μm)	0.33 (av.)
Contents of B_4C (%)	5–10
Heat treatment (°C)	2000
Thermal conductivity (W/m/K)	~ 160 (at RT)
Base CFC	
Type	UD CFC (Tonen co.)
Diameter of fibers (μm)	~ 10
Fibers ratio (V_f) (%)	60
Density (g/cm³)	1.7
Thermal conductivity (W/m/K)	~ 500 (at RT)
	(after heat treatment at 3200°C)

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. Irradiation conditions for samples in this experiments are summarized in Table 1.

The fabrication method and major material properties of B_4C doped CFCs are summarized in Table 2. B_4C doping into the matrix of CFCs was carried out by sticking B_4C on the carbon fibers in the fabrication process of CFCs. The solution containing B_4C powder, whose diameter was approximately 0.33 μm, was used in the sticking process. The contents of B_4C in the CFCs were controlled by the quantity of B_4C powder in the solution. In this experiment, the B_4C contents in CFCs were 5 and 10 wt%. The ratio of the fibers weight to the total weight of this

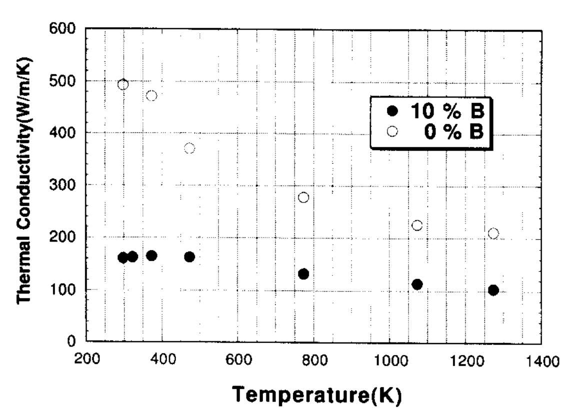

Fig. 2. Temperature dependence of thermal conductivity of newly developed B_4C doped CFC and non-doped CFC. Heat treatment temperature of B_4C doped CFC and non-doped CFC are 2000 and 3200°C, respectively.

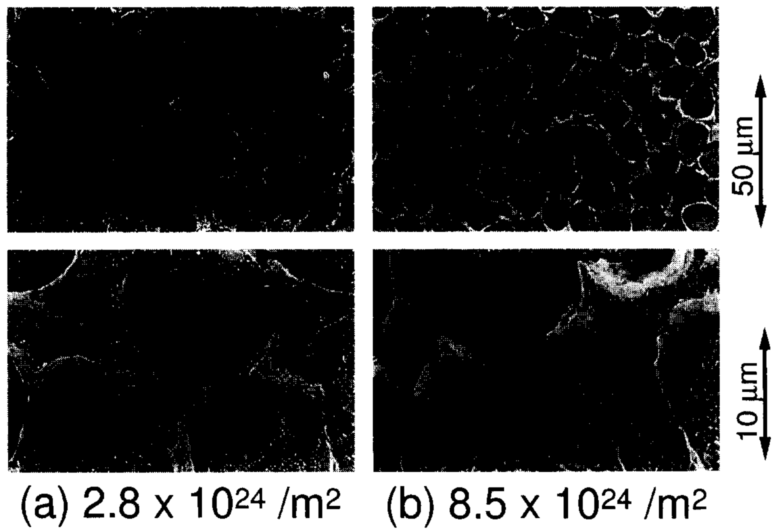

Fig. 3. Photographs of 5% B_4C doped CFCs after hydrogen beam irradiation with different particle fluence. Particle energy and flux were 50 eV and 2.5–2.8 × 10^{20}/m^2/s, respectively. (a) Particle fluence: 2.8 × 10^{24}/m^2, weight loss: 1.18 mg, sputtering yield: 0.034 and (b) particle fluence: 8.5 × 10^{24}/m^2, weight loss: 2.63 mg, sputtering yield: 0.025.

base CFC was around 60% and the diameter of the fibers was ~ 10 μm. Although the graphitization temperature of non-doped CFC was 3200°C for getting the high thermal conductivity, that of B_4C doped CFCs were limited to less than 2000°C for avoiding degradation of the thermal conductivity by the diffusion of doped B_4C from the matrix part to the fibers. The temperature dependence of the thermal conductivity of B_4C doped and non-doped CFCs are shown in Fig. 2. The thermal conductivity in parallel with fibers direction was measured up to 1000°C by a laser flash method. The material has the high thermal conductivity of ~ 160 W/m/K at room temperature and still has 100 W/m/K at around 1000°C. Typical dimensions of the sample were 25 mm in width, 25 mm in length and ~ 2 mm in height, which is sufficiently small compared with the uniform beam area of 40 × 60 mm. All samples were washed in the acetone bath with an ultrasonic cleaner for 10 min to completely remove carbon powder from the

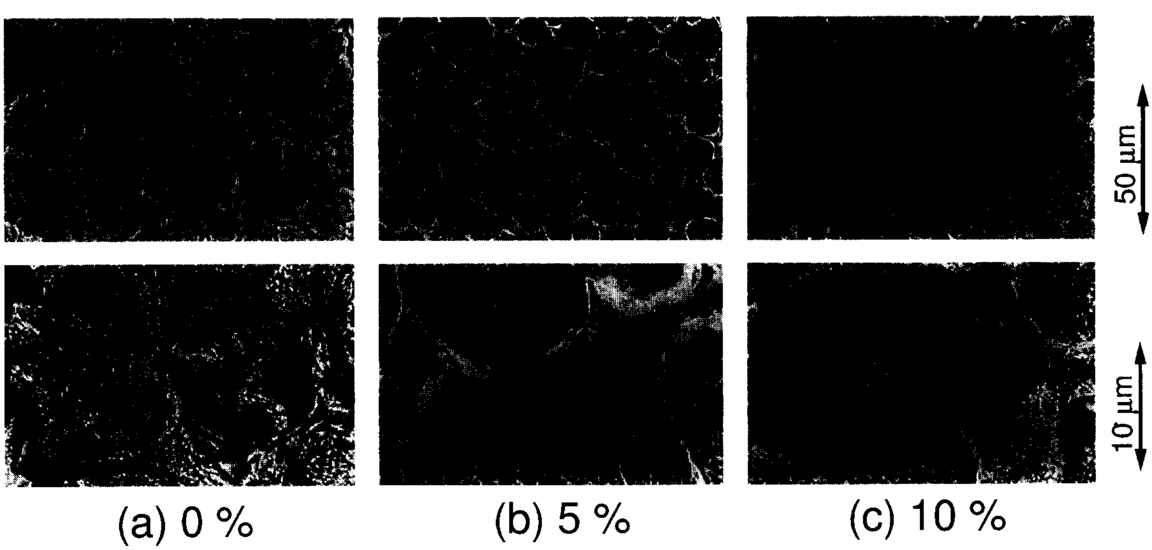

Fig. 4. Photographs of B_4C doped CFCs and non-doped CFC after hydrogen beam irradiation with similar particle fluence. Particle energy and flux density were 50 eV and 1.9–2.5 × 10^{20}/m^2/s, respectively. (a) particle fluence: 7.0 × 10^{24}/m^2, weight loss: 2.39 mg, sputtering yield: 0.027, (b) particle fluence: 8.5 × 10^{24}/m^2, weight loss: 2.63 mg, sputtering yield: 0.025 and (c) particle fluence: 7.5 × 10^{24}/m^2, weight loss: 2.94 mg, sputtering yield: 0.031.

samples. After washing, all samples were baked up to 1000°C by an infrared heater in vacuum for 30 min.

3. Results and discussions

The sputtering experiments of B_4C doped CFCs were carried out under the hydrogen particle flux of $1.9–2.8 \times 10^{20}/m^2/s$ and the hydrogen particle fluence of $2.8–8.5 \times 10^{24}/m^2$ at 50 eV. The bulk temperature of the sample reached up to 200–300°C during irradiation. The micrographs of the 5% B_4C doped CFCs after irradiation with the different fluences are shown in Fig. 3. In the surface of both samples, partial erosions in the matrix were observed, which would be attributable for the irregular distribution of B_4C particles. This fact implies that further improvement of sticking procedure of B_4C powder onto the fiber would be necessary for uniform distribution of B_4C particles in the matrix to reduce sputtering erosion of matrix effectively. At the particle fluence of $8.5 \times 10^{24}/m^2$, fine structures of the erosion were clearly observed on the top of the fibers. Although the reason of the creation of the fine structures is not clear, it might be related with the irregular distribution of the chemical activity in the fibers.

The micrographs of the 5–10% B_4C doped CFCs and non-doped CFC after irradiation at the particle fluence of $7.0–8.5 \times 10^{24}/m^2$ are shown in Fig. 4. In the surface of the non-doped CFC, preferential erosion of the matrix was observed. On the other hand, the matrix of the 5–10% B_4C doped CFCs were hardly eroded and many remaining B_4C particles were observed in the matrix parts. This means that the existence of the B_4C particles effectively suppresses the sputtering erosion of the matrix by hydrogen beam. However, the carbon fibers of the B_4C doped CFCs are preferentially eroded and also the fine structures can be seen. The fine structures in the fibers of the 10% B_4C doped CFCs is much more than that of 5% B_4C doped CFCs.

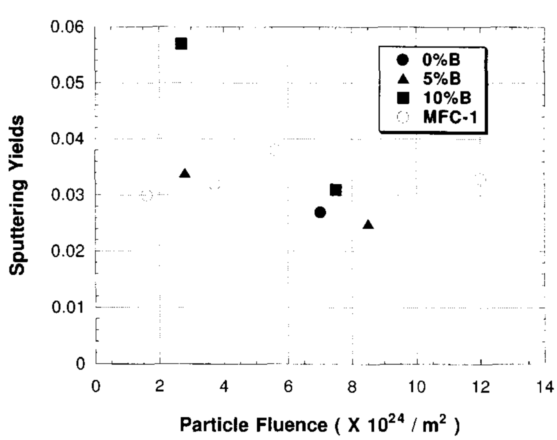

Fig. 5. Sputtering yields of B_4C doped CFCs and non-doped CFC with different particle fluence. Particle energy and flux density were 50 eV and $1.9–2.8 \times 10^{20}/m^2/s$, respectively. Sputtering yields of MFC-1 were also plotted as Ref. [2].

The sputtering yields of 5–10% B_4C doped CFCs and the non-doped CFC are shown as a function of the particle fluence in Fig. 5. In the present experiments, the sputtering yields of 0.025–0.034 are obtained for both the B_4C doped CFCs and the non-doped CFCs except 10% B_4C doped CFC at a particle fluence of $2.8 \times 10^{24}/m^2$. The sputtering yields of MFC-1 are also shown in Fig. 5, which was measured in the previous experiments [2]. As mentioned above, the SEM observations show that the sputtering erosion of the B_4C doped CFCs are suppressed in the matrix because of B_4C doping, but the carbon fibers are preferentially eroded. This fact implies that the reduction of the erosion of the matrix by the B_4C doping would be compensated with the increase of the erosion of the fibers. Further it should be noted that the grade of the graphitization of the fibers of B_4C doped CFCs is lower than that of non-doped CFC because the graphitization temperature of B_4C doped CFCs is lower than that of non-doped CFC. If the different grade of the graphitization of the fibers might influence the sputtering erosion, it would be possible that the fiber erosion of B_4C doped CFCs become larger than that of non-doped CFC.

4. Conclusions

The sputtering experiments on B_4C doped CFCs were carried out by means of the high particle flux facility, SLEIS, in JAERI and the results can be summarized as follows;

(1) The sputtering yields of 0.025–0.034, which are measured by weight loss method, are obtained for both the B_4C doped CFCs and the non-doped CFCs except for 10% B_4C doped CFC at a particle fluence of $2.8 \times 10^{24}/m^2$. It could be attributable to the fact that the reduction of the erosion of the matrix by the B_4C doping would be compensated with the increase of the erosion of the fibers.

(2) With the SEM observation, a reduction of the matrix erosion of CFCs by B_4C doping is observed, while the preferential erosion of the CFC carbon fibers are found.

(3) Fine structures in the fibers of B_4C doped CFCs after irradiation were observed.

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