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Temperature and angular dependences of sputtering yield of B_4C-carbon fiber composite irradiated with low energy deuterium ions

R. Jimbou [a,b,*], K. Nakamura [b], V. Bandourko [b], M. Dairaku [b], Y. Okumura [b], M. Akiba [b]

[a] *Hitachi Research Laboratory (On loan to JAERI), Hitachi Ltd., Ohmika-cho, Hitachi 319-1292, Japan*
[b] *NBI Research Laboratory, JAERI, 801 Mukohyama, Naka Fusion Research Establishment, Naka-machi, Ibaraki-ken 311-0193, Japan*

Abstract

The sputtering conditions at the divertor in next fusion experimental reactors such as ITER were simulated by using a super low energy ion source (SLEIS). B_4C-carbon fiber composite was sputtered with low energy and high flux deuterium ions from SLEIS. The flux was as high as 1–2 × 10^{20} $D^+/m^2/s$ with D_3^+ of 200 eV. The target temperatures were 200–700°C, namely, in the chemical sputtering region, the incident angles were 0–60° and two kinds of surface finish of samples were used. The results were compared with those of SiC doped CFC and 2D (two directional)-CFC.

The chemical sputtering yields of 38% B_4C-carbon fiber composite at temperatures of 200–700°C were smaller than those of 2D-CFC, showing a temperature dependence similar to those of 10% SiC doped 1D-CFC. The chemical sputtering yields at 500°C of 38% B_4C-carbon fiber composite and 10% SiC doped 1D-CFC decreased with the incident angle. The sputtering yields of 38% B_4C-carbon fiber composite were lower than those of 10% SiC doped 1D-CFC and 2D-CFC (CX-2002U). The surface structures of the samples after sputtering examined with SEM have greatly differed from those before sputtering, showing a pattern of needle-like leaves.

Keywords: Angle of incidence; Boron carbide tiles; Carbon fiber composite; Chemical sputtering; Deuterium irradiation; Erosion of graphite; Low energy deuterium incidence; Plasma facing material; Plasma material interaction; Sputtering yield

1. Introduction

Carbon fiber-reinforced carbon composites (CFCs) are candidate materials for the armor tiles of the plasma facing components in next fusion experimental reactors such as ITER, though CFC has a relatively low erosion resistance to hydrogen ions in the physical and chemical sputtering temperature regions. In order to improve this lower erosion resistance, 38% B_4C-carbon fiber composite and 10% SiC doped 1D-CFC have been recently developed [1,2].

In this paper, B_4C-carbon fiber composite and SiC doped CFC with machined and polished surfaces were sputtered with SLEIS in the chemical sputtering temperature region, including angular dependence measurements of the sputtering yield. These results were compared with the sputtering yields of 2D-CFC [3].

2. Experimental

SLEIS has been constructed to simulate the sputtering conditions at the divertor in ITER (energy: 50–100 eV, flux: 10^{22}–10^{23} $D^+/m^2/s$) and has a wide irradiation area (100 × 90 mm) to cover the test sample (25 × 25 mm) with a uniform flux density [4]. A schematic of SLEIS is shown in Fig. 1.

* Corresponding author. Tel.: +81 29 282 6956; fax: +81 29 282 6305; e-mail: jinbou@popsvr.tokai.jaeri.go.jp

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Two kinds of material, 38% B_4C-carbon fiber composite (abbreviated as the B_4C–CF composite hereinafter in this paper) and 10% SiC doped 1D-CFC (abbreviated as the SiC doped CFC), as shown in Table 1, were sputtered by SLEIS with high fluxes of $1{\sim}2 \times 10^{20}$ $D^+/m^2/s$ at 200 eV, as shown in Table 2. The B_4C–CF composite was made with a hot-pressing process and has a higher density (rare porosity) and uniform distribution of B_4C [1]. All samples, which include ones machined from raw materials and ones polished after machining, were washed two times in an ethyl alcohol bath with an ultrasonic vibrator for 10 min to remove carbon powder from the samples. After washing, the samples were heated in a vacuum furnace up to 900°C to remove the ethyl alcohol. Test samples were placed about 10 cm downstream from the ion source and sputtered at temperatures ranging from 200°C to 700°C with the incident angle of 30°. The angular dependence of the sputtering yields was measured at 500°C with the incident angle of 0° (normal to sampe surface), 30°, 45° and 60°.

Since more than 85% of the total charged particles were analyzed to be D_3^+ with the magnetic mass analyzer [4], the beam energy of almost particles was one third of the energy of D_3^+ (200 eV), that is, 67 eV. Sputtering yields were calculated from the weight loss of irradiated samples and the fluences, where the error in the weight loss method was less than ±4%.

The surfaces of test samples before and after sputtering at 500°C with an incident angle of 45° were examined with SEM to see the change in the surface structure after sputtering.

3. Experimental results

Fig. 2 shows the temperature dependence of sputtering yields of the B_4C–CF composite and the SiC doped CFC at chemical sputtering temperatures of 200–700°C with machined and polished surfaces, where one dotted curve of 2D-CFC (CX-2002U) sputtered at the same conditions was cited as a reference from the previous paper [3]. From Fig. 2, the additions of B_4C to carbon fibers or SiC to CFC can be seen to have reduced their sputtering yields to 50–75% of those of 2D-CFC (CX-2002U) with the machined surface, where the B_4C–CF composite showed a temperature dependence similar to that of the SiC doped CFC. The sputtering yields of

Table 1
Materials for experiments

Materials	Density (relative value)	
38% B_4C-Carbon fiber composite	2.18 g/cm^3	(0.96)
10% SiC doped ID-CFC	1.74	(0.77)
CX-2002U (2D-CFC)	1.71	(0.77)

Table 2
Irradiation conditions

Ion source species	D_3^+
Acceleration voltage	200 V
Flux	$1\text{–}2 \times 10^{20}$ $D^+/m^2{\cdot}s$
Fluence	$1\text{–}2 \times 10^{20}$ D^+/m^2
Target temperature	473–973 K (200–700°C)
Incident angle	0° (normal), 30°, 45°, 60°

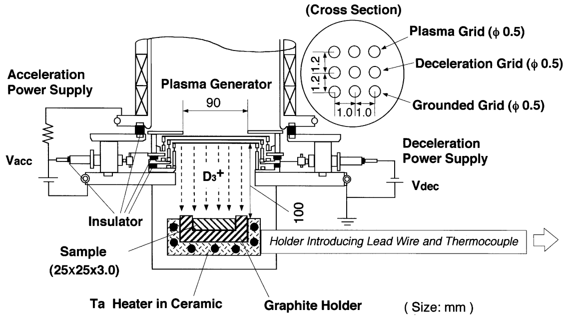

Fig. 1. Schematic of super low energy ion source (SLEIS) for high particle flux.

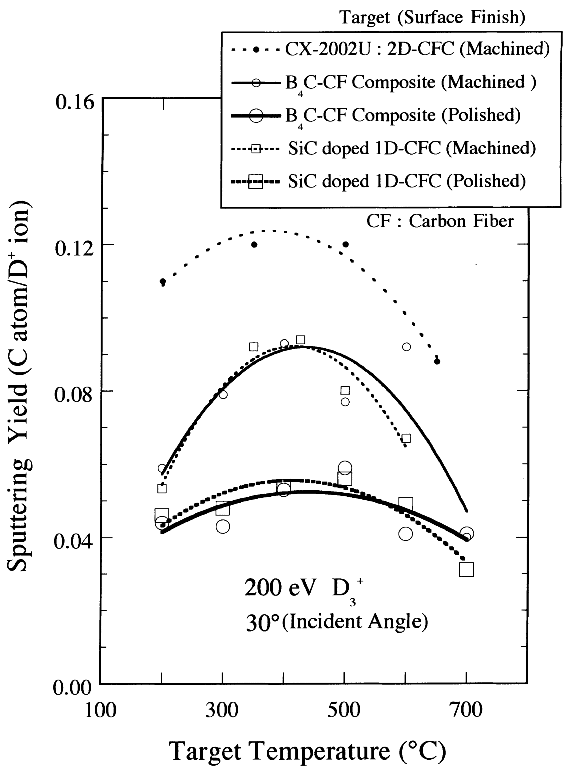

Fig. 2. Temperature dependence of sputtering yields for the target materials with different surface finishes by D_3^+ ions, where the graph for 2D-CFC was cited from a paper [3].

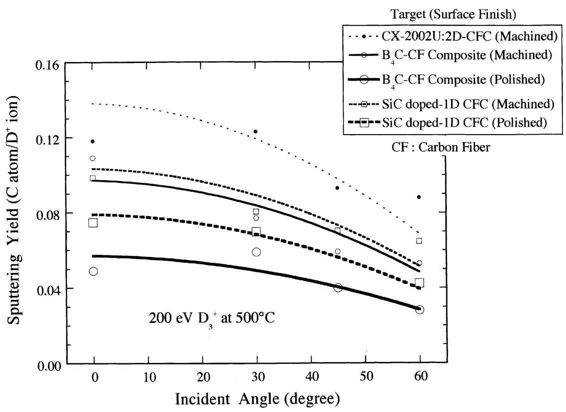

Fig. 3. Angular dependence of sputtering yields for the target materials with different surface finishes by D_3^+ ions at 500°C.

the B_4C–CF composite and the SiC doped CFC decreased furthermore by 20–50% with the polished smooth surface.

Fig. 3 shows the angular dependence of the sputtering yields at incident angles of 0–60° for three kinds of the above mentioned samples at 500°C. These sputtering yields decrease with the cosine value of the incident angle, where the sputtering yields of the B_4C–CF composite are lower than those of the SiC doped CFC and remarkably lower than those of 2D-CFC (CX-2002U).

Figs. 4 and 5 show the change in the surface structures of the polished surface of the B_4C–CF composite and the SiC doped CFC before and after sputtering at 500°C with an icident angle of 45°, where the surfaces of both samples are perpendicular to carbon fibers. From Fig. 4 and Fig. 5, it is clearly seen that the surfaces of the sputtered samples have greatly differed from the surfaces before sputtering. Magnified SEM images on the rights of Fig. 4(a) and Fig. 5(a) for sections of car-

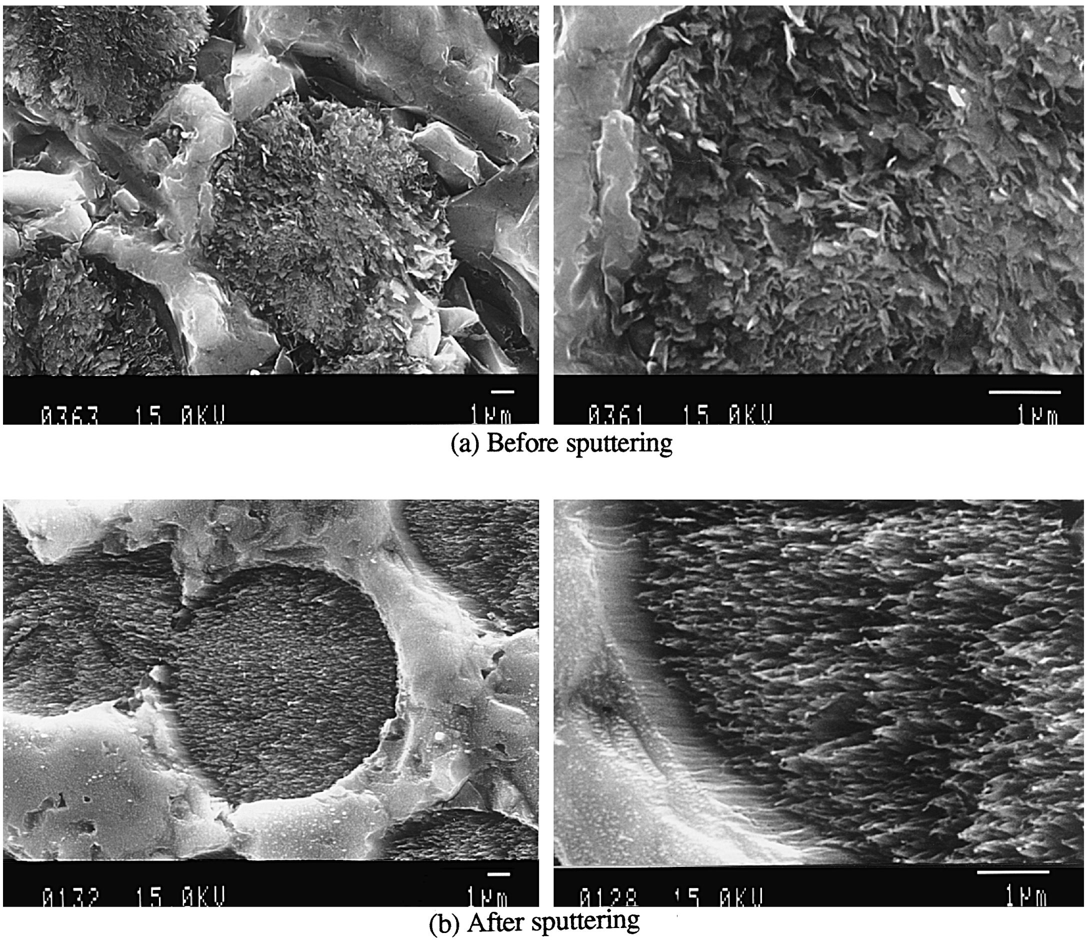

Fig. 4. Surface (polished) of 38% B_4C-carbon fiber composite before and after sputtering, photographed with SEM (sputtering: 1.2×10^{20} D^+/m^2 s for 4 h with incident angle of 45° at 500°C, SEM: 15 kV).

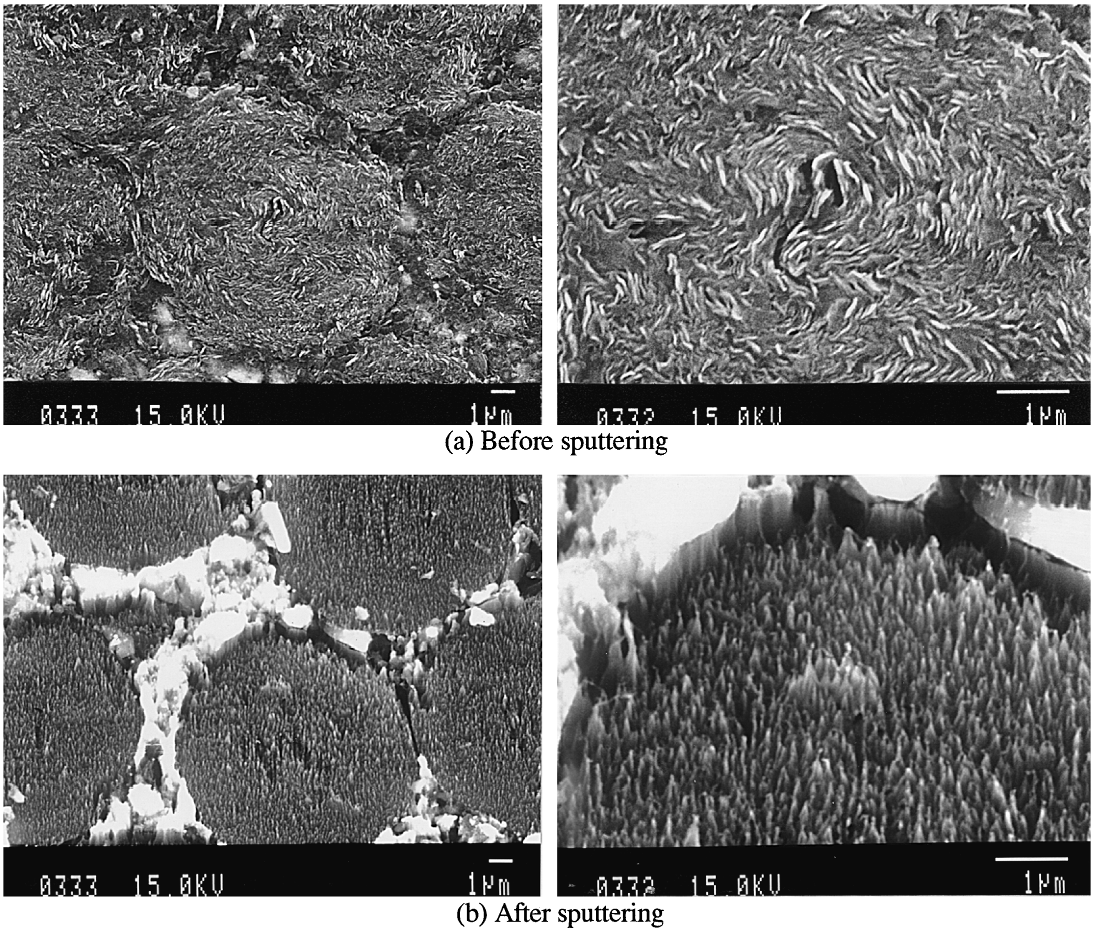

Fig. 5. Surface (polished) of 10% SiC doped 1D-CFC before and after sputtering, photographed with SEM (sputtering: 1.0×10^{20} D^+/m^2 s for 4 h with incident angle of 45° at 500°C, SEM: 15 kV).

bon fibers before sputtering seemed to be random structures like fallen leaves in the B_4C–CF composite and wavy paths in the SiC doped CFC. After sputtering, these structures changed to a similar pattern of needle-like leaves on the rights of Fig. 4(b) and Fig. 5(b) for both samples and showed the sharp difference in level, around 1 μm, between carbon fiber surface and B_4C or SiC matrix.

4. Discussions

The sputtering yields of 2D-CFC (CX-2002U) in Fig. 2, which were cited from a previous paper, can be representative of other CFCs because they were nearly the same as the values of other CFCs (1D: MFC-1, 3D: NIC01) at the chemical sputtering temperatures of 200–700°C [3]. The sputtering yields of the B_4C–CF composite and the SiC doped CFC decreased to 50–75% of those of 2D-CFC (CX-2002U) with the machined surface. This decrease would be owing to the additions of B_4C to carbon fibers or SiC to CFC. These sputtering yields decreased furthermore by 20–50% with the polished smooth surface than with the machined rough one, probably owing to the decrease of effective surface area.

The chemical sputtering yields of all samples with the incident angles of 0–60° at 500°C in Fig. 3 decrease with the cosine values of the incident angles. This might have resulted from the increase in reflectivity or the decrease in absorptivity of the incident deuterium ions in samples with increasing incident angles [5]. In Fig. 3, the sputtering yields of the B_4C–CF composite at 500°C were lower at the incident angles of 0–60° than those of the SiC doped CFC with machined and polished surfaces. This would have resulted from the higher relative density (smaller porosity) of the B_4C–CF composite, 0.96 than that of the SiC doped CFC, 0.77, as shown in Table 1, because the smaller effective surface area which results from the higher relative density might have realized the lower sputtering yields.

A similar pattern of needle-like leaves in the B_4C–CF composite and the SiC doped CFC after sputtering, as shown in Fig. 4(b) and Fig. 5(b), might have revealed an inherent structure of highly oriented graphite crystal in carbon fibers. On the other hand, the surface structures of carbon fibers before sputtering in Fig. 4(a) and Fig. 5(a) might have been affected by random stress along arbitrary direction in polishing, showing random structures.

The level difference around 1 μm between carbon fiber surface and B_4C or SiC matrix after sputtering in Fig. 4(b) and Fig. 5(b) shows the higher sputtering yield of the carbon fiber than B_4C or SiC. A roughly calculated fluence to make 1 μm erosion depth of graphite is 10^{24} D^+/m^2 with a sputtering yield of 0.1 at 500°C for 2D-CFC (CX-2002U) in Fig. 2 and graphite density of 2 g/cm^3, where the sputtering yields of B_4C and SiC were supposed to be zero. These real fluences were 1.4 and 1.7×10^{24} D^+/m^2 in these experiments that is well agreed with the calculated results. So, around 1 μm erosion depth of graphite or CFC was estimated to be formed with the fluence of 10^{24} D^+/m^2 and the sputtering yield of 0.1.

5. Conclusions

The chemical sputtering yields at temperatures from 200°C to 700°C of 38% B_4C–carbon fiber composite and 10% SiC doped 1D-CFC with an incident angle of 30° decreased to 50% to 75% of those of 2D-CFC (CX-2002U) with the machined surface, where 38% B_4C-carbon fiber composite showed a temperature dependence similar to that of 10% SiC doped 1D-CFC. These sputtering yields decreased furthermore by 20–50% with the polished smoother surface.

The angle dependence measurement showed that the chemical sputtering yields at 500°C of 38% B_4C-carbon fiber composite and 10% SiC doped 1D-CFC decreased with the cosine values of the incident angle, where the sputtering yields of 38% B_4C-carbon fiber composite were lower than those of 10% SiC doped 1D-CFC and 2D-CFC (CX-2002U).

The surface structures of 38% B_4C-carbon fiber composite and 10% SiC doped 1D-CFC examined with SEM showed a similar pattern of needle-like leaves.

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