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Journal of Nuclear Materials 258–263 (1998) 724–728

journal of nuclear materials

Temperature dependence of sputtering yield of carbon fiber-reinforced carbon composites with low energy and high flux deuterium ions

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Abstract

A super low energy ion source (SLEIS) was used to simulate the sputtering conditions at the divertor in next fusion experimental reactors such as ITER. One directional:1D-, two directional:2D-, three directional:3D-CFCs, isotropic graphite, and B_4C doped 1D-CFC were sputtered by SLEIS with high fluxes ($1–2 \times 10^{20}/m^2/s$) of 200 eV D_3^+ ions. Test samples were sputtered at temperatures of 200–800°C, where chemical sputtering dominates, with an incident angle of 30° and also at 500°C with incident angles of 0–60°. Sputtering yields were calculated from the weight loss of irradiated samples and the fluences. The sputtering yields of 1D-, 2D- and 3D-CFCs showed nearly the same temperature dependence as an isotropic graphite, though the yield of B_4C doped-1D CFCs was smaller than those of these CFCs. The angular dependence of the chemical sputtering yield is determined by the cosine value of the incident angle in each CFC, where the yield of B_4C doped-1D CFC is lower than those of MFC-1 (1D) and CX-2002U (2D).

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. The armor tiles are eroded by hydrogen isotopes from the plasma. It is important to evaluate the sputtering yield of CFCs at irradiation conditions similar to those in ITER for estimating the lifetime of the armor tiles. SLEIS has been constructed to simulate the sputtering conditions at the divertor in ITER (energy: 50–100 eV, flux: $10^{22}–10^{23}/m^2/s$) and has a wide irradiation area to cover the test sample (25×25 mm) with a uniform flux density [1].

In this paper, the difference in sputtering yields of isotropic graphite, 1D-, 2D- and 3D-CFCs, and B_4C doped 1D-CFC was investigated in the temperature range of chemical sputtering, including angular dependence of the sputtering yields.

2. Experimental

SLEIS has a wide irradiation area of 90×100 mm to cover the test samples ($25 \times 25 \times 3$ mm) with a uniform flux density less than 5% variation [1]. A schematic of SLEIS is shown in Fig. 1.

One directional:1D- (MFC-1), two directional:2D-(CX-2002U), three directional:3D-(NIC-01) CFCs, isotropic graphite (IG-430U) and 5% B_4C doped 1D-CFC shown in Table 1 were sputtered by SLEIS with high fluxes of $1–2 \times 10^{20}$ ions/m^2/s at 200 eV, as shown in Table 2. All samples, which were machined from raw materials, were washed two times in an ethyl alcohol bath with an ultrasonic vibrator for 10 min to remove completely 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

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0022-3115/98/$19.00
PII: S0022-3115(98)00252-9

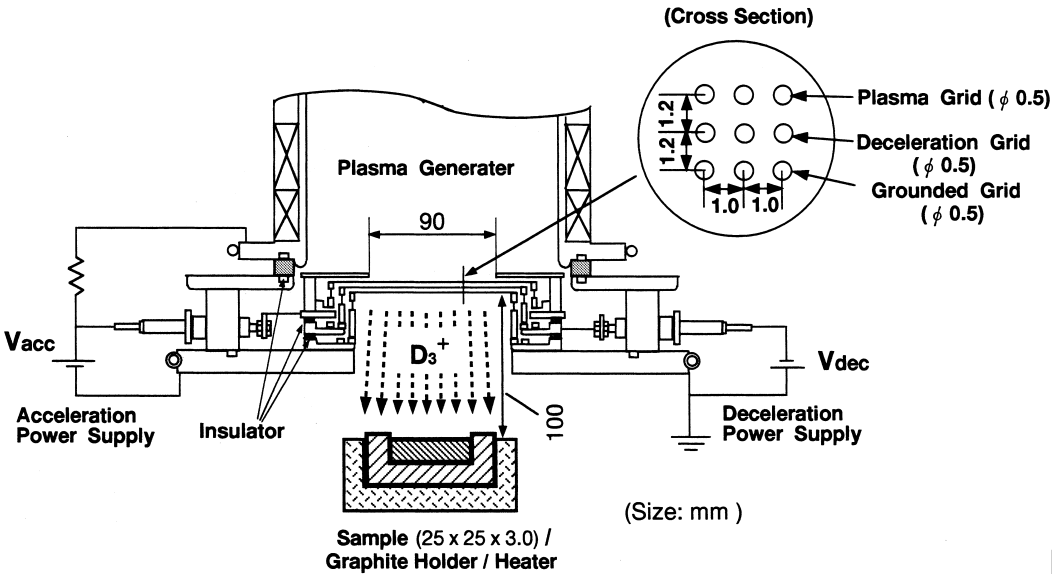

Fig. 1. Schematic of super low energy ion source (SLEIS) for high particle flux.

were placed about 10 cm downstream from the ion source and sputtered at temperatures ranging from 200°C to 800°C. The angular dependence of the sputtering yields was measured at 500°C with the incident angle of 0°, 30°, 45° and 60°.

Since more than 85% of the total charged particles were analyzed to be D_3^+ with the magnetic mass analyzer [1], 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.

Table 1
Materials for experiments

Materials	Density (g/cm^3)	Matrix Contents (wt%)
IG-430 (isotropic graphite)	1.80	–
MFC-1 (1D-CFC)	1.89	60
CX-2002U (2D-CFC)	1.71	10
NIC-01 (3D-CFC)	1.94	
5%B_4C doped 1D-CFC	1.85	50

Table 2
Irradiation conditions

Ion source species: D_3^+
Acceleration voltage: 200 V
Flux: $1–2 \times 10^{20}/m^2$ s
Fluence: $1–3 \times 10^{24}/m^2$
Target temperature: 473–1073 K (200–800°C)
Incident angle: 0° (normal), 30°, 45°, 60°

The surfaces of test samples before and after sputtering at different temperatures were examined with SEM to see the change in the surface structure by the sputtering.

3. Experimental results

Fig. 2 shows the angular dependence of the sputtering yields for two kinds of CFCs (MFC-1 and CX-2002U) and 5% B_4C doped 1D-CFC at 500°C. These sputtering yields decrease with the cosine value of the incident angle, where the sputtering yield of 5% B_4C doped 1D-CFC is lower than those of MFC-1 and CX-2002U.

Fig. 3 shows sputtering yields of three kinds CFCs (1D to 3D) to which are added one 1D-CFC doped with 5% B_4C, and one isotropic graphite in the temperatures from 200° to 800°C which correspond to the chemical sputtering region. In Fig. 3, two dotted curves of pyrolytic graphite irradiated at the normal incident angle with low (50 eV) and high (1 keV) energies of deuterium ions are shown [2]. The isotropic graphite of IG-430 and three CFCs show nearly the same temperature dependence which are higher than the sputtering yield of pyrolytic graphite irradiated with the energy of 50 eV. The sputtering yield of 5% B_4C doped 1D-CFC shows a lower value than those of the other CFCs and isotropic graphite. The peak temperatures of the sputtering yields for isotropic graphite and CFCs with energies of 50 and 67 eV in Fig. 3 are around 400–500°C which are 50–100°C lower than that of the pyrolytic graphite irradiated with the energy of 1 keV.

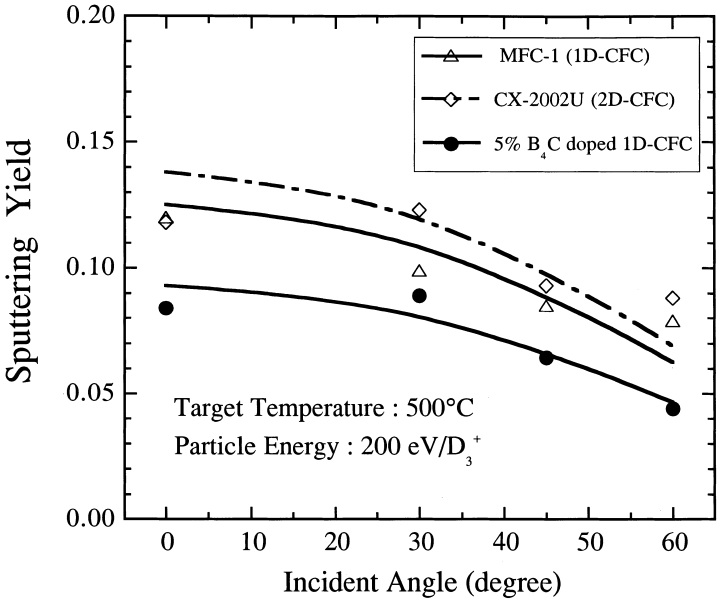

Fig. 2. Angular dependence of sputtering yields for 1D- and 2D-CFCs and B_4C doped 1D-CFC, simulated by the cosine function.

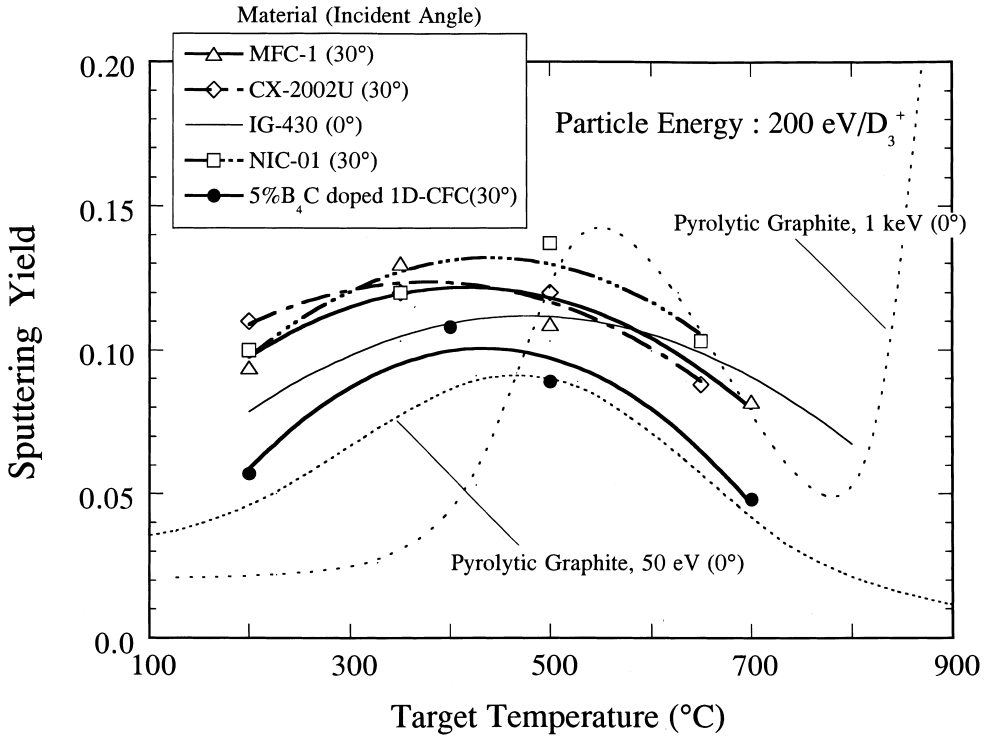

Fig. 3. Temperature dependence of sputtering yields for several CFCs in the chemical erosion temperature with D_3^+ ions of 200 eV, showing pyrolytic graphites sputtered at the normal incident angle with low (50 eV) and high (1 keV) energies of deuterium ions [2].

Fig. 4 shows changes in the surface structures of IG-430, NIC-01 (3D-CFC) and 5% B_4C doped 1D-CFC before and after sputtering tests at different temperatures, where the surface of NIC-01 (3D-CFC) is parallel to carbon fibers and the surface of 5% B_4C doped 1D-CFC is perpendicular to carbon fibers. From Fig. 4,

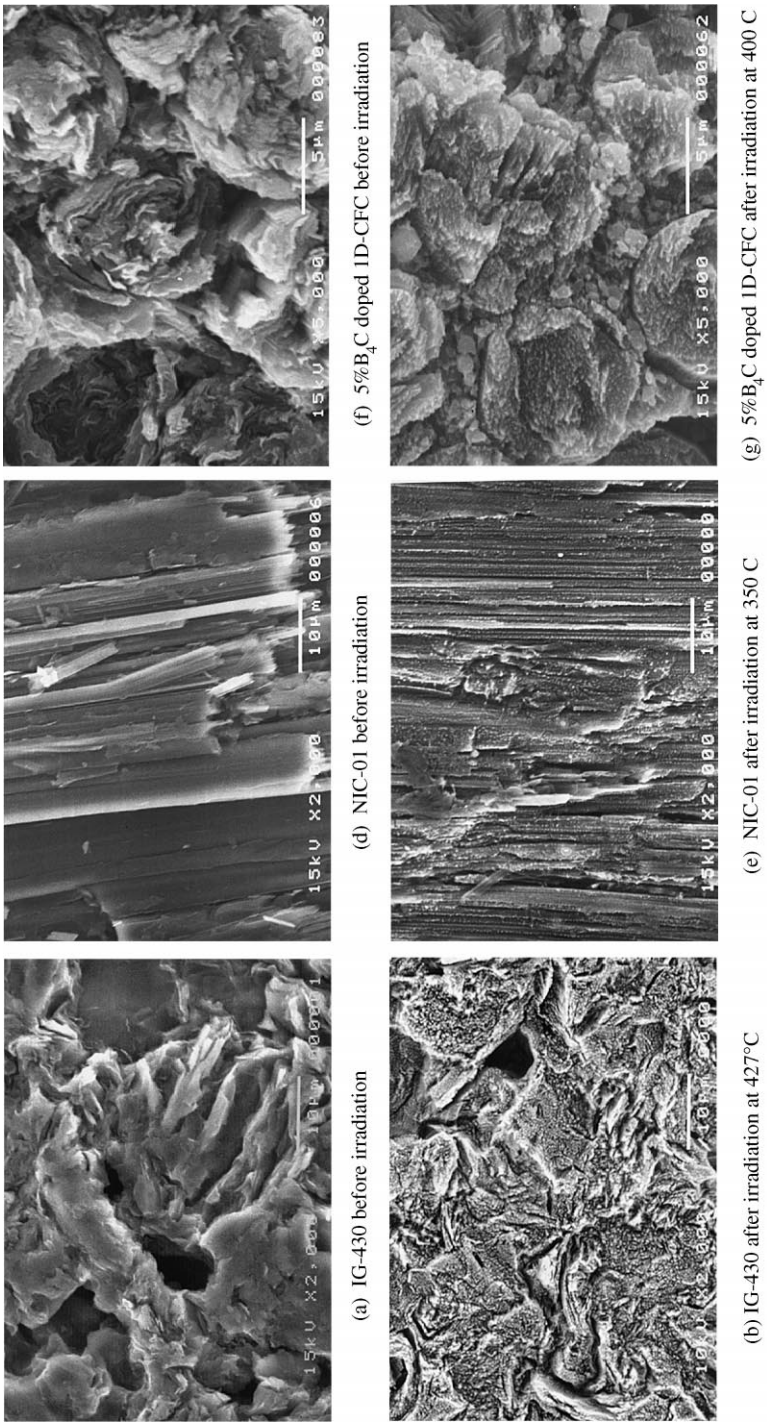

Fig. 4. Surface structures of several CFCs before and after irradiation with deuterium ions (D_3^+ of 200 eV) in SLEIS.

many small pits with dimensions below 1 μm cover most of the surfaces of samples (b, e and g) sputtered at the temperatures around 400°C which are in connection with the both structures of graphite matrices and carbon fibers, that is, both the isotropic graphite (IG-430) and CFCs have been sputtered similarly. Such kind of pitted area are observed to be smaller at a higher temperature of 800°C (c) where the value of the sputtering yield becomes lower.

4. Discussions

The sputtering yields of three kinds of CFCs with and without B_4C have shown an angular dependence as shown in Fig. 2. This angular dependence could be explained in the following way: since the sputtering by the chemical reaction is caused by the formation of hydrocarbons (C_xD_y) such as methane (CD_4) [3], the sputtering value in each sample will be controlled by the cosine value of the incident angle (cosine law) because the flux of deuterium ions in the constant area of target samples decrease with the cosine value of the incident angle. In the case of 5% B_4C doped 1D-CFC in Fig. 2, the angular dependence obeys the cosine law and is smaller than those of other two CFCs, because the existence of B_4C with a lower chemical sputtering yield results in a lower sputtering yield of this CFC in the chemical sputtering region [4–6].

The sputtering yields of the CFCs with a higher (MFC-1) and a lower matrix contents (CX-2002U:) in Table 1 show nearly the same characteristics in the temperatures of 200–700°C in Fig. 3. The reason for these can result from the phenomena that the chemical sputtering or the chemical reaction of deuterium with carbon atoms to form hydrocarbons controls this sputtering where the matrices (graphite grains) and the carbon fibers have been eroded or sputtered to form pits to nearly the same extent as seen in Fig. 4 (b and e).

The lower peak temperatures in the sputtering yields with a lower particle energy of 67 eV in Fig. 3, in comparison with a higher particle energy of 1 keV, as in the other research [6], could result from a shallower intrusion of the deuterium ions into the samples that causes a quick landing of hydrocarbons at lower temperatures, as well as the smaller and lower damage in samples caused by a lower flux or a lower energy of deuterium ions and recovered to make hydrocarbons at lower temperatures [6].

5. Conclusions

The sputtering yields of MFC-1 (1D), CX-2002U (2D), NIC-01 (3D) CFCs and IG-430 (isotropic graphite) by deuterium ions with a particle energy of 67 eV showed nearly the same temperature dependence in the chemical sputtering region of 200–800°C, while 5% B_4C doped 1D-CFC showed a lower sputtering yield than those of these materials. The sputtering yields of all samples with a lower particle energy of 67 eV had the lower peak temperatures by 50–100°C than that with a higher particle energy of 1 keV.

The angular dependence of the chemical sputtering yield in each CFC is determined by the cosine value of the incident angle, where the yield of B_4C doped-1D CFC is lower than those of MFC-1 (1D) and CX-2002U (2D).

Acknowledgements

The authors would like to thank Drs. M. Ohta and Y. Ohara in JAERI, and Y. Kozono and K. Takahashi in Hitachi Ltd for their continuous support.

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