



## Sputtering of carbon–tungsten mixed materials by low energy deuterium

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### Abstract

In the divertor of next step fusion experimental devices such as ITER, simultaneous presence of carbon and tungsten material will lead to their mixing during the redeposition process. In the present paper, sputtering experiments on C–W mixed material by low energy hydrogen beam had been performed. The C–W mixed material was simulated by the deposits during the D<sub>2</sub> arc discharge between carbon and tungsten electrode. Five types of C–W mixed materials having different W concentrations which ranged from 0 to 75 wt% were used as the specimens for the sputtering experiment. It was found that the sputtering yield strongly depends on the W concentration in the C–W mixed materials. From the results of electron probe micro-analysis, it was indicated that carbon material is preferentially sputtered by chemical sputtering. The formation of W–C bonding is considered to be the reason for the low sputtering yields of C–W mixed material layers.

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### 1. Introduction

In the divertor of next step fusion experimental reactors such as ITER, carbon composite material and tungsten will be used simultaneously. Carbon will be used on the highest heat load components in the divertor and tungsten will be used in other areas where the particle load is expected to be high. As the eroded materials due to high heat load and/or sputtering will be redeposited on the surface of the armor, carbon and tungsten will be mixed to form carbon–tungsten (C–W) mixed materials during the redeposition process. In fact, C–W mixed materials are observed in many tokamak devices such as TEXTOR [1] and ASDEX-U [2,3], where part of the plasma facing surface was covered with a tungsten

layer. Due to the material mixing, the surface property of the armor material could be changed compared with the original material. Especially, sputtering properties of the carbon material will be drastically changed by the presence of a mixed material layer because the sputtering erosion of carbon is dominated by the chemical sputtering in the criteria of the ITER divertor. The formation of C–W mixed materials and their erosion by low energy hydrogen were investigated by many authors [4–6]. In these works, the fundamental data for the sputtering of C–W mixed materials such as the energy dependence of the sputtering yield and the sputtering threshold energies were obtained experimentally. However, the effect of the W addition on sputtering characteristics of carbon film is not fully understood. Therefore, in the present work, we have prepared the C–W mixed materials with various W concentrations. The sputtering experiments on C–W mixed materials by low energy deuterium beam had been performed. The C–W mixed materials were prepared by a D<sub>2</sub> arc discharge

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between carbon and tungsten electrodes. Carbon and tungsten evaporated from the electrodes were deposited on a molybdenum substrate. The properties of the deposited C–W mixed materials were examined by electron probe microanalysis. Five types of C–W mixed materials having different W concentrations which ranged from 0 to 75 wt% were used as the specimen for the sputtering experiment. The effect of W on the sputtering of carbon material was examined.

## 2. Experimental

### 2.1. Preparation of the C–W mixed materials

The C–W mixed materials were prepared by a  $D_2$  arc discharge between carbon and tungsten electrodes. Fig. 1 shows the schematic of the arc discharge apparatus used in this work. After  $D_2$  gas was introduced into the chamber, arc discharge was turned on between the electrodes. Carbon (C) and tungsten (W) evaporated from the electrodes were deposited on a molybdenum substrate, which was placed in the discharge chamber. During the discharge,  $D_2$  pressure in the chamber was kept at around 1.0 Pa. The size of the Mo substrate was  $25 \times 25$  mm in width and 0.3 mm in thickness. The vertical distance between the electrodes and the substrate was 60 mm. During an arc discharge, the Mo substrate is heated by radiation and the temperature of the substrate increased to be a constant value of about 400 K. After the discharge, the W electrode was found to be melted as well as the carbon electrode. Therefore, the

deposited material in the chamber is considered to contain W and C deposits. The W concentration of the C–W mixed materials was expected to be controlled by changing the condition of the arc discharge. For this purpose, the arc current was controlled from 20 to 70 A, and the arc power was changed from 360 to 1260 W. The W concentration in the obtained C–W mixed material was investigated by the electron probe micro-analyzer.

### 2.2. Sputtering experiment by super low energy ion source

Sputtering experiments on these test specimens were performed using the super low energy ion source (SLEIS) facility in JAERI [7,8]. The SLEIS facility consists of an ion source and a sample holder, which locates 10 cm downstream from the ion source. The ion source produces mainly low energy  $D_3^+$  beam (200 eV) with a large irradiation area of  $90 \times 100$  mm which covers the whole surface of the test specimens ( $25 \times 25$  mm). The maximum deuterium ion beam flux is  $1 \times 10^{20}$  ion/m<sup>2</sup>/s with an almost uniform flux density. The variation of the flux density over the irradiation area is less than 5%. The composition of the beam was measured by the magnetic mass analyzer. More than 95% of the total charged particles were found to be  $D_3^+$ , and the rest of the beam was  $D^+$ . Impurity ions such as oxygen were less than 0.3%. The total fluence of the deuterium beam incident on the sample was estimated from the beam current. The temperature of the sample during the irradiation can be controlled by a heater equipped just below the sample holder. In the present work, the irradiation temperature was kept at 673 K because the

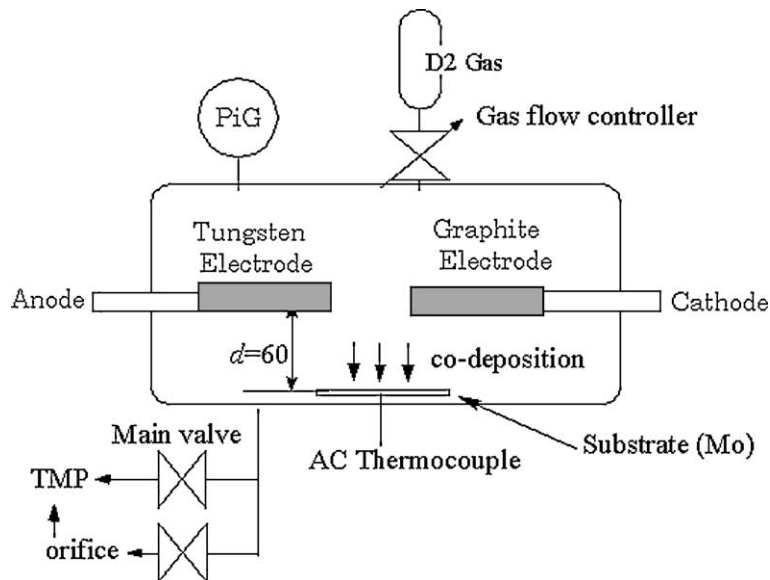


Fig. 1. Schematic of the discharge chamber.

Table 1  
Irradiation conditions

|                             |  |
|-----------------------------|--|
| Ion species                 | D <sub>3</sub> <sup>+</sup> 95%, D <sup>+</sup> < 5% |
| Acceleration energy (eV)    | 200  |
| Temperature (K)             | 673  |
| Flux (n/m <sup>2</sup> /s)  | 5.0 × 10 <sup>19</sup>                               |
| Fluence (n/m <sup>2</sup> ) | 1.8 × 10 <sup>24</sup>                               |

chemical sputtering of carbon shows the maximum at this temperature [9]. Table 1 summarizes the conditions of the sputtering experiments performed in the present study.

### 3. Results and discussions

#### 3.1. Properties of simulated C–W mixed materials

Fig. 2 shows the typical surface morphology of the C–W mixed materials deposited on a Mo substrate observed by scanning electron microscope (SEM). The sample has a smooth surface but a lot of small particles (particle size  $\ll 1 \mu\text{m}$ ) can be observed. The number of these small particles tends to increase with increasing arc power. This indicates that the particle emission from the electrode occurs as well as the evaporation under the conditions of high arc power. These are considered to be the carbon particles, which were cracked from the carbon electrode during the formation of the C–W mix. The properties of the C–W mixed materials were investigated by electron probe microanalysis (EPMA). EPMA was performed with an acceleration electron energy of 15 keV. The diameter of the probe electron beam was 50  $\mu\text{m}$ . Fig. 3 shows the W concentration (wt%) at several points on the surface of the C–W mixed materials produced in various arc conditions. The C–W mixed ma-

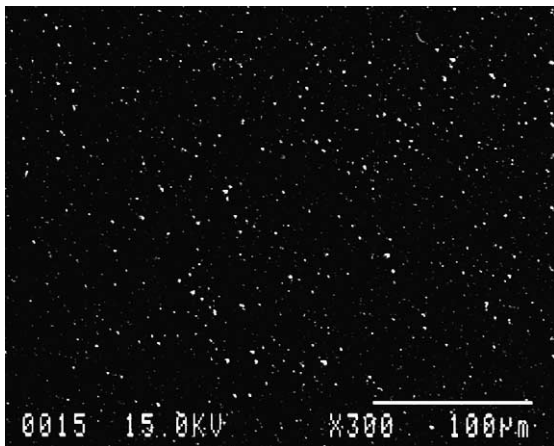


Fig. 2. SEM image of the C–W mixed material.

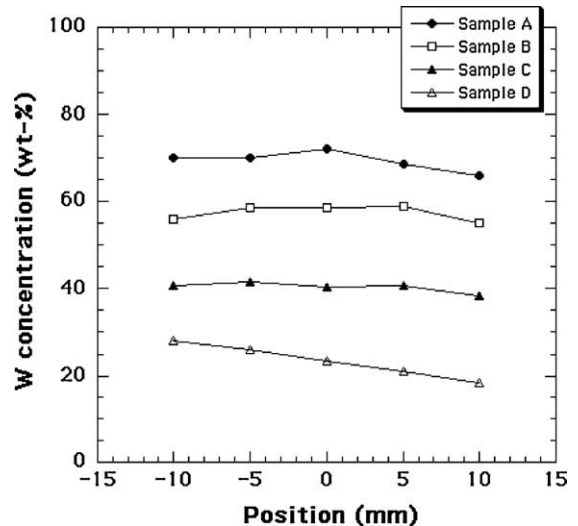


Fig. 3. Tungsten concentration of C–W mixed materials.

terials with various W concentrations were obtained and the W concentrations were almost uniform in each sample. As for the arc power dependence of the W concentration, it was found that the W concentration increases with increasing arc power. This is considered to be due to the high melting temperature of the tungsten. The thickness of the C–W mixed materials deposited on Mo was evaluated by a laser profile meter. The thickness of the C–W layer was estimated to be 15–20  $\mu\text{m}$ .

#### 3.2. Sputtering experiments on C–W mixed materials

The sputtering experiments were performed in the SLEIS facility. The sample of C–W mixed materials having five different W concentrations which ranged from 0 to 75 wt% were used as the specimen for the sputtering experiment. Fig. 4 summarizes the results of the sputtering experiments on C–W mixed materials with various W concentrations. The amount of sputtering was evaluated by the weight loss per unit irradiation time measured by the microbalance. The data for the 100% carbon film prepared by the same method using the carbon–carbon electrode is also plotted. The period of the irradiation was 10 h so that the thickness of the C–W layer (15–20  $\mu\text{m}$ ) is considered to be sufficient throughout the experiment. As a result, it was found that the sputtering yield decreases with increasing W concentration in the C–W mixed materials. In Fig. 4, results of other authors for pure C [6] and C–W layer [4] are also plotted. The data obtained in this work seems to coincide with these data.

In the case of 25 wt% W contained sample, the weight loss was about 1/3 of the 100% carbon film. Sputtering of the carbon material by low energy hydrogen is

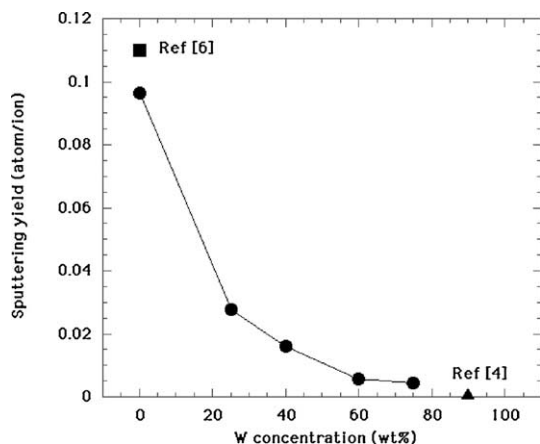


Fig. 4. Dependence of sputtering erosion on W concentration. The data by other authors for pure carbon [6] and W–C layer [4] are also plotted for comparison.

Table 2  
Change in W concentrations before and after the irradiation

| Sample no. | Before (%) | After (%) |
|------------|------------|-----------|
| A          | 70         | 77        |
| B          | 60         | 74        |
| C          | 40         | 62        |
| D          | 25         | 55        |

dominated by chemical sputtering. In the C–W mixed material, tungsten carbide is considered to be formed. The formation of C–W bonding is considered to suppress the chemical sputtering of the C–W mixed materials.

After being taken from the sputtering apparatus, the irradiated area of the test specimens were again investigated by EPMA. Table 2 summarizes the results of EPMA after the deuterium beam irradiations. It was observed that the W concentration of the surface increases after the deuterium beam irradiation. This fact

indicates that the preferential sputtering of carbon occurs in C–W mixed materials.

#### 4. Conclusion

Sputtering experiments on C–W mixed materials were performed in the SLEIS facility at JAERI. The C–W mixed materials were prepared by using the arc discharge between carbon and tungsten electrodes. As a result, it was found that the sputtering yield decreases with increasing W concentration in the C–W mixed materials. From the results of the EPMA study, it was indicated that the carbon is preferentially sputtered by chemical sputtering. The formation of W–C bonding is considered to be the reason for the low sputtering yields of the C–W mixed material layer. These results indicate that the redeposition of W on carbon based armor material might reduce the sputtering erosion.

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#### References

- [1] D. Hildebrandt et al., *J. Nucl. Mater.* 290–293 (2001) 89.
- [2] H. Maier et al., *J. Nucl. Mater.* 266–269 (1999) 1008.
- [3] D. Hildebrandt et al., *J. Nucl. Mater.* 266–269 (1999) 533.
- [4] Ch. Linsmeier et al., *J. Nucl. Mater.* 290–293 (2001) 25.
- [5] M.I. Guseva et al., *J. Nucl. Mater.* 266–269 (1999) 222.
- [6] H. Plank et al., *Nucl. Instrum. and Meth. B* 124 (1997) 23.
- [7] K. Nakamura et al., *J. Nucl. Mater.* 220–222 (1995) 890.
- [8] R. Jimbou et al., *J. Nucl. Mater.* 258–263 (1998) 724.
- [9] J. Roth, *J. Nucl. Mater.* 266–269 (1999) 51.