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Journal of Nuclear Materials 258–263 (1998) 735–739

journal of nuclear materials

Boron ion particles sputtered from boron films deposited on graphites

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Abstract

Boron films were deposited on pyrolytic and isotropic graphites by vapor deposition at different deposition rates. The particles sputtered from these films were measured directly by the QMS during 5 keV D_3 beam irradiation with a flux of 2×10^{21} D/m^2 s. It was found that the sputtered particles were in the form of boron ion and neutral. The boron ion showed a different behavior from the neutral. The ion signal had a peak value at the beginning of each shot. It also depended on the conditions of the substrates and the deposition rate. These dependencies might be associated with the existence of oxygen on the surface.

1. Introduction

The wall conditioning in fusion devices plays an important role to realize a stable and high purity plasma. The boronization of the inner wall in the vessel has been successfully applied to the current fusion devices [1–4], since the high gettering effect of boron is efficient in reducing the plasma contamination such as oxygen impurity.

The boronization usually has been prepared by a chemical vapor deposition using DC glow discharge in hydrogenated boron compounds such as decaborane ($B_{10}H_{14}$) or diborane (B_2H_6). This boron film on the inner wall is always eroded by ions escaped from the core plasma, and neutrals with very high flux more than 10^{23}/m^2 s. In this way, the understanding of the erosion behavior of the boron film is important for the core plasma behavior. Few data for the properties of the pure boron film, however, has been presented [5]. Therefore it seems to be necessary to investigate the ion irradiation effect on boron film under high flux density where the film suffers from the particle loads of fusion plasma.

The properties of boron films depend on the conditions of the substrate and the deposition. Recently, it was found that the internal stress in the boron films on Mo substrate changed from compressive stress to large tensile stress with increasing deposition rate [6]. This would indicate that the deposition rate is one of the significant parameters to determine the properties of the boron film. In this study, the boron films were produced on graphites by vapor deposition with different deposition rates. The irradiation effects were directly estimated from the particles sputtered by the high flux beam.

2. Experimental

We used a high flux beam apparatus with a direct measurement system of sputtered particles as shown in Fig. 1. The apparatus consisted of a bucket type ion source, a target chamber, a measurement chamber, and a load-lock chamber. In the ion source, a plasma was produced by arc discharge using tungsten hot cathodes. The ions were extracted from the ion source and the most of them were neutralized by the charge exchange processes with neutral gas flowing out from the ion source. Since the ion source was equipped with spherical electrodes, this beam was focused geometrically near the center of spherical curvature. At the focal point, the maximum flux was with 1×10^{22}/m^2 s for a deuterium beam.

The beam was limited to 8 mm diameter by an orifice, which was 30 mm apart from a sample in the

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0022-3115/98/$19.00
PII: S0022-3115(98)00253-0

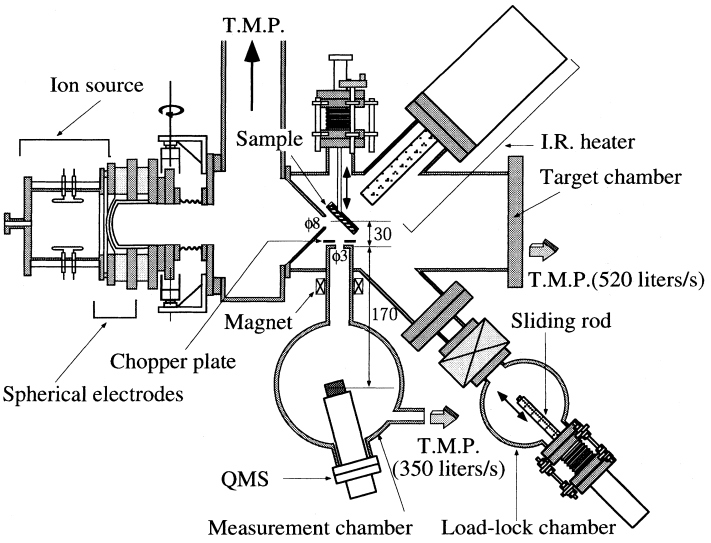

Fig. 1. Schematic of the high flux beam apparatus with a direct measurement system of sputtered particles.

target chamber. The sample was irradiated at an angle of 45° with respect to the sample normal. During the beam irradiation, the particles sputtered from the sample were directly detected by quadrupole mass spectrometer (QMS) where the ionization zone was positioned at 200 mm from the sample and at an angle of 45° with respect to the sample normal. In order to prevent the energetic particles reflected on the sample from producing false signals during the beam irradiation, the QMS was set at 15° with respect to the flight direction of the particles coming into the measurement chamber. In this way, the contribution of the energetic particles was almost removed from the QMS signals. There were a chopper plate and a removable magnet between the sample and the QMS. The sputtered particles were chopped by this plate in front of an orifice at a distance of 30 mm from the sample. Therefore the signals of the sputtered particles and the residual gas can be separated. In order to avoid collisions between the sputtered particles and the residual gas, the target chamber and the measurement chamber were evacuated with turbo molecular pumps with a pumping speed of 520 l/s and 350 l/s, respectively. During the irradiation, the pressure of the target chamber was increased up to 2×10^{-4} torr. It is expected that the most of the boron particles would enter the measurement chamber without any collisions.

The sample holder, which can be installed at the end of a sliding rod, moves between the irradiation chamber and the load-lock chamber. A calorimeter consisting of a $10 \times 10 \times 5$ mm^3 copper block was used for the estimation of the beam flux. The temperature rise of the calorimeter was monitored by a thermocouple set in the block. In this work, a D_3 beam and an Ar beam at the energy of 5 keV irradiated the samples with a flux of 2×10^{21} D/m^2 s and 8×10^{20} Ar/m^2 s, respectively. The pulse duration of the beam was 2 s per shot and the irradiation interval was 90 s. The beam contained a small amount of impurities such as oxygen. In the case of the Ar beam, the total concentration of impurities was less than 6%. For the D_3 beam, it was less than 2% after He discharge cleaning in the ion source. The samples were heated by the infrared heating lamp. The surface temperature was monitored by the pyrometer. The total erosion yield was estimated from the weight loss measured in the atmosphere by a microbalance with weight resolution of 10 μg for the Ar beam irradiation.

Isotropic graphite ISO-630 and IG-430 (Toyo Tanso Co.) and pyrolytic graphite (Union Carbide) were used as the substrate for the deposition of boron. The size of the substrates was $20 \times 10 \times 0.2 \sim 0.3$ mm^3. Boron films were prepared on these substrates by the vacuum deposition. The substrates were mounted at 14 cm apart from boron powder with a purity of 99%. The two values of the deposition rate were determined by the power of EB gun (4 keV, 500 mA) used for the boron evaporation. The fast deposition rate was over 0.5 nm/s, while the slow case was below 0.1 nm/s. Before the deposition, the substrates were washed with distilled water in an ultrasonic washing machine to remove the dust on the surface, and then were heated at 570 K to release the absorbed gas. During the deposition, the substrate temperature, which was measured with a thermocouple, was kept at 570 K and the film thickness was monitored by a quartz-crystal oscillator. The deposition and irradiation conditions were summarized in Table 1.

Table 1
The deposition and irradiation conditions of the boron films

Substrate	Deposition rate (nm/s)	Thickness (μm)	Beam particles	Method
Pyrolytic graphite	0.48 0.06	0.4	D_3	QMS
	0.66 0.09	~1.5	Ar	Weight loss
IG-430U	0.48 0.06	0.4	D_3	QMS
ISO-630	0.46 0.08	~1.5	Ar	Weight loss

3. Results and discussion

Fig. 2 shows the signals of the sputtered particles measured by the QMS when the boron film at the temperature of 730 K was irradiated by 5 keV D_3 beam. The surface temperature was increased up to about 900 K during the beam irradiation. In the case of no magnet along the flight path of the sputtered particles, the QMS signal ($M/e = 11$) showed a peak value at the beginning of the shot and thereafter decreased exponentially. The signal was almost the same regardless of the emission current of the QMS. However, it could not be detected at the mass selection such as $M/e = 12$. For the reflected particles, the QMS signals would not be influenced by the mass selection as well as the emission current. Therefore the QMS signal ($M/e = 11$) without the magnet along the flight path mainly corresponded to that of the sputtered boron ion. On the other hand, with the magnet, the QMS signal decreased to an order less than that of boron ion. This signal was independent of the temperature during the irradiation. The small similar profile due to the reflected particles was measured when the emission current was cut off. Therefore the signal of the sputtered boron neutral was obtained by the substraction of the reflected particles from the QMS signal where the magnet was used. As described above, it indicated that the boron films irradiated by D_3 beam were sputtered in the form of boron ion and neutral. However, the quantitative ion flux and neutral flux sputtered from the boron films cannot be estimated because of the differences in the detecting process. In order to detect the signal, the boron neutrals must be ionized at the ion source in the QMS, while the ions enter directly into the area of quadrupole. Therefore we limit the discussion to the qualitative boron ion and neutral, but recognize that more detailed studies should be performed.

Fig. 3 shows typical signals of boron ions sputtered from boron film deposited on the isotropic graphite IG-430U with the deposition rate of 0.5 nm/s. The signals were gradually decreased with increasing the number of

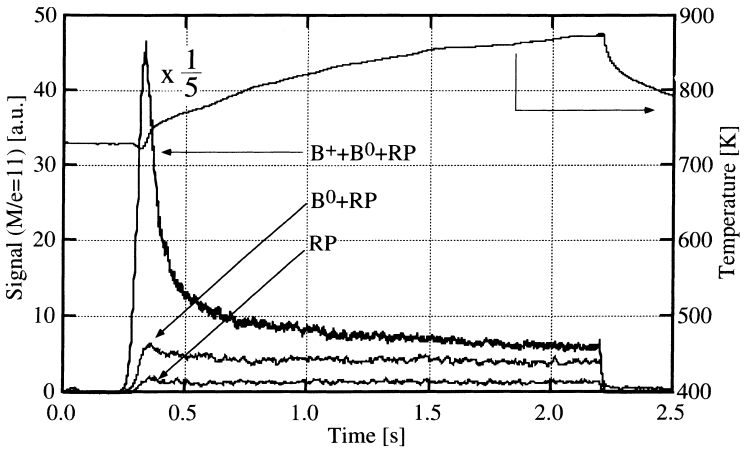

Fig. 2. The typical QMS signal of $M/e = 11$ while the 5 keV D_3 beam irradiated the boron film at 700 K. In the case of no magnet along the flight path, boron ion (B^+), neutral (B^0) and the reflected particles (RP) were measured. With the magnet, the reflected particles were detected when the emission current was cut off. In addition, the neutral was also detected when the current was turned on.

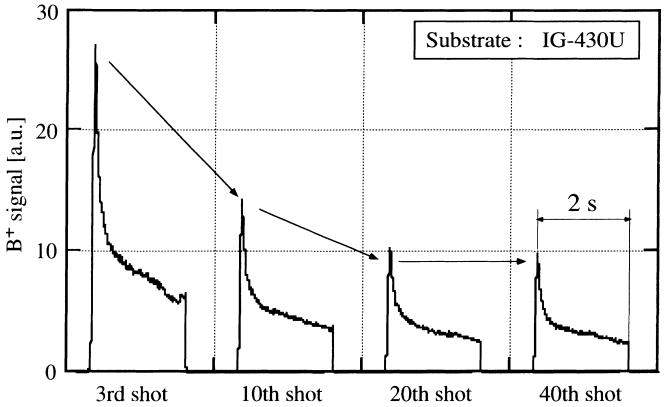

Fig. 3. The typical boron signal sputtered from boron films deposited on IG-430U with a deposition rate of 0.5 nm/s.

shots, and eventually showed the same time evolution. In any shot, boron ion signals showed very sharp peaks at the beginning. It was reported that the production rate of secondary ions was enhanced by using oxygen in SIMS [7]. From this result, it is possible that the presence of oxygen on the surface of born film would increase ion ratio of sputtered boron. Between each successive irradiation pulse, the sample was not irradiated and it could adsorb oxygen existed in the residual gas because of high oxygen gettering effect of boron. Therefore the first sharp peaks of ion signals might be associated with the existence of oxygen on the surface.

The results for the boron films deposited on the different substrates with the different deposition rates were shown in Fig. 4. These films were irradiated up to a fluence of 8×10^{22} D/m^2. In the case of boron ion, the signal intensity just before turning off the beam was plotted. The signal from the film on the isotropic graphite was about 1.5 times larger than that on the pyrolytic graphite. For the same substrate, the signal with the low deposition rate was larger than the high case. Fig. 5 shows the SEM photographs of boron films taken before and after D_3 beam irradiation. The unirradiated surface seemed to be flat for the film on pyrolytic graphite, while it was uneven for that on isotropic graphite. The surface modification could not be seen even after the high fluence irradiation. The ion signals sputtered from boron films on different substrates show some relationship between the surface configuration and the production rate of boron ion. The signal also depends on the deposition rate, with which the amount of the contamination in the films could change. Although the ion production might depend on the condition such as the surface morphology and the existence of oxygen on the surface, a more detailed study would be required to confirm this. On the other hand, for the neutral signal as shown in Fig. 4, there were only slight variations with substrate conditions and deposi-

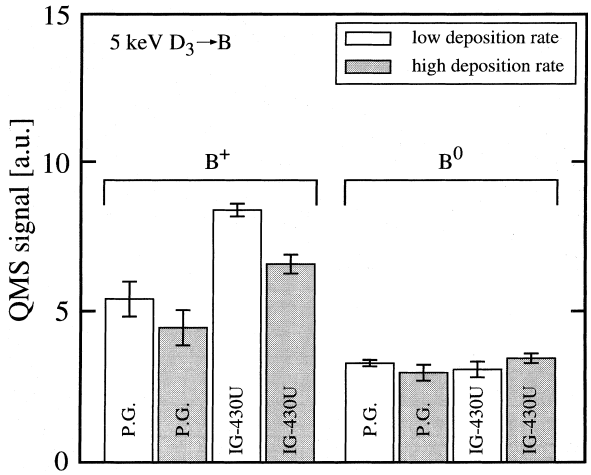

Fig. 4. The signal of the boron ion and neutral at the steady state.

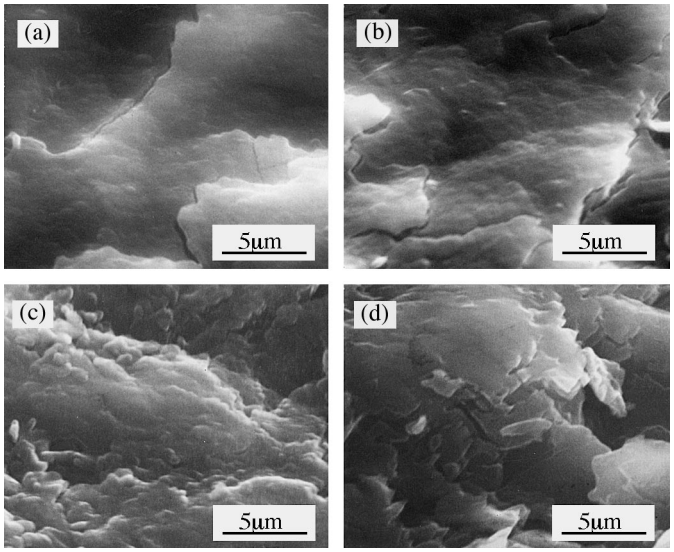

Fig. 5. The surface morphology of the boron films before and after D_3 irradiation, (a) unirradiated film on pyrolytic graphite, (b) irradiated film on pyrolytic graphite, (c) unirradiated film on IG-430 and (d) irradiated film on IG-430.

Table 2
The physical sputtering yield of boron films on the graphites irradiated by 5 keV Ar beam

Deposition rate (nm/s)	Yield	
	P.G.	ISO-630
0.08–0.09	1.0 ± 0.3	1.0 ± 0.2
0.46–0.66	0.9 ± 0.3	1.1 ± 0.5

tion rates. The similar results were obtained in the case of 5 keV Ar beam irradiation to the boron films. Table 2 shows the physical sputtering yield of the boron films estimated by the weight loss method. The yield was found to be independent of the substrate and the deposition rate. Therefore the sputtered boron neutrals would indicate the behavior of physical sputtering process for boron films.

It will be severe problem that the erosion would cause the depletion of the boron atoms deposited on the plasma facing components. The sputtered boron ion, however, could not enter into the core plasma due to the magnetic field in the scrape off layer and could be redeposited on the first wall. In order to evaluate the boron film performance, it will be important to investigate quantitatively the flux of ion or neutral sputtered from the boron films.

4. Summary

The boron films deposited on pyrolytic and isotropic graphites were irradiated by a D_3 beam with a flux of 2×10^{21} D/m^2 s and an Ar beam with a flux of 8×10^{20} Ar/m^2 s at an energy of 5 keV. The particles sputtered from the boron films were directly detected by the QMS during the D_3 beam irradiation. These particles were in the form of boron ion and neutral. The sputtered boron neutral indicated the behavior of physical sputtering process for boron films. On the other hand, the ion signal had its peak value at the beginning of each shot. It also depended on the substrate conditions and the deposition rate. These dependencies might be associated with the existence of oxygen on the surface.

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