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Surface modification of thick SiC gradient coatings on doped graphite under long pulse plasma irradiation

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Abstract

Selection of candidate plasma facing materials (PFM) in experimental fusion device is very important due to its direct influence on the plasma performance. To meet the requirements of high power and steady state operation, one kind of multi-element doped graphite GBST1308 (1%B_4C, 2.5%Si, 7.5%Ti) with thick SiC gradient coatings was chosen as the main limiter materials for the HT-7 device. Surface modification of the SiC coatings on doped graphite under long pulse plasma irradiation was carefully investigated by examining the surface morphology, structure, and atomic composition. The obtained results showed the crystal structure of SiC was replaced by lamellar matter or film with plentiful oxygen and some other elements existed in the surface of graphite tiles after the plasma irradiation.

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Keywords: SiC; Long pulse; Plasma facing material

1. Introduction

Carbon-based materials have long been recognized as possessing many properties such as low atomic number (Z), high operating temperatures, superior thermal shock resistance and potentially high thermal conductivities that make them attractive candidates for applications in experimental fusion devices. However, pure carbon materials shows enhanced erosion duo to chemical interaction with hydrogen and oxygen resulting in a relatively high oxygen impurity concentration in the plasma [1]. The chemical erosion rate could be reduced by some dopants, e.g. boron carbide, silicon or titanium [2–4]. One kind of multi-element doped graphite GBST1308 (1%B_4C, 2.5%Si, 7.5%Ti) with ~100 μm thick SiC gradient coatings was chosen as the main limiter materials for the HT-7 device. The belt limiters, two poloidal limiters, up and down toroidal limiters in HT-7 were covered with this material. The change to the new carbon limiter materials turned out to be very successful [5,6], the hard X-ray and high-Z impurities central radiation problem have been alleviated.

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As the result of high thermal loads and a large flux of particles bombardment, the surface morphology, structure and atomic composition of PFM will be changed, the erosion and deposition can be clearly found on the surface of PFM frequently. In the last experimental campaign of 2005, the longest discharge duration of HT-7 device has been more than 5 min. After this campaign, some graphite limiter tiles were found differently damaged. Erosion and deposition were clearly found on the surfaces of several graphite tiles. To investigate the surface modification of SiC coatings on doped graphite under long plasma irradiation, two typically different changed tiles and a new one as the comparable sample were chosen to examine the surfaces by means of SEM, EDS and XPS, and to contrast the results together.

2. Experimental set-up

The three specimens were respectively got from a new carbon tile, a surface eroded tile from toroidal limiter and an obviously deposited tile from poloidal limiter. A new carbon tile with SiC coatings was prepared by a special siliconization technique, a course of chemical vapor reaction (CVR) combined with chemical vapor infiltration (CVI) in the temperature range of 1600–1800 °C for 3 h [7], the thickness of SiC coatings was about 100 μm. The eroded tiles mainly came from the end of heat sink of toroidal limiters, where the thermal conduction was not so good as other places, and thus the temperature was also higher than other tiles. The first specimen is from a new carbon tile with SiC coatings, the second one was cut from the eroded carbon tiles, and the third specimen got from the deposition dominated area where looks like metal shine.

Surface morphologies and structures of the specimens were examined by scanning electron microscope (SEM). Surface atomic compositions were characterized by means of energy dispersive spectrometer (EDS). And comparatively precise composition and the chemical valence of the elements were measured by the means of X-ray photoelectron spectroscopy (XPS). Further more, the distribution in the certain deepness from surface was measured by XPS with argon ion (Ar^+) bombardment. In this article, the parameter of Ar^+ bombardment was 3 kV of accelerative voltage, 1.4 μA of target electric current, 200 s of duration, the sputtering deepness is about 12 nm.

3. Results and discussion

3.1. Surface morphology and structure

The SEM results as Fig. 1 clearly showed that the surface morphologies and structure of the eroded tile and deposited tile were quite distinct from the new one. Surface morphology of the new tile is a regular SiC crystal as Fig. 1(a), the lamellar struc-

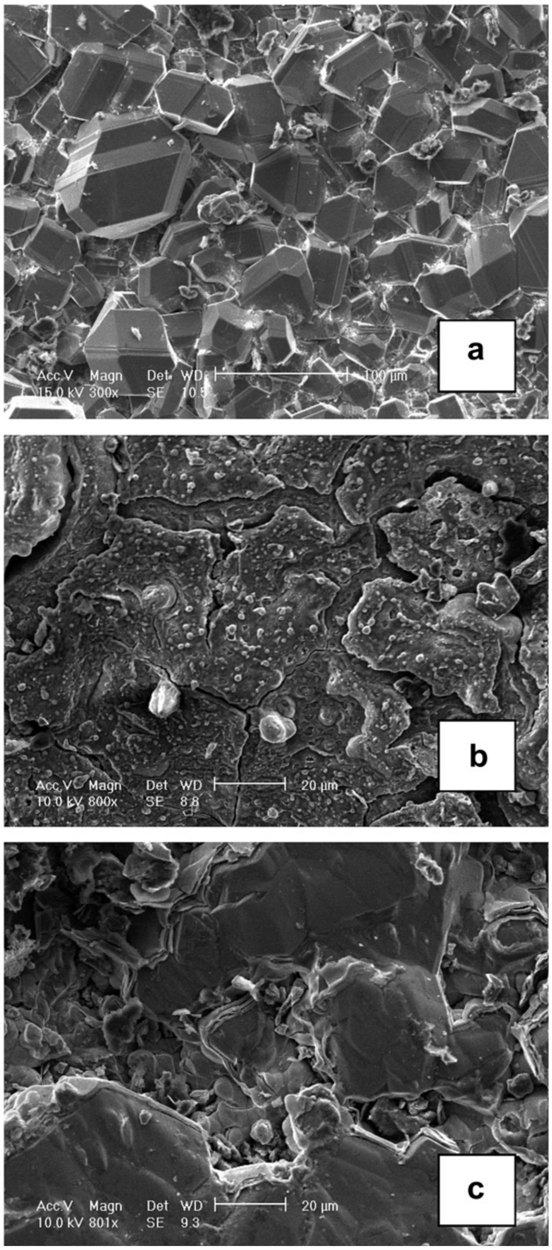

Fig. 1. SEM surface morphologies of the new, eroded and deposited carbon tiles with thick SiC coatings.

ture with little spots and cracks on the surface of eroded tile was shown as Fig. 1(b), while the profile of SiC crystal with thin film was seen on the surface of deposited tile as Fig. 1(c). Thus it can be seen that the structure of SiC crystal on the graphite tile was damaged clearly different and the coating morphology was changed in various degrees under long pulse plasma irradiation.

3.2. Surface atomic composition

The surface compositions of the three tiles characterized by EDS were listed as Table 1. The surface composition of new tile is nearly as stoichiometric composition of SiC (50.72%C, 49.28%Si). But the atomic percent of silicon in surface of eroded tile or deposited tile was reduced greatly. At the same time, oxygen and metal elements such as iron, molybdenum, chromium and nickel etc. were also found in the two tiles.

Decline of silicon percent showed the damage of the SiC structure on the surface of eroded tile and deposited tile also. Metals in the two tiles all came from the process of deposition, but oxygen and metals in the two tiles only can come from the deposition. So it took place simultaneously that the erosion and co-deposition occurred on the graphite limiter under irradiation with the plasma discharge.

3.3. Surface composition and the chemical state

Survey of the surface from new limiter tile by XPS as Fig. 2 showed that oxygen beside the carbon and silicon existed. The surface composition of new tile was: 34.51%C, 36.19%O and 29.30%Si. Analysis of the binding energy subdivision showed that 29.86%C was from SiC or graphite, 4.65%C was from carbon oxide; 22.41%O was from O_2, 13.78%O was from carbon oxide or silicon oxide; 20.67%Si was from SiC, 8.63%Si was from silicon oxide. The composition changed obviously after Ar^+ bombardment. Survey as Fig. 3 showed that oxygen percent was reduced to 10.42% and the peak binding energy was 533.5 eV which was mainly from SiO_2; silicon percent was 33.53% and the peak binding energy was 100.6 eV which was from SiC or SiO_2; carbon percent was 50.05% and the peak binding energy was 283.9 eV, from SiC or graphite.

Ar^+ bombardment of 12 nm reduced the oxygen percent obviously, so we considered that oxygen

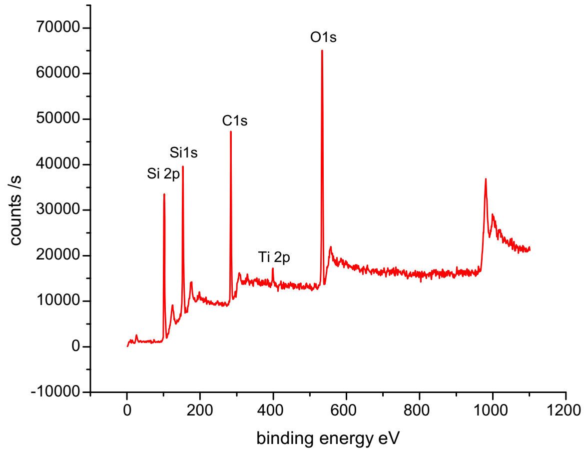

Fig. 2. XPS survey of the new carbon tiles with thick SiC coatings.

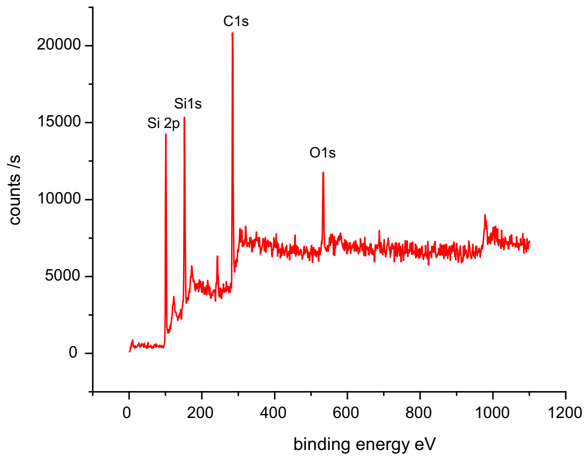

Fig. 3. XPS survey of the new carbon tile with thick SiC coatings after Ar^+ bombardment.

Table 1
Change of the surface atomic composition with different specimens by EDS

Specimens	Surface atomic composition (%)							
	C	Si	O	Fe	Mo	Cr	Ni	Ca
New tile	50.72	49.28						
Eroded tile	50.40	6.41	25.88	11.36	0.37	4.78	0.80	
Deposited tile	36.19	21.17	36.90	3.78	0.70	1.07		0.19

percent was gradient distributing from surface, and that most of the oxygen in surface of new tile is O_2 adsorbed. The residual oxygen percent after Ar^+ bombardment possibly came from the oxygenation in course of siliconization.

Survey of the surface from eroded limiter tile by XPS as Fig. 4 showed the surface composition of new tile: 26.29%C, 49.03%O, 15.05%B, 3.46%Fe, 1.25%Cr, 0.51%Ni 0.29%Mo, and 4.11%N. According to the binding energy subdivision, 11.73%C was from graphite, 14.56%C was from carbon oxide or B_4C; 35.33%O was from O_2, 13.70%O was from carbon oxide or metal oxide; boron was from B_4C or BN; all the metal existed as metal oxide. Survey after Ar^+ bombardment as Fig. 5 showed that the composition had changed from original surface and oxygen percent reduced to 25.43%. So the large part of oxygen in the surface of eroded tile was O_2 adsorbed and the residual oxygen percent after Ar^+ bombardment possibly came from the oxygen gettering in this campaign.

The composition of deposited tile tested by XPS as Fig. 6 was: 63.76%C, 25.54%O, 2.56%Si, 2.20%B, 1.69%Fe, 0.46%Cr, 0.30%Ni 0.39%Ti, and 3.00%N. In the same way, the binding energy subdivision showed that carbon was from graphite, SiC and carbon oxide; oxygen was from O_2, carbon oxide or metal oxide; boron and nitrogen was BN; all the metal existed as metal oxide. Survey after Ar^+ bombardment as Fig. 7 showed the oxygen percent reduced to 21.39% and was mainly from carbon

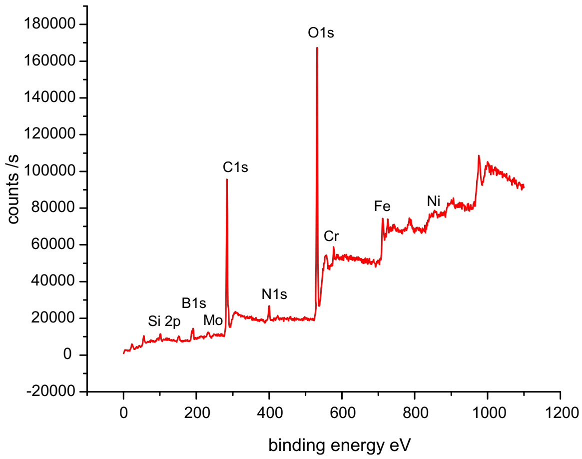

Fig. 4. XPS survey of the eroded carbon tile with thick SiC coatings.

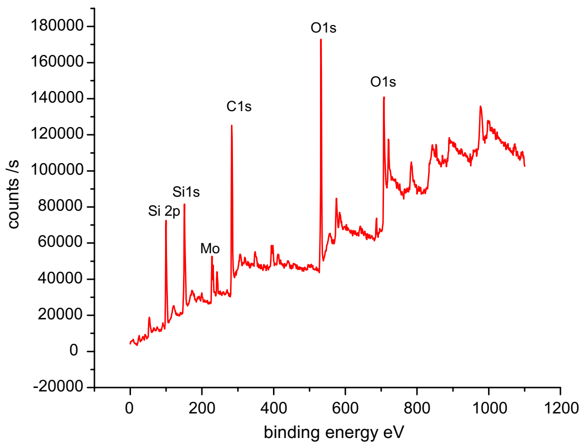

Fig. 5. XPS survey of the eroded carbon tile with thick SiC coatings after Ar^+ bombardment.

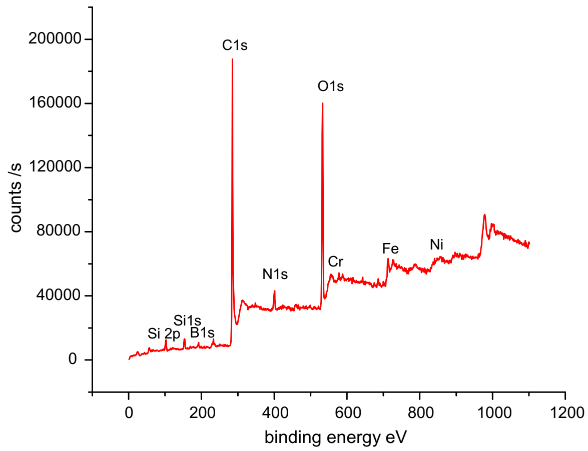

Fig. 6. XPS survey of the deposited carbon tile with thick SiC coatings.

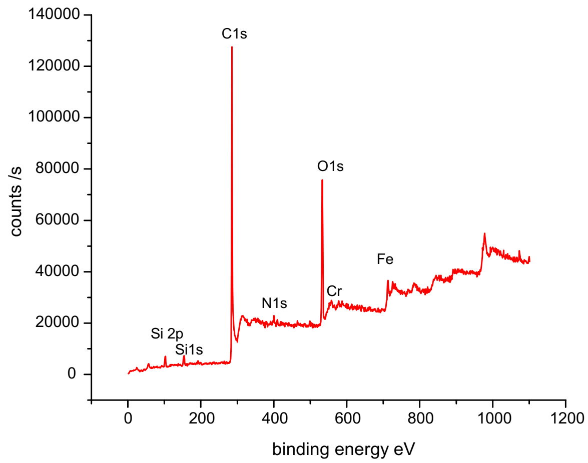

Fig. 7. XPS survey of the deposited tile with thick SiC coatings after Ar^+ bombardment.

oxide or metal oxide. The other elements remained with a little change of percent and peak binding energy.

By contrasting the result of XPS surveys and the binding energy subdivision of each specimen, we found that the surface composition of eroded tile and deposited tile was alike. Some metal elements existed in the two tiles but not existed in new tile. These elements came from the co-deposition during the plasma surface interaction. Oxygen was found in every specimen, and Ar^+ bombardment reduced the oxygen percent. So part of the oxygen was O_2 adsorbed and part came from oxygen gettering.

4. Conclusion

Thick SiC gradient coatings on the surface of limiter tiles were changed under long pulse plasma irradiation. Erosion and deposition were found on the surface. The surface morphology showed that lamellar matter or film replaced crystal structure after plasma irradiation. Plentiful oxygen and some other elements existed in the surface of graphite tiles after the plasma irradiation. Partial oxygen in tiles was O_2 absorbed and the rest was oxide which may came from the oxygenation in the experimental campaign. Composition result of Ar^+ bombardment sputtering showed that oxygen was distributed in the near surface of limiter tiles.

Acknowledgements

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