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# Gas desorption properties of TiC/C compositionally graded coatings on doped graphite

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#### ABSTRACT

To mitigate carbon contamination from doped graphite (1% B, 2.5% Si, 7.5% Ti) by irradiation of plasma, TiC/C compositionally graded coatings (CGCs) were prepared by chemical vapor deposition. Scanning electron microscopy (SEM) results indicated that the TiC/C CGCs were characterized by avoidances of cracks and loosely bonded particles. Gas desorption results suggested that out gas amount increased with the increase of temperatures. However, it was much lower than that of pure TiC coatings after irradiation for 200 s

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#### 1. Introduction

Carbon-based materials are characterized by light weight, high thermal conductivity, low-Z, excellent thermo-mechanical properties and good physical/chemical sputtering resistance. Therefore, they are not only widely used in current fusion devices exampled by HT-7 in China, but also of the primary choice of future fusion devices [1,2]. In a divertor of a fusion reactor, impurity control is one of the most crucial requirements [3], and plasma facing materials (PFMs) are an important factor to get high-plasma performance [4]. Therefore, many studies have been carried out to investigate impurities introduced into plasma environments [5–7]. In the next generation of fusion devices, higher heat and particle fluxes will be exerted on surface of PFMs. Persisting at obtaining materials with good physical, thermo-mechanical and sputtering resistance properties, many studies have been carried out [8–12].

Some ceramic coatings had been deposited on carbon materials to reduce C contamination [13]. However, thermal stresses arise at interfaces of coatings and substrates during thermal cycles, which led to cracks or even peeling off of the coatings. Our laboratory

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had prepared doped graphite named BSTDG1308 (1% B, 2.5% Si, 7.5% Ti), which were successfully used as the limiter plates in the last two campaigns of HT-7 tokamak experiments [14]. Chen et al. investigated good properties of the doped graphite [15].

For further enhancement of properties and efficiency of plasma facing materials, TiC/C CGCs were prepared by chemical vapor deposition (CVD). This paper focus on gas desorption properties of the as-prepared CGCs. Moreover, microstructure changes of coatings were also studied.

#### 2. Experimental

#### 2.1. Preparation of TiC/C compositionally graded coatings

Doped graphite with the size of  $10\,\mathrm{mm}\times10\,\mathrm{mm}\times10\,\mathrm{mm}$  was polished, and then ultrasonically cleaned subsequently in distilled water, ethanol and acetone. The TiC/C functionally graded coatings were prepared by thermal CVD at 1373 K. Ar (99.99%) and H<sub>2</sub> (99.99%) gas were used as protective and carrier/diluent gas, respectively. Moreover, titanium tetrachloride (TiCl<sub>4</sub>, 99.9%) and acetylene (99.9%) was used as sources of Ti and C, respectively. The amount of TiCl<sub>4</sub> transferred into the reactor was regulated by the flow rate of carrier gas H<sub>2</sub> and the temperature of the evaporator.

Composition of TiC/C coatings was conducted by adjusting the temperature and flow rates of the TiCl<sub>4</sub> and  $C_2H_2$  gas at intervals from 0 to 35 sccm and 100–65 sccm, respectively. Thickness of each layer was achieved by adjust deposition time. All the coat-

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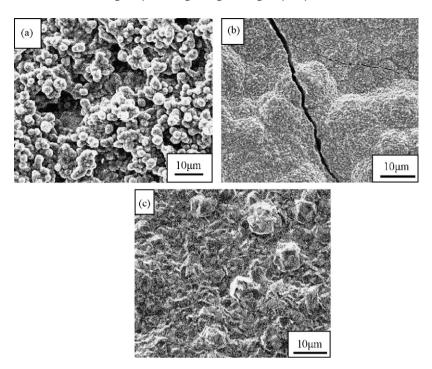


Fig. 1. Microstructures of different coated graphite: (a) TiC/C composite coatings; (b) pure TiC coatings; (c) TiC/C compositionally graded coatings.

ings in this paper were about  $120\,\mu m$ , which had been described in [16].

#### 2.2. Evaluation of gas desorption properties

After evacuation of the reactor with a volume of about  $0.2~{\rm m}^3$  for 5 h, gas desorption properties were evaluated by bombardment of electron beam at about  $2\times 10^{-5}$  Pa. Surface temperature of samples was controlled by equilibrium of cooling water and electron beam. The specific temperature was obtained from an infrared thermometer, and current of electron was adjusted in 0–1 A. Desorbed gases were analyzed by a QMS after bombardment of 300 s. Other specific parameters were listed in Table 1.

**Table 1**Parameters of plasma

| Plasma energy (eV)              | 200               |
|---------------------------------|-------------------|
| Voltage (keV)                   | 10-12             |
| Water pressure (MPa)            | 2                 |
| Plasma area (cm²)               | 0.2-1             |
| Pressure (Pa)                   | $25\times10^{-5}$ |
| Frequency of electron beam (Hz) | 200               |

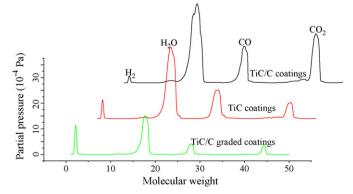


Fig. 2. Gas desorption of different coatings at 1773 K.

#### 3. Results and discussion

#### 3.1. Microstructures of coated graphite

Microstructures of coated graphite were given in Fig. 1. Compared to TiC/C composite coatings, pure TiC coatings were

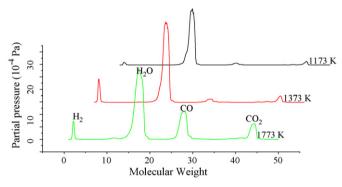


Fig. 3. Gas desorption of TiC coatings at different temperatures.

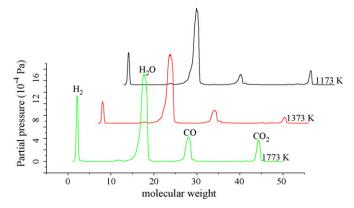


Fig. 4. Gas desorption of TiC/C graded coatings at different temperatures.

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