



# Selection of candidate doped graphite materials as plasma facing components for HT-7U device

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## Abstract

Selection of candidate materials for plasma facing material (PFM) in HT-7U device and plasma–wall interactions are critically important to reach high plasma performance. Based on concentrated research on multi-element doped graphite containing B, Si and Ti, two kinds of doped graphites have been chosen as candidates for PFM in HT-7U. Doped graphite GBST1308 with the dopant concentration of 1% B, 2.5% Si, 7.5% Ti was developed as low-Z PFM for reducing the chemical sputtering and suppressing the radiation enhanced sublimation, and successfully used as the new limiter material in last two campaigns of HT-7 tokamak experiments. Doped graphite with the composition of 2.5% Si, 7.5% Ti has improved mechanical properties and thermal conductivity of 314 W/m K at room temperature. TDS and high heat flux experiments results demonstrated that such doped graphites are promising candidate plasma facing components for HT-7U.

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## 1. Introduction

HT-7U is a large size tokamak with superconducting toroidal coils, which is being built in China. The main purpose of the device is to explore high performance plasma operation under steady-state conditions. Selection of candidate materials for plasma facing material (PFM) in HT-7U and plasma–wall interactions are critically important to reach high plasma performance under steady-state conditions. In the previous studies [1,2], we have concentrated on the research of microstructure and properties of multi-element doped graphite containing B, Si and Ti and optimized design of doped graphite with improved mechanical, thermal

properties and interaction behavior with plasma. Among many types of doped graphite developed, two kinds of doped graphites have been chosen as candidates for PFM for HT-7U. Doped graphite BSTDG with the dopant concentration of 1% B, 2.5% Si, 7.5% Ti was developed as low-Z PFM for reducing chemical sputtering (CS) and suppressing the radiation enhanced sublimation (RES), and successfully used as the new limiter material in last two campaigns of HT-7 tokamak experiments. Doped graphite STDG with the composition of 2.5% Si, 7.5% Ti has improved mechanical properties and thermal conductivity of 314 W/m K at room temperature [2]. It seems to be a promising high flux material for the divertor in HT-7U.

The aim of this paper is to present the properties and microstructure of two kinds of doped graphite and the primary tokamak experimental results with the doped graphite BSTDG as new limiter material in HT-7.

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Combined with TDS and high heat flux experiments, the possibility of the doped graphite to be used as plasma facing components in HT-7U under steady-state operation was discussed.

## 2. Optimized design and selection of doped graphite for plasma facing components

Boronized graphite has the excellent mechanical properties and low chemical erosion during plasma interaction [3]. However, the low thermal conductivity of boronized graphite restricts the application as PFM under steady-state conditions. Ti-doped graphite can reduce RES significantly when it is operated under high thermal fluxes and the surface temperature exceeds 1200 K [4]. Nevertheless, the reduction of chemical erosion by Ti was not so effective as B-doping [5]. Optimized design of doped graphite with improved mechanical, thermal properties and plasma interaction behavior can be realized by doping multi-element of B, Si and Ti into the carbon matrix [2]. From our previous studies of the multi-element (B, Si, Ti) doping system, it was found that a small concentration about 1 wt% of B can improve mechanical strength of doped graphite while the influence of thermal conductivity due to the formation of a B-C solid solution and increased phonon scattering [6] can be reduced to a limited level. Some researchers found that 1–3 wt% boron doping is effective to reduce chemical erosion drastically [5]. In our studies [2], by Ti-doping with a concentration around 7 wt% and high temperature heating treatment (2600 °C), doped graphite has high thermal conductivity; a concentration below 3 wt% of Si can lower the heat-treatment temperature and promote graphitization of carbon matrix as a catalyst, which is necessary for high thermal conductivity of doped graphite. It should be noted here that high concentration of Si dopant will produce cavities due to the depletion of Si during heat-treatment and led to the degradation of mechanical strength and microstructure, hence should be strictly controlled [1,2]. Among many types of multi-element doped graphite, we selected two kinds namely BSTDG with the composition of 1% B, 2.5% Si, 7.5% Ti and STDG of 2.5% Si, 7.5% Ti as two candidates for plasma facing components in HT-7U.

## 3. Tokamak experimental results with the doped graphite as new limiter in HT-7

HT-7 is a superconducting tokamak with the limiter configuration designed to operate with high power and long duration discharge. The last closed flux surface is defined by the main poloidal limiter, which receives the highest heat loads and largest flux of energetic particles bombardment from the plasma. In past operations with



Fig. 1. BSTDG coated with SiC gradient coating as new limiter in HT-7.

Mo limiter, due to energetic particles bombardment and overheating problem, the plasma discharge was usually terminated by the very strong hard X-ray radiation, hot spot and high-Z impurities. In order to alleviate above problems, a larger and fully circular carbon limiter was designed and installed to replace the smaller and discrete Mo limiter.

The new carbon limiter was made of doped graphite BSTDG with 200  $\mu\text{m}$  SiC gradient coating by chemical vapor reaction method. Fig. 1 shows the configuration and installation of new limiter in HT-7. Before the installation, performance evaluations of CS have been performed with BSTDG. The erosion experiment indicates that the CS yield of multi-element doped graphite at 1 keV and 50 eV  $\text{D}^+$  bombardment was decreased by factor of 5% and 30% respectively in comparison with that of pure graphite.

The move of limiter from Mo to the doped graphite BSTDG in HT-7 device turned out to be very successful [7]. There is no obvious surface damage on the surface of the new carbon limiter after tokamak experiment. Very stable and reproducible discharges were obtained. The operation parameter space is largely expanded, and the edge recycling, plasma density and impurity can be easily handled.

## 4. TDS and high heat flux test of the doped graphites

### 4.1. Thermal desorption spectrum of the doped graphites

In order to determine the out-gassing behavior of the doped graphite, TDS experiments were carried out with BSTDG and STDG, as well as one type of pure graphite as the reference material. For TDS experiments, each

material was tested with two runs of heating and the temperature was raised from RT to 1400 °C and held for 30 min at 1400 °C in Fig. 2. Total out-gassing amount after heating was calculated in each run for the materials and the results are shown in Table 1. It was noticed that in the first heating the out-gassing amount of pure graphite and BSTDG was nearly equal, while the out-gassing amount of STDG was about three times that of for pure graphite and BSTDG. While in the second heating the out-gassing amount of pure graphite,

BSTDG and STDG decreased by a factor of 3, 40, and 100 respectively. It reveals that pre-heat-treatment of doped graphite up to 1400 °C under vacuum is helpful to improve vacuum properties of doped graphite tiles. Based on the results of partial pressure of TDS and mass spectrum at peak desorption temperature, the main components of the desorbed gases released during heating were analyzed for each type of material, as shown in Table 2. For pure graphite, BSTDG and STDG, H<sub>2</sub>O, CO, CO<sub>2</sub> and C<sub>x</sub>H<sub>y</sub> are the dominant de-

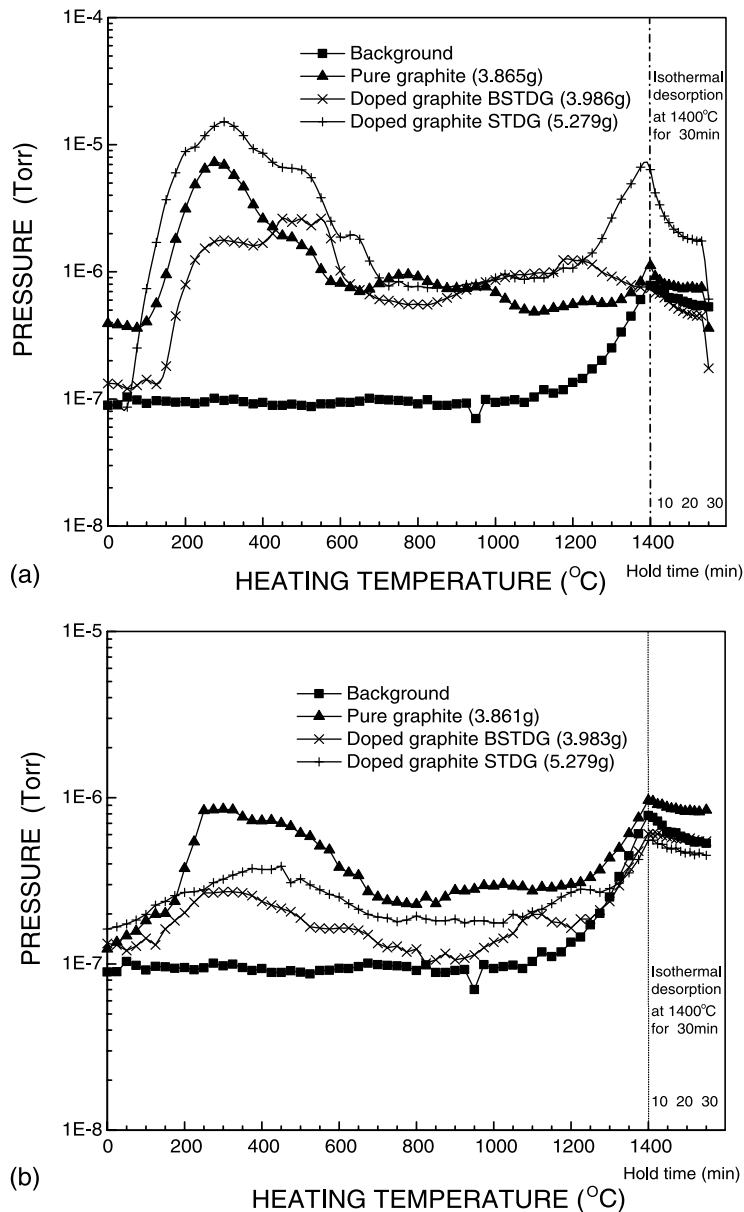


Fig. 2. TDS spectrum of carbon-based materials during (a) first heating and (b) second heating.

Table 1  
Out-gassing amount of carbon-based materials

Material type	Out-gassing amount of carbon-based materials <sup>a</sup>	
	First run	Second run
Pure graphite	0.1380 Torr l/g	0.0521 Torr l/g
BSTDG	0.1278 Torr l/g	0.0035 Torr l/g
STDG	0.3619 Torr l/g	0.0036 Torr l/g

<sup>a</sup> Background has been subtracted for out-gassing amount of carbon-based materials.

Table 2  
Analysis of the major components of desorbed gases during thermal desorption

Material type	Heating number	Out-gassing amount of desorbed species ( $\times 10^{-4}$ Torr l/g)			
		H <sub>2</sub> O	CO	CO <sub>2</sub>	C <sub>x</sub> H <sub>y</sub>
Pure graphite	First run	25	11	3	25
	Second run	59	12	6	6
BSTDG	First run	14	15	7	14
	Second run	0	24	1	0
STDG	First run	45	53	30	45
	Second run	0	15	1	0

sorbed gas specimen in the first heating. While in the second run H<sub>2</sub>O dominates for pure graphite and CO does for BSTDG and STDG.

#### 4.2. High heat flux test of doped graphites

High heat flux test of doped graphite BSTDG, together with pure graphite, IG-430U and CX-2002U composite to have a comparison, was performed on the active cooling teststand of National Institute for Fusion Science [8]. The test specimens in the size of 70 × 30 × 10 mm were mechanically bolted to copper heat sink with a torque of 20 kg cm. In order to have a tight contact, a graphite sheet was placed between the specimens and copper heat sink. Uniform electron beam at 30 keV was irradiated on the surface of the carbon-based materials through a beam limiter with an aperture of 29 mm × 30 mm. Beam duration during ramp-up, plateau, ramp-down and rest time were 25, 100, 1 and 20 s, respectively. Heat flux changed from 1 to 8 MW/m<sup>2</sup>. Thermal response tests were carried out by stepwise increase of the heat flux. The surface temperature of the central part about 5 mm in diameter was measured with an optical pyrometer. Two thermocouples were inserted into two deep holes to measure the temperature difference. The distance between two holes is 3 mm. Time evolution of heat flux and thermal response of carbon-based materials under electron beam irradiation is shown in Fig. 3. Thermal conductivity of carbon-based materials can be calculated from heat flux and temperature difference between the upper hole and the lower

hole measured with two thermocouples. Dependence of thermal conductivity of carbon-based materials on heat flux and temperature is shown in Fig. 4.

It seems that the surface temperature of carbon-based materials detected with an optical pyrometer under irradiation is not only governed by thermal conductivity of bulk materials, but also controlled by surface smoothness. For doped graphite BSTDG, its surface temperature is the lowest during irradiation when heat flux is below 6 MW/m<sup>2</sup>. When heat flux is increased from 6 to 8 MW/m<sup>2</sup>, a rapid increase of surface temperature of BSTDG can be observed in Fig. 3. It

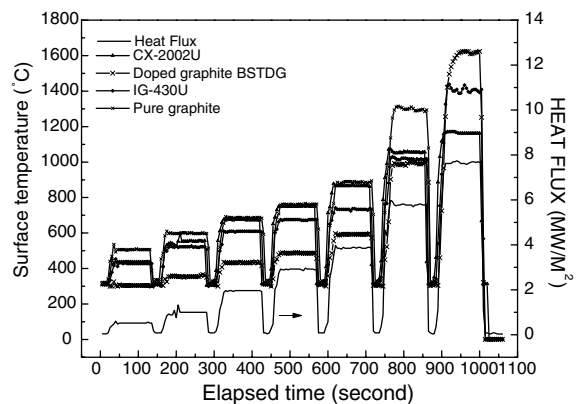


Fig. 3. Time evolution of heat flux irradiated and thermal response at surface of test material.

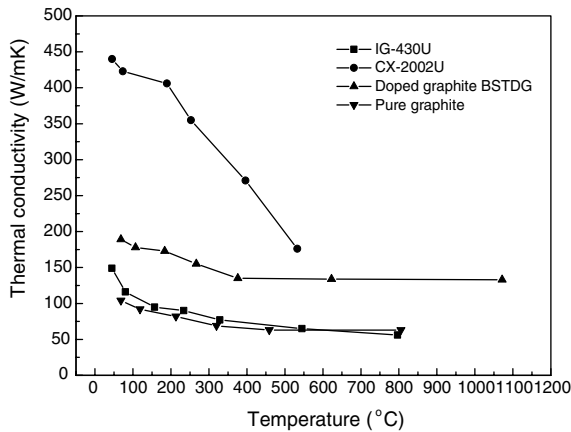


Fig. 4. Dependence of thermal conductivity of carbon-based materials on bulk temperature during irradiation.

might be due to the strong interactions between electron beam and dopant imposed at the surface.

It should be highlighted here that thermal conductivity of BSTDG has a weak dependence on the bulk temperature from 400 to 1100 °C, which is different from that of CX-200U, IG-430U and pure graphite. It was proposed that vapor shielding played a significant role in the energy process during irradiation [9–11]. Under high heat flux, a partial loss of thermal contact due to the mismatch of coefficient of thermal expansion between doped graphite and copper heat sink might lead to quick increase of surface temperature. We assume that dopant also plays an important role. At high temperatures TiC dopant, which is mainly distributed on the grain boundaries and introduces imperfections there, can make a contribution to specific heat of bulk doped graphite [12]. Considering the low electrical resistivity of TiC, the contribution of electron as charge carrier to thermal conductivity should be also taken into account.

## 5. Summary

We select two doped graphites BSTDG and STDG as candidate plasma facing components for HT-7U device

under steady-state conditions. These doped graphites have improved mechanical, thermal and vacuum properties as well as plasma–surface interaction behavior. BSTDG has been successfully used as limiter materials in HT-7 device. TDS experiments demonstrated that pre-heat-treatment of the doped graphites under vacuum up to 1400 °C can reduce the out-gassing amount to a very low level. High flux test of the doped graphites reveals that they can withstand 8 MW/m<sup>2</sup> without surface damage and the thermal conductivity has a weak dependence on higher heat flux or bulk temperatures.

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