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High heat flux test of SiC coated doped graphite

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Abstract

Doped graphites containing B, Si and Ti dopants with improved mechanical and thermal properties are developed as divertor plates in experimental advanced superconducting tokamak (EAST) device. SiC coatings with a grading distribution along the depth up to 100 μm from the surface were applied on the doped graphites by chemical vapor reaction (CVR) method in order to reduce the carbon impurities emitted from the surface of doped graphites during long pulse plasma discharge. High heat flux test on SiC coated graphites were carried out with an active cooling test (ACT) facility. Results show that SiC coated doped graphites can handle heat flux up to 6 MW/m^2 while keeping structural integrity. The abrupt increase of surface temperature and the eroded pot on the surface implied that SiC coatings acted as a heat transfer barrier when irradiated with heat flux of 6 MW/m^2.

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1. Introduction

Experimental advanced superconducting tokamak (EAST) [1] with superconductive coil systems has been built at Institute of Plasmaphysics, Chinese Academy of Sciences (ASIPP). EAST is designed with double null-divertor and its mission is to explore high performance of plasma discharge at steady state [2]. For that, the device needs several thousands of plates as first wall for heat and particle removal. The heat flux on the divertor plates can be determined with the input heating power. The maximum heat flux on the divertor plate of EAST is designed to be 5 MW/m^2 with long pulse discharge operation up to 1000 s [3].

Multi-element doped graphites containing B, Si, Ti with improved mechanical, thermal properties have been developed in China [4,5]. These types of doped graphites are to be used as divertor plates in EAST device. In order to reduce the carbon impurities emitted from the surface of doped graphites during plasma discharge, grading distributed SiC coatings were prepared on the surface of doped graphite with CVR methods [6]. Such materials have been successfully installed as carbon limiter

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in HT-7 device to handle the heat of 31 MJ injected, with the longest plasma discharge duration up to 239 s [7,8].

In order to evaluate thermal behavior of SiC coated doped graphite tiles under high heat flux, mechanically bolt-jointed plates with doped graphites and copper heat sink were developed. High heat flux test of the plates was carried out with a facility named ACT with a 100 kW electron gun at National Institute for Fusion Science [9]. The aim of this work is to investigate thermal response of SiC coated doped graphites under electron beam irradiation at different heat flux and long duration, to reveal the effect of SiC coating on heat-conducting and the surface microstructure evolution during irradiation.

2. Experimental

2.1. Materials and properties

2.1.1. Doped graphite BSTDG

In this study, one type of doped graphite named BSTDG was selected as the base material for SiC coating. This material has the dopant concentration of 1 wt% B, 2.5 wt% Si and 7.5 wt% Ti. The thermophysical properties were tabulated in Table 1 and one isotropic graphite, named IG-430U and produced in Toyo Tansu corporation was chosen as the reference material.

2.1.2. Grading SiC coating on doped graphite

The doped graphite was placed in a high-temperature vacuum electric furnace. Si (vapor) was used to siliconize the substrate materials in the temperature range of 1500–1600 °C. During the siliconization process, silicon carbide coating with some free silicon was formed by CVR of Si (vapor) with carbon at the surface of the substrate materials. The pore existed in the substrate materials provided a path for Si to diffuse inside and the SiC coating showed a grading distribution. The thickness of SiC coating was estimated to be 100 μm, as indicated in Fig. 1(a). SiC coating was composed of large crystal of SiC grains, shown in Fig. 1(b). Thermal shock resistance of SiC coated doped graphite was carried out by water-quenching test with a electrical muffle furnace. The critical temperature difference (ΔT) was 1000 °C. Due to the in situ reaction of Si (vapor) with carbon substrate, the SiC coatings showed very strong adhesion with carbon substrate. Only some microcracks can be observed in Fig. 1(c) and no spallation occurred for the SiC coating.

2.2. Thermal conductivity measurement

Thermal conductivity measurement of doped graphite before and after coating was performed with a Nano-Flash-Apparatus LFA447 in the temperature range from 25 to 300 °C. The Nanoflash instrumentation can be used to measure the thermal diffusivity, specific heat and thermal conductivity of graphite and other materials. The size of the samples is 12.7 mm in diameter and 2.05 mm in thickness. The measurement of the thermal diffusivity and specific heat allows the calculation of the thermal conductivity with an additional measurement of the bulk density of the samples. One type of graphite named PocoAXM was used as the calibration material to measure the specific heat.

2.3. High heat flux test

2.3.1. High heat flux test facility ACT

A high heat flux test facility called ACT at National Institute for Fusion Science in Japan was used to evaluate the thermal response of the selected materials under electron beam irradiation. The facility consists of a vacuum vessel of 400 l in volume, 100 kW electron gun, vacuum pumping system, water-cooling system, and data acquisition.

2.3.2. High heat flux test

Heat load test was performed on ACT facility of National Institute for Fusion Science (NIFS). Uni-

Table 1
Properties of doped graphite BSTDG and reference material of IG-430U

Material	Bulk density (g/cm^3)	Electrical resistivity (μΩ m)	Flexural strength (MPa)	Compressive strength (MPa)	Tensile strength (MPa)	Elastic modulus (GPa)	CTE (10^{-6} K^{-1})	Thermal conductivity (W/mK)
BSTDG	2.02	6.4	56.0	94	47	11.5	4.2	180
IG-430U	1.82	9.2	53.9	90.2	37.2	10.8	4.8	150

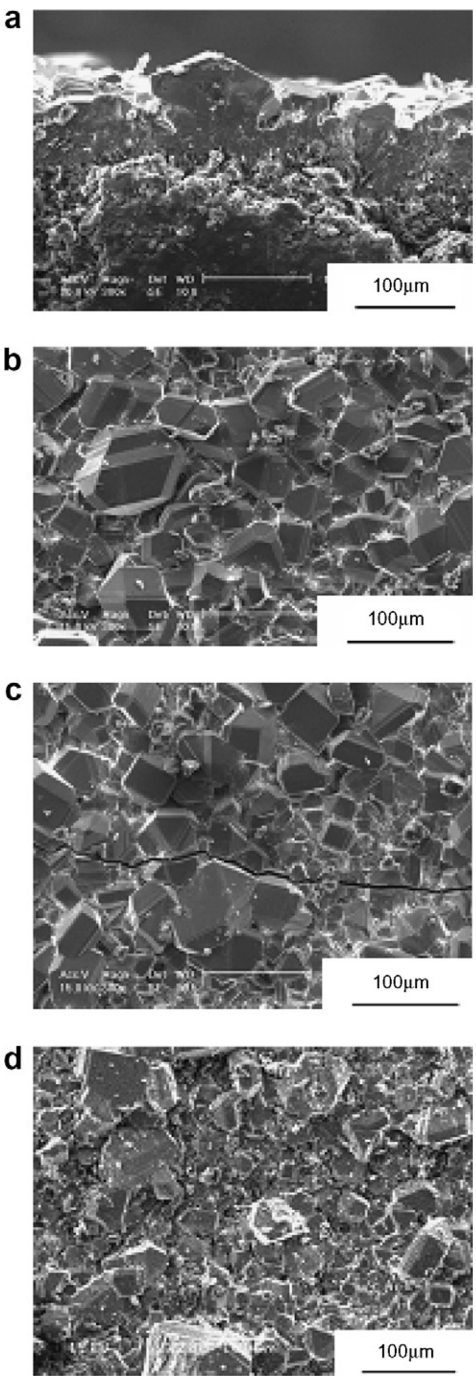

Fig. 1. SiC coatings on doped graphite BSTDG (a) SiC grading distribution, (b) surface morphology, (c) microcrack after water-quenching test and (d) thermal erosion of SiC coating.

form electron beam at 30 keV was irradiated on the surface of doped graphite with or without SiC coating through a beam limiter with an aperture of 20 × 20 mm. The mechanically bolt-jointed plates of doped graphites and copper heat sink were performed. The heat flux tests were carried out under the condition of high vacuum more than 1.0×10^{-5} Torr, and the water flow velocity, pressure and temperature were 15.0 m/s, 0.5 MPa and 293 K, respectively. After the irradiation of the samples, the surface of the samples was observed with a scanning electron microscope to investigate surface damage due to electron beam irradiation.

3. Results and discussion

3.1. Dependence of thermal conductivity on temperature

Dependence of thermal diffusivity and thermal conductivity on temperature was measured with NETZSCH LFA447 and indicated in Fig. 2. The effects of SiC coatings on thermal properties of the test plates can be summarized as three aspects. First,

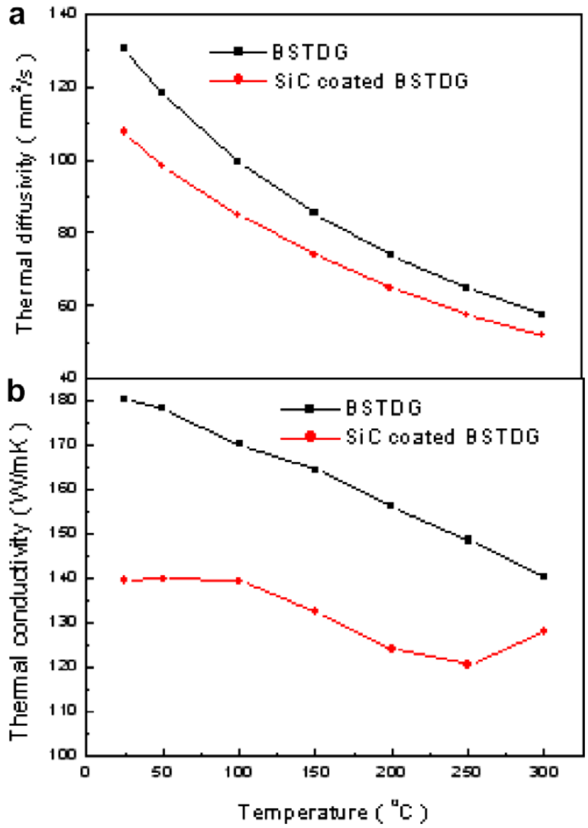

Fig. 2. Dependence of thermal properties on temperature for BSTDG with and without SiC coatings (a) thermal diffusivity and (b) thermal conductivity.

due to the formation of SiC coating on the surface of doped graphite, thermal diffusivity showed a decrease, and the gap between with and without coatings became more and more less with the rise of temperature. Second, due to the formation of SiC coating, the heat capacity of coated plates showed an increase in contrast with thermal diffusivity. Third, bulk density of coated samples increased from 2.02 to 2.10 g/cm^3 after coating. It can be observed that thermal conductivity of SiC coated samples show a weak dependence on temperature, which makes it one promising candidate as high heat flux component.

3.2. Thermal response under high heat flux test

Heat load tests were carried out with ACT facility. The test specimens in the size of 70 × 30 × 10 mm were mechanically bolted to copper heat sinks with a torch of 20 kg cm. In order to have a tight contact, a graphite sheet was placed between the specimens and copper heat sink. By adjusting the beam current, heat flux ranging from 1 to 8 MW/m^2 was released and irradiated on the surface of the specimens. Beam durations during ramp-up, plateau and ramp-down were 20, 100 and 0 s, respectively. Surface temperature (T_s) of the specimens is measured with an optical pyrometer. Temperatures of the upper side (T_u) and the lower side (T_l) are also measured with thermocouples. The positions for T_u and T_l are 1.5 mm upper/lower away from the joined interface.

Fig. 3(a) showed thermal response of BSTDG and reference material IG-430U under high heat load. It is obvious that the surface temperatures of these materials increase steadily with heat flux. For both materials, surface temperature saturated quickly and kept stable for heat flux reached different plateau level ranging from 1 to 6 MW/m^2. Surface temperature was in the level of 820 °C, revealing excellent capacity for heat removing. Surface temperature difference between BSTDG and IG-430U was very small when heat flux was below 4 MW/m^2, due to the very near thermal conductivity and well designed tiles/heat sink with high heat removing capacity. While when heat flux was increased to 6 MW/m^2, there was a large difference of surface temperature for BSTDG and IG-430U. The difference for the heat-removing capacity can be ascribed to the less decrease of thermal conductivity for BSTDG under high temperatures. Such results have been observed in our previous studies [5].

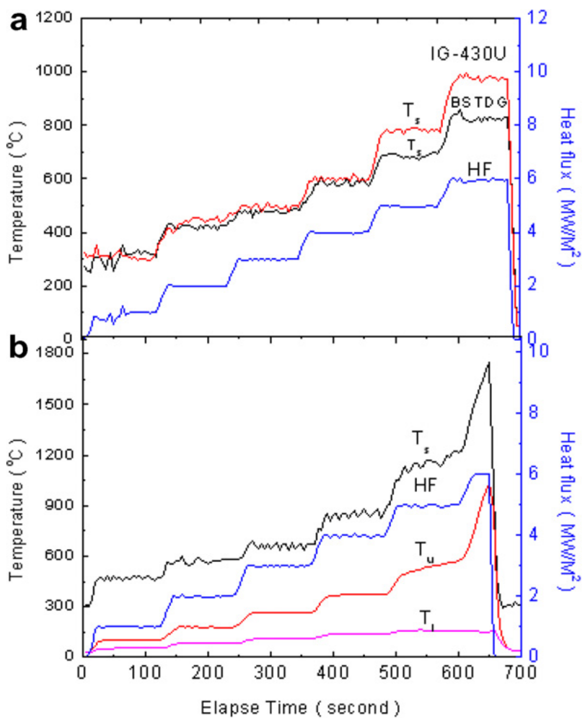

Fig. 3. Thermal response of the tested plates under high heat load (a) BSTDG and reference material IG-430U and (b) SiC coated BSTDG.

Thermal response of SiC coated BSTDG under heat load was shown in Fig. 3(b). It can be obviously observed that surface temperatures for the coated material were significantly higher than that without coating. Under heat flux irradiation of 6 MW/m^2, surface temperature was in a nearly uncontrolled state and showed a sharp increase to 1800 °C. Two reasons are responsible for this, one is that the coating of SiC on doped graphite leads to the decrease of bulk thermal conductivity for specimens, and the other reason is due to the hardness and coarseness of SiC coating, the contact between specimen and copper heat sink was less tight than doped graphite, leading to poor thermal conductance at the interface at high temperatures. This effect can be also confirmed with the fact that T_u increase rapidly in accordance with surface temperature.

3.3. Surface microstructure evolution after irradiation

After electron beam irradiation, the surface morphology was observed with SEM. For the doped graphite without coating, no obvious damage can be found. While for SiC coated samples, there was an obvious dun part changed from light green on the center of irradiated area. Comparison between Fig. 1d and Fig. 1b showed that SiC particles became much more finer and surface became rough due to the depletion of SiC under high heat load, when exposed to heat flux of 6 MW/m^2 and surface temperature reaches 1800 °C. Nevertheless, the microstructure of SiC coating after irradiation still keep the integrity and no microcracks can be observed on the surface of coating.

4. Conclusion

The grading SiC coatings distributed in the range of 100 μm on the surface of doped graphite have excellent thermal shock resistance. High heat load test demonstrated that doped graphite without SiC coatings can bear heat flux up to 6 MW/m^2 without obvious surface damage, while SiC coating on doped graphite led to a quick increase of surface temperature when heat flux is increasing to 6 MW/m^2. SiC coating might act as a barrier for heat transfer and SiC particles in the irradiation area became more finer due to thermal erosion.

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