

Research on SiC coating for graphite by chemical infiltration sintering

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Abstract

In this study, SiC coating for the graphite was prepared by using chemical infiltration sintering method. The effect of sintering temperature on microstructure and thickness of SiC coating was investigated. The crack-free SiC coating with small crystal size was achieved, and the thickness of the coatings were in most cases about 150 μm with a comparatively gradient distribution, when the sintering was performed at the temperature about 1773-1873K. However, the extreme grain growth as well as obvious cracks of SiC coating was observed, and the thickness of SiC coating was less than 120 μm when the sintering temperature was about 1973-2073K. In addition, the grain boundaries between coating and carbon substrates were also observed. X-ray plate diffraction analysis showed that the phase of the coatings was SiC and Si-SiC when the sintering temperature was 1773-1873K and 1973-2073K, respectively.

Key words: Coatings; Doped carbons; Diffusion

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1. Introduction

Carbon materials have been widely used as plasma facing components in fusion devices. Due to exposure high heat load and strong energetic particle bombardment, sputtering and erosion are inevitable for carbon materials. In order to reduce carbon impurities, carbon materials by doping some elements (such as B, Si, Ti, etc) can effectively reduce chemical sputtering (CS) yield and suppress the radiation enhanced sublimation (RES) [1,2]. Moreover, some refractory carbide coatings on carbon substrates are stable in fusion plasma and prevent the strong interactions between hydrogen plasma and carbon substrates to a great degree [3]. For example, in order to suppress the erosion of the graphite due to the oxygen, boronization and siliconization with Si- and B-containing organic compounds and helium through in situ glow discharge [4-7] in JT-60U and HT-7 tokamak facilities, resulting in Si- and B-based films can effectively reduce the contamination of light impurities in plasma due to their strong affinity ability for oxygen. However, the thickness of such films was limited within the range of 700-1000Å, its lifetime can only survive 140-200 seconds [8] of discharge so that this kind of film is ineffective for higher heat flux components as well as the next generation (quasi-steady state) tokamak machines, since such film will be too rapidly eroded in plasma exposure. Therefore, thick carbide coating for carbon substrates is necessary and indispensable.

In this study, thick SiC gradient coating on the doped graphite surface is developed by a chemical infiltration sintering method. The effect of sintering temperature on microstructure and thickness of SiC coating was investigated.

2. Experimental

2.1. Preparation of SiC coating

A new kind of doped graphite named BSTDG, was used as carbon substrate in this work, and now commercially as limiter material in Experimental Advanced Superconducting Tokamak (EAST) built in China. Its properties were listed in many references [9,10]. The pre-coating specimens were firstly mechanically cut from the BSTDG, then polished and ultrasonically washed in ethyl alcohol for 15 min.

In order to obtain a homogeneous and stable Si infiltration slurry, some additives were applied in such a way that polyethylene was the dispersant and distilled water was the solvent. The dispersant of polyethylene was firstly dissolved into the distilled water, then the Si powder (mean particle size 13 μ m, purity, 99.3 wt.%) were added into the blending solvent to keep slurry in a ball-milling machine, and mixed about 30 min. The solid-to-liquid ratio of the slurry was adjusted to produce a viscosity suitable for the application of slurry on the specimens. Then the pre-coating specimens were dipped into the Si infiltration slurry (In this work, the ratio of the polyethylene, Si powder and distilled water was 0.5:5:94.5 wt. %). After drying, the As-coated graphite was sintered in a vacuum inductive furnace to 1773, 1873, 1973 and 2073K, and dwell of 2 h at final temperature, and the resulting samples were named BSTDG-1, BSTDG-2, BSTDG-3 and BSTDG-4, respectively.

2.2 Microstructure and properties evaluation

The surface and cross-section images of the specimens were observed by scanning electron microscopy (SEM). X-ray plate diffraction patterns (XRD) with a Cu K α radiation was used to determine the crystalline phases.

3. Results and discussion

Surface phase composition analysis of the coating by XRD (Fig. 1) showed that Si reacted with carbon to form SiC. What was interesting that there is also small diffraction peak for Si when the sintering temperature was higher than 1873K. On the basis of the results, the phase of the coatings were SiC and Si-SiC when the sintering temperature was 1773-1873K and 1973-2073K, respectively. Fig. 2 showed the surface morphologies of SiC coating at different sintering temperature. It can be seen that the formation of small crystal size and crack-free SiC coating is achieved when the sintering temperature was performed at 1773 and 1873K, in addition, the coating obtained at 1873K showed a more perfectly compact structure compared with that of 1773K. However, the extreme grain growth as well as obvious cracks of SiC coating was also observed when the sintering temperature was around 1973-2073K. These facts implied that there were some interrelations between microstructure of SiC coating and the sintering temperature.

The cross-section images of the SiC coatings prepared at different sintering temperature by SEM were all shown in Fig. 3. The thickness of the SiC coatings, which were in most cases

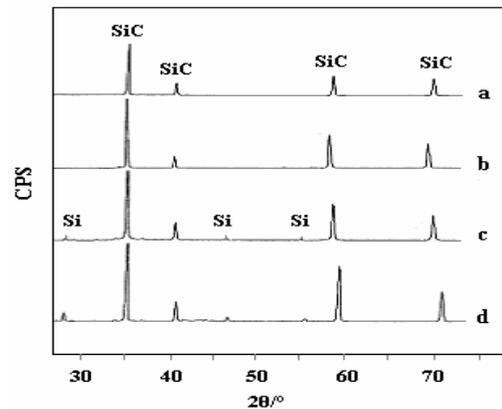


Fig. 1. Surface phase composition analysis of the coatings (a: Sintering temperature at 1773K; b, c and d: Sintering temperature at 1873K, 1973K and 2073K, respectively).

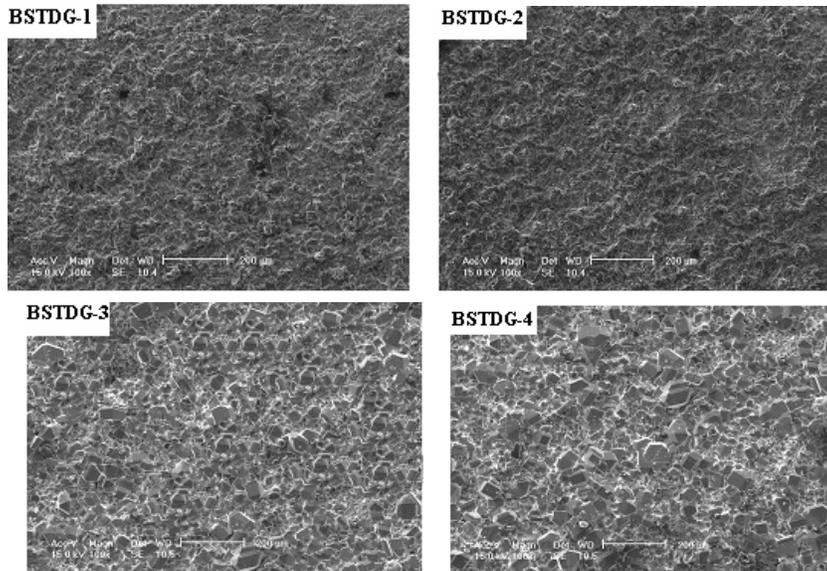


Fig .2. Surface morphology graphs of SiC coatings at different sintering temperature.

about 150 μm with a comparatively gradient distribution at 1773 and 1873K. However, the thickness of SiC coating was less than 120 μm when the sintering temperature was about 1973-2073K, in addition, grain boundaries between coating and carbon substrates for such coatings were also observed. Based on the above analysis, we can conclude that certain crystal structure and the thickness of SiC coating could be achieved by controlling the sintering temperature.

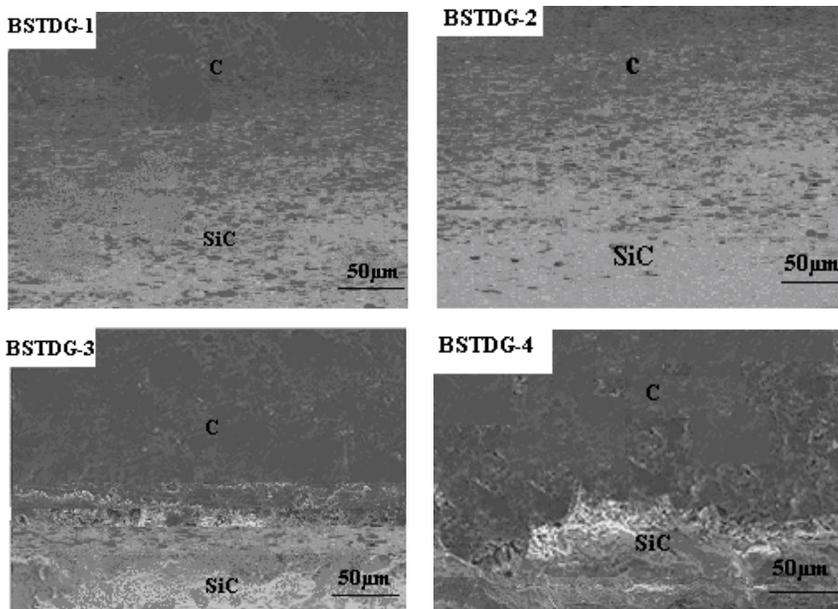


Fig .3. Section-cross morphology graphs of SiC coatings at different sintering temperature.

When the sintering temperature was above melting point of Si (1410°C), liquid silicon was gradually formed and flowed along the surface of the BSTDG. The chemical reaction simultaneously took place as follows: Si (liquid) + C (solid) \longrightarrow SiC (solid). It occurred not only on the surface, but also in the interior of carbon substrates due to the infiltration of liquid silicon through the open pores by the capillary force. As was well known, the chemical reaction rate of Si and C was a function of temperature, only when the infiltration rate of liquid Si was smaller than the chemical reaction rate, a gradient distribution of SiC coating could be formed [11]. A gradient distribution of SiC coating was only formed when the sintering temperature was around 1773-1873K. The major reason was probably that the chemical reaction rate was bigger than the infiltration rate of liquid Si during this temperature range, which had an advantageous of the infiltration of liquid silicon through the pores of the BSTDG. The ability to produce a dense, crack-free as well as gradient SiC coating demonstrates a clear technological accomplishment of this work when the sintering temperature was at 1873K. On the contrary, if the sintering temperature was excessive high, such as 2073K, a sudden SiC coating can be formed due to high chemical reaction rate. The primarily SiC layer on the surface of substrates acted as a bigger diffusion barrier against Si-mass transfer by the liquid phase, which not only resulted in extreme grain growth and obvious grain boundary, also restrained the increase of the thickness of SiC coating. Such coating layer easily causes fracture or crack at the interface by residual thermal stress, attributed to their mismatch of the thermal expansion coefficients (CTE).

As mentioned above, a little Si was determined when the sintering temperature was around 1973-2073K. We suggest there may be the two reasons as follows. On one hand, some exterior silicon dipped could not wholly penetrate through the pre-formed SiC coating due to its barrier effect, and did not effectively react with the carbon. This result was similar to the one reported by Zhu et al [12]. On the other hand, a part of silicon before reacted with carbon was probably vaporized at comparatively high temperature, and deposited the surface of the coating during

cooling stage.

4. Conclusions

The present work can be summarized as follows:

(1) The coating obtained at 1773-1873K has not only small crystal size and crack-free, but also a comparatively gradient distribution. However, the extreme grain growth as well as obvious cracks of SiC coating was also observed when the sintering temperature was higher than 1973K.

(2) The thickness of the coatings, which was about 150 μm at 1773-1873K, however, 120 μm at 1973-2073K. The phase of the coatings was SiC and Si-SiC when the sintering temperature was 1773-1873K and 1973-2073K, respectively.

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