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Renewable boron carbide coating in plasma shots of tokamak T11-M

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ABSTRACT

Experimental results on boronization in plasma shots of the tokamak T-11M are presented. Non-toxic and not explosive metacarborane $C_2H_{12}B_{10}$ was used in the boron deposition process. Experiments have been carried out in shots with parameters: toroidal field ~1–1.2 T, plasma current I_p = 70 κA, average shot duration $t_p \sim 150$ ms and electron density along the central chord $n_e \sim 2.5 \times 10^{13}$ cm^{-3}. As a result of experiment, a dense film of ~0.2 microns thickness with good adhesion to a surface has formed on the reference specimens after 8 s boronization. After boronization the impurities in wall areas have been suppressed. High vacuum characteristics of the discharge chamber were stabilized. Working vacuum was reached without a preliminary induction heating and cleaning by a glow discharge, and stabilization of the plasma filament has improved. Shot duration without disruption at densities of $n_e = 1.3 \times 10^{13}$ cm^{-3}, I_p = 70 κA was 350 ms and $n_e = 4.64 \times 10^{13}$ cm^{-3}, I_p = 70 κA was 250 ms. High repeatability of experimental results has appeared. Developed technology opens an opportunity of practical production of renewable structured boron–carbon coatings with use of plasma shots in large-scale tokamaks, such as DIII-D, JET, JT-60 UP, ITER, DEMO.

1. Introduction

Crystalline boron carbide coating B_4C, produced by the method of chemical vapor deposition in a reactor from the fluoride phase at the temperatures to ~2000 °C, is widely used in the Russian tokamaks [1,2]. The coating has a number of substantial advantages in comparison with graphites: the small chemical and physical sputtering, low ion-stimulated desorption and radiation-accelerated sublimation. As a result, the rate of the film erosion and sputtering at ion and plasma irradiation in existing accelerating and thermonuclear facilities is much lower than those of graphites (Fig. 1) [3]. These characteristics vary little up to the temperatures of ~1400 °C. Hydrogen capture in boron carbide in several times is less, than in fine-grained, dense graphites and CFC composites (Fig. 2) [4]. This difference is increased with radiation dosage, hydrogen capture in B_4C is saturated at doses about 10^{23} at/m^2. Thermal conduction of boron carbide is not high (20 W/mK), but a coating of thickness up to 100 μm, deposited on graphites with high thermal conduction, withstands high heat loads without its destruction and integrity losses. Coatings of thickness up to 100 μm, deposited on various types of graphites (MPG-8, RGT, ATJ, pyrolitic graphites), were examined under an irradiation in various plasma-physical installations simulating disruptions and divertor conditions in tokamaks. Experiments were carried out under electron and ion beams irradiation in the Sandia National Laboratories, in the PLADIS plasma gun facility in the University of New Mexico [5], in the DIII-D tokamak divertor plasma («General Atomic», San Diego) [6–8], under real conditions in the T-11M tokamak,in the plasma facility МК-200 in TRINITI, Troitsk [5,9] and in the tokamak T-10 in RRC «Kurchatov institute», Moscow [10,11]. In all experiments the boron carbide coating showed a high resistance to heat loads without destruction and integrity losses, and without changes in the chemical composition and material structure. The best coatings were for deposition on graphites with a high thermal conduction (RGT or pyrolitic graphite) [5,10,11]. Boron carbide coating produced by chemical vapor deposition has obvious advantages. However, because of complex technology of production at high temperatures a coating can be used in tokamaks only at the stage of initial mounting, reconstruction and modification of a discharge vessel. Boronization in a glow discharge in-situ results in to formation of thin amorphous films of thickness up to 100 nm [12,13]. After this process, impurity atoms of carbon, oxygen and metals are suppressed. However such films erode during several tens of seconds of plasma shots, reducing the effectiveness of the thin boron films from deposition in a glow discharge. Recently, boron carbide films with a composition close to stochiometric B_4C have been obtained on the PISCES-B facility in the University of California, San Diego [14]. Deposition was performed in plasma with parameters similar to the DIII-D tokamak divertor plasma. Non-toxic, non-explosive metacarborane $C_2H_{12}B_{10}$ was used as initial substance. Deposition rate was extremely high and achieved of ~30 nm/s, that approximately in 1000 times exceeds a rate of film deposition in a glow discharge. Thickness of deposited layer

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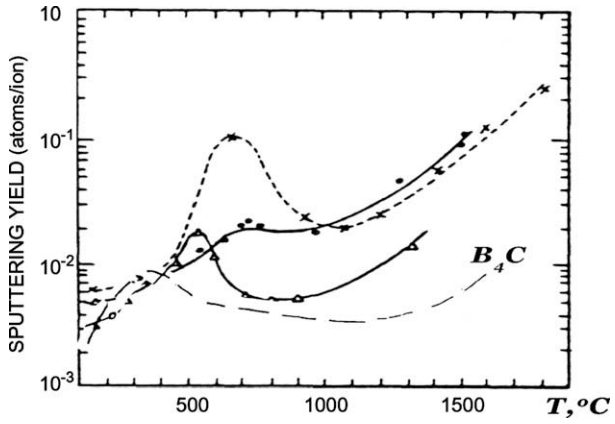

Fig. 1. Temperature dependence of the sputtering coefficients for graphites and B_4C. x – pyrolittic graphite, 3 κeV H_3^+; ● – C–SiC general atomic, 6 keV H_3^+; Δ – C/B USB – 15, 10 keV H^+; B_4C – 10 keV H^+.

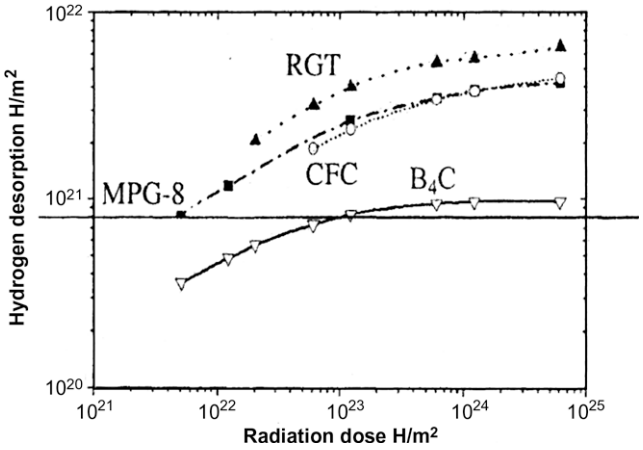

Fig. 2. Hydrogen capture in the graphites and B_4C.

depended on discharge time and achieved up to 40 μm. The coating was dense, without pores, with high hardness and good adhesion to the surfaces of samples. The high film deposition rate is apparently linked to the high degree of carborane ionization and dissociation caused by the ~40 eV PISCES-B plasma with high density, compared with electron temperature <1 eV plasmas of glow discharge. Therefore, it is of interest to investigate a boronization of the discharge chamber in tokamak plasma shots with higher values of density and electron temperature.

2. Experiments at tokamak T-11M

Boronization of the tokamak T-11M discharge chamber in plasma shots was carried out after experiments with a lithium limiter [15]. The placement of the lithium and graphite limiters, diagnostics, transport device with reference specimens and the batcher with carborane in the T-11M tokamak chamber is shown in Fig. 3. Non-toxic, non-explosive metacarborane ($C_2H_{12}B_{10}$) was used in boronization experiments. Carborane is a clear crystal substance at room temperature. Its molecular structure is a dodecahedron for the B/C atoms, with an H atom attached externally to each B/C band. Metacarborane has a low melting point ~265 °C, and readily sublimates at room temperature. Crystalline metacarborane was placed in a glass container connected to a high vacuum electromagnetic valve. The electromagnetic valve was connected to a diagnostic port of the T-11M tokamak discharge chamber through a vacuum gate valve. The temperature of the container (T = 20–150 °C) was regulated by a thermostatically controlled heater. The electromagnetic valve is opened synchronously with the tokamak toroidal field. Start time of its opening and closing could vary in a wide range. The plasma current was stabilized at 70 κA, toroidal field on axis was 11.5–12 kG. The deuterium and hydrogen mixture (H/D ratio 1:1) was used in plasma shots. Behavior of the lithium, boron emission, deuterium and hydrogen light lines was defined by an optical method. During the boronization experiments the lithium limiter was moved into "shadow" of the graphite limiter, which was located 20 cm from the plasma center.

3. Plasma – carborane interaction

Boronization in the T-11M tokamak was carried out after operation with the lithium limiter without preliminary induction heating and cleaning of chamber by a glow discharge. In shots before boronization, atomic lithium and other impurities came into the plasma due to ion sputtering from the chamber walls. In Fig. 4(a) a radiation spectrum of plasma before boronization is shown by red colour.[1] There is a bright line of Li ion in the plasma radiation spectrum. However, already after several shots with carboran, the Li line and impurities lines practically vanish from the plasma radiation spectrum (blue line in Fig. 4(a)), and a B ion line appears. In the plasma radiation spectrum there are also deuterium and hydrogen lines. During lithium experiments D:H ratio in peripheral plasma was ~1:1.3. During boronization D:H ratio in peripheral plasma changed up to ~1:4 (Fig. 4(a)). When the valve for carborane injection was closed, B emission vanished from the plasma radiation spectrum, at the same time lines of impurities have completely vanished, D:H ratio increased (Fig. 4(b) – blue line). The carborane injection valve has been opened normally at the most for 50 ms before the plasma shot start and the injection time could be varied in a wide range down to plasma shot disruption. In all there were about 50 shots with average duration ~150 ms and the total plasma operation time during these shots was ~8 s. Carborane injection at the valve opening was well detected by optical diagnostics on B line of the plasma radiation spectrum. Shot duration has increased from t_p = 150 ms up to 250 ms at the density of $n_e = 4.65 \times 10^{13}$ cm^{-3} and up to 350 ms at the density of $n_e = 1.3 \times 10^{13}$ cm^{-3}. Boronization results in to an essential decrease of the volt-second consumption rate (and, correspondingly, to an increase of shot duration). Thus, injection of carborane in the tokamak plasma shots has improved a stabilization of plasma filament, the impurities in wall area have been suppressed, high vacuum characteristics of the discharge chamber were stabilized.

4. Coating at boronization in plasma shots

As a result of boronization in plasma shots, a boron–carbon coating has been formed on the polished substrate from AISI 321 stainless steel. The coating images on the sample in an electron microscope is presented in Fig. 5(a), and in optical one in Fig. 5(b). The coating had a cellular structure, the size of cells in the center of a sample is less than at the periphery. IN order to measure the coating thickness, a coating layer was removed from the sample surface and the coating thickness was measured by an optical interferometer. In the center of the sample the coating thickness was ~2000 Å, and at the periphery ~1200 Å. Boronization in tokamak plasma shots occurred during of about 50 impulses with total exposure time ~8 s, thus a film deposition rate was ~25 nm/s, close to the rate of the boron film deposition in the PISCES-B experi-

[1] For interpretation of color in Fig. 4, the reader is referred to the web version of this article.

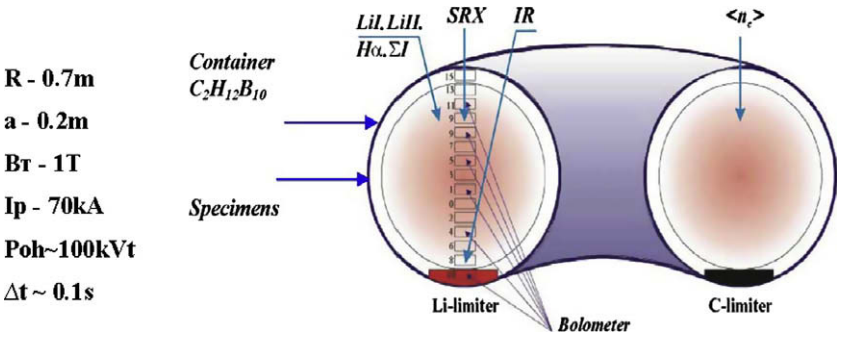

Fig. 3. Location of diagnostics, limiters and carborane container at the tokamak T-11M.

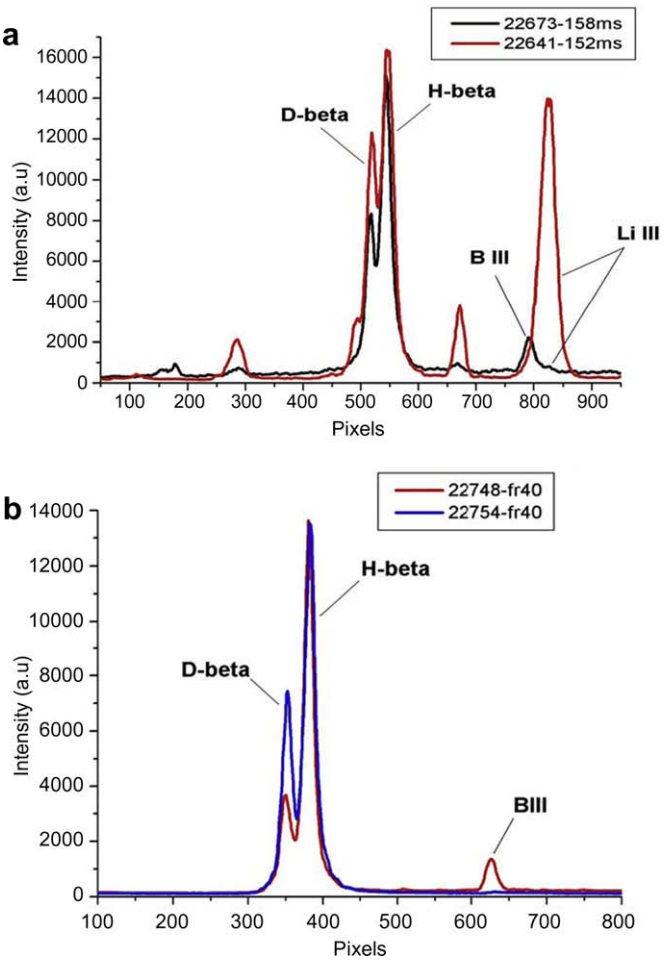

Fig. 4. Emission spectrum of the T-11M tokamak plasma: shot 22641 before boronization (with lithium limiter) and shot 22673 at the beginning of boronization (a); shot 22748 (at the end of boronization) and shot 22754 (valve for carborane injection was closed) (b).

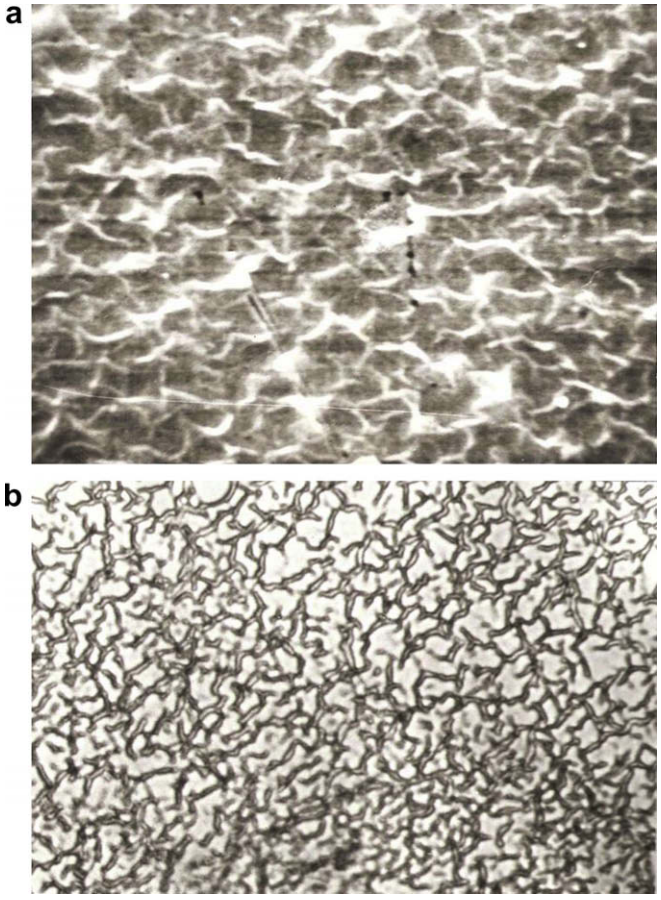

Fig. 5. Images of the boron–carbon film surface, SEM × 1400 (a) and optic microscope × 1500 (b).

ments. The film surface microhardness was measured by a microhardness meter PMT-3. The substrate microhardness was H10-250 and the microhardness of the formed boron containing film H10-600, indicating a good degree of crystallinity of the produced coating (the microhardness of the CVD B_4C films was H_{100}-1800).

5. Conclusions

Experiments on boronization in-situ in the tokamak T-11M plasma shots using metacarborane were carried out. As a result of boronization, the film with a microhardness H10-600 and the thickness up to 0.2 μm at deposition rate ~25 nm/s was produced. The impurities in wall areas were suppressed, high vacuum characteristics of the discharge chamber were stabilized, and stabilization of a plasma filament has improved. Plasma shot duration without disruption increased by more than twofold. At the density of $n_e = 1.3 \times 10^{13}$ cm^{-3}, $I_p = 70$ κA a shot duration was 350 ms and at the density of $n_e = 4.65 \times 10^{13}$ cm^{-3}, $I_p = 70$ κA was ~250 ms. High repeatability of experimental results has appeared. Proposed technique opens the possibility of practical production of renewable structured boron–carbon coating with use of plasma shots in large-scale tokamaks, such as DIII-D, JET, JT-60 UP, ITER, DEMO.

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