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Bulk-Boronized Graphites for Plasma-Facing Components in ITER

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ABSTRACT

Newly developed bulk-boronized graphites and boronized C-C composites with a total boron concentration ranging from 1 wt% to 30 wt% have been evaluated as plasma-facing component materials for the International Thermonuclear Experimental Reactor (ITER). Bulk-boronized graphites have been bombarded with high-flux deuterium plasmas at temperatures between 200 and 1600 °C. Plasma interaction induced erosion of bulk-boronized graphites is observed to be a factor of 2-3 smaller than that of pyrolytic graphite, in regimes of physical sputtering, chemical sputtering and radiation enhanced sublimation. Postbombardment thermal desorption spectroscopy indicates that bulk-boronized graphites enhance recombinative desorption of deuterium, which leads to a suppression of the formation of deuterocarbon due to chemical sputtering. The tritium inventory in graphite has been found to decrease by an order of magnitude due to 10 wt% bulk-boronization at temperatures above 1000°C. The critical heat flux to induce cracking for bulk-boronized graphites has been found to be essentially the same as that for non-boronized graphites. Also, 10 wt% bulk-boronization of graphite hinders air oxidation nearly completely at 800°C and reduces the steam oxidation rate by a factor of 2-3 at around 1100 and 1350°C.

Key words: PISCES, ITER, Boronized graphite, Erosion, Thermal desorption, Tritium inventory, Thermal shock, Oxidation.

1. INTRODUCTION

Graphites have been extensively used for plasma-facing components in major tokamaks including TFTR, DIII-D, JT-60, JET and TEXTOR. However, it is also true that in general graphite suffers from weaknesses such as high tritium inventory, chemical sputtering and radiation enhanced sublimation. In fact, graphite components in JET [1] and TEXTOR [2] have recently been modified with beryllium and boron coatings, respectively. Due to the strong oxygen gettering effects of these coatings, a significant improvement in the plasma confinement has been observed in these tokamaks [1,2]. Following this trend, similar boronization is presently applied in TFTR for improved plasma confinement [3].

Parallel to these experimental activities, a major effort has been made in designing ITER [4] over the last few years. Currently, graphite is selected as the plasma-facing material in the physics phase of ITER [4]. As demonstrated in existing tokamaks, it is possible to modify and improve surface characteristics with coatings. However, no such coatings can be expected to perform well as plasma-facing surfaces in ITER because of their incompatibility with high-flux and steady-state plasmas. It follows directly from these arguments that for the application in ITER, graphite should be bulk-modified to improve on its weaknesses.

In the present work, newly developed bulk-boronized graphite has been evaluated as a graphite-alternative material in several aspects related to critical issues pointed out in the design study of plasma-facing components for ITER.

2. EXPERIMENTAL

2.1. Test Materials

Materials tested in this work include: (1) isotropic graphites (POCO: AXF-5Q and Toyo Tanso: GB-100); (2) pyrolytic graphite (Pfizer and Union Carbide); (3) bulk-boronized graphites (Toyo Tanso: GB-101, GB-103, GB-110, GB-120 and GB-130 with a total boron concentration of 1 wt%, 3 wt%, 10 wt%, 20 wt% and 30 wt%, respectively). These bulk-boronized graphites have essentially the same matrix structure as that of the non-boronized graphite, GB-100.

In addition, a boronized carbon-carbon (C-C) composite (Toyo Tanso: CCB-407) with a total boron concentration of 7 wt% has been tested. This boronized C-C composite consists of chopped carbon fibers attached with a boronized resin.

2.2. Experimental Facilities

A variety of facilities have been utilized to evaluate the bulk-boronized graphites listed above. These facilities include: (1) the PISCES-B [5] and outgassing facilities at the University of California, Los Angeles (UCLA) for deuterium plasma bombardment and postbombardment thermal desorption spectroscopy, respectively; (2) the TPX-facility [6] at the Sandia National Laboratories, Livermore (SNLL) for tritium inventory measurements; (3) the EBTS-facility [7] at the Sandia National Laboratories, Albuquerque (SNLA) for electron beam induced thermal shock tests; and (4) the air and steam oxidation facilities [8,9] at Toyo Tanso Co. Ltd. and the Idaho National Engineering Laboratory (INEL), respectively. Details of these facilities can be found elsewhere.

3. RESULTS AND DISCUSSION

3.1. Critical Issues on Plasma-Facing Components for ITER

Critical issues in designing plasma-facing components for ITER are addressed here to discuss the possible application of bulk-boronized graphites. These critical issues are: (1) thermal stability at elevated temperatures; (2) neutron radiation damage; (3) plasma interaction induced materials erosion; (4) tritium inventory; (5) thermal shock induced cracking; and (6) air and steam oxidation due to accidental vacuum losses.

In the physics phase design of ITER, operational temperatures of plasma-facing components are: (1) up to 1600-1800°C for the radiation-cooled first wall armor; (2) up to 1000°C for the conduction-cooled first wall armor and the actively cooled divertor plate [4]. Because of these high operational temperatures, the thermal stability of materials must be investigated. According to the phase diagram of the carbon-boron system [10], bulk-boronized graphites used here consists of two phases: (1) the solid-solution phase; and (2) the boron carbide phase, presumably B_4C . This two-phase structure is stable from room temperature up to 2475°C, the melting point of B_4C .

Boron is well known to be an excellent absorber of thermal neutrons due to the reaction: $^{10}B(n, \alpha) ^7Li$. One must be aware that fast recoil α and Li particles generated in this reaction cause atomic displacements. However, the cross section for this reaction decreases by about 4 orders of magnitude towards the DT-fusion neutron energy range [11]. Therefore, as demonstrated in earlier data [12], the fast neutron damage will not be the fatal concern about bulk-boronized graphites. This is particularly true for the physics phase of ITER in which the neutron load is limited to 0.2 dpa [4]. To ensure this point, low-dpa reactor experiments are under way [13]. If necessary, boron can be enriched with

an isotope of ^{11}B , the natural abundance of which is 80%, for the production of boronized graphites for fusion applications.

Perhaps the most critical issue in the current design of ITER is the erosion of the divertor plate due to high-flux plasma bombardment. The divertor plate will be subject to plasma bombardment at ion fluxes from 10^{18} to 10^{19} ions $\text{cm}^{-2} \text{s}^{-1}$ at ion bombarding energies around a few hundred electron volts. Under these conditions, the estimated rate of recession of a divertor plate made of graphite can be as high as 27 cm/year even if an effect of redeposition is considered [14]. Obviously, new materials need to be developed.

From a safety point of view, it is desirable to reduce the tritium inventory in a reactor. The tritium retention in graphite generally decreases as the temperature increases. However, tritium can diffuse into grains of graphite rapidly at temperatures above 1000°C, which then leads to an increase in the total tritium uptake [15]. Unfortunately, plasma-facing components in ITER are designed to operate at these high temperatures.

Thermal shock effects have received increasing attention recently as most damage to graphite components in operating tokamaks is found to be due to high-heat fluxes associated with off-normal events such as plasma disruptions [16]. In ITER, a thermal energy of about 500 MJ will be released in the event of a thermal quench with a duration less than 1 ms [4]. Although it is difficult to simulate such off-normal events, high-power electron or ion beam bombardment allows us to conduct first-order materials screening.

In the plasma-surface interactions research community, little attention has been paid to safety concerns about accidental losses of vacuum. The reaction of graphite with air-moisture at high temperatures results in a generation of huge mass of hydrogen. Therefore, accidental vacuum losses can be followed by a spectacular explosion of hydrogen mixed with air. Also, hot graphite surfaces will react directly with air, leading to massive combustion.

3.2. Plasma Interaction Induced Erosion

In our earlier experiments [5], 3 wt% bulk-boronized graphite (GB-103) was bombarded with hydrogen plasmas in PISCES-B and showed an improved erosion resistance by 30-40%, relative to non-boronized isotropic graphites (GB-100 and POCO AXF-5Q). Here, bulk-boronized graphites with higher boron concentration and boronized C-C composites have been bombarded with deuterium plasmas at an ion bombarding flux of 2×10^{17} ions $\text{cm}^{-2} \text{s}^{-1}$, an electron temperature of 10 eV, an effective ion bombarding

energy of 250 eV and a magnetic field of 600 gauss. The ion bombarding flux was measured with a Langmuir probe positioned in front of the target.

The erosion yield was obtained by the weight loss method. As demonstrated in PISCES-A [17], the weight loss due to erosion is strongly affected by the degree of redeposition. The present plasma bombarding conditions were set in such a way that little redeposition occurs. In fact, the erosion factor [18] is evaluated to be around 90%, meaning the effect of redeposition is only 10% on the erosion yield. Therefore, the erosion yield data obtained here can be compared with those from ion beam studies although the ion flux dependence laws [19,20] must be considered, as will be seen later.

Shown in Fig.1-(a) are the erosion yield data in the temperature range from 200 to 1600°C. The surface temperature was measured with 2-color infrared pyrometers. Notice that boronized graphites show a reduced erosion by a factor of 2 to 3, relative to the basal plane of pyrolytic graphite (Pfizer), in regimes of physical sputtering, chemical sputtering and radiation enhanced sublimation (RES).

As to boronized C-C composites (see section 2.1), no improved erosion resistance was found. This indicates that boron must be uniformly distributed over the surface to be effective in reducing erosion. Also, there was no clear difference observed in the erosion yields measured for 10 wt%, 20 wt% and 30 wt% bulk-boronized graphites. This is presumably because once the boron concentration reaches the level corresponding to the two-phase region in the boron-carbon system, additional boron will be segregated as the boron carbide phase, the volumetric fraction of which is small compared with that for the solid solution phase. From these evidences, one can postulate that boron in the solid solution phase plays a crucial role in suppressing erosion of bulk-boronized graphites.

The chemical sputtering yield at temperatures from 200 to 850°C for pyrolytic graphite is apparently smaller (by about a factor of 2-3) than the corresponding data from ion beam experiments [21]. However, ion fluxes in beam facilities are typically about 2 orders of magnitude lower than those used in this work. Therefore, one must take into account the flux effect on the erosion yield. Using the flux dependence data [19], the yield at 10^{17} ions $\text{cm}^{-2} \text{s}^{-1}$ is expected to be about a factor of 2 smaller than that at 10^{15} ions $\text{cm}^{-2} \text{s}^{-1}$ at 527 °C. Clearly, this correction makes the present data fit more reasonably in the existing database.

At temperatures around 900 °C, one generally assumes physical sputtering to be dominant. Therefore, little difference was predicted for the erosion yields for bulk-boronized graphites and pyrolytic graphite. However, as shown in Fig. 1-(a), the data contradict this prediction. At present, it is not possible to explain this finding consistently. Similar unexplainable data have been obtained for boron carbide coat-mix carbon [22]. In

these data, the chemical sputtering yield of this material at 600°C is found to be smaller than the elemental physical sputtering yields of boron and carbon. These peculiar findings might be specific for boron-carbon compounds. Obviously, further investigation is needed.

In Fig. 1-(a), one clearly sees the effect of boron on RES erosion even at 1600°C which is the first trigger point to carbon blooms in TFTR [23]. However, earlier data on TEXTOR-type boronized amorphous carbon (a-C/B:H) films with a boron concentration of 33 % indicated no effect of boron at temperatures above 1500°C [24]. One suspects that this is due to the depletion of boron from the film. In the present work, postbombardment surface analysis showed no depletion of boron for bulk-boronized graphites after plasma exposure at 1600°C. Details of the surface analysis data will be reported separately [25].

From the Arrhenius plots (see Fig.2-(b)) of the erosion yield data at temperatures above 1000°C, the activation energies for RES are evaluated to be 1.1 eV and 1.4 eV for pyrolytic graphite and bulk-boronized graphites, respectively. The activation energy for pyrolytic graphite is about 40% higher than the earlier data of 0.78 eV [20]. Due to this large activation energy for the RES process, a steep temperature dependence of the erosion yield is seen in Fig. 2. This might be due to the high-flux plasma bombardment, yet details are still unknown.

In our previous work [5], it was hypothesized that boron might cause some chemical pinning effect to slow down the migration of carbon atoms associated with RES. The difference in the RES activation energies obtained here clearly indicates an effect of boron on the kinetics of carbon migration. More interestingly, the activation energy obtained here for bulk-boronized graphites is in excellent agreement with the interaction potential between boron and interstitial carbon of 1.45 eV [26]. This agreement strongly supports our hypothesis on the chemical pinning effect of boron. Also, these arguments are perfectly consistent with our postulation of the role of boron in the solid solution phase.

In earlier work [20], it was predicted that high-flux ion bombardment might result in a reduction of RES-erosion in the manner that $Y \propto \Gamma^{0.91}$ where Γ is the ion flux. This power law predicts that the RES-erosion yield at 10^{17} ions $\text{cm}^{-2} \text{s}^{-1}$ is about 34% smaller than that at 10^{16} ions $\text{cm}^{-2} \text{s}^{-1}$. Contrary to this prediction, the erosion yield data obtained here at 1500°C for pyrolytic graphite agree well with those at an ion flux of about 1×10^{16} ions $\text{cm}^{-2} \text{s}^{-1}$ [27]. Also, the data taken here at 1350°C were reproduced within 10% at fluxes of 10^{17} and 10^{18} ions $\text{cm}^{-2} \text{s}^{-1}$. From these evidences, one concludes that the flux dependence prediction will not hold for high-flux plasma bombardment conditions to be seen in the divertor region of ITER.

Shown in Fig.2 are the surface morphologies of graphites bombarded with deuterium plasmas at 1600°C. Surface grains of the plasma-bombarded GB-100 appears to be relieved as opposed to the virgin surface (not shown here). No strong surface modification is observed for GB-110 and GB-120. For pyrolytic graphite, one notices a characteristic pattern reminiscent of "Martian canals", the formation mechanism of which is still unclear.

3.3. Postbombardment Thermal Desorption

In this section, the thermal desorption from bulk-boronized graphites bombarded with deuterium plasmas in PISCES-B is described. The deuterium plasma bombardment was conducted in PISCES-B at an ion bombarding energy of 300 eV at temperatures between room temperature and 800°C. The total ion bombarding fluence is around 3×10^{20} ions cm^{-2} which well exceeds the minimum fluence to saturate the surface layer [28]. Thermal desorption spectroscopy measurements were carried out using the outgassing facility with a computer controlled infrared furnace and residual gas analyzer. The sample temperature was programmed to ramp from room temperature to 720°C at a rate of 20 degs/min.

Deuterium containing species detected during thermal desorption are HD, D₂, CD₄ and C₂D₄. Shown in Fig.3 are the thermal desorption spectra of deuterium (D₂) and deuteromethane (CD₄) from 30 wt% bulk-boronized graphite (GB-130) and POCO-graphite (AXF-5Q), both bombarded with a deuterium plasma at 320°C. Notice that the desorption of deuterium from boronized graphite is clearly enhanced, relative to that from POCO-graphite. The opposite is true for deuteromethane. A similar trend has been observed for HD and C₂D₄, the desorption spectra of which are not shown in Fig.3. Clearly, these observations are indicative of a catalytic effect of boron.

Importantly, for the same total deuterium retention (see section 3.4), the amount of deuterium consumed due to the enhanced desorption is found to agree well with that saved from the reduced desorption of deuterocarbons, equivalent to chemical sputtering [25]. Also, in recent studies metallic impurities such as iron on graphite have been found to enhance the desorption of preimplanted hydrogen and suppress chemical sputtering [29, 30]. From these evidences, one concludes that the enhanced recombinative desorption of deuterium is responsible for the reduced chemical sputtering observed for bulk-boronized graphite in the present work (see Fig.1-(a)).

3.4. Deuterium and Tritium Retention

The total deuterium uptake due to plasma bombardment can be estimated by time-integrating partial pressures of deuterium containing species during thermal desorption (see Fig. 3) in the manner that $Q = \int S p(t) dt$ where Q is the total throughput, S is the pumping speed and $p(t)$ is the time-dependent partial pressure. The total deuterium retention is shown in Fig.4-(a) as a function of surface temperature during plasma bombardment. One sees that the deuterium retention tends to maximize at around 300°C. This result contradicts earlier ion beam data taken at relatively high energies [31] but agrees well with recent measurements using thermal atomic hydrogen [32]. Pyrolytic graphite (Union Carbide) shows the smallest deuterium retention, indicating the effect of surface porosity.

Possible explanations for these findings are as follows. Low energy deuterium such as Frank-Condon neutrals is weakly bound to the surface and needs to be thermally activated to migrate deeper into the bulk via diffusion. However, above a certain temperature the thermal desorption becomes predominant, which then decreases deuterium retention. Also, such weakly bound deuterium atoms can be liberated upon the exposure to air [33], which also reduces deuterium retention. This air-exposure effect is particularly important for the plasma exposure at room temperature for which the depth of migration of deuterium is relatively shallow.

As mentioned earlier, the volumetric dissolution of tritium in grains of graphite becomes important in determining the total uptake at elevated temperatures. For tritium inventory measurements, 3 wt% and 10 wt% bulk-boronized graphites and non-boronized graphite (GB-100) have been exposed to deuterium+tritium plasmas (99% D and 1% T) at an ion bombarding energy of 100 eV at temperatures above 800°C, each for 1 hour in the TPX-facility at SNLL [6]. Plasma-bombarded samples were first dissolved in a chromosulphuric acid solution at 160°C, then the tritium activity was measured with a liquid scintillation counter.

Shown in Fig.4-(b) are the resultant tritium retention data as a function of temperature. Notice at temperatures above 1100°C, the tritium retention in bulk-boronized graphites is considerably lower than that in non-boronized graphite. In fact, the tritium retention in bulk-boronized graphites at these high temperatures is close to that in pyrolytic graphite [15]. Generally, tritium tends to be trapped by crystalline defects with unsaturated bonds if graphite is not well graphitized [15]. One possible explanation for the reduced retention of tritium is that boron dissolved in the lattice (i.e. the solid-solution phase) might preoccupy these trapping sites or deactivate unsaturated bonds. Also, the dissolution of

tritium into grains might be hindered due to the boron catalysis described in section 3.3. Nevertheless, further investigation is necessary.

3.5. Thermal Shock Induced Cracking

Because of the brittle nature of boron carbide, one might be concerned about a deterioration effect of bulk-boronization on thermomechanical properties of graphite. To investigate this point, thermal shock tests were conducted at SNLA using the EBTS-facility [7]. Used in these tests are 3 wt% and 10 wt% bulk-boronized graphite and isotropic graphites (POCO AXF-5Q and Toyo Tanso GB-100). These samples are subject to the standard treatment [34] prior to electron beam bombardment. The electron beam bombardment was conducted for 1.5 s and the power was increased in increments of 1 kW/cm² until the target was seen to crack.

The critical heat flux has been found to be 5 to 6 kW/cm² for all the materials tested. This means that bulk-boronization does not affect the thermal shock resistance of the base graphite material. The maximum surface temperature during electron beam bombardment was measured to be around 3000°C, yet postbombardment surface analysis indicated no sign of the depletion of boron from bulk-boronized graphite.

3.6. High Temperature Air and Steam Oxidation

The oxidation of boronized graphites in air and steam at an atmospheric pressure is described in this section. Using the Toyo Tanso facility [8], the air oxidation reaction rates were measured at 800°C. At INEL[9], the steam oxidation reaction experiments were conducted at about 1100 and 1350°C.

The results are shown in Figs. 5-(a) and (b). Notice that in Fig. 5-(a) the air-oxidation reaction appears to be inhibited completely for 10 wt% and 30 wt% bulk-boronized graphites. These surprising data were not only measured with weight loss but were also confirmed with gas analysis of reaction products such as CO. More detail about the air-oxidation behavior of bulk-boronized graphites will be reported separately [35]. As shown in Fig. 5-(b), bulk-boronized graphites (particularly GB-110) clearly exhibit suppressed reaction with steam, relative non-boronized graphite (GE-100). Although steam oxidation experiments have been done only at a limited number of temperatures, the data obtained so far are seen to follow a similar trend to that for GraphNOL.

The improved oxidation resistance in air is partly due to the formation of a passive protective surface layer of boron oxide (B₂O₃) [36]. Obviously, this protective layer

argument does not completely explain the present data because the melting point of boron oxide is 450°C. One speculates that the molten oxide layer may be effective in reducing the oxidation rate until the temperature approaches its boiling point of 2250°C. Nevertheless, if bulk-boronized graphites are used in present-day fusion devices, the formation of surface oxides will be important in reducing the contamination due to oxygen.

On the other hand, boron is known to enhance graphitization during the final baking process [37]. The oxidation rate is generally expected to be lower for better graphitized structures. Consistent with this, in Fig. 5-(b) pyrolytic graphite shows appreciably lower oxidation rates than other graphites. Therefore, the enhanced graphitization in bulk-boronized graphites is considered to be the key to the improvement of oxidation resistance.

4. CONCLUSION

As a possible plasma-facing material for ITER, bulk-boronized graphites have been proposed and evaluated systematically in the present work. Results demonstrate that bulk-boronization noticeably improves high temperature characteristics of graphite in critical aspects related to the current design of ITER. These aspects include: (1) plasma interaction induced erosion; (2) tritium inventory; and (3) air oxidation resistance. However, bulk-boronization generally reduces the thermal conductivity of graphites. Perhaps C-C composites with boronized fibers need to be developed for more improved performance.

As to the applicability in existing short-pulse fusion devices, one can take advantage of the strong affinity of bulk-boronized graphites to oxygen-containing gases such as H₂O and O₂. Due to this chemical affinity, a strong oxygen gettering effect is expected similarly to boronized films in TEXTOR [2]. It is, therefore, highly possible that if bulk-boronized graphites are used for surface components, the plasma confinement performance will be improved due to the oxygen gettering effect and the reduced plasma erosion. In fact, 3 wt% and 10 wt% bulk-boronized graphites (GB-103 and GB-110) are presently planned to be used for the mushroom limiter in Tokamak de Varennes [38].

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FIGURE CAPTIONS

- Fig. 1 (a) Plasma interaction induced erosion of non-boronized and boronized graphites under identical deuterium plasma bombardment conditions in the temperature range from 200 to 1600°C. Notice that the gap between the erosion curves remains nearly constant, meaning that the ratio of the erosion yields remains constant. (b) Arrhenius plots of the erosion yields at temperatures above 1000°C.
- Fig. 2 Surface morphologies of plasma-bombarded graphites: (a) non-boronized graphite (GB-100); (b) 10 wt% bulk-boronized graphite (GB-110); (c) 20 wt% bulk-boronized graphite (GB-120); and (d) pyrolytic graphite (Pfizer). The deuterium plasma bombardment was done at an ion energy of 250 eV at a surface temperature of 1600 °C. The total ion fluence is about 5×10^{20} ions cm^{-2} .
- Fig. 3 Thermal desorption from plasma-bombarded graphites. Notice that the desorption of deuterium is clearly enhanced for bulk-boronized graphite (GB-130), relative to non-boronized graphite (POCO AXF-5Q). The total deuterium retention in these two materials is found to be the same (see Fig. 4-(a)). Therefore, the enhanced deuterium desorption observed for bulk-boronized graphite is not due to a larger deuterium retention.
- Fig. 4 (a) The deuterium retention in plasma-bombarded graphites. The data were obtained from similar thermal desorption spectroscopy to that shown in Fig. 3. Obviously, the deuterium retention depend on the surface temperature during plasma bombardment. Interestingly, the data indicate that the deuterium retention maximizes at around 300°C. (b) The tritium retention data at high temperatures above 800°C. Notice that bulk-boronization can considerably reduce the tritium retention in graphite.
- Fig. 5 Oxidation of various graphites:
(a) air-oxidation at 800°C. Surprisingly, the data indicate that the reaction is completely inhibited for 10wt% and 30wt% bulk-boronized graphites.
(b) steam-oxidation at temperatures between 1100 and 1600°C. Notice that the reaction rate decreases as the boron concentration increases to 10 wt%.

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Fig. 1

(a) Non-boronized graphite (GIB-100)

(b) 10 wt% boronized graphite (GIB-110)

(c) 20 wt% boronized graphite (GIB-120)

(d) Pyrolytic graphite

Fig. 3

Fig. 4

Fig. 5