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BORON CARBIDE-BASED COATINGS ON GRAPHITE FOR PLASMA FACING COMPONENTS

by

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ABSTRACT

In the effort to evaluate boron-rich coatings as plasma facing surfaces in fusion devices, a new process for applying boron carbide (B_4C) coatings to graphite was developed. The process entails eutectic melting of the carbon (C) substrate surface with a precursor layer of B_4C particles. Adherent coatings were achieved which consisted of two layers: a surface layer and a graded penetration zone in the outer portion of the substrate. The surface-layer microstructure was multiphase and ranged from reaction-sintered structures of sintered B_4C particles in an eutectic-formed matrix to that of hypereutectic carbon particles in a B_4C -C eutectic matrix. Because of high surface energy, the coating generally developed a non-uniform thickness. Quantitative evaluations of the coating were performed with limiters in the TEXTOR fusion device and with coupons in electron beam tests. Test results revealed the following: good adherence of the coating even after remelting; and, during remelting, diagnostics detected a corresponding interaction of boron with the plasma.

1. INTRODUCTION

Methods of applying boron carbide (B_4C) coatings on high thermal conductivity carbon-based materials are of special interest for use on plasma-facing components (PFC's) in present and near-future fusion devices to provide a low-Z, stable refractory surface of high boron content. Preferred are surfaces which release predominantly boron which will aid the control and performance of a confined plasma, as described by Winter [1] and Jackson [2]. Studies by Linke [3], Ando [4], and Bolt [5] have introduced, evaluated, and examined the behavior of many types of boron- and B_4C -based coatings on carbon-based substrates to determine applicability in tokamak devices. Methods to develop a B_4C -based surface have included: chemical vapor deposition (CVD), chemical vapor reaction (CVR) conversion, bulk boronization, low pressure plasma spray (LPPS), and composites. The deposition of thinner coatings of boron (e.g., 100 nm) by plasma-assisted CVD processes is at present a frequent in situ operating procedure for some tokamak devices.

The objectives of this study were (1) to develop processing parameters for applying B_4C -based coatings to graphite, by the reaction-sintering coating method, a high temperature method not explored previously for PFC applications; and (2) to characterize and evaluate the resulting coatings through microstructural examinations and energy deposition evaluation tests.

The reaction-sintering coating fabrication method was selected because previous efforts by Valentine *et al.* [6] demonstrated that reaction-sintered coatings containing metal borides and carbides are very adherent to carbon materials and have excellent high temperature and rapid heating performance characteristics. Reaction-sintered coatings can be applied to graphite and carbon-carbon (C-C) composites using a brush-on technique which makes use of powdered boride and carbide refractory compounds (in the present study, B_4C) and an organic binder. The reaction-sintering process is a combination both of (a) substrate carbon (and binder carbon) reacting with the refractory compounds, and of (b) sintering of the boride and/or carbide particles. The coating microstructure consists of carbide or boride particles surrounded by lower melting point eutectic material. Depending upon the material system, the eutectic may be a binary or ternary eutectic. Processing conditions can be varied to influence the coating microstructure and composition with respect to the amount of carbon.

Furthermore, the opportunity to develop and control multiphase coating microstructures offers an attractive means for developing unique coating designs for PFC's.

2. MATERIAL AND EXPERIMENTAL PROCEDURES

MATERIAL

Two-phase coatings comprised of B₄C and graphite [*i.e.*, carbon (C)] were applied to graphite substrates using various processing parameters. The precursor coating mixtures consisted of B₄C powder, ethyl alcohol, and an organic binder (hydroxypropyl-cellulose). Two B₄C precursor coating mixtures were investigated: (1) a powder with particles $\leq 45\text{ }\mu\text{m}$ (typical average particle size was $\leq 20\text{ }\mu\text{m}$), and (2) a powder with particles of $\leq 45\text{ }\mu\text{m}$ and $\leq 250\text{ }\mu\text{m}$ (CERAC, Inc., Milwaukee, WI, USA). Initial coating trials used Grade AXF-5Q1 graphite (Poco Graphite, Inc., Decatur, TX, USA) and Grade ATJ graphite (Union Carbide Corp., Cleveland, OH, USA). Then coated limiters and electron beam test coupons were fabricated using Grade EK 98 graphite, an isotropic fine grain graphite (Ringsdorfwerke, Bonn, Germany). Test coupons evaluated were $2.5 \times 2.5 \times 1.0\text{ cm}$ and $5.0 \times 5.0 \times 1.5\text{ cm}$. Three limiters with complex curvature geometries (nominally $10.0 \times 6.0 \times 5.0\text{ cm}$) were coated.

EXPERIMENTAL PROCEDURES

Coating Fabrication. Although many coating process parameter variations were examined, the basic coating fabrication method was relatively straightforward, consisting of the following five steps: (1) the substrate was given a specific surface finish, typically by abrasive grinding with 240 grit silicon carbide metallographic grinding paper; (2) the substrate material was ultrasonically cleaned in ethyl alcohol; (3) the substrate material was heat treated in vacuum (at $\leq 1.3 \times 10^{-3}\text{ Pa}$) at 1000°C for 1 hr. to remove volatiles and high vapor pressure impurities; (4) a precursor coating was applied by a brush-on technique to the substrate material and cured at 125°C for at least 0.25 h; and (5) the precursor-coated substrate was heat-treated at the coating formation temperature (greater than 2375°C) for at least 15 s, and up to 210 s, using a 3 kHz induction furnace with rapid heating and cooling capabilities. Because of the formation of very thick, localized, coating regions or nodules (through liquid phase coating contraction), four additional processing steps were performed with the limiters: (a) large nodule regions were ground, with a rotating tool which contained diamond particles, to achieve a thickness of approximately 1 mm, (b) secondary grinding of the nodules was performed using abrasive paper (SiC particles), (c) each tile was ultrasonically cleaned in ethyl alcohol, and

(d) the tiles were further cleaned by undergoing a 1-hr. 1000°C vacuum (at $\leq 1.3 \times 10^{-3}$ Pa) heat treatment.

Coating Fabrication Process Parameters. Fabrication of B₄C-based coatings, by reaction-sintering, proved to be quite complex. Coatings of multiple thicknesses and microstructures were developed (see Fig. 1). A narrow processing temperature range exists because of the small difference between the melting point of B₄C (2450°C) and the eutectic point for B₄C + C (2375°C). The moderate surface tension of the liquid, formed during processing, favored partial coating dewetting, after reaction, and the formation of very thick nodules (of $\leq 45^\circ$ contact angle). Regions experiencing partial coating dewetting retained a penetration/conversion coating on the substrate surface; thickness of this conversion coating was approximately 25 μm . As carbon diffused into the coating material from the substrate substantial changes occurred in the coating composition and its associated microstructure. Therefore, a number of process parameters were investigated, with the aim always of applying adherent, uniform-thickness, B₄C + C coatings to the EK 98 graphite test coupons and limiters.

The primary coating fabrication process parameters investigated are presented below. Included is information on the range or variation in the parameters used for the test coupons and limiters.

1. B₄C particle size in precursor coating: $\leq 45\text{-}\mu\text{m}$ powder (typical average particle size was $\leq 20\text{ }\mu\text{m}$) was used for the limiters; for the other test coupons, $\leq 250\text{-}\mu\text{m}$ powder was also used;
2. Amount of precursor coating material applied: ranged from 1×10^{-4} to 8×10^{-4} gm/mm² in the investigation, and 1×10^{-4} to 4×10^{-4} gm/mm² on the tested specimens and limiter;
3. Time at maximum temperature: ranged from 15 to 120 s;
4. Maximum temperature: ranged from slightly above the B₄C-C eutectic temperature (2375°C) to slightly less than the B₄C melting point (2450°C);

L-015(5)
9-20-93

Fig. 1. Range of coating microstructures achieved using a precursor coating consisting of B_4C powder particles and an organic binder. Secondary-electron SEM images of cross-sectioned specimens are shown. Coating (a) was formed primarily through sintering of B_4C particles. Coatings (b), (c), and (d) are the result of eutectic reaction. Coating (b) has a hypoeutectic composition; coating (c) has a hypereutectic composition. Specimens with coated regions of types (b) and (c) also had large regions with thin coatings of type (d), which result from B_4C penetration/conversion of the graphite substrate.

5. Surface finish (or texture) prior to application of the precursor coating material: four surface finishes were investigated (as-machined, 240-grit ground, 60-grit ground, and grit blasted with fine-size grit); no significant differences were observed;
6. Surface orientation (vertical vs. horizontal): test coupons were coated with their coated surface oriented horizontally during coating fabrication because gravity was found to influence the flow behavior of the liquid phase at the higher temperature; the limiter surfaces coated included both horizontal and vertical surfaces;

7. Pre- and post-reaction-sintering holds at elevated temperatures: none for the test coupons; a 5.0-min. hold between 2050°C and 2150°C was used with the coated limiters to improve coating adherence.

The resulting coatings completely covered the desired portions of the test specimens, but a substantial variation in the coating thickness occurred over the surfaces of each specimen. For each specimen, approximately 40 %–70% of the coated surface(s) had a coating of 25–50 μm thickness; the remaining coated surface area consisted of discrete nodules or thick coating regions, some of which were several millimeters in thickness. In all of the specimens coated, over a range of processing conditions, no coating spallation was observed. Some through-the-thickness microcracking was observed in the coating.

Microstructural Characterization Methods. Examinations were conducted of both the surfaces and cross-sections of the coated graphite specimens. Scanning electron microscopes (SEM's), with energy dispersive X-ray analysis (EDX) capabilities, and reflected-light microscopes were employed. Coated specimens were examined both in the as-coated condition and after undergoing energy deposition tests.

Energy Deposition Tests. The coating evaluation tests conducted included the following: (1) disruption simulation experiments, to assess erosion, in the JUDITH (Jülich Divertor Test Facility in Hot Cells) electron beam facility at KFA, (2) plasma-heating simulation experiments in the same electron beam teststand (Bolt [5]), and (3) plasma plus neutral beam injection heating of a coated limiter in the TEXTOR (Tokamak Experiment for Technology Oriented Research) tokamak fusion device at KFA (Bolt [5], Philipps [7]).

The disruption simulation experiments were conducted at pulse durations of 2 ms and with energy densities of the incident electron beam of up to approximately 8 MJm^{-2} . For comparison purposes, tests were also conducted with uncoated EK 98 graphite and with a LPPS B_4C on graphite. Test parameters were as follows: (1) 3×3 mm (9 mm^2) irradiated area; (2) a rastered beam of 50 kHz in the x-direction and 5 kHz in the y-direction; (3) an acceleration voltage of 140 kV (penetration depth of 130 μm); and, (4) the beam current was increased in 20 mA steps, and the zone of beam deposition on the test specimen was changed from shot to shot, until significant degradation was observed.

The plasma-heating simulation experiments were conducted, using electron beam pulses of several seconds duration at reduced power, both to assess the performance of the coatings

under heating conditions similar to those in TEXTOR, and to enable a comparison with the thermal response of the test limiter. The test parameters used included the following: (1) the irradiated area was 20×20 mm (400 mm^2) on the specimen [no. GA-07 ($24 \times 24 \times 10$ mm)] that was evaluated, (2) the electron beam was rastered, (3) the electron beam voltage was 140 kV, while beam currents of 20, 24, 28, 32, 36, 40, 45, 50 mA were employed, and (4) the heating profile used consisted of a linear ramp-up for 0.5 s, a hold for 3 s, and a linear ramp-down for 0.5 s. The surface temperature vs. time behavior was compared for both the coated specimen and for an uncoated EK 98 graphite specimen, at incident power densities of 12 MWm^{-2} (40 mA beam current) and 15 MWm^{-2} (50 mA beam current).

A coated limiter was exposed to multiple plasma discharges of increasing power with added heating from a neutral beam in TEXTOR. Description of the limiter attachment positioning, operation condition, and diagnostic measurements are presented in a companion paper by Bolt [5]. The last discharge (no. 54421) deposited the highest heat flux, a calculated value of 26 MWm^{-2} .

3. RESULTS AND DISCUSSION

COATING MICROSTRUCTURAL CHARACTERIZATION: AS-COATED CONDITION

A range of coating microstructures were obtained; the microstructure type formed depended primarily on the maximum temperature and on the length of time at temperature. These two variables determined the kinetics and diffusion rates associated with formation of the coating. Once eutectic material formed at the processing temperature, the amount of carbon ingress in the coating increased rapidly, often achieving concentrations greater than that of the carbon content at the B_4C -C eutectic (31 wt.-% C). Thus, the coating microstructures ranged from: $B_4C + C$ eutectic plus sintered B_4C particles, to $B_4C + C$ eutectic plus B_4C phase, to $B_4C + C$ eutectic plus C phase. Scanning electron micrographs of these microstructures are shown in Fig. 1.

A very thin ($<1\text{ }\mu\text{m}$), noncontinuous carbon surface film (white areas) developed on the reaction-sintered surface [Fig. 2(a)]; at higher temperatures and longer processing times, a more substantial carbon layer ($10\text{--}20\text{ }\mu\text{m}$) completely covered the B_4C -based coatings [Fig. 2(c)]. The thin dewetted coating regions exhibited a $25\text{--}50\text{ }\mu\text{m}$ thick B_4C penetration/conversion layer on the surface of the graphite substrates [Fig. 2(d)].

The planned or targeted coating microstructure was a mixture of sintered B_4C particles surrounded by a matrix of $B_4C + C$ material formed by the eutectic reaction. In fact, the coating microstructures achieved ranged from a sintered structure with some consolidation [Fig. 1(a)], to a structure formed from solid B_4C particles and liquid B_4C -C eutectic material [Fig. 1(b)], to a structure formed from hypereutectic carbon particles and liquid B_4C -C eutectic material [Fig. 1(c)]. In general, most of the coatings fabricated had the third type of coating microstructure, consisting of hypereutectic carbon in a "matrix" of fine eutectic B_4C and C grains. Also, because of the large amount of liquid phase present during processing, and because of the moderate surface energy of the B_4C , most of the resulting coatings developed both very thick regions and very thin regions [Fig. 1(b-d); Fig. 3]. The thin coating regions ($25\text{--}50\text{ }\mu\text{m}$) consist of the outer portion of the graphite substrate which has been partially converted to B_4C through the inward diffusion of B and the penetration of liquid B_4C . The

L-015(6)
9-20-93

Fig. 2. Range of coating surface morphologies achieved using a precursor coating consisting of B_4C powder particles and an organic binder. Secondary-electron SEM images of specimen surfaces are shown. Surface (a) is typical of a reaction-sintered coating with a noncontinuous carbon surface film; the energy dispersive X-ray spectrum for surface (a) is shown in (b); surfaces (c) and (d) are typical of coatings formed through eutectic reaction. A thin, continuous carbon outer layer is shown in (c); B_4C penetration of the graphite substrate is shown in (d).

thick coating regions (150–1,500 μm) consist of three layers: the inner-most layer is the same penetration/ conversion layer also observed in the thin coating regions; the middle layer is the primary surface layer which consists of B_4C and C; the outer-most layer is a very thin layer of C [the result of the decomposition of B_4C (and the loss of the B, with the C remaining) and/or the redeposition of vaporized C (without the deposition of B)]. From an examination of coated surfaces, in conjunction with the associated coating microstructures, it was determined that the carbon layer observed on thick coating regions forms very rapidly. Only coating regions which have a sintered/reaction-sintered structure of limited eutectic melting did not exhibit a

Fig. 3 Variable, nonuniform topography coatings (typical) resulting from eutectic melting of boron carbide and carbon on EK 98 graphite surfaces: (a) a test coupon used for the JUDITH electron beam disruption simulation tests; (b) the limiter tile used for the coating evaluation tests in TEXTOR. Both (a) and (b) show pre-test surfaces.

carbon outer layer (Fig. 2). The surface carbon layers examined in cross-sections ranged from less than a few microns thick to 20- μm thick. The latter was observed on coated specimens which had been held at the higher process temperatures for the extended times of 210 s.

All of the coatings fabricated were found to be very adherent to the three different graphite materials, which covered a range of coefficients of thermal expansion (CTE's) from 3×10^{-6} to $8 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$. No evidence of crack or void formation in the penetration/conversion layer was observed for any of the graphites. Although CTE-induced cracks were observed in the coatings on the EK 98 graphite ($\text{CTE} = 3 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$), the coatings remain adherent because the cracks are through-the-thickness as opposed to interface cracks (spalling); also, because the substrate has the lower CTE (compared to the coating material), the crack surfaces appear closed.

ENERGY DEPOSITION EVALUATION TESTS

Disruption Simulation in JUDITH Electron Beam Teststand. Two B_4C -coated specimens with significantly different microstructures and carbon concentrations were tested in the JUDITH facility. One of the specimens was B-rich, with respect to the eutectic composition, and exhibited a sintered/reaction-sintered microstructure (Fig. 1). The other specimen

was C-rich, with respect to the eutectic composition, and exhibited a hypereutectic-carbon-particle/eutectic microstructure. Under the severest heat load conditions ($\geq 6.2 \text{ MWm}^{-2}$), the performance of the B₄C-C coatings was quite good and more resistant to erosion and melting when compared with uncoated fine grain graphite and with plasma-sprayed B₄C coatings heated less severely (Table 1 and Fig. 4). Furthermore, the melted coating resolidified without spallation. This finding is comparable with disruption simulation tests, at up to 5.5 MJm^{-2} using electron beam heating, reported by Ando [4] for graphite and C-C substrates coated with B₄C by a CVR method.

TABLE 1
COMPARISON OF JUDITH ELECTRON BEAM TEST RESULTS

Material	Lowest I_{beam} Resulting in Material Degradation (mA)	Incident Energy Deposition Over a Period of 2 ms (MJm^{-2})	Material Degradation Observed By SEM Examination
C-Rich ¹ B ₄ C-C Melted-Eutectic Coating	280 ²	8.7	Portions of C surface film spall. Melting and resolidification of B ₄ C-C occurs; "droplets" form.
B-Rich ¹ B ₄ C-C Reaction-Sintered Coating	> 200 ³	6.2	No significant damage was observed (NOTE: maximum I_{beam} used = 200 mA).
Uncoated EK 98 Graphite	140 ⁴	4.3	An erosion crater forms. Microcracks form.
Low Pressure Plasma-Sprayed B ₄ C Coating	80	2.5	An erosion crater forms. Microcracks form (NOTE: cracks occur at $I_{\text{beam}} \geq 60 \text{ mA}$).
¹ Refers to the side of the B ₄ C-C eutectic composition on which a specific material lies. ² At lower beam currents (ex.: 200, 220, 240 mA), some limited spallation of the outer thin carbon surface film was observed. ³ For this material, at the maximum I_{beam} (200 mA), significant damage did not occur. ⁴ Although significant erosion does not occur for $I_{\text{beam}} < 140 \text{ mA}$, when $I_{\text{beam}} \geq 100 \text{ mA}$ a substantial loss of the binder phase in the graphite occurs (observed by SEM), along with an associated weakening of the effected graphite material. I_{beam} : Electron beam current.			

In Table 1, four materials are examined, two from the present coating development effort, an uncoated graphite sample, and a plasma-sprayed B₄C coating (Bolt [5]). Plots of I_{abs} versus time indicated that when significant drop-off in the value of I_{abs} was observed after only 1 ms, erosion and/or emission of thermal electrons or charged particles had commenced. The B₄C-C reaction-sintered coatings showed the best high heat flux performance in these disruption simulations. Even at beam currents of 200 mA, which correspond to incident energy densities of 6.2 MJm^{-2} , no significant coating degradation was observed in the SEM

Fig. 4. Comparison of surface features after disruption simulation tests using 2-ms pulse electron beam. Backscattered-electron [(a), (b), and (d)] and secondary-electron [(c)] SEM images of specimen surfaces are shown. Carbon-rich hypereutectic B₄C-C coating (a) shows evidence of exfoliation of ~10 μ m carbon outer layer of the B₄C-C coating; boron-rich hypoeutectic B₄C-C coating (b) shows no evidence of degradation; uncoated EK 98 graphite (c) shows evidence of near-surface erosion; a low pressure plasma-sprayed B₄C coating (d) shows evidence of micro-cracking and formation of a melt crater.

examinations. Under such conditions, severe erosion of the uncoated graphite did occur; in plasma-sprayed B₄C, melting, which occurred at a relatively low energy density, caused most of the damage. However, due to the relatively large penetration depth (approximately 130 μ m) of 140 keV electrons in low atomic number materials, volumetric heating effects must also be considered in PFC design. For example, in coatings with thicknesses of less than 100 μ m, such as the thin 25–50 μ m coating regions on the EK 98 graphite, part of the incident energy deposited would be absorbed by the underlying graphite.

Plasma Heating Simulation in JUDITH Electron Beam Teststand. During the heating of the B₄C-coated test specimen to 12 MWm⁻², melting of the coating commenced at

the end of the 3-s flat top hold condition. Heating at the 15 MWm^{-2} level caused melting of the coating after 1.8 s. During the remaining 1.2 s hold time, the gradient of temperature rise changed slightly and was similar to the rate for the uncoated graphite. After exposure, the beam-heated zone of the B_4C coating material was characterized as a flat droplet covering about half of the area; the other half was covered by a thinner coating; this topography is similar to the pre-test coating surface. The thermal conductivity would appear to be higher than the $4 \text{ Wm}^{-1}\text{K}^{-1}$ value reported for a LPPS B_4C coating by Bolt [5]. Similar evaluation of these B_4C coating designs using the same electron beam method is discussed in the Bolt reference.

TEXTOR Plasma Exposure Tests of a Coated Limiter. The B_4C coated limiter (GA-31) was exposed to 15 plasma discharges. A description of the final discharges (ending with TEXTOR shot no. 54421) were reported and discussed by Bolt [5]. Surface heating rate up to eutectic melting was comparable to the electron beam heating test. At the onset of melting the limiter surface temperature remained near constant in contrast to the continued rise observed for the electron beam tests. An explanation for this is still under study. Diagnostic spectroscopy revealed no detection of species other than boron and carbon emanating from the melted coating. Post-exposure microexamination of the coated surface indicated adherence of the coating of both the 1 mm thick nodules and the less than $50 \mu\text{m}$ thick coating regions prevailed following coating resolidification. The microstructure observed, typical of the carbon-rich $\text{B}_4\text{C} + \text{C}$ eutectic composition presented earlier in Fig. 1, was characteristic for the thick, nodular zones of coating.

4. CONCLUSIONS

A new process for applying boron carbide (B_4C) coatings to graphite was developed and evaluated on test specimens by electron beam heating and by exposure of a limiter tile in the TEXTOR tokamak. B_4C -based coatings can be produced by methods of reaction sintering and by eutectic melting of a B_4C particle precursor layer. However, eutectic melting occurs rapidly and results in a non-uniformity in coating thickness and topography for thick coatings and appears not controllable with variation in temperature and time of the process thermal cycle step. A recommendation would be to establish a greater temperature range of partial melting by addition of other borides and/or carbides into precursor mixture. At present new research and development is in progress with a $B_4C + SiC$ mixture precursor.

The B_4C -based coatings exhibited eutectic melting near $2400^\circ C$ under rapid heating in beams or plasma. Adherence of coating was maintained, with a reduced release of carbon at high temperature. Therefore, the coating method is attractive for: PFC's in near-future fusion devices, where a stable refractory surface of high boron-content is specified; and for developing multiphase microstructure and multi-component compositions for coatings on substrates of graphite and carbon-carbon composite materials. Therefore, it would appear timely to investigate the reaction of precursor coatings on substrates by a variety of external heating methods (i.e., laser beam heating; or in situ plasma/NBI heating to recoat local tile regions) to provide new design options for PFC's in tokamaks.

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