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Carborane Films: Applications to First-Wall Problems in Tokamaks*

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ABSTRACT

RF plasma-assisted CVD and sputter deposition of amorphous boron-carbon layers similar to those being used in the TFTR tokamak at the Princeton Plasma Physics Laboratory have been performed. The initial stoichiometry has been determined using Rutherford backscattering spectrometry and elastic recoil detection. Films have also been implanted with deuterium in order to determine H-isotope pumping capacity. These studies, in addition to characterizations made of layers collected on probes in TFTR, have been used to optimize the boronization parameters and to better understand the effects of boronization on TFTR.

INRODUCTION

In-situ plasma-assisted chemical vapor deposition (PCVD) of thin hydrogenated carborane (a-BC:H) films[1-4] is being combined with He/C conditioning discharges and He glow-discharge cleaning (GDC) to modify the near surface of plasma-facing components on the TFTR Tokamak[5,6]. This is being done to improve plasma performance to levels required for DT-fusion breakeven.

In this paper we outline the properties of both PCVD and sputter deposited thin amorphous hydrogenated carborane films (a-BC:H) with emphasis on their impact on plasma performance in TFTR. Laboratory measurements of the as-deposited composition and measurements of the capacity of these materials to pump and trap H-isotopes are correlated with the use of similar films in TFTR.

LABORATORY SIMULATIONS OF BORONIZATION

1. Deposition of a-BC:H films

a-BC:H films were deposited on Si substrates using two different techniques: PCVD and Ar sputtering (SPD) of boron carbide/carbon targets. All films were deposited at or near (T<40°C) room temperature.

The PCVD deposition conditions were chosen to emulate as closely as possible those originally used in TFTR--Gas: 10% B_2H_6, 10% CH_4, 80% He; pressure: 100 mtorr; flow: 3 sccm; temperature: 38C; RF power: 0.4 W/cm^2. The deposition rate was ~.01 nm/s. Films varying from ~15 to ~80nm were deposited in this manner.

Sputter deposition was performed in a magnetron sputter system. In order to produce sample films with varying B/C ratios, half of the sputter cathode surface was covered with polycrystalline B_4C, the other half with pure carbon (Papyex). Additional films were created using a pure B_4C sputter target. Deposition parameters were: base pressure: 4×10^{-7}torr; flow: 56 sccm Ar; deposition pressure: 1.5 mtorr; RF power: ~1kW; bias: ~700V; current: 1-2A.

2. Analysis and characterization of deposited films

Following deposition, the composition of a-BC:H films deposited by the two different techniques were measured using ion beam analysis (IBA) techniques: (1) 2.7MeV He^+ elastic recoil detection (ERD) to detect H-isotopes and (2) 3.7MeV He^+ enhanced Rutherford backscattering spectrometry (RBS) for the detection of B,C,O and other impurities. The results of this analysis are summarized in the bar graphs shown in Figure 1.

Analyses of the PCVD boronized films using ion-beam analysis and other techniques resulted in the following determinations-- Average Composition: 13% C, 25% B, 53% H, 9% O (see Fig. 1); Density: ~1.78 g/cm^3, Resistivity: 10^{11} ohm-cm; Refractive Index: 1.8. The films blistered and lost H when exposed to water vapor, indicating that the as-deposited H-content may have been even higher than 53% and that some of the 9% oxygen could have resulted from environmental

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contamination. It is well known that the stability of carborane films is improved if the deposition temperature is increased[3] to ~300C, however, the walls of TFTR can not exceed a temperature of 130C.

The a-BC:H films produced in the laboratory by PCVD and those produced during the initial deposition in TFTR have similar densities and total hydrogen-isotope content (Figure 1), but the B/C ratios are reversed. The TFTR films contain twice as much carbon as boron. Deuterium is also present in the TFTR films which implies that some interaction with the graphite wall (which contains deuterium) of the tokamak occurred during film deposition. TFTR has reduced the amount of methane (to 2%) in its deposition gas in an attempt to reduce the carbon content in the deposited films.

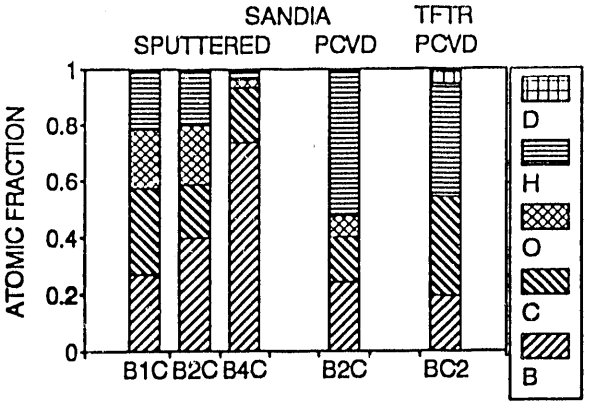

Figure 1.
Composition of as-deposited a-BC:H films.

Identical IBA techniques were employed in the analysis of the sputter deposited a-BC:H films. Boron to carbon ratios ranged from 1:1 to almost 4:1 with the highest ratios stemming from sputtering the polycrystalline B_4C target. The mixed B_4C/C targets produced B:C ratios in the 1:1 to 2:1 range. Surprisingly, neither extremely boron-rich nor boron-poor films were produced by sputtering the mixed target as was originally expected. Oxygen content of these SPD films was also high as in the PCVD films, but the hydrogen content was significantly lower, at ~25%. The high amounts of O in the mixed target films is an indication of the excellent gettering properties of a-BC:H films which is discussed below.

3. H isotope saturation

Experiments to determine the hydrogen saturation levels of the sputter deposited a-BC:H films were also performed. 2keV D_2^+ (1keV/D) ions were implanted into the films, while periodically monitoring the hydrogen and deuterium areal densities with 2.4MeV ^{4}He ERD. 3.7MeV RBS following implantation showed that some boron was lost from the films during the implants.

Figure 2 shows the hydrogen saturation concentrations of several a-BC:H samples plotted, along with the those of carbon, boron and polycrystalline B_4C measured in previous studies[7,8] against the proportion of boron in the sample. The loss of boron in some of the films is indicated by arrows in the figure. The H saturation concentration of sputtered (and TFTR's PCVD) a-BC:H films is ~0.7 H/host which is ~50% higher than for pure carbon. This fact, combined with the lower initial H concentration of sputtered films suggests that a first wall coating of sputtered a-BC:H films would pump H more efficiently than the PCVD

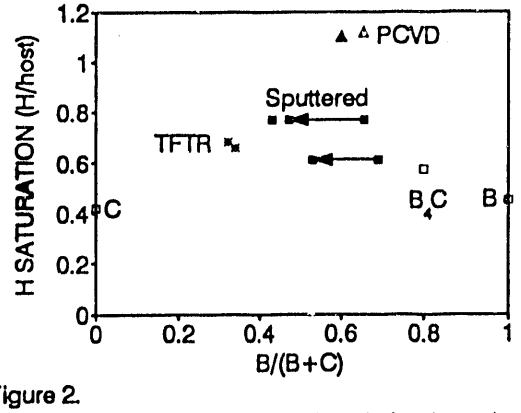

Figure 2.
H isotope saturation concentrations in implanted samples (elements/compounds and sputtered-films) and as deposited films (TFTR and PCVD).

a-BC:H films now used in TFTR. Indeed, the PCVD a-BC:H films deposited on the heated walls of TEXTOR with very low initial H concentrations do exhibit superior H pumping capacities[1]. The lab-prepared PCVD films were saturated as deposited at extremely high H concentrations; clearly unsuitable for use as first wall coatings.

TFTR EXPERIENCE WITH BORONIZATION

Plasma-enhanced CVD of thin (40-100 nm) amorphous carbon films has been applied several times to the first wall surfaces of the TFTR device[3]. The deposition technique was similar to the technique first applied to the TEXTOR tokamak at KFA-Jülich.[1] The original intent of these tokamak first-wall modifications was to take advantage of the decreased sputtering of carbon which has been observed with a-BC:H films in comparison to sputtering of pure C or a-C:H films.[9] In the case of TFTR, the evolution and confinement properties of the highest performance discharges, the so-called "supershots" are dependent very sensitively on the ohmic target density which is in turn dominated by carbon ions sputtered from the graphite first-wall structures[10].

Suppression of carbon impurities was observed in TFTR after a-BC:H deposition, however, due to the high ion fluxes on the inner wall limiter and the relatively thin deposition, the suppression of carbon influx was short-lived. Figure 3 shows the intensities of CII radiation before and after the deposition of a 40 nm and 100 nm a-BC:H film. The CII light is suppressed initially a factor of four with the 100 nm deposition compared to the pre-boron case, but after exposure to 10-20 discharges the CII radiation is near pre-boron levels. It is possible that these transient benefits of C/B materials with respect to carbon erosion could be extended by employing much thicker films or incorporating boron within the lattice of the base graphite material.

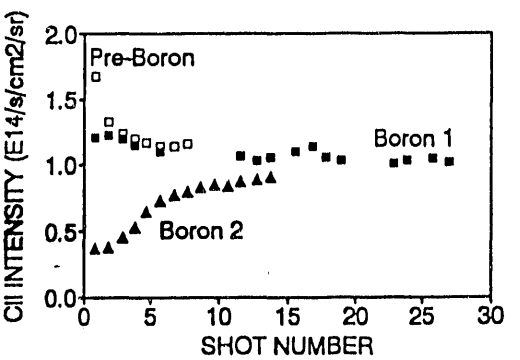

Figure 3

Intensity of CII radiation in TFTR ohmic discharges - before and after boronization.

Longer-term benefits of a-BC:H deposition in TFTR were observed with respect to the oxygen gettering properties. Oxygen is not usually a significant impurity in TFTR discharges in comparison to carbon except following a major disruption of the plasma. A disruption event occurs when plasma confinement is lost (due to exceeding plasma pressure of MHD limits), and the plasma column collapses onto the first wall over a short time scale (<1 ms). This event thermally desorbs oxygen from the bulk of the graphite first-wall.

The oxygen gettering properties of the a-BC:H film enabled a much more rapid recovery from the effects of disruptions than was possible with the pure carbon wall[3]. In addition, the average oxygen plasma impurity levels were depressed by a factor of two following a-BC:H deposition and remained depressed for exposures of several hundred high power discharges.

The a-BC:H depositions on TFTR also protects the first wall from the contaminating effects of major oxygen incursions such as H_2O or air leaks from loss-of-vacuum accidents,[3] and significantly shortens the start-up wall conditioning time following pump-down from atmospheric exposure.

There were no observed harmful effects of the a-BC:H deposition on TFTR operations. After the first two depositions, the vessel was vented to atmosphere for two months of in-vessel maintenance activities. With extended exposure of the deposited films to atmosphere, significant flaking of the thicker film deposits (>50 nm) from the graphite substrate was observed.

CONCLUSIONS

TFTR will be operated with deuterium and tritium in the middle part of this decade in an attempt to demonstrate scientific breakeven of a fusion plasma (i.e. equal amounts of externally applied power used to heat the plasma and t(d,n) fusion power). Paramount to the success of this demonstration will be the improvement and even the control of plasma-edge conditions which are present in the Tokamak. One aspect of the approach which is being undertaken at PPPL to provide wall conditions more conducive to DT-breakeven is boronizing the entire vacuum vessel of TFTR.

Carborane coatings appear very promising for controlling certain aspects of the plasma-wall interaction. The three boronizations of TFTR to date have resulted in significantly reduced recovery times from disruptions; however, with the exception of transient improvements, the plasma parameters have yet to experience long-term benefits through the use of boronization.

Experiments in the laboratory indicate that carborane films possess a significantly higher inherent pumping capacity for H than do the C films present in graphite-wall machines. This greater pumping capacity would be of benefit to TFTR in sustaining supershots. The fact that TFTR has not witnessed persistent benefits from boronization may indicate that the films are simply sputtered away during the supershot conditioning process. This conditioning is required to desaturate the carborane of H-isotopes. We note here that if films could be prepared in TFTR which were not saturated with H as deposited (e.g. PCVD at ~300C or sputter deposition), then the conditioning step could be either eliminated or at least minimized. This would increase the chances that the film deposited in high flux regions would survive, and be present for improved wall-pumping of supershot discharges.

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