

Chemical erosion of DIII-D lower divertor tiles

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Abstract

Spectroscopic measurements in the DIII-D lower divertor have shown an order of magnitude decrease in the brightness of the CD emission band over the course of eight years of plasma discharges. These observations could imply a reduction of the chemical erosion of the carbon component in the divertor. To test this hypothesis, chemical erosion measurements of specimens from the DIII-D lower divertor inner and outer strike points were performed using a low-energy D_2^+ ion beam, with energies extending down to $\sim 15\text{eV/D}^+$. The results indicate that the erosion characteristics of the DIII-D lower divertor plasma-exposed specimens are similar to those of pyrolytic graphite and the virgin DIII-D tile material. The total chemical erosion yields for all specimens peak at $\sim 2\text{--}4\%$ at 700 K for 15eV/D^+ , and reach $\sim 7\%$ for 200eV/D^+ .

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1. Introduction

Carbon has long been a preferred material for divertor use in tokamak fusion devices. This is mainly due to its superior thermo-mechanical properties, low Z , availability, low cost, and its ease of machinability. However, carbon's chemical reactivity with hydrogen can lead to erosion – affecting component lifetime – and tritium inventory build-up in the reactor through codeposition of the eroded C-containing molecules with the tritium fuel. The issue of tritium build-up in the torus has raised concerns about the use of carbon-based materials in next-step fusion devices such as ITER, triggering at-

tempts to mitigate the extent of codeposition and to develop techniques for removing tritium from the codeposits [1,2]. One such technique is the thermo-oxidative removal of the codeposits, e.g., [3].

Spectroscopic analysis in the DIII-D tokamak fusion reactor (operated by General Atomics, San Diego) has shown that the brightness of the CD emission band in the lower divertor has decreased by an order of magnitude over the course of eight years of plasma discharges (~ 23000 plasma shots corresponding to $\sim 10^5\text{s}$ of plasma exposure) [4]. Over this time period the B_{CD}/B_{CII} ratio has also decreased by a factor of 4 [4]. Both of these observations imply that the role of chemical erosion as a carbon source has been progressively reduced over the eight years. A further implication of these observations is that the carbon tiles in the lower divertor have undergone some surface modification, resulting in a reduced chemical reactivity. Possible causes for the surface changes might be the effect of surface boronizations that

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have taken place in DIII-D or some sort of *plasma conditioning* of the tiles [4].

The influence of boronizations on the chemical reactivity of DIII-D tiles removed from the midplane and lower divertor inner strike point has been investigated by Wright and co-workers [5,6]. Results of a variety of surface diagnostics of the midplane tile surface indicated that the surface material consisted of a boron-rich layer of $\sim 2\mu\text{m}$ thickness – formed during boronizations – covered with a thin layer of codeposited C/B/D, with 20–40 at.% C [5]. Chemical erosion yields (measured subsequent to removal from DIII-D) of the midplane specimens at 300 K and 500 K gave initial erosion yields ($0.001\text{--}0.015\text{ C/D}^+$) which are similar to both pure and B-doped graphite under similar conditions [5]. The yields were found to decrease with fluence, consistent with the depletion of C from the thin surface layer [5].

The surface composition of specimens removed from the inner strike point of the DIII-D lower divertor was primarily carbon with trace amounts of boron; the film thickness was estimated to be $\geq 5\text{ nm}$ [6]. Chemical erosion yields (measured subsequent to removal from DIII-D) very quickly reached steady-state values comparable to pure graphite [6]. It is likely that this tile was located in an area of net erosion just prior to its removal [6].

The objective of the present investigation was to perform a further controlled study of the chemical erosion of lower divertor tile specimens from both the inner and outer strike point positions, and consequently, determine whether the extensive reactor plasma exposure of the DIII-D lower divertor graphite tiles has affected their chemical erosion characteristics.

2. Experiment

2.1. Specimens

Four graphite specimens were used in this investigation: pyrolytic graphite (HPG99) as reference, specimens from the DIII-D lower divertor inner (ISP) and outer (OSP) strike point locations, and an unexposed (virgin) tile specimen planned for installation into DIII-D. The DIII-D tiles were made of UCAR-TS-1792 (ATJ) graphite. The ISP and OSP specimens were present in the DIII-D tokamak during the 1993–1999 operational period; see Fig. 1 for their locations. These specimens had also been exposed to air for about two years and special care was taken to outgas the specimens slowly. The virgin DIII-D graphite specimen has never been exposed to a plasma environment but has undergone all preparation treatments performed with tiles prior to installation into DIII-D. This specimen has also experienced prolonged air exposure.

The specimens were cut from larger tiles into $\sim 4\text{ cm} \times 1\text{ cm}$ pieces. They were sanded down to a thick-

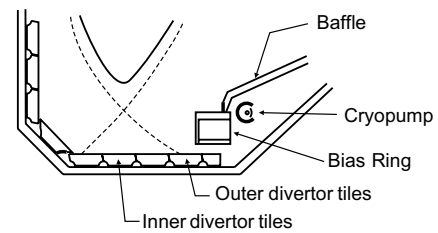


Fig. 1. Schematic of the DIII-D divertor showing the location of the tiles from which the test specimens were cut.

ness between 0.40 and 0.45 mm so that Ohmic heating could be used to vary the specimen temperature. Temperature measurements were made by optical pyrometry. Care was taken to avoid altering the plasma-exposed faces during the cutting and sanding procedures.

2.2. Surface analysis

Surface analysis was performed on the OSP and virgin DIII-D divertor tile specimens at Surface Interface Ontario using time-of-flight secondary ion mass spectrometry (SIMS). Secondary ions were produced with 25 keV ^{69}Ga ions, and depth profiling was achieved by sputtering with a 3 keV Ar^+ beam at 45° angle of incidence to the surface. A $500\mu\text{m}^2$ area was sputtered from the specimen while secondary ions were obtained from a $200\mu\text{m}^2$ area centered in the sputtered crater. One measurement was made *on* and one was made *off* the D_2^+ ion beam spot.

2.3. Chemical erosion due to D_2^+

To test the hypothesis that the reduction of the chemical reactivity of the carbon tile surface in the DIII-D divertor was due to some sort of plasma conditioning process, chemical erosion measurements of specimens from the DIII-D lower divertor, in the vicinity of the inner and outer strike points, were performed at the University of Toronto using a mass-analyzed, low-energy D_2^+ ion beam. In this study, the D_2^+ beam was not meant to simulate divertor plasma conditions, but rather, to serve as a probe for characterizing the erosion behaviour of the tile surface as it existed after eight years of DIII-D plasma exposure.

The measurements were performed under ultra-high vacuum conditions (base pressure $< 1 \times 10^{-8}$ Torr) achieved by baking the target chamber for more than 24 h at 500 K. The test chamber was further conditioned by bombarding the walls with atomic deuterium produced by dissociation of D_2 gas on a hot ($\sim 1700\text{ K}$) W filament. During ion beam operation the test chamber pressure was $2\text{--}3 \times 10^{-6}$ Torr, mainly D_2 . The D_2^+ ion beam ($2.5\text{--}3.5\mu\text{A}$ current) impacted on the specimen at normal incidence, over a 4 mm diameter spot

($\sim 12\text{mm}^2$ area) with a flux density of $\sim 3 \times 10^{18}\text{D}^+/\text{m}^2\text{s}$. (Here we denote the incident D atoms as D^+ even though the D_2^+ molecular ion is only singly charged.) Erosion experiments were performed for all of the specimens using 200, 50, and 15 eV/ D^+ beam energies at specimen temperatures ranging from 300 to 1000 K. The hydrocarbon reaction products resulting from the D_2^+ irradiation (CD_4 , C_2D_2 , C_2D_4 , C_2D_6 , C_3D_6 , and C_3D_8) were detected by quadrupole mass spectroscopy (QMS) in the residual gas. The quadrupole was calibrated in situ with leaks of CD_4 , C_2D_4 , and C_3D_6 while relative sensitivities for C_2D_2 , C_2D_6 , and C_3D_8 were estimated from previous calibrations performed by Mech et al. [7]. Beam-induced background signals (wall contributions) were evaluated and subtracted as in Ref. [7]. Depending on the specimen temperature and the incident ion energy, the wall contribution can be 30–90% of the total beam-produced QMS signal. Since the wall contribution has such a strong effect on the QMS signal, uncertainty in the wall contribution is considered to be the main component of the error in the calculated erosion yields.

3. Results and discussion

3.1. Surface analysis

SIMS analyses *on* and *off* the D_2^+ beam spots of the OSP and virgin tile specimens revealed the presence of impurity clusters containing aluminum, boron, hydrogen, and trace amounts of oxygen, silicon and deuterium. The impurities were more abundant on the beam spots, probably due to preferential erosion of the carbon. The impurity clusters occupied only a small fraction of the surface area ($<5\%$ for the virgin specimen and $<10\%$ for the OSP specimen) and have not affected the measured chemical erosion yields of the specimens. Since similar impurities and impurity distributions were found for both specimens, it is suspected that the impurities were introduced during the original tile preparation; e.g., polishing prior to installation in the reactor. If the impurity clusters were excluded from the surface analysis data, the surface would be essentially pure carbon with trace amounts of oxygen (most likely due to prolonged atmospheric exposure). (We note that previous surface analysis of an ISP specimen indicated the presence of a surface layer of $\geq 5\text{nm}$ thickness, which was primarily carbon with trace amounts of boron [6].)

3.2. Chemical erosion due to D_2^+

Measured chemical erosion yields as a function of specimen temperature are presented in Figs. 2–4, for ion energies of 200, 50, and 15 eV/ D^+ , respectively. Methane yields and total chemical erosion yields,

$\text{C}_{\text{chem}}/\text{D}^+$ (where C_{chem} represents the sum of the C atoms contained in the C_1 , C_2 , and C_3 hydrocarbons) are shown. For all specimens, the yields – both CH_4/D^+ and $\text{C}_{\text{chem}}/\text{D}^+$ – increase with increasing temperature up to a peak yield (Y_{max}) occurring at a temperature (T_{max}). For specimen temperatures greater than T_{max} , the yields decrease with increasing temperature. The observed yields and their temperature dependence are consistent with previous measurements for pyrolytic graphite [7].

The error bars shown in Figs. 2–4 are based on variations in the measured wall contributions. We note that the error bars become relatively larger for the lower energies and for low and high temperatures. This is due to the relatively lower hydrocarbon formation rates under these conditions, in comparison with the respective wall contributions.

For the most part, the observed differences in the erosion yields of all specimens, both CH_4/D^+ and $\text{C}_{\text{chem}}/\text{D}^+$, are within the error bars shown in Figs. 2–4. The only notable exception is the 200 eV/ D^+ irradiation of the ISP specimen in Fig. 2; this specimen exhibits yields at T_{max} – both methane and total chemical – which are about a factor of two lower than the corresponding yields for the other specimens. Another observation to

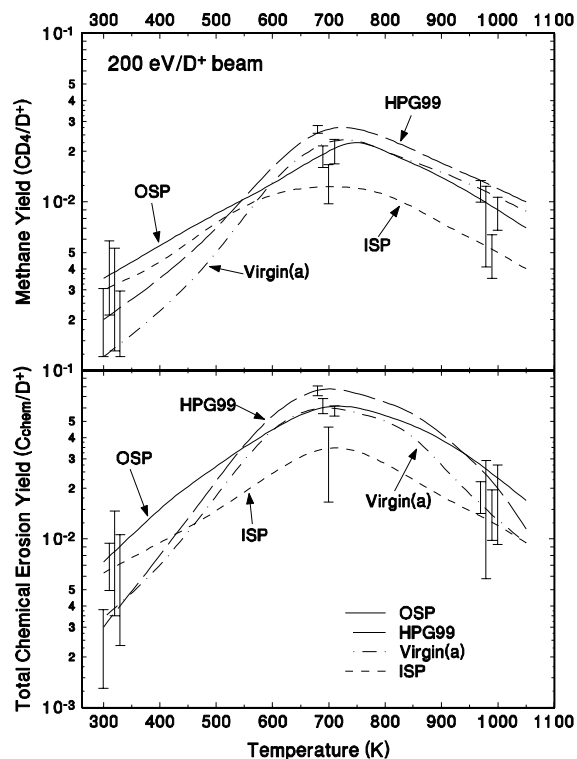


Fig. 2. Methane yields (CD_4/D^+) and total chemical erosion yields ($\text{C}_{\text{chem}}/\text{D}^+$) as a function of temperature for specimen erosion by 400 eV D_2^+ (200 eV/ D^+).

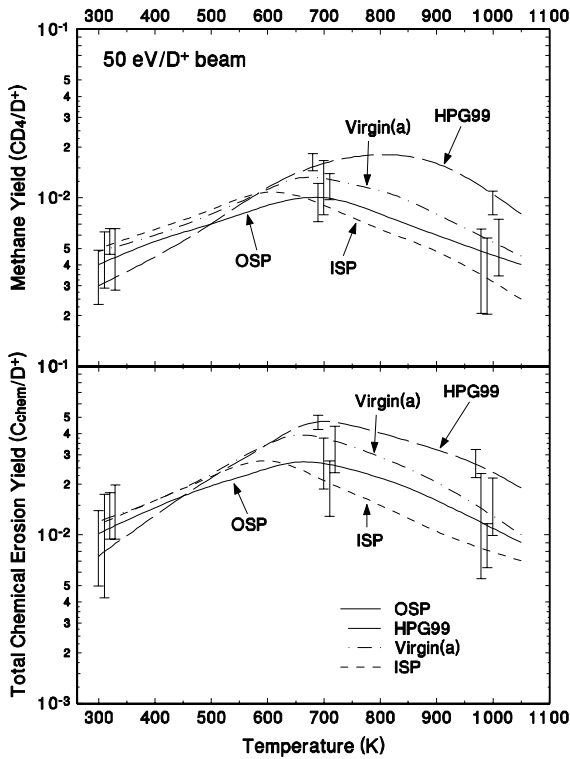


Fig. 3. Methane yields (CD_4/D^+), and total chemical erosion yields (C_{chem}/D^+) as a function of temperature for specimen erosion by $100\text{ eV } D_2^+$ ($50\text{ eV}/D^+$).

note is the difference (\sim factor of 2) in the high temperature segment ($>600\text{ K}$) of the erosion yield curves for two virgin tile specimens (shown as a and b in Fig. 4), which can be assigned to sample-to-sample variation and experimental reproducibility.

Considering the fact that it is the outer strike point that has seen relatively higher plasma fluences in DIII-D, the expectation was that the OSP specimen would experience reduced chemical reactivity due to plasma conditioning. Our findings do not support this hypothesis.

DIII-D divertor tile specimens were also exposed to a deuterium plasma in the PISCES-B plasma device at the University of California, San Diego [8]. While in our experiment the D_2^+ beam was used simply as a probe (flux: $\sim 3 \times 10^{18} D^+/m^2s$), the plasma flux density in PISCES-B was sufficiently high ($\sim 10^{22}\text{--}10^{23} D^+/m^2s$) to cause substantial erosion of the surface. During a 10 min exposure in PISCES-B, the CD emission band observed via optical emission spectroscopy was seen to decrease by a factor of 2, corresponding to a factor of 2–4 decrease in the chemical erosion yield [8]. Auger emission spectroscopy revealed a surface composition of 87% C, 5% B, and 6% O before plasma exposure and a surface composition of 11% C, 55% B, and 32%

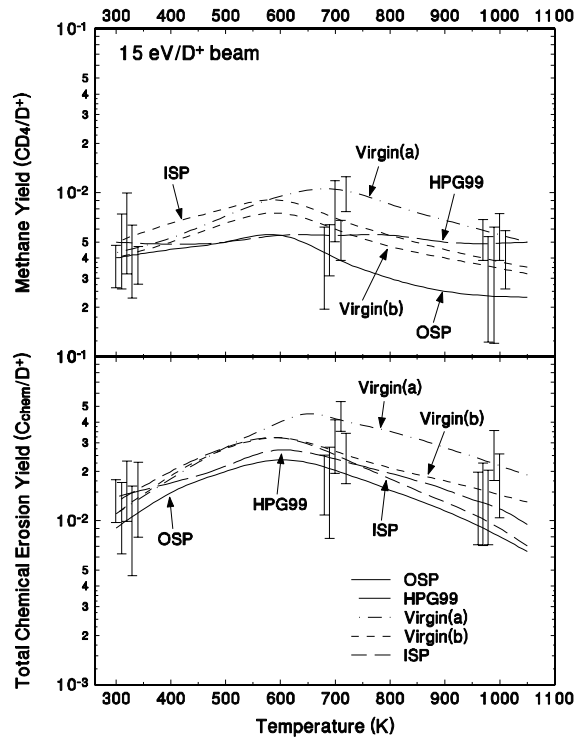


Fig. 4. Methane yields (CD_4/D^+) and total chemical erosion yields (C_{chem}/D^+) as a function of temperature for specimen erosion by $30\text{ eV } D_2^+$ ($15\text{ eV}/D^+$). The erosion yield curves for two virgin tile specimens shown as (a) and (b) indicate a difference of about a factor of 2 in the high temperature segment ($>600\text{ K}$), which can be assigned to sample-to-sample variation and experimental reproducibility.

O after plasma exposure. It was concluded that preferential erosion of the carbon led to a boron-rich surface, which resulted in the reduced chemical erosion [8]. For the current specimens from the DIII-D divertor, no such accumulation of B was observed, and thus we cannot make a direct comparison with the PISCES result.

4. Conclusions

It was the hypothesis of this investigation that some sort of physical change has occurred in the lower divertor tiles during eight years of DIII-D plasma exposure that has affected the chemical erosion properties of the material – leading to the observed reduction in the CD emission signal. Were this the case, one would expect to see significant reductions in the chemical erosion yields of the plasma-exposed DIII-D tiles, when compared with pyrolytic graphite, or with a virgin specimen of the same material and similar preparations as the plasma-exposed DIII-D tiles. However, our measurements do not show significant differences in the yields

of the specimens studied, which appears to rule out any plasma conditioning effect. The similar chemical erosion yield of pyrolytic graphite and the DIII-D virgin specimen further implies that the DIII-D divertor tiles have not experienced any significant changes to their physical properties due to surface preparation prior to their installation into the lower divertor.

SIMS surface analyses performed *on* and *off* the D_2^+ beam spots of the OSP and virgin tile specimens revealed the presence of impurity clusters containing aluminum, boron, hydrogen, and trace amounts of oxygen, silicon and deuterium, possibly introduced during tile preparation. These clusters cover only a small fraction of the surface (<5% for the virgin and <10% for the OSP specimen) and have no discernable effect on the chemical erosion of the tiles.

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