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Control of High-Z PFC Erosion by Local Gas Injection in DIII-D*

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Abstract

Suppressed erosion of a high-Z PFC divertor surface was observed in DIII-D with local injection of methane and deuterium gases. Molybdenum-coated silicon samples were exposed in the lower divertor of DIII-D using DiMES under plasma conditions previously shown to cause significant net erosion of Mo. Three exposures featured $^{13}CH_4$ and one exposure D_2 gas injection about 12 cm upstream of the samples located within 1–2 cm of the attached outer strike point. Suppression of Mo erosion was evidenced *in-situ* in all four exposures by the disappearance of MoI line radiation at 390.2 nm once the gas injection was turned on. Post-mortem ion beam analysis found the erosion of molybdenum near the center of the samples exposed with $^{13}CH_4$ injection being below the measurement resolution of 0.3 nm, corresponding to a rate of ≤0.02 nm/s. Compared to the previously measured erosion rates, this constitutes a reduction of more than 20X.

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1. Introduction

High-Z materials such as tungsten will be used for plasma facing components (PFCs) in the divertor of ITER [1] and very likely in devices beyond ITER. PFC erosion will produce high-Z impurities that may lead to unacceptably high radiation losses if they enter the plasma core. It is envisaged that erosion of the main divertor plates will be controlled by detachment [2] induced by injection of radiating gas. However, this may leave some critical PFCs such as divertor baffles, startup/protection limiters, etc., vulnerable to excessive erosion and surface damage including melting, particularly during transients such as Edge Localized Modes (ELMs) and disruptions. A sacrificial low-Z coating deposited on a high-Z PFC surface can protect the surface from erosion and damage. However, a thin coating will quickly erode, so it has to be renewable *in-situ*, preferably during a plasma discharge. Injection of a gas containing low-Z impurities such as B, C, Si, or Li through capillaries in a PFC surface can lead to local deposition of a low-Z coating on the surface due to a local decrease of the electron temperature (T_e) and increase of the low-Z impurity content (see Section 2). Previous work in TEXTOR [3] demonstrated the effectiveness of *in-situ* coatings produced by a local injection of silane gas (SiH_4) for controlling erosion of the leading edge of a graphite limiter. In the experiments reported here, we successfully suppressed erosion of molybdenum (Mo) by using methane gas injection upstream of Mo-coated samples in the DIII-D divertor.

While creating a protective low-Z coating *in-situ* is certainly an attractive concept, it has certain disadvantages: eroded low-Z impurities would re-deposit elsewhere inside the vacuum vessel, potentially leading to tritium retention and accumulation of dust and debris from exfoliating re-deposited layers. In case of carbon, this problem may potentially be alleviated by thermal oxidation [4]. However, this solution is unlikely to be acceptable for ITER. Therefore, we also tested an alternative concept, where Mo erosion was reduced by a local injection of D_2 gas that does not create substantial coating on the sample. This technique, while probably less effective for PFC protection from transients (for lack of protective coating, gas injection would have to be triggered on the transient event pre-cursor), may possibly be used for alleviating erosion of PFCs where critical heat loads may exist for relatively short and known periods of time, e.g. ITER startup limiters [5].

2. Background: balance between erosion and deposition

It can be shown from simple first-principle considerations that local injection of a gas containing low-Z impurity can shift the balance between erosion and deposition from net erosion to net deposition. We will use carbon in deuterium plasma as an example. Let the D ion flux density onto the solid surface be Γ_D and the C ion flux density onto the solid surface be Γ_C. Let the sputtering yield for D on C be Y_{DC} and for self-sputtering be Y_{CC}. With a C-containing gas injection, $f_C \equiv \Gamma_C / \Gamma_D$ is externally adjustable. Neglecting backscattering, the gross rate at which C particles deposit on the surface is $f_C \Gamma_D$ and the rate at which they are removed by sputtering is $Y_{DC} \Gamma_D + Y_{CC} f_C \Gamma_D$. Thus for steady state, where the layer thickness remains constant, one has:

$$f_C \Gamma_D = Y_{DC} \Gamma_D + Y_{CC} f_C \Gamma_D \tag{1}$$

$$f_C = Y_{DC} / (1- Y_{CC}) \tag{2}$$

Note that f_C is a function of T_e since Y_{DC} and Y_{CC} are, thus eqn. (2) defines $T_{e,th}(f_C)$, the temperature at which the thickness of the carbon layer neither increases nor decreases.

Figure 1 shows a calculated dependence of $T_{e,th}(f_C)$ ([6], details of the calculation will be published elsewhere). Physical sputtering yields from [7] and chemical sputtering yields from [8] for 300K, 500K, 800K and 1000K were used. The curves in the figure correspond to balance between erosion and deposition. The regions to the right of the curves correspond to net erosion and the regions to the left to net deposition. Thus, for a given surface temperature, the transition from net erosion to net deposition of carbon can be achieved by either increasing f_C or decreasing T_e. Injection of a gas containing carbon (e.g. methane) is likely to cause both of those effects, thus, when performed near a high-Z PFC surface, it may reduce high-Z surface erosion via creating a protective coating. Injection of a gas not containing carbon (e.g. D_2) is likely to locally decrease both f_C (through dilution) and T_e, thus its effect is harder to predict. Even if low-Z coating is not formed on a high-Z surface, its erosion may be sufficiently reduced due to the reduction of T_e.

3. Experimental approach

Exposures of Mo-coated samples were conducted in the lower divertor of the DIII-D tokamak [9]. Molybdenum was chosen over tungsten for its more well known inverse photon efficiency coefficient (equal to number of ionizations per photon), S/XB [10]. In order for net erosion to be measurable by ion beam analysis (IBA), 15-75 nm thick Mo films deposited on 1 cm diameter silicon disks were used. Films were deposited in a magnetron sputter deposition system and pre-characterized by IBA prior to plasma exposures. For the exposures, the samples were installed in graphite casings and inserted in the lower divertor of DIII-D using the Divertor Material Evaluation System (DiMES) manipulator [11]. During the exposures, the top surface of the DiMES probe was flush with the divertor tile plasma-facing surface within 0.1–0.2 mm. For the first two exposures, the Mo-coated samples were mounted in the center of the DiMES head ~5 cm in diameter [Fig. 4(a,b)]. For the latest two exposures, a DiMES head featuring an embedded Langmuir probe (LP) downstream of the samples was fabricated [Fig. 4(c)]. The LP allowed measurements of the local plasma density and electron temperature before and during the gas injection.

All experiments were performed in deuterium discharges, in a lower single null (LSN) magnetic configuration. Figure 2(a) shows a poloidal cross-section of DIII-D with an equilibrium last closed flux surface (LCFS) used during the sample exposures. Also shown are the location of DiMES and arrangement of the optical diagnostics. In the exposure discharges, the outer strike point (OSP) was kept off DiMES sample during the plasma current ramp-up and ramp-down. Once stable conditions were achieved at ~1 s into the discharge, the OSP was moved to the inboard edge of the samples and dwelled there for 3-4 s in each exposure discharge. The samples were imaged by an absolutely calibrated digital CMOS camera and a high resolution MDS spectrometer [12]. Gas was injected through a capillary opening through a hole in a floor tile ~12 cm upstream from the center of the DiMES head at the same major radius [Fig 2(b)]. The gas injection rate was increased stepwise till the MoI signal at 386.3 and 390.2 nm measured by the MDS fell below its detection threshold [Fig. 4].

Net erosion of Mo and deposition of carbon were measured by post-mortem IBA [13]. Mo erosion was measured by Rutherford backscattering spectroscopy (RBS) with 2 MeV ^{4}He; C and D deposition were measured by nuclear reaction analysis (NRA) using an analysis beam of 2.5 MeV ^{3}He . In order to discriminate between the carbon originating from the gas injection and that from the background plasma, stable, isotopically enriched $^{13}CH_4$ methane (99% ^{13}C) was used for the injection.

4. Experimental results

Four exposures were performed in two experiments. In the first experiment both exposures were with a $^{13}CH_4$ gas injection, first in four L-mode discharges for ~14 s total exposure time (exposure #1), and second in two H-mode discharges for a total of ~7 s (exposure #2). $^{13}CH_4$ gas was injected at a low rate that did not cause measurable perturbation of the global discharge and divertor plasma parameters. Figure 3(a) shows temporal evolution of the MoI signal at 390.2 nm measured by the MDS spectrometer with $^{13}CH_4$ gas injection at a rate of 0.5 Torr*l/s in L-mode. At that injection rate the measured emission was reduced by about a factor of 3. In subsequent discharges with a gas injection rate of 1.5 Torr*l/s, the MoI signal fell below the detection level. In the following H-mode exposure the injection rate had to be increased to 2.8 Torr*l/s in order to achieve MoI signal suppression.

Post-mortem RBS analysis found the net erosion of molybdenum near the center of the samples being below the measurement resolution of 0.3 nm, corresponding to a rate of ≤0.02 nm/s. Compared to the net erosion rates of 0.4–0.7 nm/s previously measured in L-mode discharges similar to those used in exposure #1 [14, 15], this constitutes a reduction of more than 20X.

Visible carbon deposits were observed on both samples upon removal [Fig. 4(a,b)]. On the sample exposed in L-mode the deposits formed a well-defined toroidal stripe, while on the sample exposed in H-mode the deposition pattern was less regular, possibly due to effect of ELMs. OSP position control was also not as good in H-mode as in L-mode, so the sample was in the private flux region for part of the exposure. Post-mortem NRA measured C deposition rates of ~32 nm/s in L-mode and ~8.5 nm/s in H-mode, assuming a deposited C density of 2 g/cm^3. Measured coverage of both ^{13}C and ^{12}C on the sample exposed in L-mode is shown in Fig. 5. Carbon coverage on the sample exposed in H-mode was about 9 times lower. The ratio of ^{13}C/^{12}C carbon in the deposits was about 3-4 on both samples, indicating that the deposition was largely from the gas injection. The significant amount of the intrinsic ^{12}C carbon in the deposits is likely an indicator of the local T_e decrease due to the gas injection causing the carbon erosion/deposition balance to shift towards net deposition [Fig. 1].

Post-exposure deuterium coverage on the exposed samples was also measured by NRA. The average D/C ratio on the sample exposed in L-mode was 0.47. The IBA left visible dark spots on the sample, characteristic of a hydrogen-rich low density film. This indicates that the deposition occurred from a relatively low temperature plasma. On the sample exposed in H-mode, the average D/C ratio was ~0.1 and no discolored spots were observed. This indicates harder deposits formed at a higher plasma temperature.

Since the local plasma parameters at DiMES sample were not measured in the first experiment, the L-mode discharges of exposure #1 were later reproduced with similar gas injection and a DiMES head featuring a Langmuir probe in place of the material sample. LP measurements of T_e showed that it stayed comparatively high, at 8-10 eV during the gas injection. Therefore, it was confirmed that Mo erosion suppression was mostly due to the carbon deposition rather than a local detachment.

In the second experiment, the first exposure (#3) was completed with D_2 upstream gas injection, and the second one (#4) was with a $^{13}CH_4$ gas injection, both in three L-mode discharges with the plasma parameters similar to those of exposure #1. During exposure #3, D_2 injection started at 2.5 s into the discharge, then at 3.5 s the injection rate was doubled. The initial injection rate was increased in each subsequent discharge until MoI emission was eventually suppressed [Fig 3(b)]. The suppression occurred at the injection rate of ??? T*l/s, which still had no measurable effect on the global discharge parameters. The first exposure discharge spontaneously transitioned into H-mode for the last second of the sample exposure.

Exposure #4 was conducted in three reproducible discharges with $^{13}CH_4$ gas injection starting 0.5 s into the discharge, before OSP was placed on DiMES. Gas injection was at the rate of 1.5 Torr*l/s, shown to suppress Mo erosion during exposure #1. The aim of this exposure was to repeat exposure #1 at constant reproducible conditions and with the local T_e measured by the embedded LP. This data set should be of good enough quality to benchmark future modeling of the local erosion/deposition.

The exposed samples were characterized by RBS and NRA. Net surface-average molybdenum erosion of ~9.3 nm was measured on the sample exposed with D_2 injection. This is not surprising, since the sample was exposed at the OSP for about 1.5 seconds before the start of the D_2 injection in each of the three exposure discharges. If assumed that Mo erosion only occurred before the D_2 injection, the average net erosion rate would be ~2 nm/s, which is a factor of 3-5 higher than previously reported for similar conditions without gas injection [14, 15]. Considering that molybdenum erosion was still occurring at reduced rate during the lower rate D_2 injection in the first two exposure discharges, the pre-injection erosion rate was probably about 1-1.5 nm/s, which is still higher than reported previously [NEEDS TO BE EXPLAINED]. Carbon deposition with an average thickness of ~7 nm was also measured on the sample. This layer could help reducing molybdenum erosion rate, but good correlation of the MoI light reduction with the start of the D_2 injection [Fig 3(b)] suggests that erosion reduction was primarily due to the gas injection.

Molybdenum erosion on the sample exposed with $^{13}CH_4$ gas injection was found to be below the RBS measurement resolution. Deposition of ^{12}C and ^{13}C was measured by NRA with the corresponding surface-average deposition rates of ~4 nm/s and ~42 nm/s. Therefore, the ratio of $^{13}C/^{12}C$ in the deposits was even higher that in exposures #1 and #2, confirming that ~90% of the deposited carbon originated from the gas injection. D/C ratio in the deposits was measured at 0.35.

5. Summary

We have demonstrated that a local gas injection in the vicinity of a small area high-Z PFC surface (molybdenum in our case) in a tokamak divertor can reduce or suppress the surface erosion without globally disturbing the plasma discharge. When a protective carbon coating was created by the methane gas injection, the erosion of Mo, both gross and net, fell below our diagnostic resolution. However, creating an *in-situ* low-Z coating that would eventually erode and redeposit elsewhere may have long-term adverse effects in the future machines, so we also tested erosion suppression with a non-depositing deuterium gas injection. While net erosion of Mo was probably not fully suppressed during D_2 gas puff, spectroscopic measurements confirmed that the gross erosion was strongly reduced. Future experiments will be designed to further quantify high-Z surface erosion reduction with non-depositing local gas injection.

Acknowledgment

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Figure captions

Fig. 1. Threshold electron temperature for transition from net deposition to net erosion of carbon as a function of the relative carbon concentration at different surface temperatures.

Fig. 2. Experimental setup showing LCFS, DiMES, and views of CMOS camera and MDS (a); image of the sample taken during exposure #1 by the camera with CD/CH filter (b)

Fig. 3. Temporal evolution of MoI signal at 390.2 nm measured by MDS spectrometer with $^{13}CH_4$ gas injection at 0.5 Torr*l/s in exposure #1 (a) and with D_2 gas injection at [rate?] in exposure #3 (b).

Fig. 4. Post-exposure photographs of the samples exposed with $^{13}CH_4$ gas injection in L-mode (a, c) and H-mode (b). DiMES sample holder in (c) features an embedded LP.

Fig. 5. Post-exposure ^{12}C and ^{13}C coverage of the DiMES sample from L-mode exposure #1.

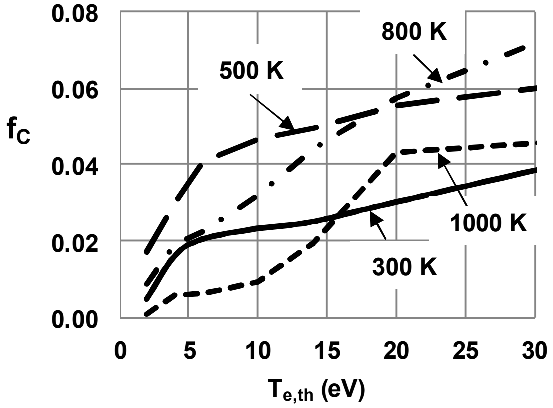

0.08
0.06
0.04
0.02
0.00
f_C
0
5
10
15
20
25
30
$T_{e,th}$ (eV)
800 K
500 K
1000 K
300 K

Fig. 1

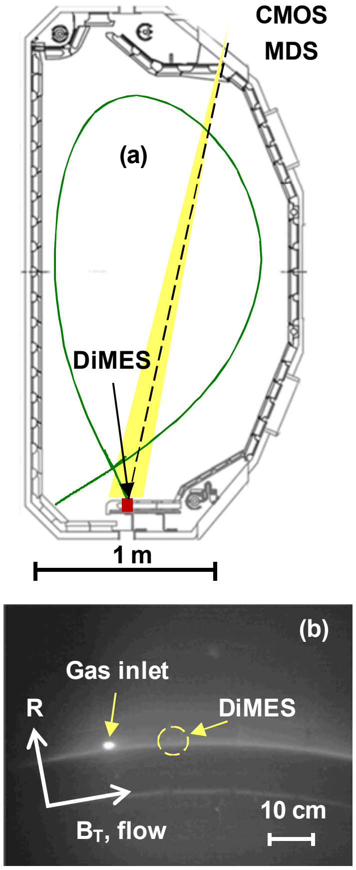


Fig. 2

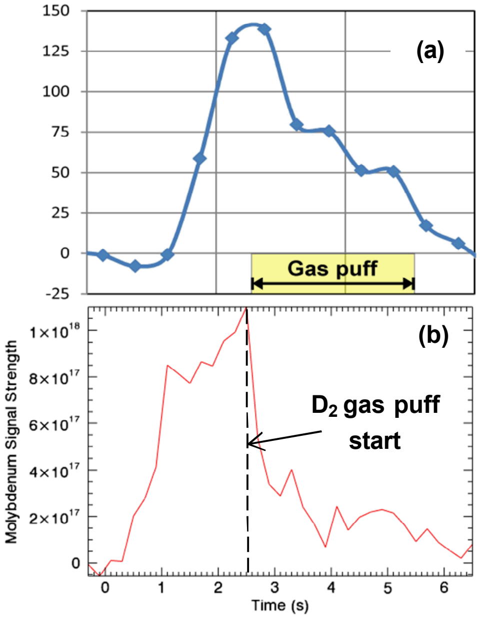

150
125
100
75
50
25
0
-25
(a)
Gas puff
Molybdenum Signal Strength
1×10^{18}
8×10^{17}
6×10^{17}
4×10^{17}
2×10^{17}
0
(b)
D_2 gas puff start
0
1
2
3
4
5
6
Time (s)

Fig. 3

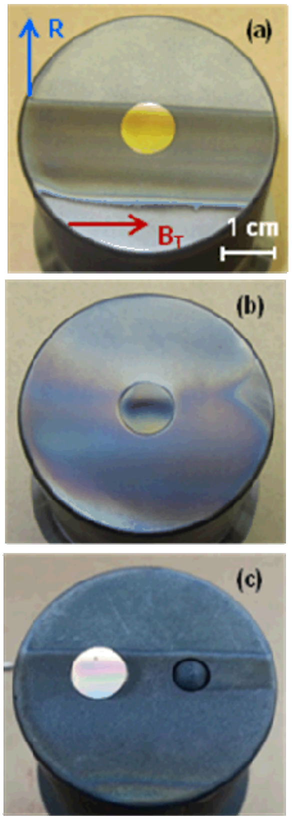
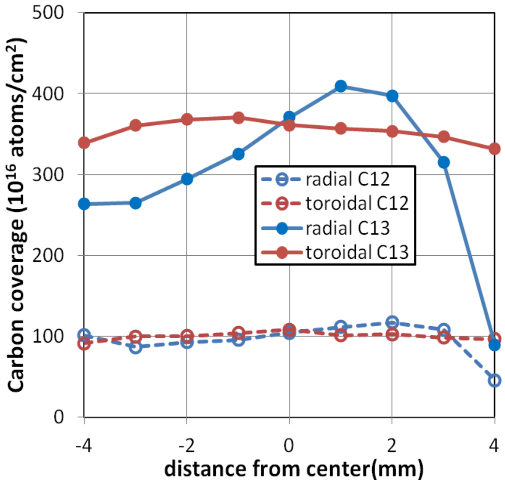


Fig. 4



500
400
300
200
100
0
Carbon coverage (10^{16} atoms/cm^2)
-4
-2
0
2
4
distance from center(mm)
radial C12
toroidal C12
radial C13
toroidal C13

Fig. 5