Measurements of gross erosion of Al in the DIII-D divertor

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Abstract

Aluminum (Al) is a convenient proxy for beryllium (Be) plasma material interaction studies since they have a number of physical and chemical similarities. Al samples were exposed at the lower outer strike point of an L-mode divertor plasma in DIII-D (conditions $7\text{--}11 \times 10^{18}$ D-ions $\text{cm}^{-2} \text{ s}^{-1}$, $T_e = 12\text{--}47$ eV). The gross erosion rate was directly measured using post-mortem ion beam analysis of small 1 mm-sized samples where local re-deposition was determined to be negligible. The gross erosion rate was also calculated using spectroscopic methods, but these rates greatly underestimate the direct (i.e. non-spectroscopic) measurement. The direct measured erosion yields were within the range of published $\text{D}^+ \rightarrow \text{Al}$ ion beam sputtering yields. The ionizations per photon (S/XB) coefficients used in the spectroscopic analysis were determined in separate experiments using He plasmas at the PISCES-B linear plasma facility at UCSD. The measured S/XB coefficients were on average ~6X higher than the theoretically calculated values.

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1. Introduction

The erosion of the low-Z element aluminum (Al) in the DIII-D divertor using the Divertor Material Evaluation System (DiMES) is being studied as a proxy for beryllium (Be), which will be the primary plasma facing material in the ITER first wall and secondary (upper) divertor plasma-facing components. Al is a useful proxy for Be since it shares similar sputtering and chemical characteristics [1] without having the severe chemical toxicity restrictions of Be, and is easily distinguishable from background sources in DIII-D. In this study we compare spectroscopic and direct (non-spectroscopic) measurements of Al erosion, and we present the measurement of the ionizations per photon (S/XB) coefficients that were used for the spectroscopic determination.

2. Experimental setup

In two separate experiments, thin coatings of Al were exposed on the DiMES probe at the outer strike point of repeated simple-as-possible deuterium (D) plasma discharges in DIII-D that have been well-characterized and previously modeled [2]. The average plasma conditions during each sample exposure are listed in Table 1. The dominant impurity was carbon (C), and although it was not measured, estimates for similar plasmas place it at 1% of the D content with an average charge state of +3 [3]. The toroidal field strength was 2.0 T, and the magnetic field vector was inclined $\sim 2.2^\circ$ to the surface tangent at the sample. Steady exposure conditions were achieved by controlling the strike point position and keeping the sample in the private flux region until the steady phase of the discharge.

The Al coating geometry for both samples, described in Fig. 1, included two areas: a large area onto which significant re-deposition is likely to occur; and an area smaller than the ionization mean free path and located on the *upstream* side of the larger area, thus minimizing any redeposition. This combination enabled measurement of both the net erosion

(i.e. erosion reduced by redeposition) and gross erosion (i.e. erosion without redeposition) rates using pre and post-mortem helium (He) Rutherford backscattering (RBS) film thickness measurements of the large and small areas, respectively. For the first case sample, a 93 nm Al layer was deposited on a smooth, carbon coated silicon (Si) planchet and mounted in a graphite DiMES sample holder. For the second case sample, an 80 nm Al layer was applied directly onto the polished graphite probe surface.

Spectroscopic measurements of Al erosion were made by measuring AlI emission at 394.4 nm and 396.1 nm with both an absolutely calibrated and band-pass filtered CCD camera and an absolutely calibrated visible spectrometer viewing the sample. The filter transmission curves are shown in Fig. 2 along with the observed emission spectra. The ionizations per photon (S/XB) coefficients for AlI emission lines used to interpret the DIII-D spectroscopic data were measured in low temperature He plasmas in the linear divertor plasma simulator PISCES-B and scaled up to the DIII-D electron temperature (T_e) and density (n_e).

3. Non-spectroscopic gross erosion measurement

The non-spectroscopic gross erosion measurement relies on the assumption that the majority of atoms eroded from within and near the small sample area are re-deposited outside the area such that net erosion becomes equivalent to gross erosion. In the case of a Al erosion in the DIII-D plasmas described herein, this condition is satisfied for the 1 mm spot and radial stripe regions due to the long ionization mean free path (~ 3 mm, calculated using ionization rate coefficients from ADAS [4]) for a sputtered Al atom with average energy 1.7 eV, compared its ion gyro radius (~ 0.3 mm) and the magnetic pre-sheath thickness (D+ gyro radius ~ 0.25 mm).

Film thickness measurements on both the small and large area samples were made using He RBS before and after sample exposure. For the second case sample, the thin (<100 nm) film was deposited on a relatively rough substrate with dense porosity intersecting the surface (surface was polished to ~1 μm , but pores measuring ~35 μm in size uniformly covered 15% of the surface). The effect of this substrate may have led to inhomogeneous erosion and deposition rates [5], and the resulting measured change in film thickness represents an average of the film thickness distribution over the 0.5 mm beam measurement spot size. The net and gross erosion rates and erosion yields are summarized in Table 1. For the first case sample, the entire 1 mm spot had eroded away along with a portion of the 1 cm spot, so only a lower limit estimate of the gross erosion rate was possible and the net erosion rate may be over-estimated. For the second case sample, the the net and gross erosion rates were found to be similar, to within the measurement error. In both cases, almost no re-deposited Al was found on the DiMES substrate, indicating that the average Al redeposition length was larger than the probe length.

The RBS measured erosion yields for both cases were within the range of published [6] sputtering yields for $\text{D} \rightarrow \text{Al}$. However, the measured erosion yield includes the contribution of sputtering by background C ions which can vary widely based on the C ion charge distribution, concentration, and sputtering yield, all of which are unknown. A rough estimate of the $\text{C} \rightarrow \text{Al}$ sputtering contribution, made using background carbon ion distribution of similar, previously modeled SAPP discharges [2,3] and TRIM calculated sputtering yields [6], finds that sputtering by carbon may account for 50% of the observed erosion in case 1 and 90% of the observed erosion in case 2.

4. Spectroscopic erosion measurements

Spectroscopic erosion measurements were accomplished by measuring AlII emission. The emission intensity for a particular AlII line (I_{AlII} [photons/ $\text{cm}^2\text{-sr-s}$]) was calculated by scaling

the line-integrated photon flux ($I_{\text{All}}^{\text{spec}}$) measured by a spectrometer viewing the sample by the ratio of the ~ 30 mm diameter view spot area (A_{spec}) to sample area (A_{sample}), reduced by an estimated $F_{\text{mis}} \sim 60\%$ due to a slight misalignment of the spectrometer view. A combined intensity from both All lines passed by the filter ($I_{\text{All}}^{\text{pass}}$) was calculated from the CCD image data. Using a ~ 40 mm diameter integrating area, the count rate centered on the sample ($C_{\text{Sample}}^{\text{CCD}}$) was scaled to the original sample area and adjusted for the sample reflectivity, R_s . The minor C and D emission also passed by the filter was removed by subtracting the area integrated intensity 50 mm toroidally upstream of the sample ($C_{\text{Background}}^{\text{CCD}}$). The inverse sensitivity of the camera (S^{-1} [photons/cm²-ster-count]) was calibrated through the vessel windows with all filters and optics in place. Figure 3 shows an annotated example of the All filtered CCD camera view of the sample.

$$I_{\text{All}}^{\text{pass}} = 4\pi S^{-1} \frac{(C_{\text{Sample}}^{\text{CCD}} - C_{\text{Background}}^{\text{CCD}})}{A_{\text{sample}}(1 + R_s)}, \quad (1)$$

$$I_{\text{All}} = 4\pi \frac{I_{\text{All}}^{\text{spec}} A_{\text{spec}}}{F_{\text{mis}} A_{\text{sample}}(1 + R_s)}. \quad (2)$$

The total Al ionization influx $\Gamma_{\text{Al} \rightarrow \text{Al}^+}$ was calculated using the S/XB coefficient and emission intensity described in Sec. 5. For the combined emission line intensity passed by the filter, an average S/XB was calculated using the S/XB_n and the normalized filter transmission (T_n) for each line n .

$$\Gamma_{\text{Al} \rightarrow \text{Al}^+} = I_{\text{All}} \left(\frac{S}{\text{XB}} \right) = I_{\text{All}}^{\text{pass}} \left(\sum_n \frac{T_n}{S/\text{XB}_n} \right)^{-1}. \quad (3)$$

The resulting spectroscopic erosion rates for the second case sample are plotted vs time for each exposure discharge in Fig. 4. Average values for both exposure cases are summarized in Table 1, along with averages for the different case 2 sub-regions (a–e) labeled in Fig. 4. The spectroscopic erosion rates under-estimated the non-spectroscopic erosion rate measurements by a factor of ~ 10 . The spectroscopic erosion rate increased by 15%–50% during the steady phase of each shot and continued to increase with each subsequent exposure. This could be an indication of surface oxide removal or thermal enhancement of erosion. Also surprisingly, the spectroscopic erosion rate appeared to increase strongly with n_e , and was less sensitive to changes in T_e .

5. Measurement of Al S/XB in PISCES-B

The S/XB coefficients for AlI emission at 394.4 nm and 396.1 nm were measured in He plasmas in PISCES-B over a range of $T_e = 5\text{--}8\text{ eV}$ and $n_e = 5\text{--}10 \times 10^{12}\text{ cm}^{-3}$ using a technique similar to that described in Ref. [7]. Bulk Al samples 22 mm in diameter positioned at the center of the plasma column, radial profiles of the plasma were measured with a plunging Langmuir probe at $Z = 25\text{ mm}$, and photon emission intensities were measured with an absolutely calibrated spectrograph viewing through one of two lines of sight LOS-A or LOS-D (schematic shown in Fig. 5). Ion impact energy was controlled by sample biasing.

The $\text{Al} \rightarrow \text{Al}^+$ ionization flux ($\Gamma_{\text{Al} \rightarrow \text{Al}^+}$) is calculated from the sputtered Al influx ($\Gamma_{\text{Al}}^{\text{Spt}}$), reduced by the fraction of non-ionized atoms lost to the wall (radial loss fraction) which can be estimated as $\Gamma_{\text{Al}}^{\text{Loss}} / \Gamma_{\text{Al}}^{\text{Spt}} = \exp(-L/2\lambda_{\text{mfp}})$. Here L is taken as the plateau radius of the plasma parameters ($\sim 14\text{--}18\text{ mm}$), and λ_{mfp} is the ionization mean free path for sputtered Al atoms ($4\text{--}12\text{ mm}$) as estimated from the decay length of the axial emission profiles viewed through LOS-A.

The sputtered Al atomic influx was calculated using probe-measured ion flux (Γ_i) and a He→Al sputtering yield (Y_{Al}) measured in a separate mass loss experiment. A mass loss of 2.8 mg was recorded after a 4000 s, 22.6×10^{22} ions/cm² exposure at 35 eV incident He energy resulted in a sputtering yield of 7.3×10^{-4} . This mass loss measured sputtering yield was a factor of ~30 lower than the TRIM calculated sputtering yield [6], and while similarly reduced sputtering yields have been seen before in Be erosion experiments in PISCES-B [8], the reason is not well understood. An under-estimate of the sputtering yield due to redeposition would result in an under-estimate of S/XB, but this was not expected here due to the long ionization mean free path for sputtered Al atoms and the large Al⁺ Larmor radius (~40 mm) compared to the sample radius.

The photon flux [Γ_{ph} (photons-m⁻² s⁻¹)] was calculated by integrating measured axial profile of spectral emissivity in the direction of the sputtered influx.

Thus, S/XB can be evaluated as:

$$\frac{S}{XB} = \frac{\Gamma_{Al \rightarrow Al^+}}{\Gamma_{ph}} = \frac{\Gamma_{Al}^{Spt} - \Gamma_{Al}^{Loss}}{\Gamma_{ph}} \approx \frac{\Gamma_i Y_{Al} \left[1 - e^{(-L/2\lambda_{mfp})} \right]}{\Gamma_{ph}} . \quad (4)$$

The resulting S/XB values are plotted in Fig. 6 along with theoretical S/XB (T_e , n_e) curves calculated using ionization cross-sections data available in the Atomic Data and Analysis Structure (ADAS) [4] and plane wave Born (PWB) scaling of excitation cross-sections [9]. The uncertainty in the measurement is due to the combined uncertainties in the plasma ion flux ($\pm 25\%$), sputtered atom radial loss fraction ($\pm 20\%$), and variations in the sputtering yield ($\pm 25\%$) over the course of the exposure due to an evolving nano-scale surface roughness and poor control of the surface temperature (25°–300°C). The S/XB was

extrapolated to the T_e range used in DIII-D by scaling the theoretical S/XB curves to fit the measured values.

6. Conclusions

Both net and gross erosion rates for Al were found to be similar, to within the RBS measurement error. The RBS measured Al erosion rate in DIII-D was found to be >10x higher than the erosion rate determined spectroscopically by using S/XB values measured in PISCES-B He plasmas to sample exposures in DIII-D D plasmas. Uncertainties in the measured S/XB coefficient cannot account for this discrepancy, and using the theoretically (ADAS) calculated S/XB coefficients would further increase this discrepancy. Some hypotheses proposed for this discrepancy include (i) the Al was eroded in a molecular form (such as by chemical sputtering in D plasmas) and dissociated to Al ions, (ii) the energy dependence of S/XB used to scale the PISCES-B values is steeper than predicted by ADAS, and (iii) the effect of collisional de-excitation of sputtered atoms by ions and neutrals was stronger in DIII-D than PISCES-B. There is no available collisional de-excitation cross-section data to test hypothesis (iii). Future work is proposed test hypothesis (i) and (ii) to repeat these DIII-D exposures in He plasmas, and to repeat the PISCES-B S/XB measurements in D and He plasmas over a wider range of T_e .

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Table 1. Summary of plasma parameters and erosion rates observed for Case 1 and Case 2 samples. Case 2(a-e) are sub-sets of the total sample exposure where plasma conditions, correlating to the sub-regions in Fig. 4(a-e).

Sample Case	Background Plasma			Al Erosion Summary					
	T_e (eV)	$n_e \times 10^{12} \text{ cm}^{-3}$	$\Gamma_i \times 10^{19} \text{ cm}^{-2} \text{ s}^{-1}$	Exposure Time (s)	Spect. Gross Erosion Rate (nm/s)	RBS Net Erosion Rate (nm/s)	RBS Gross Erosion Rate (nm/s)	Spect. Erosion Yield (atoms/ion)	Actual Erosion Yield (atom/ion)
Case 1 avg.	21	9.6	1.31	14.00	1.49	5.4±0.3	>6.7	0.0068	>0.02
Case 2 avg.	21	9.7	0.85	12.60	0.18	1.87±0.7	2.01±0.7	0.0013	0.014
Case 2a	46	6.7	0.81	3.15	0.13			0.0009	
Case 2b	12	10.8	0.85	8.10	0.20			0.0014	
Case 2c	17	7.8	0.90	0.45	0.17			0.0013	
Case 2d	16	10.2	1.04	0.45	0.17			0.0010	
Case 2e	12	10.9	0.90	0.45	0.14			0.0009	

Fig. 1. Coating geometries for Case 1 and Case 2 samples. Dark regions indicate Al coating areas.

Fig. 2. Peak-normalized transmission curve (dashed line) for the CCD camera All filter and a typical emission spectrum (solid line) seen by the spectrometer during Al exposure in DIII-D.

Fig. 3. False color image CCD view of DiMES, Case 2, through All filter. Bright Al emission is clearly visible from the rectangular Al coating area (dotted line) and background DI light inboard of the separatrix (dash-dot line). Dashed circles indicate regions of integration for CCD data and approximate location and size of the spectrometer view spot.

Fig. 4. Real-time spectroscopic erosion rates based on narrow band filtered CCD camera and spectrometer data for Case 2. Total exposure time is split into sub-regions of constant background plasma conditions which are summarized in Table 1.

Fig. 5. Schematic diagram of PISCES-B sample and spectrometer view geometry.

Fig. 6. S/XB for All 394.4 nm and 396.1 nm as measured in PISCES-B He plasmas. Solid lines show ADAS calculated values at $n_e = 7.5 \times 10^{-12} \text{ cm}^{-3}$ to 10^{-12} cm^{-3} , and dashed lines are the curves scaled by 5.8X to fit the average measured value.

Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5

Fig. 6