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Comparison of chemical sputtering yields for different graphites at high ion flux densities

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Abstract

Graphite is widely used inside the vacuum vessel of magnetic fusion devices and is proposed as target material for future machines like ITER. There are, however, uncertainties concerning the erosion of the material by chemical sputtering via hydrocarbon formation at high ion flux densities. We report on experiments at the plasma generator PSI-1 using a stationary quasi-neutral plasma beam. The ion flux densities used cover the range from $4 \cdot 10^{20}$ to $1.2 \cdot 10^{22}$ m^{-2} s^{-1}. They are thus filling the gap between the upper limit of ion beam experiments (10^{20} m^{-2} s^{-1}) and tokamak relevant values ($> 10^{23}$ m^{-2} s^{-1}). To suppress impurity-induced erosion the hydrogen discharges were carefully conditioned and checked for possible impurities, especially oxygen. Samples of different advanced carbon fiber composites (CFC) – including a silicon-doped one – were exposed to various plasma conditions. A calibrated mass spectrometer monitored the C_xH_y-formation in situ and the axial dependence of the CH-band intensities at 431 and 432.4 nm in front of the target was detected. We have studied the temperature dependence (250–700°C) of the erosion yields at ion flux densities up to more than 10^{22} m^{-2} s^{-1} in hydrogen discharges. Weight loss measurements and scans with an optical profilometer were used to determine the mass loss. For Si-doped CFC an erosion yield of 1% was found, which is a factor of two less than for pure CFC.

Keywords: Plasma–wall interaction simulator; Chemical erosion

1. Introduction

Using carbon as plasma facing material for fusion devices the erosion of the material by chemical sputtering via hydrocarbon formation is a crucial issue. Especially the erosion yields for high ion flux densities will be decisive for the choice of the material. There is only few experimental data to support the expected saturation of chemical erosion due to increasing recombination of hydrogen at high ion fluxes [1,2], since the upper limit for ion flux density of monoenergetic ion beams is in the order of 10^{20} m^{-2} s^{-1}. As discussed in [3,4] the very high flux densities of a fusion device can only be provided in ITER or possibly in high current arc facilities generating a quasi-neutral plasma beam.

We have performed specific experiments in the plasma generator PSI-1. First results were achieved at flux densities comparable to ion-beam experiments, but without an external control of the target temperature [5]. We have now determined the temperature dependence of the chemical erosion yields at ion flux densities above 10^{22} m^{-2} s^{-1} under controlled temperature conditions.

2. Experimental

The plasma in the PSI-1 device (Fig. 1) is generated between a heated LaB_6-cathode and an anode made from copper. It expands through the hollow anode along the axial magnetic field (0.1 T) into the target chamber onto the floating target. The plasma parameters in the target chamber are controlled by the magnetic field configuration, the neutral gas pressure (absolute pressure and pressure ratios between cathode, differential pumping stage and target chamber) and the electrical power input of the discharge.

In the experiments under discussion the electron temperature T_e was kept nearly constant at about 4–6 eV, so the particle impact energy onto the samples was smaller then 30 eV, i.e. near the threshold for physical sputtering

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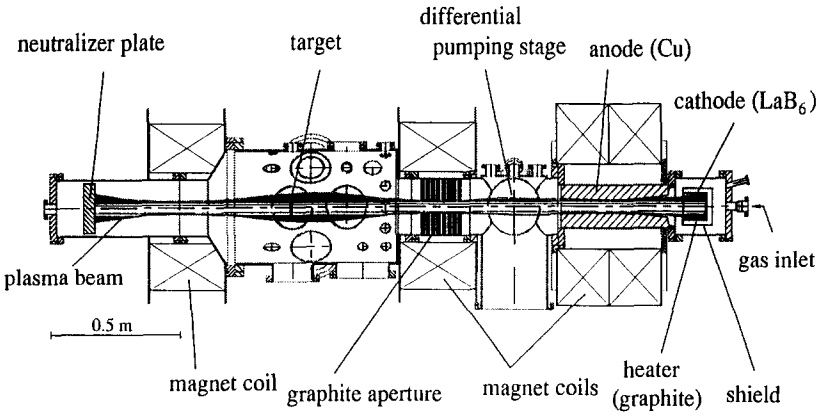

Fig. 1. Side view of the plasma generator device PSI-1.

[6]. The electron density n_e was varied between $(0.3–6) \cdot 10^{17}$ m^{-3} by changing the discharge electrical power input while keeping the neutral gas pressure constant ($1.2 \cdot 10^{-3}$ mbar in the target chamber), thus varying the ion flux density in the range $(0.08–1.2) \cdot 10^{22}$ m^{-2} s^{-1}. Under these conditions the ionization lengths for hydrocarbons are comparable to or larger than the discharge dimensions, and redeposition is not expected to play a role. Radial profiles of T_e and n_e (Fig. 2) were routinely measured 10 mm in front of the target (with and without target) by a fast scanning Langmuir probe. Measurements of the ion saturation current to the biased target confirmed the particle flux determined by the Langmuir probe. The density of neutral atomic hydrogen in the plasma column was derived from space-resolved H_α-line intensities to be less than 20% of the electron density.

Samples of different advanced carbon fiber composites — Concept II (DUNLOP) and silicon-doped NS 31 (SEP)-were exposed to various plasma conditions. The samples (20 mm diameter) were placed either concentric to the plasma column or in the edge region for 120 min each. Their temperature was controlled actively using a gas cooled sample holder and was measured by an attached thermocouple. The surface temperature distribution was monitored by IR-thermography (Fig. 3) and (in some cases) by a pyrometer. The hydrocarbon formation was monitored permanently with a residual gas analyzer. For calibration of the gas analyzer a known flux of methane was blown through a hole in a molybdenum target into the discharge under identical conditions as for graphite target exposure.

The axial dependence of the CH-band intensity at 430 nm was measured in front of the target. Weight loss measurements and scans with an optical profilometer over the etching crater area were carried out to determine the eroded mass. Additionally, surface analysis methods (SIMS, AES and SEM) were used to check for possible impurities deposited on the target surface.

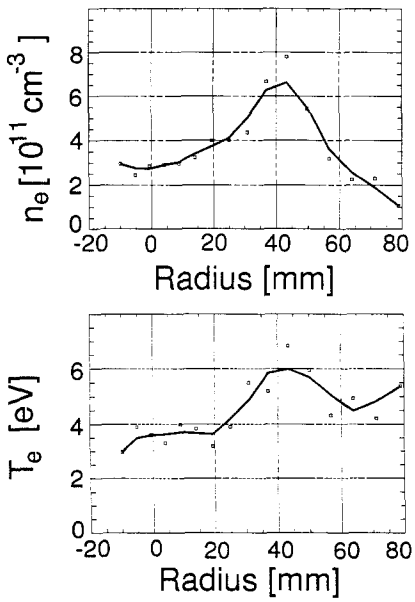

Fig. 2. Radial profiles of the electron density and temperature.

3. Results and discussion

A comparison of the erosion yields of the two CFC-materials mentioned above was performed. Fig. 4 shows

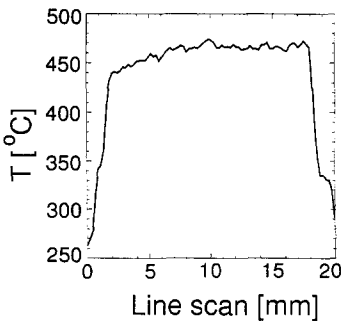

Fig. 3. Temperature scan across the sample surface.

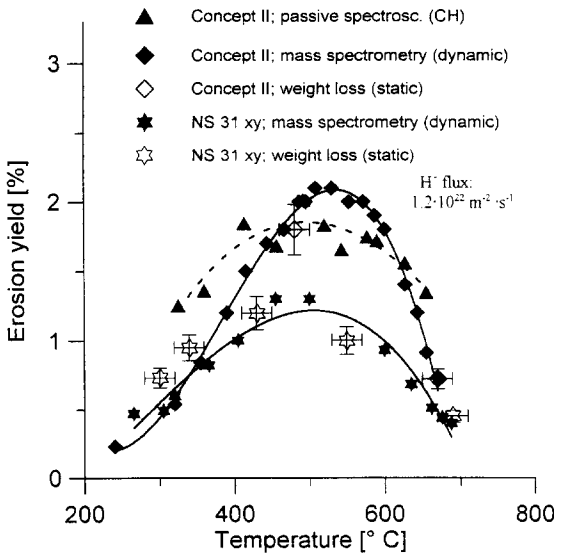

Fig. 4. Temperature dependence of the chemical erosion yield at an ion flux density of $1.2 \cdot 10^{22}$ m^{-2} s^{-1}.

the temperature dependence of the erosion yields at an ion flux density of $1.2 \cdot 10^{22}$ m^{-2} s^{-1}. The open symbols represent the values derived from mass loss measurements after long term exposure at constant temperature for both materials. The curves with the corresponding full symbols were recorded during temperature rise of the samples with the mass spectrometer ($m/e = 16$, according to CH_4^+). The curve with the triangles shows the CH-band intensity during temperature rise of the sample; it was normalized to the value of the mass spectrometer and is indicating a similar evolution with temperature as the CH_4^+ signal. All curves reveal the known maximum of the chemical erosion yields at about 500°C. The maximum values differ by about a factor of two. The lower value of approximately 1% was measured for the silicon-doped CFC NS 31.

Fig. 5 shows the temporal development of a set of mass-spectrometer traces (m/e-values) during the whole time of a NS 31 sample exposure. These measurements are necessary to distinguish the proportions of the various hydrocarbons. The final target temperature of 700°C is reached after 30 min of exposure. A matrix analysis (including further m/e-values) revealed the proportion of CH_4:C_2H_4:C_2H_6 = (85–86%):(10–12%):(2–4%). No indication for heavier hydrocarbons was found. These proportions are neither dependent on the sample temperature nor on the ion flux density. During exposure of the silicon-doped CFC no indication for SiH_4-formation was observed.

There is a tendency of reduction of the chemical erosion yields for both materials with increasing ion flux density, but further measurements are needed to confirm this statement.

Information concerning the purity of the plasma was obtained from gas analysis and spectroscopy. The residual gas pressure of the vacuum system is $3 \cdot 10^{-8}$ mbar. Spectroscopic data give no indication for any impurities. For a further assessment of the purity of the plasma, sputtering experiments with a biased tungsten-target were carried out. The threshold energies for W-sputtering are 447 eV and 209 eV for hydrogen and deuterium, respectively. For all heavier ions the threshold energy is even less. Biasing the target to −350 V, no tungsten could be detected by laser-induced fluorescence (LIF) and passive spectroscopy at 400.8 nm in hydrogen discharges, whereas at bias voltages over −200 V in deuterium discharges the tungsten-signal increased with increasing bias voltage (Fig. 6). A similar effect would have been expected for traces of heavier ions at even lower bias voltage.

Because of the high yields for chemical sputtering of carbon by oxygen, the influence of this impurity was studied separately. An addition of 0.2% oxygen into the H-discharge was clearly detected by visible spectroscopy

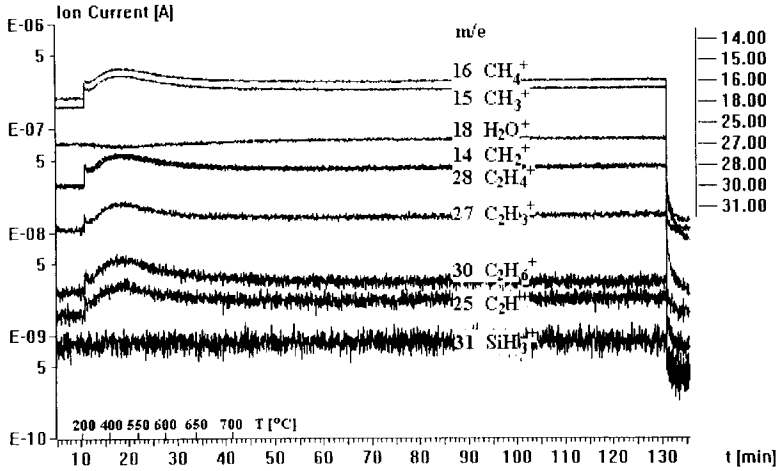

Fig. 5. Temporal development of m/e-values during sample exposure. The sample was inserted 11 min after the start of the recording.

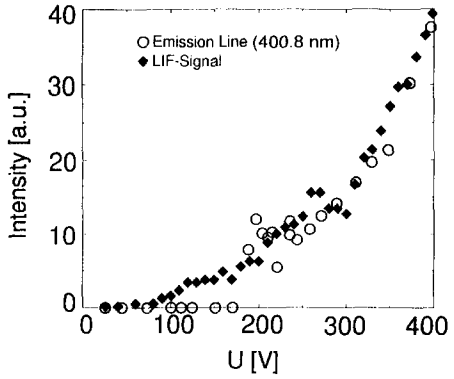

Fig. 6. LIF and emission-line signals of tungsten as a function of the bias voltage in a deuterium discharge.

and changed the intensities and the ratio of the signals at $m/e = 16$ (CH_4^+) and 28 ($C_2H_4^+$ and CO^+) in the gas analysis remarkably. It is thus concluded, that the oxygen content in the hydrogen discharge is normally well below 0.1%. and hence of no importance. However, the oxygen content is kept as low as possible during the measurement.

4. Summary

Advanced Carbon Fiber Composites, DUNLOP Concept II and silicon-doped SEP NS 31, chosen for possible application in ITER, were examined concerning their chemical sputtering yields in a stationary high power hydrogen plasma. At an ion flux density of $1.2 \cdot 10^{22}$ m^{-2} s^{-1} the temperature dependence of the chemical sputtering yield was measured dynamically (during temperature rise) with a calibrated mass spectrometer and the CH-band intensity as well as during the stationary regime (at constant temperature) by the mass loss method. Both values show good agreement. The absolute erosion yields of the investigated materials differ by almost a factor of two, being maximal about 2% for Concept II (DUNLOP) and 1% for NS 31 (SEP) at a temperature of 500°C. Further measurements at higher densities (10^{23} m^{-2} s^{-1}) are planned to investigate the reduction of chemical sputtering with increasing influx.

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