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# Removal of carbon layers by oxygen glow discharges in TEXTOR

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## Abstract

Glow discharge cleaning (GDC) in mixtures of  $O_2$  and He was applied in TEXTOR for about 4 h to study the removal of redeposited carbon layers. The behaviour of the formation of volatile carbon oxides (CO and CO<sub>2</sub>) and its dependence on the  $O_2$  injection rate and on the addition of He into the  $O_2$  GDC are described. Carbon (5.22 g) was removed from TEXTOR in about 4 h GDC corresponding to a carbon removal rate of ~2.1 × 10<sup>19</sup> C/s. Plasma recovery was obtained after wall cleaning in H<sub>2</sub> GDC and finally by boronisation. © 2007 Elsevier B.V. All rights reserved.

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## 1. Introduction

The development of methods to remove fuel from in-vessel components [1] and the demonstration of their tokamak compatibility is of highest priority in fusion research. Removal of redeposited C-layers was done previously in TEXTOR by molecular oxygen venting [2] but this requires wall temperatures in excess of ~550 K while oxidation by energetic ion impact does not depend significantly on wall temperatures [2]. This contribution reports the removal of carbon layers from TEXTOR by conventional glow discharge conditioning (GDC) in He/O<sub>2</sub> gas mixtures and the behaviour of plasma recovery afterwards. An overview on oxidation of carbon layers can be found in [3]. In a parallel activity, carbon removal by ICRH plasma induced oxidation in TEXTOR is under investigation and reported in [4]. Removal of C layers by GDC and ICRH is being intensively investigated in HT-7 [5].

### 2. Experimental

The TEXTOR wall is formed by various limiters made from fine grain graphite with a total area of about  $10 \text{ m}^2$  and an inconel liner acting as first wall with an area of about  $35 \text{ m}^2$ . All plasma facing surfaces are routinely covered with a boron film of 150-200 nm thickness. The liner temperature was

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set to ITER-like temperatures of 200 °C. An RF at 13.2 MHz with a power of ~250 W was applied on four antennas (acting as anodes) on which a DC bias voltage was overlaid (RF supported GDC) and the TEXTOR wall represents the cathode. A stabilized discharge current of 6 A (~10<sup>14</sup> ions/ cm<sup>2</sup>) was used. The DC bias voltage was typically between 400 V and 500 V. TEXTOR was pumped with the ALT pump-limiter at resulting pumping rates of 485, 517, 413 l/s for O<sub>2</sub>, CO and CO<sub>2</sub>, respectively. The total pressure in TEXTOR was measured using a Baratron capacitance manometer with an estimated accuracy of about 5%.

The formation of volatile reaction products was measured using several differentially pumped quadrupole mass spectrometers (QMS). The overall sensitivities of the QMS were determined from measured QMS ion currents and TEXTOR pressures from Baratron gauges with different gases injected in TEXTOR. Removal rates of volatile reaction products were determined from the partial pressures in TEXTOR and the pumping rates.

### 3. Results

Fig. 1 shows the behaviour of the injection of  $O_2$ and He and the partial pressures of O<sub>2</sub>, CO, CO<sub>2</sub> both during GDC switched on and off for the whole GDC treatment. At the beginning of the GDC, all the injected  $O_2$  is consumed and CO appears suddenly, while the  $CO_2$  and  $O_2$  pressures rise slowly with progressing GDC. With increasing O<sub>2</sub> flow rate (all at given total ion current), the  $O_2$  partial pressure increases also. As seen in Fig. 2, the CO production does not depend on the O<sub>2</sub> flow rate while the CO<sub>2</sub> production increases with increasing O<sub>2</sub> flow rate . Since the O ion flux was kept constant during the increase of O<sub>2</sub> injection, this result indicates that the CO production is mainly induced by the O ion flux (kept constant) while the increase of the  $CO_2$  production indicates the importance of oxygen atoms which increase with increasing  $O_2$ pressure. The sum of CO and CO<sub>2</sub> production saturates at a moderate O injection of about  $8 \times 10^{19}$  O/s rates showing an advantage of operation at lower O flow rates at which most of the O<sub>2</sub> is converted to CO and CO<sub>2</sub>.

He gas was added to the  $O_2$  GDC to investigate the influence of He on the GDC efficiency. In the range of He/O<sub>2</sub> pressures investigated, no dependence of the CO and CO<sub>2</sub> production on He injection could be observed, as seen in Fig. 3(a) and (b).



Fig. 1. Temporal evolution of the  $O_2$  and He injection rates and the partial pressures of CO,  $CO_2$  and  $O_2$ . The shadowed areas indicate GDC on/off.



Fig. 2. Dependence of CO,  $CO_2$  and  $O_2$  partial pressures on the O injection rate.

The balance of oxygen appearing in partial pressures of CO, CO<sub>2</sub> and O<sub>2</sub> can account within the accuracy for all the injected oxygen, except at the very beginning of the GDC. Thus no significant other oxygen sinks except O<sub>2</sub>, CO and CO<sub>2</sub> need be assumed indicating that no significant amount of oxygen is converted into water molecules.



Fig. 3. (a): Dependence of CO (a) and CO<sub>2</sub>; (b) production on He-injection for different O injection rates. The total GDC ion current is kept constant.

However, this conclusion needs further confirmation in future experiments.

The release of HD (only HD could be measured precisely while  $D_2$  is overlaid by He and  $H_2$  has a large background) accounts for about 2–3% of the sum of CO and CO<sub>2</sub> release and about 10% of the mass 19 signal (HDO). The fraction of H, D retained in C layers in TEXTOR is typically 0.1–0.2, indicating that the preferred release of D, H during removal of the C layer occurs in form of water molecules, as observed also in oxygen beam experiments [3].

The raw water signals exhibit mass 19 (HDO) of about 20% of the H<sub>2</sub>O signal while TEXTOR codeposits contains typically 1/1 H/D. This shows that a significant part of the H<sub>2</sub>O partial pressure rise during the GDC is not due to the removal of C-layers but produced on other areas and/or in the mass spectrometer itself. The HDO signal is much more sensitive to changes in GDC conditions, indicating indeed, that it is to a large extent produced by O<sub>2</sub> GDC removal of C layers.

The integral of CO and CO<sub>2</sub> production sums up to about 5.22 g of carbon giving a carbon removal rate of  $2.1 \times 10^{19}$  C/s. This corresponds to an averaged removal of  $6 \times 10^{13}$  C/cm<sup>2</sup> s. On the re-deposition areas of the TEXTOR limiters, averaged C deposition rates are about  $1.5 \times 10^{16}$  C/cm<sup>2</sup> s (2.5 nm/s) [6], about a factor of 250 above the averaged C-removal rate measured here.

Si probes have been coated ex situ with an amorphous carbon layer (a-C:H) and exposed at the wall position together with an amorphous boron layer taken from TEXTOR routine boronisations. a-C:H layers of 190 nm have been totally removed while the amorphous boron layer was eroded by about 20–30 nm and showed some uptake of oxygen.

Fig. 4 shows the sputter Auger depth profiles of the a-C:H samples cleaned with glow discharge in TEXTOR and the identical coated samples, which were not exposed to the glow. These data give a



Fig. 4. Sputter Auger depth profiling of witness probes exposed to GDC treatment in TEXTOR and non-exposed reference coated samples.

lower limit of the C-removal rate of  $1 \times 10^{14}$  C/ cm<sup>2</sup> s, extrapolating to  $3.8 \times 10^{19}$  C/s for the whole wall area of TEXTOR. This lower erosion limit is in agreement with the rate deduced from the mass spectroscopy.

## 4. Wall cleaning after the O<sub>2</sub> GDC treatment

After the GDC, immediate plasma recovery has failed (only one attempt was done though), similar as observed already in previous oxygen experiments in TEXTOR. Wall cleaning was done in H<sub>2</sub> GDC for 66 h, followed by a short GDC in He for about 30 min. An overall amount of  $4.9 \times 10^{22}$  O-atoms was pumped out in form of CO and CO<sub>2</sub>, corresponding to an averaged O-retention of about  $1.7 \times 10^{17}$  O/cm<sup>2</sup>, significantly larger than the amount of O-retention estimated from O-pressure balances. This indicates that a significant amount of released CO and CO2 during the H2 GDC does not originate from the GDC or that the amount of retained oxygen from pressure balance during O<sub>2</sub> GDC is significantly underestimated. A definitive answer cannot be given here and require additional experiments.

### 5. Plasma recovery

After the H<sub>2</sub> GDC wall cleaning, standard ohmic TEXTOR current ramp up was achieved, but with C and O impurity levels about 3–5 times higher than before. This is shown in Fig. 5. However, the ohmic discharges disrupted in a later stage of the discharge, which was attributed, however, to a failure in the TEXTOR plasma positioning system leading to unwanted wall contact. The influence of enhanced impurity levels from the O<sub>2</sub> GDC could not clearly be separated. The repair of this failure and boronisation of TEXTOR was performed in parallel resulting in successful plasma operation with carbon and oxygen impurity levels very similar as observed before, see Fig. 5.

## 6. Summary

Carbon layers have been removed from TEX-TOR PFCs by glow discharge cleaning in mixtures of  $O_2$  and He. The  $O_2$  GDC transforms injected



Fig. 5. Normalised  $O/H_{\alpha}$ , fluxes before, after  $H_2$  GDC cleaning and after boronisation in the ohmic early phase of the discharges.

oxygen to CO and CO<sub>2</sub> with an efficiency of 100% at lower injection rates with the conversion efficiency decreasing with time and increasing oxygen injection. CO formation reaches saturation at low oxygen flow rate while the CO<sub>2</sub> production continuously increases. The addition of He to the O<sub>2</sub> GDC does not influence the CO and CO<sub>2</sub> production. Carbon (5.22 g) was removed from TEXTOR in about 4 h GDC corresponding to a rate of  $\sim 2.1 \times 10^{19}$  C/s. The averaged C removal rate is in good agreement with the lower limit of erosion deduced from the erosion of C-coated witness samples. Immediate plasma built up after the O<sub>2</sub> GDC treatment failed but ohmic plasma start up was possible after 66 h of H<sub>2</sub> GDC cleaning accompanied by about 3-5 times increased O and C levels. After boronisation, the oxygen and carbon content decreased to the routine levels observed in TEX-TOR. No obvious damage to any component inside TEXTOR has been observed.

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