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### Oxygen glow discharge experiment to remove deposited layers and to release trapped hydrogen isotopes in HT-7 superconducting tokamak

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#### Abstract

An experiment to remove re-deposited layers and to release hydrogen using a glow discharge in oxygen (O-GDC) has been performed in the HT-7 superconducting tokamak. In the absence of magnetic fields, the O-GDC wall conditioning had produced rapid, controlled co-deposit removal. Average removal rates,  $5.2 \times 10^{22}$  H-atoms/h,  $5.65 \times 10^{21}$  D-atoms/h and  $5.53 \times 10^{22}$  C-atoms/h, respectively, were obtained during 145 min O-GDC experiment in the pressure range 0.5–1.5 Pa. The corresponding removal rate of co-deposited films was ~1.19 µm/day (26.5 g/day for carbon) based on an area of 12 m<sup>2</sup>. Compared to thermo-oxidation and O-ICR experiment, high pressure O-GDC wall conditioning promoted the oxidation and improved the C and D atoms removal. In the O-GDC experiment, the removal rates of H-atoms and D-atoms as H<sub>2</sub>O, HDO and D<sub>2</sub>O were higher than that of H<sub>2</sub> and D<sub>2</sub> by factors of about 20 and 50, respectively. During the 145 min O-GDC experiment, about 14.5% O-atoms were converted into carbon oxides and hydroxides, and about  $5.37 \times 10^{22}$  O-atoms were adsorbed on the walls corresponding to a coverage of  $4.5 \times 10^{21}$  O/m<sup>2</sup> on an wall area of 12 m<sup>2</sup>. In a 100 min helium glow discharge (He-GDC) following the O-GDC experiment,  $1.53 \times 10^{22}$  O-atoms, about 28.5% oxygen retained on the walls, were removed. The removal rate of H-atoms in He-GDC cleaning after O-GDC experiment was lower than that in He-GDC cleaning before O-GDC experiment, which indicates that the O-GDC wall conditioning had effectively reduced hydrogen retention on the walls.

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#### 0. Introduction

Graphite, for its low Z and good thermomechanical properties, is the most extensively used

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as first wall material in present day fusion devices. It is used for both limiters and divertors, which are in direct contact with the plasma, and as such, shelter the remaining vessel walls. A major drawback of graphite, however, is its erosion and hydrogen retention behavior when exposed to high fluxes of plasma particles. The eroded carbon atoms, together with hydrogen isotopes from the plasma,

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redeposit on nearby surfaces to form hydrogen-rich carbon films, usually referred to as co-deposited amorphous C:H films.

The long-term retention of tritium fuel in the surface or bulk of plasma-facing materials in fusion devices is one of the major problems in fusion technology. It has been concluded from various investigations that the dominant mechanism for hydrogen retention in tokamak with carbon walls will be co-deposition of eroded carbon with deuterium on walls [1,2]. The retention of a large fraction of input hydrogen isotopes was observed in TFTR [3], TEX-TOR [4] and JET [5,6]. The ability to remove tritium from amorphous tritiated carbon layers, a-C:T, codeposited in the next generation tokamaks, such as ITER, will have an important impact on machine operation. If in situ co-deposit removal techniques are fast and effective, both in terms of T removal and plasma performance recovery after cleanup, the long-term T retention/inventory problem could be mitigated [1].

In principle, tritium could be removed from materials via thermal desorption or ion-induced desorption. However, the temperature requirement for thermal desorption from C based materials is much higher than the design temperatures for most plasma-facing components (PFCs) [1,7,8]. Ioninduced desorption at room temperature is limited to depths corresponding to the ion range, typically a few nanometers for plasma discharges, and therefore, will not reach the trapped T in the tens of micrometers thick co-deposits. Thus, the removal of T from thick a-C:T co-deposits may require the removal of the co-deposits themselves. This can be done by chemical and/or plasma assisted oxidizing reactions in the presence of oxygen or, alternatively, via abrasive/mechanical techniques [1]. Oxygen free techniques for tritium removal have been proposed but further development, such as CO<sub>2</sub> pellet blast cleaning, laser surface heating and cathodic arc cleaning [8–11], are needed.

Extensive laboratory studies of hydrogen isotope removal on exposing co-deposited films and D implanted graphite to air or oxygen have shown that it is an effectively methods to remove codeposits with a low temperature required [12– 17,19]. At these temperatures, the bulk material of graphite was not significantly attacked by oxygen. Some oxidation experiment have been performed in tokamaks, but very few. The first deliberate thermo-oxidation experiments in a tokamak to remove co-deposits on hot walls were performed in TEXTOR [2]. The behavior of the  $CO_2/CO$  ratio is agreement with laboratory observations [17]. In the TFTR tokamak, a He/O glow discharge cleaning procedure was applied to aid in the removal of tritium with minimal O contamination [20].

Very few experiments involving the injection of oxygen directly into tokamak for the purpose of removing co-deposits have been reported. The oxidation experiments performed in the HT-7 superconducting tokamak that includes oxidation associated with ion cyclotron resonance discharge [21] and glow discharge cleaning (O-ICR and O-GDC), and thermo-oxidation with molecular oxygen [22]. The objective of this series of experiments is to directly compare the removal rates of hydrogen and carbon for different oxidation procedures. O-GDC experiments have been performed in the HT-7 superconducting tokamak in absence of permanent toroidal magnetic. The O-GDC experiment has been done after the O-ICR experiment [21], but prior to thermo-oxidation [22]. Before the oxidation experiments, the HT-7 tokamak with the carbon limiter had been operated in deuterium for more than 10000 plasmas. The O-ICR experiment was performed in HT-7 in the presence of a permanent magnetic field of 1.5-2.0 T. The influence of ICRH power and filling pressure on hydrogen and carbon removal rates was analyzed. The highest removal rates of H, D and C-atoms up to  $2.64 \times 10^{22}$ ,  $7.76 \times 10^{21}$ and  $1.49 \times 10^{22}$  atoms/h, respectively, were obtained in 40 kW  $9 \times 10^{-2}$  Pa O-ICR cleaning, corresponding to the removal rate of co-deposits of about 317 nm/day (7.2 g/day for carbon). After 50 min He-ICR cleaning followed the O-ICR experiment, normal plasma discharges could be recovered after a few hours of disruptive plasma discharges.

In this paper, detail results of O-GDC experiment are introduced. Emphasis in the O-GDC experiment is on the hydrogen and carbon removal rates with different oxygen pressures. He-GDC cleanings were done before and after the O-GDC experiment with two motivations. The first was to study how to quickly remove the oxygen retained on the walls during the O-GDC experiment. The second was to compare the hydrogen removal rate during He-GDC cleaning before and after the O-GDC experiment. This may be used as a measurement of the effectiveness of O-GDC in the removal of hydrogen.

#### 1. Experiment setup and procedures

#### 1.1. Experiment setup

HT-7 is a medium sized superconducting tokamak with a major radius of 1.22 m and a minor plasma radius of 27 cm, which had been operated from 1994 in Hefei, PR China. Its main purpose is to explore high performance plasma operation under steady state conditions. The plasma is limited with two toroidal limiters (located at top and bottom in the vessel) and one belt limiter (located at mid-plane at high filed side) [23]. The total graphite plasma-facing surface area of the HT-7 limiters is about 2.35 m<sup>2</sup>. All plasma-facing materials for limiters are made from doped graphite (GBST: 1%B, 2.5%Si, 7.5%Ti) with a 100 µm SiC coating [23,24]. The rest of the plasma-facing surface is formed by a stainless steel liner within a metallic torus with r = 33 cm. The effective plasma-facing area of limiters and liner is about 12 m<sup>2</sup>. The total volume of HT-7 is about  $4.85 \text{ m}^3$ . The liner may be heated by direct current flow and the limiters are heated by thermal radiation. A temperature difference of typically  $\pm 20$  K is measured at different locations on the liner. The temperatures of the limiters are typically about 30 K lower than the liner temperature during baking.

The O-GDC experiments have been performed with pure oxygen (99.95%). The system includes a filling tank, the HT-7 vessel and a differential pump system. Three turbo pumps were used for the particle exhaust during O-GDC cleanings. The pressure of oxygen in the filling tank was monitored by a APR262 compact Piezo gauge. Two PKR251 penning gauges were used to monitor pressure in the HT-7 vessel and the differential pumping chamber respectively. A local quadruple mass spectrometer (QMS) is used to analyze the residual vacuum background at intervals between cleaning procedures. The differential pumping system is composed of a turbo pump, one PKR251 gauge and a second QMS. This QMS was used for dynamical residual gas analysis during cleaning. The differential pump system was connected to the main HT-7 chamber by a bellows tube, 1 m in length and 40 mm in diameter, which was baked to above 370 K during the oxidation experiment. The total pressure was feedback controlled. Temperatures in the limiter tiles and on the liner were measured by 12 thermocouples. All gauges were calibrated with O2, N2, Ar and He. The pump speed was measured with particle balance method. The pump speeds of  $O_2$ ,  $N_2$  are about 700 l/s and that of  $H_2$  is about 650 l/s with an error of about 20% when the pressure in the vessel is 0.5 Pa.

#### 1.2. Procedures

Before the O-GDC experiment, the liner was heated to about 460–490 K by direct current flow and the limiters were heated to about 380–400 K by thermal radiation. Due to an improvement of the heat transfer between limiters and liners with the filling gas, the temperature of the limiter is 402–425 K and that of the liners is 435–470 K during the O-GDC experiment.

The total pressure in the O-GDC experiment was scanned from about 0.5 Pa to 1.5Pa in both ascending and descending order. The O-GDC experiment was operated for about 145 min. It takes about 10 min to explore the lowest pressure of oxygen for glow discharge ignition. At each oxygen pressure the O-GDC wall conditioning lasted about 15 min. Two molybdenum cathodes were used for the O-GDC experiment, each with a voltage of about 270 V and a current of 2 A. Three turbo pumps were used for particles exhaust.

From laboratory experiments on the oxidation of carbon films, it has been concluded that the layer was removed by the formation of CO and CO<sub>2</sub> and the incorporated hydrogen is released in the form of water molecules [13–17]. The release of hydrogenic species in the form of water is expected as 18, 19 and 20 (which has to be interpreted as H<sub>2</sub>O, HDO, D<sub>2</sub>O) in case of <sup>16</sup>O<sub>2</sub> injection.

#### 2. Results

#### 2.1. Ignition of oxygen glow discharge

After filling the torus with oxygen, a typical pattern of gas pressure transients was observed. The partial pressures (defined as P.P) of oxides (such as H<sub>2</sub>O, HDO, CO e.g.) increased while those H<sub>2</sub> and D<sub>2</sub> decreased. In the torus of HT-7, 10 Pa of O<sub>2</sub> was needed to ignite the oxygen glow discharge. After ignition, if the oxygen pressure decreased below 0.2 Pa, the O-GDC would be quenched. In the range of 0.2–0.5 Pa, the light of emitted by the discharge was the brightest, but very unstable. Fig. 1 shows the temporal evolutions of the P.P of oxides and hydrogen in the differential chamber while adjusting the oxygen pressure in the range of



Fig. 1. Time evolution of the partial pressures of molecules in the differential pumping chamber during the initial period of adjusting oxygen pressure in the range of 0.2–0.5 Pa for O-GDC conditioning.

0.2–0.5 Pa. The color and brightness of the discharge light changed very fast. The changes in color may be related to the changing gas composition in the plasma. At the start of O-GDC wall conditioning, the P.Ps of released molecules except of  $D_2$ increased rapidly whereas that of oxygen decreased. At the quench of the discharge, the P.P of oxygen increased and that of other molecules decreased. This indicated that the glow discharge had promoted oxidation.

#### 2.2. O-GDC wall conditioning

During the O-GDC wall conditioning, oxygen pressures were scanned in both ascending and descending order to test the influence of oxygen pressure on the removal rates of H, D and C. For a certain oxygen pressure, O-GDC wall conditioning was maintained for about 15 min. The temporal evolutions of the P.P of molecules with oxygen pressure in both ascending and descending experiments are similar. Fig. 2 shows the temporal evolution of the P.P of molecules with increasing oxygen pressure during about 80 min of O-GDC wall conditioning. The P.P of O<sub>2</sub> during O-GDC wall conditioning is significantly higher than all other species, which means that most of oxygen was still pumped directly as O<sub>2</sub>.

The P.P of  $H_2$  and  $D_2$  decreased with increasing oxygen pressure while the P.P of HDO and  $D_2O$ increased. However, the P.P of  $H_2O$  also decreased with increasing oxygen pressure for oxygen pressure lower than 0.9 Pa and increased with increasing oxygen pressure for oxygen pressure higher than 0.9 Pa.

The P.Ps of CO and CO<sub>2</sub> had an obvious opposite trend when increasing the oxygen pressure. The rate of increase of the P.P of CO<sub>2</sub> was greater than the rate of decrease of the P.P of CO. Thus, O-GDC wall conditioning with high pressures favored the formation of CO<sub>2</sub> over that of CO and that the CO was transferred to CO<sub>2</sub>.

#### 2.3. Removal rates of molecules and H, D, C atoms

The removal rate of gases was evaluated from the P.Ps of the gases and the pumping speed (S). The pumping speed of  $D_2$  was assumed to be the same as that of  $H_2$  obtained in the calibration and the pump speeds of  $H_2O$ , HDO,  $D_2O$ , CO and CO<sub>2</sub> were calculated by the following Eq. (1):

$$S_{\rm gas} = S_{\rm N_2} \cdot \sqrt{\frac{M_{\rm N_2}}{M_{\rm gas}}}.$$
 (1)

The removal rate (RR) of gases were evaluated with the ideal gas law as following Eq. (2):

$$\mathbf{R}\mathbf{R} = \mathbf{P}.\mathbf{P}\cdot S\cdot N_0/R\cdot T. \tag{2}$$

Molar gas constant is R = 8.314 Pa m<sup>3</sup> mol<sup>-1</sup> K<sup>-1</sup>; the  $N_0$  is  $= 6.02 \times 10^{23}$  molecules/mol; *T*: gas temperature.



Fig. 2. Temporal evolution of the partial pressures of molecules during O-GDC conditioning (with oxygen pressure scanned in ascending order in about 80 min. For a given oxygen pressure the O-GDC conditioning lasted about  $\sim$ 15 min).

The removal rates of H, D, C atoms were calculated from the sum of their different forms, for example, the H atoms removal rate is the sum of the removal rates of  $H_2 \times 2 + H_2O \times 2 + HDO$  as the following Eq. (3):

$$\mathbf{RR}_{\text{H-atoms}} = 2 \times P_{\text{H}_2} \cdot S_{\text{H}_2} \cdot N_0 / R \cdot T$$
  
+ 2 × P\_{\text{H}\_2\text{O}} \cdot S\_{\text{H}\_2\text{O}} \cdot N\_0 / R \cdot T \times 2  
+ P\_{\text{HDO}} \cdot S\_{\text{HDO}} \cdot N\_0 / R \cdot T. (3)

Fig. 3 shows the relationship between the removal rates of a few molecules and the oxygen pressure during O-GDC wall conditioning. The data were taken from the entire 145 min O-GDC experiment.

The highest removal rates of H<sub>2</sub>, D<sub>2</sub>, H<sub>2</sub>O and CO were about  $2.5 \times 10^{21}$ ,  $1 \times 10^{20}$ ,  $2.25 \times 10^{22}$  and  $4.5 \times 10^{22}$  molecules/h, respectively, at an oxygen pressure of 0.35 Pa. The lowest removal rates of H<sub>2</sub> and H<sub>2</sub>O were about  $7.5 \times 10^{20}$  and  $1.76 \times 10^{22}$  molecules/h at an oxygen pressure of 1 Pa. The lowest removal rates of D<sub>2</sub> and CO were about  $2.4 \times 10^{19}$  and  $2.8 \times 10^{22}$  molecules/h at an oxygen pressure of 1.2 Pa. The removal rates of HDO, D<sub>2</sub>O and CO<sub>2</sub> increased from  $\sim 3 \times 10^{21}$ ,  $5 \times 10^{20}$  and  $1 \times 10^{22}$  molecules/h at an oxygen pressure of 0.3 Pa to  $\sim 5 \times 10^{21}$ ,  $1 \times 10^{21}$  and  $2.4 \times 10^{22}$  molecules/h, respectively, at an oxygen pressure of 1.2 Pa.

H-atoms and D-atoms were removed mainly as oxides, such as  $H_2O$ , HDO and  $D_2O$ . The removal rates of H and D exhausted as molecular hydrogen ( $H_2$  or  $D_2$ ) were lower by factors of about 20 and 50, respectively. The C-atom and D-atom removal rate increased with increasing oxygen pressure. The highest removal rate of D-atoms and C-atoms,  $7.5 \times 10^{21}$  D-atoms/h and  $5.5 \times 10^{22}$  C-atoms/h, respectively, were obtained during O-GDC wall conditioning with 1.2 Pa oxygen. Even though the removal rate of CO decreased with increasing oxygen pressure, the removal rate of CO<sub>2</sub> increased faster than the CO removal rate decreased. Similarly, the D-oxide removal rate increased faster than the D<sub>2</sub> removal rate decreased.

The H-atoms removal rate, however, was highest for the lowest pressure cleaning. Over a large range of oxygen pressure, the removal rates of H<sub>2</sub> and its oxide both decreased. The highest removal rate of H-atoms,  $6 \times 10^{22}$  H-atoms/h, was obtained during O-GDC wall conditioning with 0.3 Pa oxygen. The lowest removal rate,  $4 \times 10^{22}$  H-atoms/h, was obtained during O-GDC wall conditioning with 0.75 Pa oxygen.

Since the objective of the experiment was to remove co-deposited layers, which are proposed primarily of carbon and deuterium, the results indicate that high oxygen pressure O-GDC wall conditioning is most effective.

## 2.4. Hydrogen and co-deposited layers removal, global oxygen balance

During the total 145 min O-GDC experiment, about  $1.24 \times 10^{23}$  H-atoms,  $1.34 \times 10^{22}$  D-atoms



Fig. 3. Dependence of particle removal rates on oxygen pressure during O-GDC conditioning. (The data were directly taken from the entire 145 min O-GDC experiment directly.)

and  $1.32 \times 10^{23}$  C-atoms, corresponding to 205 mg of hydrogen, 45 mg of deuterium and 2.6 g of carbon, were removed. Corresponding average removal rates were approximately  $5.2 \times 10^{22}$  H-atoms/h,  $5.65 \times 10^{21}$  D-atoms/h and  $5.53 \times 10^{22}$  C-atoms/h, respectively. Assuming the density of co-deposited films is about 1900 kg/m<sup>3</sup>, on average, about 120 nm of codeposited film was removed over an area of 12 m<sup>2</sup>. The corresponding removal rate of the film is about 1.19 µm/day (26.5 g/day of carbon).

In the O-GDC experiment, a total of  $1.58 \times 10^{24}$ O-atoms were injected and  $1.53 \times 10^{24}$  O-atoms were removed from the torus through the vacuum pumps. The corresponding oxygen filling rate and removal rate were up to  $6.54 \times 10^{23}$  and  $6.33 \times$  $10^{23}$  O-atoms/h, respectively. About  $1.3 \times 10^{24}$ O-atoms were removed as O2, corresponding to 82.2% of the injected oxygen. About 14.5% O-atoms were removed through combining with C or H(D);  $6.11 \times 10^{22}$  O-atoms were converted to carbon oxides and  $1.69 \times 10^{23}$  O-atoms are converted into hydroxides, respectively. The remainder of the injected oxygen,  $5.37 \times 10^{22}$  O-atoms, was adsorbed on the chamber walls corresponding to 1.43 g of oxygen. The absorption corresponds to a coverage of  $4.5 \times 10^{21}$  O/m<sup>2</sup> on an wall area of 12 m<sup>2</sup>.

#### 2.5. Wall recovery by He-GDC cleaning

Standard He-GDC cleaning following the O-GDC experiment was used to remove the residual

oxygen from the walls. Fig. 4 shows the time evolution of the P.P of molecules during He-GDC cleaning. The cleaning pressure was  $5 \times 10^{-1}$  Pa; Cleaning time was 100 min. All detected gases were released immediately upon starting He-GDC and all release rates decreased during the cleaning.

In the He-GDC cleaning,  $1.53 \times 10^{22}$  O-atoms, or about 28.5% of the absorbed oxygen, was removed. Of the removed O-atoms, 78%, 28% and 3.2% was removed as carbon oxide, hydro-oxide and O<sub>2</sub>, respectively. At the end of He-GDC, there are about  $3.84 \times 10^{22}$  atoms adsorbed on the walls, and the adsorption corresponds to a coverage of  $3.2 \times 10^{21}$  O/m<sup>2</sup> on an area of 12 m<sup>2</sup>. The average removal rate of O-atoms is  $9.2 \times 10^{21}$  atoms/h.

# 2.6. Comparison between He-GDC cleanings and O-GDC wall conditioning

Two special standard He-GDC cleanings, before and after O-GDC, were done to compare the removal of impurities. Fig. 5 shows the removal rate of H-atoms and C-atoms during the two He-GDC cleanings and also during the O-GDC wall conditioning. Due to the near identical mass of helium and deuterium, the comparison of D-atoms removal rates in those cleanings was not included.

In the both He-GDC cleanings, the removal rates of H and C atoms were lower than that in the O-GDC conditioning. Specifically, the removal rate of C-atoms in O-GDC conditioning was higher than



Fig. 4. Temporary evolution of the partial pressures of molecules in the He-GDC cleaning. (The helium pressure is  $5 \times 10^{-1}$  Pa.)



Fig. 5. Removal rate of H-atoms and C-atoms during the two He-GDC cleanings and the O-GDC wall conditioning: (a) H-atoms removal rate and (b) C-atoms removal rate.

that in either He-GDC cleanings by a factor of about 25. This clearly indicates the effectiveness of O-GDC conditioning for the removal hydrogen and co-deposits as compared to He-GDC cleaning.

The removal rate of H-atoms in He-GDC cleaning before O-GDC experiment is much higher than that in the He-GDC cleaning after the O-GDC experiment. Because the same parameter cleaning were used for both He-GDC experiments, the decrease in the removal rate of H-atoms means that the hydrogen was largely removed by the O-GDC wall conditioning. On the other hand, the removal rate of C-atoms in both He-GDC cleanings was almost identical, indicating that during the O-GDC wall conditioning, the co-deposits were not completely removed.

#### 3. Discussions

The conditions for the oxidation experiment in HT-7 are very complex. The hot area of the wall components facing the plasma is about  $12 \text{ m}^2$  with a similar area not facing the plasma. The temperature distribution of the components facing the

plasma, limiters and liner, varied from 400 K to 470 K between 12 thermocouples. In addition, due to HT-7 being a superconducting device, the vacuum vessel has an even large area restricted to temperature less 370 K. Before the O-GDC experiment, the distribution and the character of the carbon deposit are not clear. After exposing the HT-7 vessel, carbon deposits were observed mainly on the surface of the graphite tiles with very little deposition observed on the liner.

In the O-GDC experiment, it can be concluded that carbon layers were removed by formation of CO and CO<sub>2</sub> and that the incorporated hydrogen was released primary in the form of water molecules. No significant release of hydrocarbons has been observed. The mass 15 (CH<sub>3</sub>) signal (due to cracking of CH<sub>4</sub> in the quadrupole ionizer) was monitored during the experiments, and no changes were observed. This result is consistent with previous results from laboratory [13–19] and tokamak [2,20] experiments.

The O-GDC experiment shows that exposing co-deposited films and D implanted graphite to oxygen plasma associated with a GDC discharge is an effective method for removing co-deposits at a low temperatures. Significant oxidation rates could be achieved in the temperature range 400-470 K during O-GDC wall conditioning. The D release rate during O-GDC is significant even below 900 K, the temperature needed for thermal desorption in vacuum [19]. This temperature is also lower than that needed to remove co-deposited films by exposure to molecular oxygen, which was reported to be effective in the temperature range between 520 and 750 K [14-17,20]. The glow discharge greatly promoted the reactions between C:D films with energetic oxygen particles. The total removal rates of H-atoms and D-atoms were higher than that of molecular hydrogen by factors of about 20 and 50, respectively, which indicates that the most hydrogen reacted with oxygen during O-GDC conditioning. Also considering the possible high oxides re-absorption of water molecules on the low temperature walls, the present results likely under estimate water production.

In the absence of magnetic fields, the O-GDC wall conditioning has produced rapid, controlled co-deposit removal. Average removal rates,  $5.2 \times 10^{22}$  H-atoms/h,  $5.65 \times 10^{21}$  D-atoms/h and  $5.53 \times 10^{22}$  C-atoms/h, respectively, were obtained during 145 min O-GDC experiment in a pressure range of 0.5–1.5 Pa. Corresponding removal rate

of the co-deposited films was  $\sim 1.19 \,\mu$ m/day (26.5 g/day for carbon) assuming an area of 12 m<sup>2</sup>. Before oxidation experiments, the HT-7 has been operated largely with deuterium, however, much more H than D was observed in the released species in different oxidation procedures, including O-ICR, O-GDC and thermo-oxidation. The possible reason is that deuterium maybe only concentrated in the co-deposits or carbon limiter whereas hydrogen would come from all walls, especially stainless steel components would provide a permanent H source.

However, the film removal was accompanied by significant contamination of wall surfaces with oxygen. About  $5.37 \times 10^{22}$  O-atoms were adsorbed on the walls corresponding to 1.43 g of oxygen. The absorption corresponds to a coverage of  $4.5 \times 10^{21}$  O/m<sup>2</sup> on an wall area of 12 m<sup>2</sup>.

During the O-GDC experiment, the oxygen pressure has a strong influence on the formation of oxides. With increasing the oxygen pressure, the removal rates of C- and D-atoms increased, whereas the removal rate of H-atoms decreased. Depending on the supply of oxygen, the formation of  $CO_2$ may be favored over the formation of CO. The possible reason for the low H-atom removal rate in the high pressure O-GDC cleaning is that the liner temperature decreased with increasing pressure due to an improvement of heat transfer between limiters and liners by filling gas (Note: the heating power is constant!). It is possible that the reduced liner temperature led to a reduction in the release of H<sub>2</sub>O, the primary H-containing reaction product. Wall recovery by He-GDC is essential to remove the oxygen contamination. By He-GDC cleaning,  $1.53 \times 10^{22}$  O-atoms, about 28.5% of the absorbed oxygen, were removed, with the adsorption decreasing from  $4.5 \times 10^{17} \text{ O/cm}^2$  to a coverage of  $3.2 \times 10^{17}$  O/cm<sup>2</sup> on an area of 12 m<sup>2</sup>. The removal rate of O-atoms during He-GDC cleaning was about  $9.2 \times 10^{21}$  atoms/h.

Compared to only thermo-oxidation of codeposits with molecular oxygen with hot wall [22], O-GDC wall conditioning has a few advantages. During the glow discharge, oxygen disassociated and ionized, forming particles (O,  $O^+$ ,  $O_2^+$ ), which would attack the co-deposits more effectively.

Compared to the oxidation conditioning associated with the ion cyclotron resonance discharge (O-ICR) experiment [21], the disadvantage of O-GDC is that it must be operated in absence of a magnetic field, which may limited its use in future devices, such as ITER. Also, the GDC power (about 1 kW) was limited, whereas the ICR power could reach about 40 kW. On the other hand, O-GDC has some advantages compared to O-ICR, such as the large coverage area and higher operating pressure. Due to the fact that plasma discharge cleaning is most effective on surface with a line-of-sight view of the plasma, the present observations pertain primarily to plasma-facing surfaces. The particles during GDC discharge can reach far position, such as the pump port, the stainless steel walls. However, due to the magnetic field, the plasma particles during the ICR discharge were confined to a toroidal column in the tokamak vessel. During O-GDC wall conditioning, the oxygen pressure can be operated in the range of  $10^{-1}$ –10 Pa whereas the pressure was to under 0.1 Pa in O-ICR conditioning. (At higher pressures, there is significant reflection of ICR waves.) These are likely the main reasons why the removal rate of co-deposit layers in the O-GDC experiment is higher than that in the O-ICR experiment.

#### 4. Conclusions

The release of reaction products from co-deposited films on plasma-facing components of HT-7 (liners and limiters) exposed to a glow discharge oxygen plasma at wall temperatures of 400–470 K was successfully investigated in the HT-7 surperconducting tokamak. Results showed the released of hydrogen (deuterium) was mainly in the form of hydroxides, as opposed to molecular hydrogen during the O-GDC wall conditioning. Higher pressures of oxygen improved the effectiveness of co-deposited film removal.

In the absence of magnetic fields, the O-GDC experiment has produced rapid, controlled codeposit removal. Average removal rates,  $5.2 \times 10^{22}$  H-atoms/h,  $5.65 \times 10^{21}$  D-atoms/h and  $5.53 \times 10^{22}$  C-atoms/h, were obtained during 145 min O-GDC experiment in a pressure range of 0.5–1.5 Pa. The corresponding removal rate of co-deposited films was ~1.19 µm/day (26.5 g/day for carbon) based on a plasma-exposed area of 12 m<sup>2</sup>. The hydrogen retention on the walls was reduced with the O-GDC wall conditioning. The O-GDC wall conditioning is a more effectively method to removal hydrogen and co-deposits than He-GDC cleaning.

However, the technique led to significant O contamination. About  $5.37 \times 10^{22}$  O-atoms were adsorbed on the walls corresponding to 1.43 g of oxygen in 145 min O-GDC conditioning. The absorption corresponds to a coverage of  $4.5 \times 10^{21}$  O/m<sup>2</sup> on an wall area of 12 m<sup>2</sup>.

He-GDC cleaning following the O-GDC experiment was found to be effective in removing retained oxygen. High removal rate of O-atoms, on average about  $9.2 \times 10^{21}$  atoms/h, were obtained during the 100 min He-GDC cleaning following the O-GDC experiment.

Given the lower surface temperatures required compared to thermo-oxidation, and the greater surface coverage obtained, and the higher pressures available compared to O-ICR conditioning, O-GDC may prove beneficial for removing tritiumcontaining co-deposits in future fusion devices.

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