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Oxidation for deposits removal and hydrogen release on HT-7

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Abstract

Oxidation experiments at wall temperatures of 400–470 K, including O-ICR, O-GDC conditioning and thermal-oxidation have been carried out for removal of deposits and hydrogen in HT-7. Thermal-oxidation seems ineffective to remove co-deposited layers and hydrogen at these temperatures. O-GDC oxidation appeared to be the most efficient on HT-7. However O-ICR was almost as good and could be applied with the magnetic field in ITER. Both O-ICR and O-GDC have a deposits removal rates ~20 times that of He-ICR and He-GDC cleaning. Higher pressures and conditioning power during oxygen wall conditioning are favorable for removal of deposits and hydrogen. The oxygen retention after O-GDC oxidations was much higher than that after O-ICR oxidations. High power low oxygen pressure and He in O-ICR oxidation are beneficial for reducing oxygen retention. To remove the retained oxygen on walls both He-ICR and He-GDC were found to be effective. Plasma discharges could be recovered after a few tens of disruptive plasmas.

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Keywords: Oxygen; Wall conditioning; Co-deposition; Tritium; ICRF

1. Introduction

Long-term and high fractions retention of tritium is unacceptable for fusion reactors, such as ITER, and the evaluation of tritium removal techniques is very important [1]. It has been concluded from various investigations, that with carbon walls the dominant mechanism for hydrogen retention is co-deposition of eroded carbon with deuterium [1,2]. Tritium removal from amorphous tritiated carbon layers, a-C:T, co-deposited in next generation tokamaks, such as ITER, is an important issue to be solved. If in-situ co-deposit removal techniques are fast and effective, both in terms of T removal and plasma recovery after cleanup, the long term T retention/inventory problem could be mitigated [1].

The removal of T from thick a-C:T co-deposits may require the removal of the co-deposits themselves, and one of the most promising techniques for this is chemical and/or plasma assisted oxidation of re-deposited carbon layers. Extensive laboratory studies have been performed on hydrogen isotope removal by exposing co-deposited films and D implanted graphite to air or oxygen [3–8]. However, only a few experiments involving the injection of

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oxygen directly into tokamaks have been reported. In TEXTOR, oxidation has been done with molecular oxygen on hot walls (O-ventilation) [9] and in TFTR oxidation with glow discharge in a gas mix of He/O (He/O-GDC) in the absence of magnetic fields has been performed [10]. Tritium removal techniques that are to be used in the presence of magnetic fields are desirable in ITER due to the permanent toroidal magnetic field.

Oxidation treatments in HT-7 include oxidation with ion cyclotron resonance discharge (O-ICR) [11,12], glow discharge cleaning (O-GDC) [13] and thermo-oxidation with molecular oxygen [14]. The main efforts concentrate on fast removal of deposits and fast plasma performance recovery after cleanup. A main objective is to compare various oxidation techniques. This paper will review the main results for co-deposit removal and hydrogen release during oxidation and compares the various oxidation techniques. Oxygen retention, removal, plasma recovery and simple predictions for ITER will be briefly discussed.

In this paper it will be assumed that the carbon removed (as CO and CO_2) is actually from co-deposits. In future, more ambitious experiments involving removal of sample tiles and surface analysis will be performed to make clear where the removed carbon came from, for example, whether most of it came from co-deposits or eroded in arc pits, carbon dust and carbon contaminants of metal surfaces.

2. Experimental setup and procedure

HT-7 is a medium sized superconducting tokamak [15]. The total graphite plasma facing surface area of the HT-7 limiters was about 2.35 m^2 in 2004 (1.88 m^2m in 2005) [16]. All plasma facing limiter materials are made from the GBST1308 (1%B, 2.5%Si, 7.5%Ti) doped graphite with a 50–100 μm SiC coating [17,18]. The effective plasma facing area of carbon limiters and stainless steel liner is about 12 m^2. The liner is heated by direct current flow and the limiters are heated by thermal radiation. RF wall conditioning techniques have been developed with a permanent toroidal magnetic field [19,20]. Temperatures in the limiter tiles and on the liner were measured by thermocouples. Three turbo pumps were used for particle exhaust during cleaning. The pumping speeds for O_2, N_2 are about 650 l/s when the pressure in the vessel is about 0.5 Pa. The pumping speed is about 850 l/s when the pressure in the vessel is 10^{-2}–10^{-1} Pa.

To avoid possible oxygen contamination of subsequent plasma discharges, oxidation experiments were specially arranged at the ends of HT-7 campaigns. A few thousand deuterium plasmas discharges with electron temperature above 1 keV have been performed typically before the oxidation experiment. Before the oxidation experiment, the HT-7 vessel wall was baked to about 370 K, and the liner and limiters were heated to 400–470 K, which was taken about 6 h from room temperature. O-ICR cleaning was performed in the presence of a permanent magnetic of field 1.5–2.0 T. Two molybdenum anodes were used for the O-GDC experiment, with a voltage of about 270 V and a total current of 4 A. Thermo-oxidation experiments with all pumping turned off were performed with filling the torus with pure oxygen to a given pressure. Pure oxygen and gas mixtures of He/O were used. After oxidation wall conditioning, oxygen removal with traditional cleaning techniques, such as He-GDC and He-ICRF, were investigated for wall cleanup. And the plasma recovery behavior after cleanup was studied. The wall temperature during cleanup and plasma recovery was the same as during the oxidation wall conditioning.

3. Results

3.1. Thermo-oxidation [14]

During the thermo-oxidation, the deposited carbon layers were removed by the formation of CO and CO_2 and the incorporated hydrogen was released in the form of water molecules. Most oxides were only formed in a short transient phase after the injection of the oxygen. Table 1 summarizes the results from the thermo-oxidation experiments of HT-7. With higher the oxygen pressure, more oxides produced. However, the largest effect was the absorption of oxygen on the walls of the vessel, accounting for as much as 94% of the O_2 admitted to the vessel. The oxygen wall coverage increased approximately linearly with the filling pressure Fig. 1.

3.2. Glow discharge associated oxidation [13]

During O-GDC wall conditioning, the release of hydrogen (deuterium) was mainly in the form of hydroxides. In the O-GDC experiment, the removal rates of H-atoms and D-atoms in form of H_2O,

Table 1
Summary of thermo-oxidation experiments performed at the wall temperature of 400–470 K in HT-7

Filling pressure (Pa)	Injected O-atoms $\times 10^{20}$	Exposure time (min)	O-atoms $\times 10^{20}$ and percent of injected O-atoms					Corresponding coverage (10^{16} O-atom/cm^2)
			Adsorbed (%)	O in CO (%)	O in CO_2 (%)	O in H(D)–O (%)	O in O_2 (%)	
0.7	12.5	46	94.4	1.3	1.6	0.3	2.5	0.98
1.1	18.7	104	75.7	6.7	9.8	2.1	5.8	1.18
9.3	222	127	62.9	2	1.3	2.0	31.7	11.6
32	630	121	48.6	0.5	0.9	0.5	49.5	26

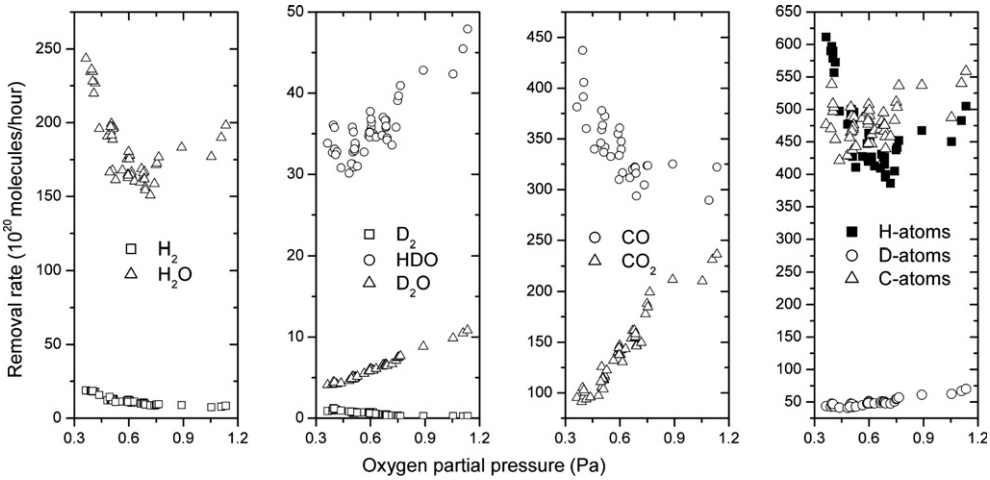

Fig. 1. Dependence of particle removal rates on oxygen pressure during O-GDC.

HDO and D_2O were higher than that of H_2 and D_2 by factors of about 20 and 50, respectively. The oxygen partial pressure has a little influence on the formation of oxides: the O_2 pressure increases nearly by ten, while the production increases by about 2, as shown in Fig. 2. Depending on the supply of oxygen, the formation of CO_2 may be favored over the formation of CO. With increasing oxygen pressure, the removal rates of C and D-atoms increased, whereas the removal rate of H-atoms is more complex to understand due to possible absorption of water formed on the walls.

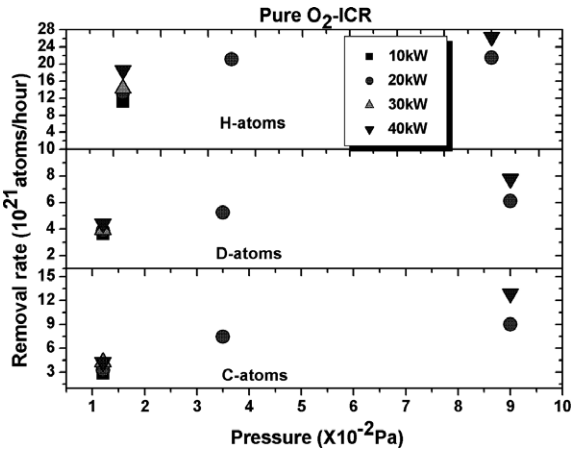

Fig. 2. Removal rate of H, D and C-atoms during O-ICR cleanings.

In the absence of magnetic fields, O-GDC wall conditioning produced rapid, controlled co-deposit removal. Average removal rates of 5.2×10^{22} H-atoms/h, 5.6×10^{21} D-atoms/h and 5.5×10^{22} C-atoms/h, respectively, were obtained during 145 min O-GDC cleaning in a pressure range of 0.5–1.5 Pa. However, this procedure led to a significant O contamination. About 5.37×10^{22} O-atoms were adsorbed on the walls in 145 min O-GDC conditioning. Compared to He-GDC cleaning, O-GDC wall conditioning has higher removal rates for H-atoms and C-atoms by a factor of about 2–4 and about 25, respectively. This indicated that the O-GDC cleaning could remove the carbon deposited layer directly whereas He-GDC cleaning has little removal efficient on deposits. During O-GDC

cleaning, H release happened while co-deposited layers were removed. However, during He GDC, H release also happened due to ion-induced release. Those may be the main reason of that a much higher relative removal rate of C was obtained during O-GDC cleaning than during He-GDC whereas not H.

3.3. ICR associated oxidation [11,12]

During O-ICR cleaning, high power and high pressure is favorable for the removal of hydrogen and co-deposits, as shown in Fig. 3. The highest removal rates of H, D and C atoms were up to 2.6×10^{22}, 7.8×10^{21} and 1.5×10^{22} atoms/h, respectively, during 40 kW 9.0×10^{-2} Pa pure O-ICR cleaning. Obviously the reduced amount of oxygen under the 4:1 He/O-ICR conditions cleaning leads to a reduced cleaning efficiency. Compared to He-ICRF cleaning, O-ICR wall conditioning has a higher removal rate for H-atoms by a factor of about 6 and for C-atoms by a factor of 20. This also indicated that the O-ICR cleaning could remove the carbon deposited layer directly whereas He-ICR cleaning has little removal efficiency for deposits. Similar as comparison of O-GDC to He-GDC cleanings, a much higher relative removal rate of C was obtained during O-ICR cleaning than during He-ICR whereas not for H.

High power and/or low oxygen filling pressure in O-ICR cleaning is beneficial for reducing oxygen retention. With the same filling rate of O_2, both the partial pressure of O_2 and oxides in the mixtures

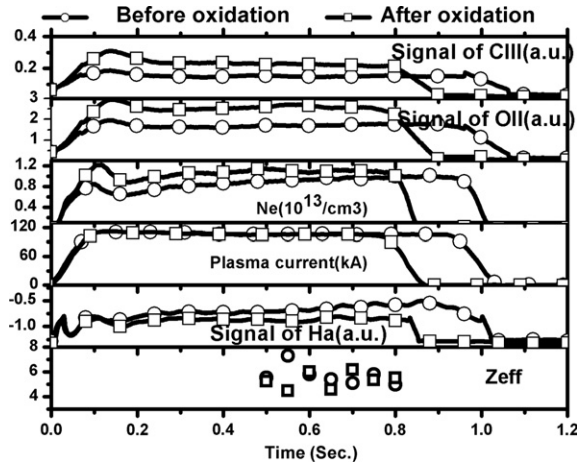

Fig. 3. Typical recovered plasma after oxidation experiment (at the end of HT-7 campaign in 2005 winter).

of He/O-ICR plasma are higher than in pure O-ICR plasma, indicating that the He in the He/O-ICR plasma could reduce oxygen retention. Normally, the O retention rate in O-ICR plasma operation is lower than that in O-GDC plasma discharge by a factor of 2–200, which depends on cleaning parameters, such as power and oxygen pressure.

3.4. Oxygen removal [21]

A few techniques using different parameters were investigated for oxygen removal after the oxidation experiments, summarized in Table 2. To remove the retained oxygen from the walls, both He-ICR and He-GDC were found to be effective. The oxygen removal rate depends on how much oxygen was retained on the walls, the ICR power and the cleaning pressure. The highest oxygen removal rates obtained were about 6.5×10^{21} and 9.2×10^{21} O-atoms/h during He-ICR and He-GDC cleaning, respectively.

3.5. Plasma recovery [11,12]

After wall cleaning, plasma discharges could be recovered but only after a few tens of disruptive plasmas (or a few hours in total time). Plasma recovery depended on the amount of oxygen retention on the walls before plasma operation. Disruptive plasma discharges are essential for removal of the remaining oxygen on the walls after cleaning and for recovering normal plasma operation. During the recovering discharges, CIII emission and OII emission is increased, and the Ha was greatly reduced, as shown in Fig. 3. On other hand, the CIII emission, OII emission and Z_{eff} normalized to the plasma density went down shot by shot. Then, plasma operation was recovered step by step, as shown in Fig. 4.

3.6. Prediction for ITER [12]

Assuming a similar power density of ICR plasma as in the 40 kW ICR used for HT-7, 6.7 MW ICR wave power would be required for ITER. Assuming in addition a similar particle flux to the walls, 4 A GDC for HT-7 would require about 220 A for ITER. Assuming that the reaction ratio of oxygen is the same or the production rate/m^2 on the wall surface is same, the pumping speed and the effective surface wall area will be the two main factors that influence the oxidation process. If the wall tempera-

Table 2
Summary of oxygen retention and removal (the cleanings were listed in the order of operation)

Oxidation wall conditioning		Pure O-ICR	4:1 He/O-ICR			1:1 He/O-ICR		O-GDC
Oxidation experiments	Total time(mins)	185	71			47		145
	O retention atoms	1.73×10^{22}	2.9×10^{21}			3.4×10^{21}		5.37×10^{22}
Oxygen removal	Cleanings	He-ICR	He-Baking1	He-Baking2	He-ICR	He-ICR1	He-ICR2	He-GDC
	Removed O-atoms	5.4×10^{21}	7.6×10^{20}	4.1×10^{20}	1.37×10^{21}	1.8×10^{21}	2.1×10^{20}	1.5×10^{22}
	Removal rate (O-atoms/h)	6.5×10^{21}	7.5×10^{20}	1.1×10^{21}	1.5×10^{21}	2.6×10^{21}	7.6×10^{20}	9.2×10^{21}
O-atoms retained on the wall		1.2×10^{22}		4×10^{20}		1.3×10^{21}		3.9×10^{22}

Wall temperatures of 400–470 K in all cleanings are as the same as that during oxidation experiments.

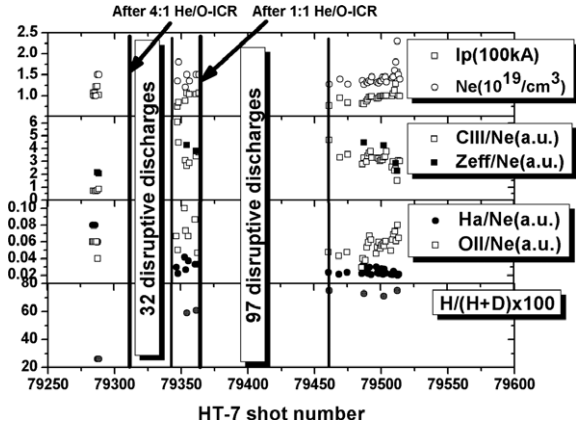

Fig. 4. Typical evolutions of (1) plasma density, (2) the behavior of CIII, OII light emission and Z_{eff}, Ha normalized to the plasma density, (3) H/(H + D) ratio, with shot number after He/O-ICR experiment (at the end of HT-7 campaign in 2005 spring).

tures in ITER is at the same as that during oxidation wall conditioning at 400–470 K, with the above assumptions, the predicted removal rates for ITER are as listed in the Table 3. It was found that 1 h O-ICR cleaning could remove about 8–25 g carbon deposition in ITER. It seems that 1 h O-ICR cleaning could remove the carbon deposited in 4–25 discharges (Carbon deposition rate was assumed up to $1–2\times10^{23}$ C-atoms/discharge in 1000 s in ITER [1]). One hour 1.5 Pa O-GDC cleaning can remove about 61–97 g carbon deposition in ITER, corresponding to carbon deposited in 30–48 discharges. The removal efficiency also depended on the wall materials and their distribution in ITER. If metal material, such as Be, W, used as wall materials, oxidation wall conditioning possibly has only little efficiency for hydrogen or carbon removal. However, carbon is currently chosen to clad the ITER divertor target and the eroded carbon would be possibly deposited with tritium on the walls in ITER. In other side, if the water coolant is not removed then ITER walls will be at 240 °C, but if the water is replaced with hot gas (which can be done infrequently) then wall temperatures up to 350 °C are achievable. Then the predicted efficiency for ITER from HT-7 experiments may be lower than actual efficiency in ITER if the walls work at a high temperature. Therefore, the oxidation wall conditioning may be useful for ITER.

4. Comparison and discussion

From the results of the different oxidation experiments, it can be concluded that carbon deposits are removed by formation of CO and CO_2 and the incorporated hydrogen is released in the form of water molecules, similar to laboratory studies [3–8]. Thermo-oxidation could remove the co-deposits only with low removal efficiency in a short transient at the admission of oxygen at the present wall temperatures of 400-470 K. Both O-GDC and O-ICR plasma are effective methods for removing of co-deposits at a low wall temperature of 400–470 K, as shown in Table 4.

While thermo-oxidation with molecular oxygen has a smaller efficiency than O-ICR and O-GDC wall conditioning, it has the advantage of being equally effective on all wall surfaces, whereas the O-ICR and O-GDC are most effective on surfaces with line-of-sight to the plasma. It may be required to remove co-deposits on less accessible places, such as tile gaps and other non-plasma facing surfaces. Additionally, thermo-oxidation could be carried

Table 3
Predicted removal rate of C-atoms in ITER for the same ICR power density as in HT-7 (P.S.: pumping speed; S.A: total plasma facing surface area)

	HT-7	ITER
Volume (m^3)	4.85	840
Plasma facing surface (m^2)	12	680
Pumping speed (m^3/h)	0.85	75
Carbon surface (m^2)	2.35(1.88)	55
GDC current	4 A	220 A
RF power	40 kW	6.7 MW
Carbon deposition		$1–2 \times 10^{23}$/1000 s plasma

Removal rate		C-atoms/h	C-atoms/h	g/h
40 kW/4.85 m^3, 9.0×10^{-2} Pa, pure O-ICR	P.S.	1.5×10^{22}	1.3×10^{24}	25.9
	S.A.		8.7×10^{23}	17.4
40 kW/4.85 m^3, 9.8×10^{-2} Pa, 4:1 He/O-ICR	P.S.	7.5×10^{21}	6.6×10^{23}	13.2
	S.A.		4.3×10^{23}	8.5
1.5 Pa O-GDC	P.S.	5.5×10^{22}	4.9×10^{24}	96.7
	S.A.		3.1×10^{24}	61.2

Table 4
Main results of oxidation experiments in HT-7

Oxidation		Thermo-O	Pure O-ICR	4:1 He/O-ICR	O-GDC
Parameters		32 Pa	40 kW, 9×10^{-2} Pa	40 kW, 9.8×10^{-2} Pa	1.5 Pa, 250 V/4 A
Highest removal rate (10^{21} atoms/h)	H-atoms	Transient	26.3	5	56.5
	D-atoms	in < 1 min.	7.76		5.65
	C-atoms		14.9	7.5	55.3
Oxygen absorption rate (10^{14} O-atoms/cm^2 min)		21.5	7.3	3.45	45.5

out in high oxygen pressure whereas the pressure is limited for O-ICR and O-GDC wall conditioning.

During GDC and ICRF discharges, oxygen is disassociated and/or ionized to energetic particles (O, O^+, O_2^+), which attack the co-deposits more effectively. Also, actively particle pumping during O-GDC and O-ICR cleaning are advantages for co-deposit removal.

Compared to O-ICR experiment, the disadvantage of O-GDC is that it must be operated in the absence of a magnetic field, which limits its use in future devices, such as ITER. Also, the GDC power (about 1 kW) is limited, whereas the ICR power could reach about 40 kW in HT-7. On the other hand, O-GDC has advantages compared to O-ICR, such as the large covered area and higher operating pressure. Plasma discharge cleaning is most effective on surfaces with a line-of-sight view of the plasma; the present observations pertain primarily to plasma facing surfaces. Also, the energetic particles (O, O^+, O_2^+) during GDC discharge can reach remote locations, such as pump ports or stainless steel walls. Due to the magnetic field, the ionized oxygen 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 limited to values lower than 0.1 Pa in O-ICR conditioning in HT-7 (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 experiments is higher than in the O-ICR experiments. However, the high removal rates in high power and high pressure O-ICR experiments under a permanent magnetic field made it to be one of important methods for removing carbon deposits in future devices, such as ITER. Compared

to O-GDC experiment, even though the efficiency for removal of co-deposits is somewhat smaller, the lower oxygen retention is beneficial for application in future device.

For removing the retained oxygen on walls, both He-ICR and He-GDC were found effective. The oxygen removal rate depends on how much oxygen is retained on the walls, ICR power and cleaning pressure. However, a few tens disruptive plasma discharges are still essential for removal of the remaining oxygen from the walls and for recovering normal plasma. After cleaning, plasma discharges could be recovered after a few tens of disruptive plasmas(or a few hours in total time). The plasma recovery depends on the oxygen retention on the walls before plasma operation. To obtain normal plasma operation, it is essential to reduce the oxygen retained on the walls before re-starting of the plasma operation.

However, frequent and lengthy oxidation wall conditioning in future devices, such as ITER, is still unacceptable. Further investigation of this topic should be carried out to obtain high removal rates of carbon deposited layers to meet the requirements of ITER. To increase the ICR power and the pressure in O-ICR wall conditioning are essential to optimize removal of tritium in ITER with permanent magnetic field. In addition, increasing the pumping speed during O-ICR and O-GDC wall conditioning is important. High power and high pressure He-ICR cleaning should be investigated to obtain a fast removal of oxygen. In addition, other oxygen removal techniques, such as D_2-ICR and D_2-GDC cleaning, and boronization should be studied. Since HT-7 is a limiter machine, similar O-ICR experiment should be carried out in a divertor superconducting tokamaks, such as EAST.

5. Conclusions

During oxidation wall conditioning, carbon deposits are removed by formation of CO and CO_2 and the incorporated hydrogen is released in the form of water molecules. Thermo-oxidation has low removal efficiency for carbon deposited layers at 400–470 K. Both O-GDC and O-ICR plasma are effective methods for removing co-deposits at low wall temperature. Then, the highest removal rate of carbon was about 1.5×10^{22} atoms/h and 1.5×10^{22} atoms/h, respectively, for O-ICR and O-GDC. Oxidation with glow discharge appeared to be the most efficient method in HT-7. However, O-ICR was almost as good and could be applied under presence of the magnetic field in ITER. Both O-ICR and O-GDC have a removal rate $\sim$20 times higher that of He-ICR and He-GDC cleaning, respectively.

Higher pressure and higher power are favorable for removal of deposits and hydrogen during oxygen wall conditioning. However, with higher oxygen pressure, the more oxygen is retained on the walls. The oxygen retention rate in O-GDC oxidation was significantly higher than that in O-ICR oxidation. Operation at high power, low pressure and the use of He in O-ICR oxidation were beneficial for reducing the oxygen retention. To remove the retained oxygen on walls, both He-ICR and He-GDC were found to be effective. Even after wall cleanings, plasma discharges could be recovered only after a few tens of disruptive plasmas, which is possibly difficult to be accepted in ITER. (It is also difficult to state is acceptable or not at this moment. If the disruptive plasmas is not full-length ITER discharges of 400 s. but shot and frequent one, the results maybe meet the requirements.) Further work should be done, such as increasing removal efficiency, the fast oxygen removal techniques and studies in a divertor device.

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