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# Oxidation of carbon deposits as a fuel removal technique for application in fusion devices

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## ABSTRACT

In tokamaks, such as ITER, with low-temperature, low-Z walls (Be or C), it is predicted that codeposition of hydrogen fuel with sputtered wall atoms will be the dominating mechanism for in-vessel tritium retention. Limits on in-vessel inventory will require the periodic removal of such tritium, and a variety of procedures have been proposed. In the case of carbon-based deposits, it is possible to use chemical reactions with oxygen to produce volatile products, which may be removed from the vacuum vessel via the vacuum pumps. Thermo-oxidation has some major advantages compared to other techniques for removing tritium from codeposits, in that it can act on all surfaces inside the vessel, including tile gaps and other non-line-of-sight surfaces, and there is no requirement for mechanical entry into the torus. This paper discusses recent experimental results and the use of oxidation in future tokamaks such as ITER.

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## 1. Introduction

The erosion of plasma-facing materials is an inevitable consequence of the interaction between a fusion plasma and adjacent solid surfaces. The rate of erosion will depend on a large number of parameters; however, the atomic mass of the wall atoms is probably the single most important factor. For low-Z wall materials, like Be and C, gross erosion yields will likely be on the order of 1%.

In a quasi-steady-state situation, the influx of impurity atoms will be roughly balanced by the outflux, meaning that there will be significant redeposition of the eroded wall material, possibly at locations far from the original source. When the surfaces on which deposition occurs are at temperatures  $\lesssim 350$  °C, hydrogen fuel from the plasma will be trapped along with the depositing impurity atoms, and lead to an inventory of trapped tritium. In ITER, with low-temperature, low-Z plasma-facing components, it is predicted that codeposition of hydrogen fuel with sputtered wall atoms, either Be or C, will be the dominating mechanism for in-vessel tritium retention [1]. This inventory will increase more-or-less linearly with reactor operating time. A further point is that much of the deposition may occur in tile gaps [2] and other non-plasma-facing surfaces, complicating the removal process.

As part of the general safety requirements for ITER, limits have been placed on the in-vessel inventory of tritium. These limits will thus require the periodic removal of tritium retained in codeposits.

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The frequency of removal will depend on the exact rate at which codeposits are built up and on the tritium concentration in the deposits. It is very important to note that when deposition surfaces are kept at higher temperatures, the concentration of tritium will be much lower [3,4]. Thus the issue of tritium codeposition is unlikely to be important in future demonstration or power reactors, where surface temperatures will necessarily be higher to improve thermal efficiencies. It is also likely that some of the codeposit locations in ITER will be similar to those measured in JT-60U, where the D/C ratio was measured to be of order 0.01 because of strong heating by the plasma [3,4].

A wide variety of procedures have been proposed for the removal of codeposits in ITER, and these have recently been reviewed e.g., [5]. Generally, for the non-chemical methods some form of mechanical intervention into the torus will be required, and a means provided to remove solid material. Similarly, dust and flakes resulting from the exfoliation of thick codeposits may require some sort of mechanical retrieval system [6].

As an alternative to concepts requiring mechanical intervention, it may be possible to remove certain types of codeposits through chemical means. Both carbon and hydrogen react readily with oxygen to form volatile products. Thus, in principle, tritium could be removed from carbon-based codeposits, and the codeposits themselves removed from the torus simply by admitting oxygen to the chamber, and pumping out the reaction products. The purpose of this paper is to review recent laboratory and tokamak results related to the oxidation of codeposits, and to look at the various issues regarding the practical application of oxidation in ITER. These include the following broad categories: (i) the effectiveness

of oxidation at temperatures available to ITER, (ii) handling of the highly tritiated water produced, (iii) collateral effects on other tokamak components, and (iv) subsequent plasma performance.

**2. Various oxidation techniques**

The various oxidation techniques might be divided into 3 categories: plasma driven, activated oxygen, and thermo-oxidation.

**2.1. Plasma**

There have been a number of plasma oxidation studies performed in various laboratories e.g., [7–9] as well as in tokamaks (see Section 4). Oxidation rates can be quite large, even at low temperature; however, when applied to a tokamak, the process is largely limited to plasma-facing surfaces. In addition, a natural consequence of producing oxygen-containing plasmas is the creation of active neutral species, as discussed next.

**2.2. Activated oxygen**

There are two particular studies, one dealing with ozone [10] and one with atomic oxygen [11], which we include as activated oxygen. In both cases, oxidation rates are increased substantially over O<sub>2</sub> exposure. For ozone, Moormann et al. [10] found that the erosion rates of TEXTOR codeposits were similar to thermo-oxidation rates, but achieved at a temperature 200 °C lower. Similarly, Drenik et al. [11] found films were removed two orders of magnitude faster with atomic oxygen than with O<sub>2</sub> at the same temperature. Erosion rates of up to ~50 μm/h were observed for lab-deposited films [11]. Activated oxygen has the advantage of not being affected by magnetic fields, and also being able to penetrate tile gaps and other hidden surfaces. Both plasma and activated oxidation techniques have the disadvantage of eroding graphite substrates [10] and possibly causing other collateral damage.

**2.3. Thermo-oxidation**

Thermo-oxidation refers to the removal of carbon-based layers through the application of heat in an oxygen environment. The classic example is of a self-cleaning domestic oven, where the oven is heated in air to ~500 °C to burn off mm-thick organic (carbon-based) layers in about an hour. There are probably millions of such “thermo-oxidation units” world-wide, operating at significantly more severe conditions (higher temperature) than are being proposed for tokamaks. The first experimental demonstration of thermo-oxidation as a means of removing tokamak codeposits was reported by Causey et al. [12] in 1990. The context was slightly different, however, and the conclusions were that tritium tapped in codeposit layers could be released during a vacuum-loss event (with the vessel hot) and pose a safety hazard. Following the initial work by Causey et al. [12], an extensive study of thermo-oxidation was started at the University of Toronto and at other laboratories.

**3. Recent laboratory results on thermo-oxidation**

In this section we will focus on recent laboratory results with important implications with regard to the application of thermo-oxidation to tokamaks.

**3.1. Temperature and pressure dependence**

Over the past few years the University of Toronto (U of T) group has performed a number of studies looking at the effect of the key parameters of temperature and pressure on oxidation rates [13,14].

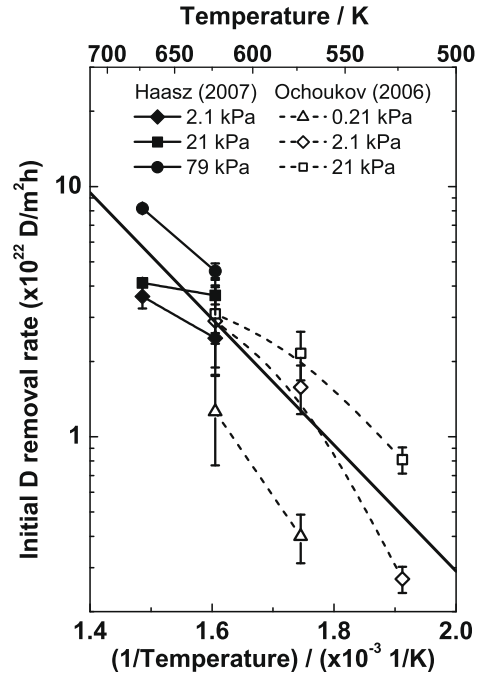


Fig. 1. Measured initial D removal rates as a function of reciprocal temperature; results from Haasz et al. [14] and Ochoukov et al. [13]. The long solid line through the data is shown as an indication of an approximate activation energy (0.5 eV) for the thermo-oxidation process.

In the range of interest for fusion devices, temperature is clearly the more important factor. A subset of temperature dependence results for DIII-D codeposits exposed to oxygen at various pressures is plotted in Fig. 1. The bottom line is that the initial oxidation rate increases by about one order of magnitude between 250 °C and 350 °C. The pressure dependence is shown in Fig. 2, and while oxidation rate does increase with increasing pressure, there is a trend toward saturation for pressures above a few kPa.

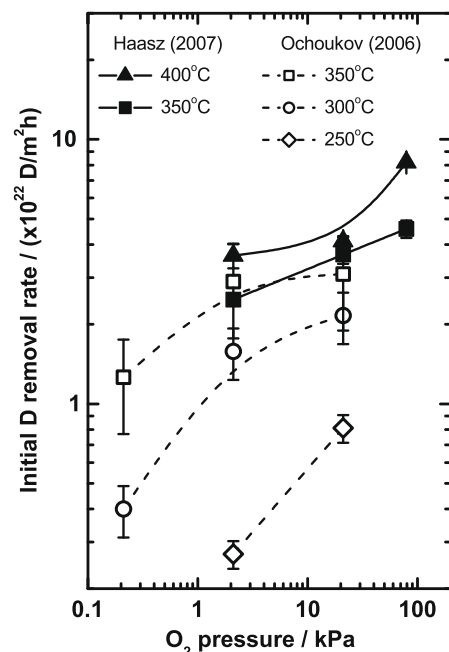


Fig. 2. Measured initial D removal rates as a function of O<sub>2</sub> pressure; results from Haasz et al. [14] and Ochoukov et al. [13].

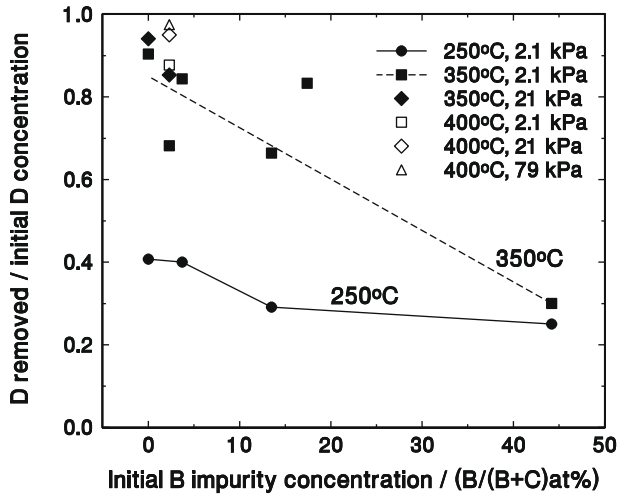


Fig. 3. Loss of D content due to oxidation normalized by the initial D content plotted against the initial boron impurity content in the near-surface of the codeposit [14]. Data from various sources [13,14,23,55,56].

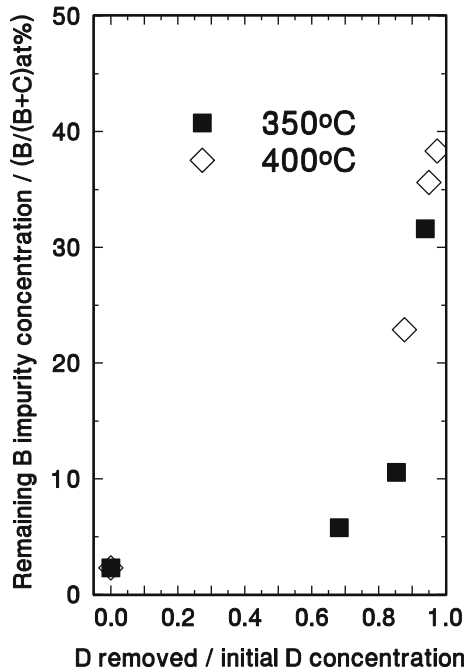


Fig. 4. The increase in boron concentration of specimens with an initial concentration of 2.3 at.% B/(B+C), following 2 h oxidation for various pressure and temperature combinations [14]. Concentrations measured by XPS.

### 3.2. The effect of impurities on oxidation

There are two main impurity elements which are found in significant concentrations in some codeposits from current tokamaks; boron and beryllium. The effect of B impurities on the thermo-oxidation of DIII-D codeposits has been summarized by Haasz et al. [14]. There is a clear decreasing trend in codeposit removal efficiency with increasing B concentration, see Fig. 3. For B concentrations ~50%, thermo-oxidation was only able to release 25–30% of the deuterium. In addition, the preferential removal of C leads to an increase in B concentration in codeposits originally containing low B concentrations, Fig. 4.

Beryllium impurities, however, do not seem to have the same effect. Fig. 5 shows the release of D as a function of Be content dur-

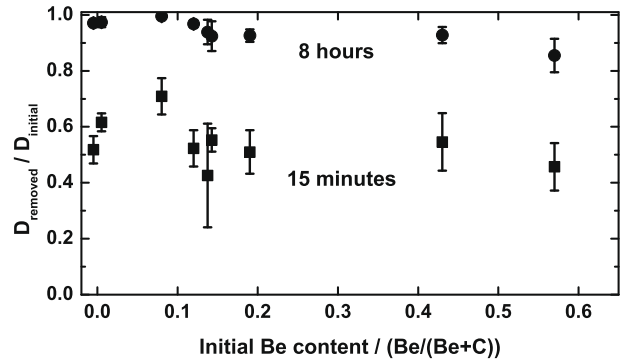


Fig. 5. The amount of D removed from JET codeposits during the first 15 min, and the full 8 h oxidation period (350 °C, 21 kPa) normalized by the initial D content, as a function of the initial Be content [15].

ing oxidation of JET codeposits with substantial Be concentrations [15]. There is no trend towards a reduction in oxidation efficiency with increasing Be concentration. While both B and Be will form oxides, B<sub>2</sub>O<sub>3</sub> somehow acts to block further oxidation of the codeposit, while BeO does not.

### 3.3. The effect of codeposit thickness

The majority of our earlier studies at U of T have been carried out with codeposits on the order of a few  $\mu\text{m}$  in thickness. Our most recent study, however, with thicker codeposits from JET [15] has shown that thicker tokamak codeposits are removed much more efficiently than thinner ones. This is contrary to the experience with laboratory-deposited a-C:H films, where the carbon content of the film appears to recede from the geometric surface [16]. Tsui et al. [15,17] found that there was a linear dependence between the initial D content of the codeposit and the rate of D removal, see Fig. 6. The significant removal of hydrogen from thick codeposits from TFTR (~50  $\mu\text{m}$ ) [12] and TEXTOR (80–170  $\mu\text{m}$ ) [18] in relatively short periods of time is strongly supportive of this result. The implication is that tokamak codeposits are porous to the point that oxygen has access to the bulk of the codeposit, and

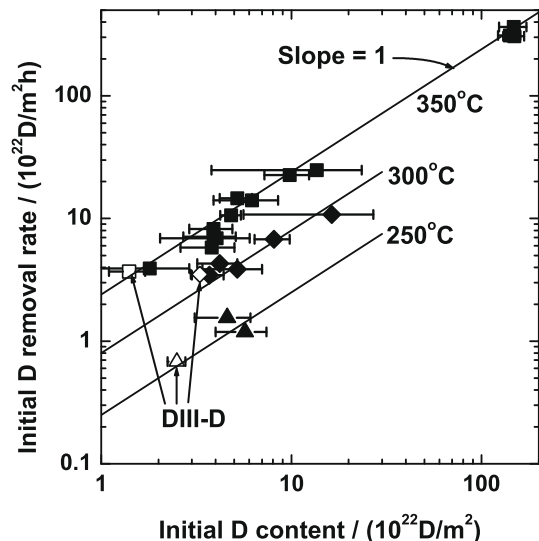


Fig. 6. The amount of D removed from JET codeposits during the first 15 min of oxidation at 250, 300 and 350 °C are plotted as a function of the initial D content [15,17]. Points for DIII-D codeposits measured under similar oxidation conditions are also shown [14].

consequently is able to act on the whole film at once. For this reason, it does not make sense to try to come up with erosion rates in terms of  $\mu\text{m}$  per hour. Recent surface analysis of eroded and partly eroded codeposits is consistent with this hypothesis [19]. The overall result is that at 350 °C, ~50% of the deuterium is removed in 15 min, and ~90% is removed after 8 h, independent of the initial codeposit thickness and Be content [15]. The implication for ITER would be the same.

#### 3.4. Are the deposits removed?

It has been a common observation e.g., [12,18], that codeposits look the same before and after oxidation. The question then arises as to whether thermo-oxidation only removes the hydrogen content, while leaving the majority of the deposit on the surface. There are several pieces of evidence to suggest that the deposits are, in fact, largely removed as well.

Firstly, ion-beam measurements of lab-produced films during the course of thermo-oxidation in the lab [16], directly show the removal of carbon. Similar measurements of film test specimens before and after plasma-oxidation in ASDEX-Upgrade [20] and TEXTOR [21,22], found the complete removal of the films from silicon substrates. For JET codeposits with very low Be content, SIMS measurements before and after oxidation also showed the near-complete removal of carbon layers [19].

Secondly, we have used mass-loss measurements, along with independent thickness measurements to estimate a codeposit density [14,23]. Values of  $\sim 1500 \pm 500 \text{ kg/m}^3$  are consistent with what we might expect for such deposits, again suggesting that the majority of deposits have been removed during oxidation.

Thirdly, we can see an increase in the concentration of impurity elements in the residual deposits remaining following oxidation indicating the preferential removal of carbon. This is clearly shown in Fig. 4, where the B concentration has increased by a factor of 15 when the D concentration has been reduced by 98% [14]. Our recent results also show an increase in Be concentrations in some JET codeposits following oxidation [19].

Where there are significant impurity concentrations, it is clear that these impurities are not removed by oxidation. In the case of Be impurities, in some specimens a significant C content was found in the residual structure, suggesting that the residual may be composed of BeC [19]. This issue leads to the question of what may happen during repeated deposition/oxidation cycles, which might occur in a tokamak. More research is needed in this area.

#### 4. Tokamak thermo-oxidation experience

It was noted from the tritium clean-up experiments following tritium experiments in JET and TFTR that one of the more effective methods of removing tritium was simply exposing the inside of the torus to air at room temperature [1,24]. For TFTR, He/O<sub>2</sub> glow discharge cleaning was also found to be effective [25]. Since then, there have been a number of attempts to test oxidation procedures in TEXTOR [21,26], HT-7 [27–31] and ASDEX-Upgrade [20]. In most cases, the evolution of CO, CO<sub>2</sub> and D<sub>2</sub>O/HDO have been observed, but there has been a limited ability to quantify the removal of codeposits.

Where thermo-oxidation has been used [26,29], there were no quantitative measurements of codeposit removal. ICR-discharges [28] and glow discharges [20,21,30], both demonstrated improved ability to remove carbon compared to thermo-oxidation. This was confirmed through the tracking of CO and CO<sub>2</sub> production, and through measurements on test film specimens. In both ASDEX-Upgrade [20] and TEXTOR [21] test a-C:H films deposited on silicon substrates were completely removed from specimens di-

rectly facing the plasma; however, boron-containing test specimens [21] and natural codeposits containing boron [20] were not removed.

An important feature of all of the tokamak experiments [20,21,26–28] has been the effect of oxidation on the restart of plasma operations. The general conclusion has been that ordinary start-up procedures, typically requiring a day of re-conditioning, were sufficient to restore normal plasma operations. Thus oxidation did not seriously affect vessel conditioning. We note that none of the tokamaks that have tested various oxidation procedures had large beryllium surfaces. While thermo-oxidation at 350 °C is not likely to cause a large increase in the oxygen inventory of a Be wall, see below, a demonstration of oxidation in a Be-containing tokamak would be recommended.

Although discussions of accidental oxidations do not ordinarily appear in publications, they also are a good indication of the effect of oxidation on plasma recovery. Air leaks of various magnitudes have probably occurred during baking or high temperature operation on most tokamaks. The bottom line here, is that a tokamak must be designed to withstand an air leak during baking, and if it can do this, then oxidation should not be a threat to any internal systems.

#### 5. Concerns associated with oxidation

##### 5.1. Tritiated water

Tritium released from codeposits during oxidation will largely be in the form of (D,T)<sub>2</sub>O [32,33]. Compared to the T<sub>2</sub> form, tritium as tritiated water is approximately 10000 times more hazardous per tritium atom for personal exposure [34], thus, the thermo-oxidation process will increase the hazard associated with the tritium. Also, highly tritiated water is corrosive; because of this it will be important to ensure that no tritiated water vapour is allowed to condense as liquid and remain in liquid form for extended periods of time.

Tritiated water is already an expected component in the ITER exhaust [35]; however, the baseline design calls for the exhaust to be predominately (D,T)<sub>2</sub> with small amounts of the other species [35]. The ITER tritium plant will include a water detritiation system [36], but this is designed to handle relatively large flowrates of comparatively dilute tritiated water; e.g., from a coolant leak. The large volume of highly tritiated water resulting from thermo-oxidation may not be appropriate for the current design of the ITER water detritiation system.

With the current 700 g T limit in the torus, the removal of T by thermo-oxidation might liberate approximately 700 g of T along with 470 g of D, leading to the production of  $\sim 4900 \text{ g}$  of tritiated water. The baseline water processing rate for the ITER tritium plant is 33 g/h [35]. At this rate it would take 150 h (6 days) to process this amount of tritiated water. Note that 6 days is not an exceptionally long time, but if the baseline tritium exhaust processing system is tied up processing this material, no ITER experimental program could be undertaken during this time. Thus, depending on the frequency of thermo-oxidation application, a scale-up of the current water detritiation system or a separate tritiated water processing system may be needed to accommodate this load.

Due to self-radiolysis, which leads to the production of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) and various radicals (and nitric acid, if nitrogen is present), tritiated water is highly corrosive [37]. Following oxidation, the tritiated water vapour must be transported through ductwork and it is important to ensure that this vapour does not condense in unwanted locations. This might necessitate additional heating of ductwork and components. However, it is already recognized that tritiated water in the normal torus exhaust will not be

diluted by (H,D)<sub>2</sub>O, so heated ductwork is required whether or not thermo-oxidation is used.

In the ITER tritium plant, tritiated water is first collected on molecular sieve beds, and then processed by a combination of catalytic reactors and permeators [36]. The catalyst promotes reactions such as  $\text{CO} + \text{T}_2\text{O} \Rightarrow \text{T}_2 + \text{CO}_2$ , and the  $\text{T}_2$  is removed by a metal membrane-based permeator. A scale-up of the storage capacity, and possibly a separate exhaust stream to avoid the torus roughing pumps, may be required to handle the increased water load associated with thermo-oxidation.

## 5.2. Oxidation of beryllium

Another possible concern that exists for thermo-oxidation in tokamaks with Be PFCs is the effect it might have on the Be first wall components. As seen in Section 3.2 above, the presence of Be, in ratios as high as  $\text{Be}/(\text{Be} + \text{C}) \sim 1$ , does not appear to have a significant impact on the release of deuterium from codeposits during thermo-oxidation. However, the oxidation of Be wall tiles is another issue. Be will form a thin surface oxide on exposure to oxygen at almost any temperature, but this layer of BeO generally blocks the formation of thicker oxide layers by blocking the transport of O inward to the interface, and Be outward to the surface. A recent study by Tomastik et al. [38] found that oxide layer growth saturates at  $\sim 20$  nm at 390 °C, and at  $\sim 40$  nm at 500–600 °C. Thus, below temperatures of  $\sim 600$  °C, the effect of thermo-oxidation on Be components should not be much more significant than exposure to air at 30 °C which would lead to an oxide layer of  $\sim 10$  nm [39,40]. At some point between  $\sim 650$  and 700 °C there is a transition between protective and non-protective oxidation [39]. In a 1997 study with  $^{18}\text{O}_2$ , Roth et al. [41] were able to show that this transition is due to the diffusion of Be through the oxide layer to react with oxygen on the surface. This process leads to the formation of thick black layers which readily exfoliate, exposing fresh Be surfaces for oxidation. Such an effect was recently observed by Grünhagen et al. [42] at the JET active gas handling facility, where oxidation was being used to release tritium from dust collected from JET. During one run, a continuous loss in mass was observed until the specimen was heated from 600 to 650 °C, at which point a doubling of mass was observed, suggesting the complete oxidation of the remaining Be.

It is noted that the tokamak environment may lead to surface conditions which might enhance the formation of more extensive oxide layers than described above. Causey et al. [43] measured the retention of hydrogen in Be following exposure to high fluxes and fluences of 100 eV ( $\text{D}^+ + \text{T}^+$ ) ions. The low values of trapped D were explained by the development of open porosity in the near-surface. Such open porosity would naturally lead to an enhancement of oxygen uptake, independent of temperature. Further experiments illustrating this process were recently published by Zalavutdinov et al. [40], where depth distributions of oxygen in Be were measured with and without ion bombardment (3 keV  $\text{D}^+$ ). The irradiation resulted in an increase of the depth at which oxygen was detected from a few 10's of nm to 200–300 nm at 30 °C. Again, this result would apply to ordinary air exposure as well as thermo-oxidation.

A further safety concern related to the use of Be PFCs in conjunction with water cooling is related to the oxidation of Be by steam. The reason this reaction is a safety concern is the evolution of hydrogen in sufficient quantities to become an explosive hazard in the event of a simultaneous air leak [44]. Hydrogen evolution will not occur for Be exposure to pure  $\text{O}_2$ . In any case, we note that the amount of oxygen that will be introduced to the ITER vessel by water/steam leaks will almost certainly greatly exceed that introduced by thermo-oxidation.

In summary, at temperatures below  $\sim 650$  °C the oxidation of Be is self-limiting, and it is not significantly greater at 350 °C than at room temperature. Ion bombardment may lead to the creation of thicker oxide layers; however, this will occur generally, and is not specifically related to oxidation.

## 5.3. Collateral damage to other tokamak components

An advantage of the thermo-oxidation technique is that codeposits on all internal surfaces may be removed. This also means that all surfaces within the tokamak will be exposed to oxygen at elevated temperature. Several studies have been done on various pure graphites and CFCs, indicating that at the temperatures suggested for thermo-oxidation,  $\sim 350$  °C, negligible erosion will occur [45–47]; oxidation rates can be  $10^4$  to  $10^6$  times higher for codeposits. In the future, it may also be possible to locally heat surfaces where the largest depositions occur, reducing the number of times the entire vacuum vessel is exposed to oxygen while hot.

For various other materials, the situation is not always as clear. Copper components oxidize readily, forming thick black layers which exfoliate, either on their own, or after exposure to air. Such components should be shielded from  $\text{O}_2$ , or kept below  $\sim 200$  °C where possible [48]. A wide range of other components have been tested in our lab at U of T, in preparation for a thermo-oxidation experiment in DIII-D. No other components tested so far have shown any evidence of being affected by oxidation. In particular, a number of very sensitive diagnostic mirrors were completely unaffected [48].

Further evidence of the minor consequences associated with thermo-oxidation comes from a number of recent accidental air leaks in JET [49] and DIII-D [50], in which air entered the vacuum vessels while they were at  $\sim 350$  °C. The only consequence observed at JET was an increase in the resistance of various insulators due to the removal of conducting deposited layers. Interestingly, while there are many exposed copper-coated bolts in JET, there has been no observation of increased Cu contamination of the plasma which might have resulted from exfoliated oxide layers [49].

## 6. Summary and application of thermo-oxidation to ITER

The material mix in ITER means that the projection of any laboratory results to ITER will be highly speculative. Plasma simulation results from the PISCES group with Be-seeded plasmas suggest that the CFC divertor plates will be coated with Be very rapidly, minimizing the erosion of carbon [51,52]. If this is the case, and codeposits are largely Be-based, it is unlikely that thermo-oxidation will be of much practical use. On the other hand, if the majority of deposits do contain a significant fraction of carbon ( $\text{C}/(\text{Be} + \text{C}) \gtrsim 0.5$ ), then laboratory results do show that thermo-oxidation can be effective in removing the tritium [15], although BeC deposits themselves may not be completely removed [19].

It is important to note that the baking temperature in the current ITER design is  $\sim 240$  °C, which is not within the temperature range where thermo-oxidation might be considered to be effective. For effective tritium removal a temperature of 350–400 °C is required. The divertor region, however, might be heated to 350 °C, and thus thermo-oxidation might be effective here, where most deposits are expected. An additional benefit of the heated divertor is that carbon-based dust might also be removed by thermo-oxidation, and this would be especially useful in the divertor region where limitations on the mass of dust allowed (i.e., on hot surfaces) are most restrictive. The most difficult deposits to remove from ITER will be those in tile gaps on the main wall; in particular, the larger gaps around the blanket modules. Due to the low baking temperatures ( $\sim 240$  °C), these codeposits would not easily be

removed by thermo-oxidation, and they would not be removable by any line-of-sight techniques either. Some other strategy will be required to deal with codeposits on these surfaces, unless the baking temperature can be increased.

As part of the over-all conclusion, it is useful to compare the temperature requirements for thermo-oxidation of carbon codeposits with the temperature required to remove tritium from other possible retention sites. To remove >90% of the hydrogen retained in Be codeposits would require temperatures >400 °C [53], and depending on the nature of the trapping sites, T trapped in neutron-irradiated tungsten may require temperatures as high as 600 °C for release [54]. From this perspective, the temperature requirements for the thermo-oxidation of carbon deposits are not as severe as may be required to remove tritium from other materials.

### Acknowledgement

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