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Tritium release from bulk of carbon-based tiles used in JT-60U

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Abstract

Tritium thermal release behavior from the isotropic graphite tile and the CFC tile used as the plasma facing material of JT-60U was experimentally examined. Whole tritium retained in the bulk of tile could not be released by dry gas purge at high temperature in such a period as one day. Utilization of the isotope exchange reaction using purge gas with hydrogen or humid gas was more effective to release the retained tritium. However, approximately 1% of retained tritium was not recovered by the isotope exchange reaction with dry hydrogen even though such high temperature as 1200 °C was applied. Combustion method with oxygen was required to recover all tritium left in the deeper site of the tile. It was observed that combustion of isotropic graphite tile and CFC tile became vigorous at higher temperature than 700 °C though the combustion rate was rather slow at 650 °C.

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1. Introduction

Quantification of tritium release behavior from the plasma facing materials is one of the key issues from the viewpoint of fuel recycling and radioactive safety of fusion reactor. After D-T or D-D plasma discharge experiment, contamination of plasma facing materials with tritium has been confirmed by the large fusion experimental devices. It has been observed in the re-deposited layer produced on the

* Corresponding author. E-mail address: take@nucl.kyushu-u.ac.jp (T. Takeishi). plasma facing materials of JET or TFTR that the amount of tritium trapped is closely related with the amount of re-deposited layer on the tiles [1,2]. Contrary, in the case of D-D experiment at JT-60U, amount of tritium was not related with the thickness of the re-deposited layer on the plasma facing materials [3,4]. These results indicated that energy of triton produced by D-D nuclear reaction gives difference on behavior of tritium in the plasma facing materials.

A plenty of studies had been performed about tritium existing in the re-deposited layer on the tile surface or in the tile bulk within a few micrometers from plasma facing surface, but discussions on

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tritium existing in the deeper site of tile bulk are few [1]. In the case of JT-60U, tritium areal distribution of isotropic graphite tiles and carbon fiber composite (CFC) tiles used at D-D plasma discharge were extensively measured by the imaging plate technique [5,6]. However, it is not well known about tritium depth profile in the plasma facing tiles because this measurement method can measure only the tritium on the surface and within a few μ m from surface of the tile.

For the next step fusion reactors, the tokamak discharge with deuterium, glow or ECR discharges with deuterium, He and He–O₂, oxygen baking etc. has been considered to the reduction method of tritium retained in the plasma facing materials [7], but the quantitative detritiation effect about these methods is not established sufficiently [8,9]. Therefore, accurate quantification for amount of all the tritium retained in the plasma facing material after detritiation operations is needed to evaluate the effect of these detritiation methods.

The combustion method with oxygen is considered to be suitable method to measure the tritium retained not only in the re-deposited layer but also in the tile bulk of plasma facing tile though it may take a little while to complete measurement. In this study, tritium retained in the isotropic graphite tile and CFC tile used at D-D experiments in JT-60U were measured using the dry inert gas purge, inert gas with hydrogen purge or inert gas with humid gas purge and combustion with oxygen. Scanning electron microscopy (SEM) observation was also carried out to see the difference in shape of the tile samples with proceeding of combustion with oxygen.

2. Experimental

2.1. Tile samples

Sample tiles used in this study are isotropic graphite tile with type IG-430U and CFC tile with type PCC-2S. The poloidal positions of sample tiles in the vacuum vessel of JT-60U during D-D discharge experiment are shown in Fig. 1. The poloidal position of tile samples 1–3 (5BP2H) are inner baffle plate (position A in Fig. 1), tile samples 4, 5 (5Md3) are inner equator (position B in Fig. 1) and tile samples 6, 7 (5DM3b) are dome top (position C in Fig. 1), respectively. Details of sample profiles and experimental conditions are listed in Table 1. These sample tiles were exposed to the deuterium plasma in the period from July 1991 to October 1998 (samples 1–5) and from June 1997 to October 1998



Fig. 1. Poloidal position of sample tiles in the vacuum vessel of JT-60U.

Table 1					
Sample	profiles	and	tritium	releasing	temperature

Sample no.	Position in the vacuum vessel (see Fig. 1)	Type of isotropic graphite or CFC	Plasma facing surface area (cm ²)	Weight (g)	Releasing temperature (°C)		
					Dry Ar	Dry H ₂	O ₂ combustion
1	A (5BP2H)	IG430U	5.5	2.55	800	800	800
2	A (5BP2H)	IG430U	4.4	1.15	600	600	600 and 800
3	A (5BP2H)	IG430U	4.1	1.04	400	400	800
4	B (5Md3)	IG430U	2.5	2.28	1200	1200	800
5	B (5Md3)	IG430U	2.7	2.46	900	**23	800
6	C (5DM3b)	*PCC-2S	5.4	0.64	800	800	800
7	C (5DM3b)	*PCC-2S	3.1	4.04	800	800	800

* CFC.

** With H₂O.

(samples 6, 7). Before replacement of graphite tiles from the vacuum vessel, various kinds of detritiation operations such as hydrogen discharge were performed, whose details were described in the reference [3]. Then, several pieces of isotropic graphite tiles and CFC tiles were stored in the container with air atmosphere at room temperature until tritium releasing experiment was performed in the present work.

2.2. Sample preparations and experimental procedure

Tritium release experiments from isotropic graphite tile and CFC tile were performed by purging with dry gas, followed by hydrogen and/or humid gas for isotope exchange reaction. Furthermore, sample tile was fully combusted with oxygen gas to recover the whole tritium left in the tile after thermal release operations. Isotropic graphite tile samples and CFC tile were cut with disc cutter into slices having about 2.5–5.5 cm² of plasma facing surface area for each piece of graphite tile. After cutting, tile samples were crushed into 12–42 meshes and inserted in the quartz tube with inner diameter of 8 mm.

Fig. 2 shows the schematic diagram of the experimental apparatus for tritium release. Crushed sample tiles were heated up to the designed temperature

by an electric furnace. Argon gas was used as the dry purge gas after dehumidification with the packed bed of molecular sieves. Approximately 1000 Pa of hydrogen in argon or water vapor in argon was used for purge gas to estimate the amount of tritium released by the isotope exchange reaction. Furthermore, approximately 20000 Pa of oxygen in argon gas were used for full combustion to estimate the residual tritium in the tile. Hydrogen and oxygen in the argon gas were dehumidified with molecular sieves at purge of tritium from tile samples. Tritium released from the sample tile was introduced to the granular copper oxide bed at 350 °C to convert the tritium of molecular form to the tritiated water. Converted tritium was collected in the dual water bubblers and a 1 cm³ of water in the bubbler was taken with time. Two water samples of 1 cm³ each were taken for one releasing operation and their radioactivity were measured by a liquid scintillation counter. After each releasing operations, sample bed and piping were purged by humid gas to recover the tritium adsorbed on the inner surface of quartz reaction tube or inner surface of piping into the water bubbler. The amount of tritium detected from the piping inner surface was added to the amount of tritium released at the previous releasing operation. In the case of combustion experiment with oxygen, weight change of the



Fig. 2. Sample preparation and flow diagram of experimental apparatus.

graphite sample was measured with microbalance in every 15 min after sample temperature was lowered to the room temperature. In addition, SEM observation was performed before and after heating for the surface region of cross-section of tile sample to know the progress of combustion situations.

3. Results and discussion

3.1. Surface SEM observations before thermal release

Prior to the thermal release experiments, surface observations of sample tile 1, 2, 4-7 were performed with SEM. The SEM images of cross-section at near the surface region of sample 1, 4 (isotropic graphite) and sample 7 (CFC) are shown in Fig. 3. After D-D discharge experiments of JT-60U, re-deposited layer of about 2 µm was found on the other position of same tile with samples 4 and 5 (i.e. position B) for the inner equator region. Alternatively, the layer of about 3 µm was found on the tile of other place with samples 1-3 (i.e. position B) for the inner first-wall region. However, the SEM observation in this study showed no formation of the re-deposited layer on the surface of any tile samples. Thus, all the tritium detected in the present study is considered to be the tritium released from the bulk of tile sample.

3.2. Measurement of surface tritium before thermal release

At first, amount of tritium left on the surface of the isotropic graphite tile was measured before the thermal release experiment. Approximately 1 g of powdered graphite sample was prepared by grinding of the plasma facing surface of tiles. Powdered graphite sample was soaked into the 20 cm³ of distilled water at the room temperatures. Tritium concentration in the water with these graphite samples gives no tritium transfer during soaking of about 14 months. It is observed in our previous study that the isotope exchange reaction between tritium on the material surface and water vapor is so fast even at the room temperature [10]. Thus, it is considered that tritium on the graphite surface was already removed during such a detritiation operation as hydrogen discharge or storage in the container after replacement from the vacuum vessel.

3.3. Tritium thermal release and combustion experiment

Fig. 4 shows the fraction of tritium released from isotropic graphite tile sample 1 with time. All releasing operations were performed at 800 °C except otherwise noted. Part A framed by dashed ellipse indicates the fraction of tritium released at purge with dry argon gas. Part a shows the fraction of tritium released at purge with argon with about 1000 Pa of hydrogen (open circle) and humid argon (solid circle) at 22 °C. Part B shows the amount of tritium released at purge with argon gas with about 1000 Pa of hydrogen at 800 °C. Further tritium release is observed at combustion of graphite tile with oxygen at 800 °C as shown in part C.

About 6000 Bq of tritium was released in 24 h at the initial release with dry argon gas purge (part A). This amount corresponds to about 1090 Bq per 1 cm^2 of unit plasma facing surface area and 26.6% of tritium released by the whole releasing operations. As shown by this figure, most of tritium released at purge with dry gas was released within initial few hours and only a little was released afterwards.

After purge with dry argon gas was continued for 24 h, sample temperature was lowered to the room temperature and purge gas was changed to argon gas with 1000 Pa of hydrogen. This purge for



Fig. 3. SEM images of cross-section at near surface region of isotropic graphite tile and CFC tile samples.



Fig. 4. Fraction of released tritium from isotropic graphite sample 1 by diffusion, isotope exchange and combustion with time.

30 min gave no detectable tritium release. However, additional 2400 Bq of tritium (10.7% of total tritium released) was released as shown in part a when purge gas was changed to the argon gas with water vapor of which partial pressure was 1000 Pa at the room temperature. The tritium release behavior from tile bulk shown in part A is interpreted as follows. Tritium existing in the deep site of tile bulk begins to move with the increase of releasing temperature from room temperature. The movement of tritium becomes more active at higher temperature than 500-600 °C [11]. Tritium diffuses from the initial trapping site of the tile grain, and some of the tritium is released to the gas phase in the form of molecular tritium through the open pore of the tile after recombination at the grain surface [12]. Other tritium is captured to the trapping site such as -C-O-H bases or -C-H bases in the shallow site of grain by isotope exchange reaction.

Isotope exchange reaction between molecular form hydrogen in the gas stream and tritium atom existing in the graphite grain is too slow at room temperature. On the other hand, as described in the above section, the isotope exchange reaction between hydrogen of water vapor in the gas stream and tritium on the graphite surface is extremely fast compared with the isotope exchange reaction between molecular hydrogen in the gas stream [10]. Thus, tritium existing in the trap site at the shallow depth of graphite tile is likely to be quickly released after purge gas is changed from hydrogen gas to humid argon gas as shown in part a of Fig. 4. This tritium releasing phenomenon corresponds to the fact that implanted tritium during the D-D discharge experiment was already released from the tritium trap sites at the shallow depth of graphite tile by such detritiation operations as hydrogen discharge performed in the vacuum vessel [13].

After tritium release by isotope exchange reaction with humid gas at room temperature, purge gas was changed to argon with about 1000 Pa of dry hydrogen gas and temperature of graphite tile was elevated to 800 °C. About 9100 Bq of tritium (40.6% of total tritium released) was released by isotope exchange reaction as shown in part B. It is considered that tritium existing in the trap site at the shallow depth of graphite tile was already replaced with hydrogen atoms of humid purge gas at the end of the previous releasing procedure at room temperature. In addition, recoverable tritium by diffusion at the high temperature was already released in the initial few hours of dry argon gas purge as shown in part A. Therefore, behavior of tritium released by isotope exchange reaction with molecular hydrogen at the high temperature shown in part B is considered to be originated mainly from the following process. Molecular hydrogen in the purge gas dissociates on the surface of graphite tile grain and penetrates to the deep site of graphite tile grain where tritium exists in the form of -C-O-T bases or -C-T bases. This tritium is replaced by the isotope exchange reaction with hydrogen and diffuses to the surface of the grain. Finally, tritium recombines

at the grain surface and leaves for the gas phase through the open pore in the graphite bulk.

Since combustion of the graphite by the water gas reaction $(C + H_2O \rightarrow H_2 + CO)$ is reported at the higher temperature than about 950 °C [14], humid gas was not used at high temperature to avoid the tritium release by reduction of carbon tile in the present study. Tritium releasing rate with humid gas at the high temperature would be faster than the release with molecular hydrogen flow at the high temperature because of the combustion of tile.

After the tritium release operations with dry argon gas and hydrogen gas, the piping between reaction tube and water bubblers were purged with approximately 1000 Pa of humid gas. If released tritium takes in the form of water vapor, a considerable amount of tritium would be adsorbed on the inner surface of piping, and desorbs from surface immediately after the purge with humid gas even at room temperature. But, in fact, detectable amount of tritium was not released by these purges. It is considered that the chemical form of tritium thermally released with dry gas purge and dry hydrogen gas purge is molecular tritium.

In the ITER design, recovery of tritium from redeposited layer is considered by combustion with oxygen [15]. Haasz et al. [16] and Wang et al. [17] reported that re-deposited layer of TFTR was combusted completely at the temperature around 450 °C. On the other hand, present authors experimentally confirmed that the isotropic graphite combusted at the temperature around 600 °C and the higher than 700 °C is required to get the smooth combustion in short period [13]. Thus, tritium left in the tile bulk after the former operations were released by combustion with oxygen at 800 °C. As shown in part C of Fig. 4, about 4800 Bq of tritium (22.1% of total tritium released) was additionally detected at combustion for 4 h. Therefore, the full combustion method with oxygen was employed in this study to quantify the all amount of tritium remained in the samples of isotropic graphite and CFC tile bulk after detritiation operations with dry gas purge and isotope exchange reaction.

The fraction of tritium released at various releasing operations and at various temperatures for the isotropic graphite tile samples 1–3 used for inner baffle plate is compared in Fig. 5. The time of the tritium releasing operations with dry argon gas and dry hydrogen gas were limited to 24 h. Releasing experiments by combustion with 20% of oxygen were performed at 800 °C and continued till tile sample disappeared completely. For the sample 2, tritium release by combustion with oxygen was applied at



Fig. 5. Fraction of released tritium from isotropic graphite samples 1-3 by diffusion, isotope exchange and combustion with time.

600 °C for initial 10 h (part A), and then sample temperature was elevated to 800 °C (part B). In the releasing operations with hydrogen performed at 600 (part D) and 800 °C (part E), it seems that further tritium release is expected when releasing period is extended to 1 week or longer because release of tritium is not completed in 24 h. Meanwhile, only about 3% of tritium was released with dry argon gas and following hydrogen gas purge at 400 °C. The achievable maximum temperature of plasma facing wall in the vacuum vessel of ITER is expected to be around 230 °C [18]. Thus, it is considered that amount of recoverable tritium in the vacuum vessel at these temperature is very small even though any kind of gas is used as purge gas. In the tritium release by combustion with oxygen, the release for the sample 2 at 600 °C (part A) and following purge at 800 °C (part B) had been performed to purge piping between the reaction tube and the water bubbler with approximately 1000 Pa of humid gas while tritium release. On the other hand, release at 800 °C for the sample 1 (part C) had been performed to purge piping without water vapor.

In the case of sample 2, tritium was quickly released when temperature was elevated from $600 \,^{\circ}$ C to $800 \,^{\circ}$ C (part B). But in the case of sample 1, several hours was required to release all tritium. It is well known that considerable amount of tritium is adsorbed on the inner surface of piping if released tritium takes form of water vapor and the desorption of tritium from piping surface is slow at dry

gas purge [19]. Therefore, it is concluded that the tritium released by combustion forms HTO.

The integral amount of combusted isotropic graphite tile or CFC tile with time in 20% of oxygen/argon gas is shown in Fig. 6, where the same size of sample (i.e. 12–42 mesh) is used as that used for tritium release by dry argon gas or about 1000 Pa of dry hydrogen gas. This figure indicates that combustion of the both tile samples became active at the temperature above 650 °C and became vigorous at higher temperature than 700 °C. However, faster release of tritium was observed in the oxygen flow at 600 °C (part A of Fig. 5) than the former hydrogen flow at 600 °C (part D of Fig. 5), slight combustion of graphite might occur.

In Fig. 7, fractional amount of tritium additionally released by combustion with oxygen from isotropic graphite and CFC is compared for all samples with amount of tritium released in the former release by diffusion and/or isotope exchange reactions. Approximately 1% of totally released tritium is obtained at combustion with oxygen at 800 °C following the release with dry argon gas and argon gas with hydrogen at 1200 °C, though 97% of totally released tritium is obtained at combustion following the purge at 400 °C.

These results for isotropic graphite and CFC tiles mean that all the tritium retained in the tile cannot be recovered only by diffusion and isotope exchange reaction at high temperature. Significant difference in tritium thermal release behavior with diffusion,



Fig. 6. Amount of combusted isotropic graphite and CFC with time.



Fig. 7. Comparison of fraction and amount of tritium released from isotopic graphite and CFC tile sample 1–7 by diffusion, isotope exchange and combustion.

isotope exchange reaction and combustion with oxygen were not found between isotropic graphite and CFC tile.

Detritiation with ultrasonic bath treatment, moist ozone, moist noble gases under RF heating etc. were performed on isotropic graphite and CFC tiles used to JET and TFTR [2]. Some of these detritiation methods were effective to eliminate the most of retained tritium in the bulk, but any methods could not eliminate all retained tritium. Thus, full combustion with oxygen at the high temperature is necessary to recover all tritium in the bulk of carbon materials.

Thermal release experiments on isotropic graphite and CFC tile of which surface was at least 2 mm far from the plasma facing surface was also performed in the same way as the tile samples which have the plasma facing surface. Because detectable amount of tritium was not released from these samples, it is considered that tritium produced by D-D nuclear reaction was not implanted directly into the graphite deeper than certain millimeters from the plasma facing side.

3.4. Progress of combustion

In the tritium release by combustion with oxygen, it is important to know the relationship between the progress of carbon combustion and the amount of tritium released. It has been reported that the tritium implanted in the tile existed within a few micrometers from plasma facing surface [5,6]. If isotropic graphite tile and CFC tile give combustion only at the surface of the plasma facing side of tile bulk, complete recovery of tritium from carbon tile bulk would become easy. Thus, SEM observation was performed to see the difference in shape of the graphite tiles with proceeding of combustion with oxygen.

A lamellar isotropic graphite tile or CFC tile samples was used for SEM observation. Outer sizes of both samples are approximately $10 \text{ mm} \times 5 \text{ mm}$ and 1 mm in thickness. After the tile sample was combusted for 15 min or 45 min with 20% of oxygen/argon gas at 800 °C, the temperature of the tile sample was lowered to the room temperature. Then sample was fractured into two pieces and SEM observation was performed at the fracture face.

Fig. 8(a) and (b) shows SEM images on top surface layer of isotropic graphite tile combusted with 20% of oxygen for 15 min and Fig. 8(c) and (d) shows for 45 min. Fig. 9(a) and (b) shows SEM images of CFC tile combusted for 15 minutes and Fig. 9(c) and (d) shows for 45 min. Change of the combustion ratio with time on isotropic graphite and CFC are plotted in Fig. 10. Although about 87% of isotropic graphite combusted for 45 min, grain size of isotropic graphite (Fig. 8(d)) seems to be almost the same as that combusted for 15 min (Fig. 8(b)), when about



Fig. 8. SEM images of isotropic graphite grain combusted for 15 min (a) and (b); 45 min (c) and (d) with 20% of oxygen at 800 °C.



Fig. 9. SEM images of CFC tile fiber combusted for 15 min (a) and (b); 45 min (c) and (d) with 20% of oxygen at 800 °C.

18% of isotropic graphite combusted. In contrast, although the diameter of CFC fiber seems scarcely changed between combusted for 45 min (Fig. 9(d)) and for 15 min (Fig. 9(b)), the size of pore scattered on the outer circumference of fiber extended and became porous throughout the depth direction of tile. Combustion ratio measured at 15 min and at 45 min were 15% and 65%, respectively. These results show that isotropic graphite and CFC do not combust only at surface of tile bulk. It seems that all grain or all fiber of tile combust uniformly within about 1 mm of depth from the surface of the tile bulk. It is conceivable that the tritium implanted by discharge

remains in the deep site of the isotropic graphite and CFC grain located at the shallow region of tile bulk from plasma facing surface. Therefore, there is the possibility that tritium retained in the grain of carbon material remains until all tile bulk combust completely.

3.5. Tritium release within a short period

The fraction of recoverable tritium from isotropic graphite tile bulk and CFC tile bulk by detritiation operation of one day is compared in Fig. 11 for three different release procedures at various temperatures.



Fig. 10. Change of the combustion ratio of isotropic graphite and CFC with time.



Fig. 11. Fraction of recoverable tritium from carbon-based tile bulk by thermal releasing operations for 1 day.

The fraction of tritium released by dry gas purge is drawn by short dashed line, released by hydrogen gas purge or humid gas purge is drawn by chain line and released by combustion is drawn in solid line, respectively. It is assumed in this comparison that no detectable re-deposited layer is formed on the plasma facing surface, and tritium existing in the shallow depth of tile was already released by detritiation operations such as hydrogen discharge.

Because the diffusion coefficient of tritium in the carbon material is so small and combustion of both tiles are not observed at lower temperature than $300 \,^{\circ}C$ [11], the amount of tritium released by any releasing operation at lower temperature than

300 °C is few. Amount of tritium released from tile bulk increases with elevated temperature but it is expected that roughly 10% of tritium remains after detritiation procedure by isotope exchange reaction with dry hydrogen gas at the high temperature of 1000 °C.

In the case of tritium release by the isotope exchange reaction with hydrogen gas and humid gas at the room temperature, tritium is not released because initially no tritium exists in the shallow region of tile bulk. However, tritium transferred from the deeper site to the shallow site of tile with diffusion at the high temperature can be released with humid gas at the room temperature because isotope exchange reaction between water vapor in the gas phase and tritium on the surface is large even at the room temperature [10,11]. In contrast, tritium exists in the near surface region is not recovered by the isotope exchange reaction with dry hydrogen gas purge because isotope exchange reaction between hydrogen in the gas phase and tritium on the surface is much slower compared to the purge with water vapor at room temperature. Since the dissolution of hydrogen occurs at the high temperature [11], it is expected that the tritium release from the deeper site of tile grain with dry hydrogen purge also occurred at the high temperature.

It was observed in this study that several percent of tritium was still left in the tile after purge with dry gas at such the high temperature as 1200 °C. Thus, as shown in the solid line, combustion with oxygen at the higher temperature than 700 °C is required to recover all tritium retained in the carbon-based tile within such short time as one day.

It is noted that combustion of carbon materials with oxygen generates carbon monoxide when temperature is higher than 1000 °C, and that tritiated methane is produced in the purge with dry gas around 500 °C. Currently, the tritium measurement system comprising copper oxide bed with water bubbler has been used. However, all tritium released from carbon material may not be detected in the system because oxygen of copper oxide could be consumed to convert carbon mono oxide to carbon dioxide at temperature is higher than 1000 °C and tritiated methane could not be oxidized with copper oxide at 350 °C.

4. Conclusions

Tritium thermal release experiments with dry gas purge, hydrogen or humid gas purge and combustion with oxygen were performed on the isotropic graphite tile and the CFC tile used in the D-D discharge experiment of JT-60U.

Tritium retained in these tile was not released by dry inert gas purge at the room temperature and the detectable tritium was observed at the temperature higher than 400 °C. Hydrogen or humid gas purge were more effective to abbreviate the time for releasing tritium by taking the advantage of the isotope exchange reaction. Tritium released with dry gas or hydrogen gas purge took molecular form of tritium. Approximately 1% of retained tritium was not recovered by isotope exchange reaction with dry hydrogen even though such high temperature as 1200 °C was applied. If detritiation period is restricted to short time as one day, oxygen full combustion was required to recover all tritium still remained in the graphite bulk after detritiation by dry gas and humid gas purge. Isotropic graphite tile and CFC tile were not combusted under 600 °C, and began to be combusted slowly at 650 °C. Combustion with oxygen at the higher temperature than 700 °C is required to recover the whole retained tritium within practical period as one day, and it seems that released tritium was in the form of HTO.

The carbon materials at least about 1 mm of depth from the oxygen facing side combusted in the form of all grain in the tile bulk combust uniformly. It is considered that recovery of all the tritium retained in the plasma facing layer by combustion with oxygen is not easy to be carried out in the vacuum vessel because it is difficult to heat the tile attached on the inner wall of the vacuum vessel to higher temperature than 700 °C.

References

- R.-D. Penzhorn, N. Bekris, U. Berndt, J.P. Coad, H. Ziegler, W. Nagele, J. Nucl. Mater. 288 (2001) 170.
- [2] N. Bekris, C. Caldwell-Nichols, L. Doerr, M. Glugla, R.-D. Penzhorn, H. Ziegler, J. Nucl. Mater. 307–311 (2002) 1649.
- [3] K. Masaki, J. Yagyu, T. Arai, A. Kaminaga, K. Kodama, N. Miya, T. Ando, H. Hiratsuka, M. Saido, Fus. Sci. Technol. 42 (2002) 386.
- [4] K. Masaki, K. Sugiyama, T. Tanabe, et al., J. Nucl. Mater. 313–316 (2003) 514.
- [5] T. Tanabe, K. Miyasaka, K. Masaki, K. Kodama, N. Miya, J. Nucl. Mater. 307–311 (2002) 1441.
- [6] T. Tanabe, K. Miyasaka, K. Sugiyama, et al., Fus. Sci. Technol. 41 (2002) 877.
- [7] G. Federich, C.H. Skinner, J.N. Brooks, J.P. Coad, et al., Nucl. Fus. 41 (2001) 1967.
- [8] P. Andrew, P.D. Brennan, J.P. Coad, J. Ehrenberg, M. Gardeberg, Fus. Eng. Des. 47 (1999) 233.

- [9] G. Federich, R. Anderl, J.N. Brooks, R. Causy, et al., Fus. Eng. Des. 39&40 (1998) 445.
- [10] M. Nishikawa, N. Nakashio, T. Shiraishi, et al., J. Nucl. Mater. 277 (2000) 99.
- [11] M. Nishikawa, N. Nakashio, T. Takeishi, S. Matsunaga, K. Kuroki, Fus. Technol. 28 (1995) 1233.
- [12] H. Atsumi, J. Nucl. Mater. 307-311 (2002) 1466.
- [13] T. Takeishi, K. Katayama, M. Nishikawa, N. Miya, K. Masaki, Fus. Sci. Technol. 48 (2005) 565.
- [14] A. Makino, H. Fujisawa, N. Araki, Combust. Flame 113 (1998) 258.
- [15] G. Federici, R.A. Anderl, P. Andrew, J.N. Brooks, et al., J. Nucl. Mater. 266–269 (1999) 14.
- [16] A.A. Haasz, J.W. Davis, J. Nucl. Mater. 256 (1998) 65.
- [17] W. Wang, W. Jacob, J. Roth, et al., J. Nucl. Mater. 245 (1997) 66.
- [18] D.G. Whyte, J.W. Davis, J. Nucl. Mater. 337–339 (2005) 560.
- [19] N. Nakashio, J. Yamaguchi, R. Kobayashi, M. Nishikawa, Fus. Technol. 39 (2001) 189.