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Surface inventory of tritium on Li_2TiO_3

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Abstract

Though lithium ceramic materials such as Li_2O, $LiAlO_2$, Li_2ZrO_3, Li_4SiO_4 and Li_2TiO_3 are considered as breeding materials in the blanket of a D-T fusion reactor, the release behavior of the bred tritium in these solid breeder materials has not been fully understood, yet. It has been pointed out by the present authors that it is essential to understand such mass transfer steps as diffusion of tritium in the grain of breeder material, absorption of water vapor into bulk of the grain, and adsorption of water on surface of the grain, together with two types of isotope exchange reactions for estimation of the tritium inventory in a uniform solid breeder blanket under the steady-state condition. The adsorption capacity and the isotope exchange capacity on the Li_2TiO_3 surface are experimentally obtained in this study. Comparison of the tritium inventory estimated for the Li_2TiO_3 blanket with the inventory for $LiAlO_2$, Li_2ZrO_3 or Li_4SiO_4 blanket using the model presented by the present authors shows that the Li_2TiO_3 blanket gives the smallest tritium inventory among the candidate ceramic breeding materials.

1. Introduction

In order to estimate the tritium inventory in a solid blanket system with sintered pebbles of micro crystal grains, it is necessary to know the contribution of such tritium transfer steps [1,2] as

1. tritium formation reaction in crystal grain,
2. diffusion of tritium in crystal grain,
3. interaction of tritium with irradiation defects in crystal grain,
4. adsorption of tritium on grain surface,
5. absorption of tritium into the bulk of crystal grain,
6. isotope exchange reaction between gaseous hydrogen, H_2, in the gas stream and tritium on grain surface (isotope exchange reaction 1),
7. isotope exchange reaction between water vapor, H_2O, in the gas stream and tritium on grain surface (isotope exchange reaction 2),
8. water formation reaction at addition of H_2 to the blanket purge gas,
9. transfer of hydrogen isotopes and water through pores of the sintered pebble,
10. transfer of hydrogen isotopes and water through boundary layer formed on the surface of a sintered pellet to the gas stream.

Steps (1), (2) and (3) decide the diffusion inventory of tritium at the steady-state condition which corresponds to tritium in crystal grains when no absorption occurs. Step (5) decides the absorption inventory due to absorption of water vapor or molecular form hydrogen, H_2. The present authors have observed that water vapor is absorbed into Li_2O and that Li_2O, $LiAlO_2$, Li_2ZrO_3 and Li_4SiO_4 have no detectable absorption capacity of molecular form hydrogen [3]. Steps (4), (6), (7) and (8) decide the surface inventory of tritium which corresponds to tritium on the surface of crystal grains. Step (9) decides the tritium inventory in micro pores of sintered pebbles.

The amount of water captured on Li_2O, $LiAlO_2$, Li_2ZrO_3 and Li_4SiO_4 have been reported in the previous papers [2–6], and the amount of isotope exchange capacity of $LiAlO_2$ and Li_2ZrO_3 have been also reported by the present authors [1]. Comparison of the

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tritium inventory in the blanket by the present authors shows that the inventory due to adsorption is much larger than that due to diffusion unless a large amount of H_2 or water vapor is added to the purge gas in the high temperature blanket when the grain size is smaller than ten microns, though the inventory due to diffusion plays an important role in the blanket at lower temperature with larger grain size. The absorption inventory in the Li_2O blanket exceeds the adsorption inventory and diffusion inventory at the higher temperature than 700 K in the case when water vapor presents in the blanket purge gas.

It has been found that the water formation reaction occurs when the purge gas with hydrogen is introduced to a bed of such ceramic breeder materials as Li_2O, $LiAlO_2$, Li_2ZrO_3 and Li_4SiO_4 [7], and the reaction rate and the capacity of water formation have been also quantified for some solid breeder materials [8].

The previous works of the present authors shows that the isotope exchange reaction 2 proceeds much faster than the isotope exchange reaction 1. Namely, effect of water vapor in the blanket purge gas cannot be disregarded in consideration of tritium behavior in blanket because the partial pressure of tritium in the purge gas is low.

The estimated tritium inventory for a $LiAlO_2$ blanket using the model by the present authors [1] considering diffusion, adsorption and isotope exchange reactions agrees well with data observed in various in situ experiments such as LILA-3 [9], TEQUILA-1 [10], CORELLI-2 [11], MOZART [12], and EXOTIC-6 [13] experiments. And the tritium inventory estimated for a Li_2ZrO_3 blanket using the same method [2] agrees well with data reported in EXOTIC-5, EXOTIC-6, MOZART and TRINE [14] experiments. It is also reported in these papers that the better agreements are observed when the effectiveness correction factor is introduced for comparison of tritium uptake by two competitive isotope exchange reactions [2], and when existence of some water vapor is assumed in the purge gas [1,2]. The same method as that used in estimation of the tritium inventory in a $LiAlO_2$ blanket [1] or a Li_2ZrO_3 blanket [2] is also applied to estimate the tritium inventory in a Li_2TiO_3 blanket in this study.

2. Inventory estimation method

Contributions of diffusion inventory in crystal grains I_D, absorption inventory in bulk of lithium ceramic I_{ab}, adsorption inventory on the grain surface I_{ad} and inventory due to the isotope exchange capacity on the grain surface I_{ex} to the total tritium inventory in a solid I_{Total} is given as

$$\begin{aligned}I_{Total} &= I_D + I_{ab} + I_{ad} + I_{ex}\\ &= G_T d_p^2/60D_T + Q_{ab}MP_{T_2O}/(P_{T_2O}. + P_{H_2O} + P_{H_2O'})\\ &\quad + Q_{ad}MP_{T_2O}/\{(P_{H_2}/\alpha_{ex}). + (P_{T_2O}. + P_{H_2O}\\ &\quad + P_{H_2O'})/\alpha'_{ex}\} + Q_{ex}MP_{T_2O}/\{(P_{H_2}/\alpha_{ex}).\\ &\quad + (P_{T_2O}.. + P_{H_2O} + P_{H_2O'})/\alpha'_{ex}\} \qquad (1)\end{aligned}$$

in the uniform solid breeder blanket under the steady-state condition, where G_T is the tritium generation rate in the whole blanket (mol T_2/s), d_p the grain diameter (m), D_T the effective diffusivity of tritium in grain (m^2/s), P_{H_2} the partial pressure of H_2 in the purge gas (Pa), P_{H_2O} the partial pressure of H_2O swamped to the purge gas (Pa), $P_{H_2O'}$ the partial pressure of residual H_2O in the purge gas (Pa), M the amount of Li ceramics in blanket (mol), α_{ex}, α'_{ex} the effectiveness correction factor (–), Q_{ab} the amount of water absorption (mol H_2O/mol Li compound), Q_{ad} the amount of water adsorption (mol H_2O/mol Li compound), and Q_{ex} is the isotope exchange capacity (mol H_2O/mol Li compound).The partial pressure of tritium vapor in the purge gas, P_{T_2O}, is given as

$$P_{T_2O} = P_{He}G_T/G_{He}\ (\mathrm{Pa}), \qquad (2)$$

where all tritium bred in the grain of a solid breeder material is considered to be released in the chemical form of water, and P_{He} (Pa) and G_{He} (mol/s) are total pressure of helium purge gas and flow rate, respectively.

It is assumed in the uniform blanket model of this study that the isotope exchange reaction 1 and the isotope exchange reaction 2 compete with each other in extraction of tritium to the purge gas from the breeder surface when both H_2 and H_2O exist in the purge gas. In such a case, the effectiveness correction factor shown in Eq. (1) is given by

$$\begin{aligned}&\alpha_{ex} = k_{ex2}/k_{ex1} && \text{when } k_{ex2} \geqslant k_{ex1},\\ &\alpha'_{ex} = 1 && \text{when } k_{ex2} \geqslant k_{ex1}\\ &\text{or} && \qquad (3)\\ &\alpha_{ex} = 1 && \text{when } k_{ex2} \leqslant k_{ex1},\\ &\alpha'_{ex} = k_{ex1}/k_{ex2} && \text{when } k_{ex2} \leqslant k_{ex1},\end{aligned}$$

where k_{ex1} and k_{ex2} mean the rate constant of the isotope exchange 1 and that of isotope exchange 2, respectively. Accordingly, the effectiveness correction factor means the relative intensity of two isotope exchange reactions.

The total pressure of water vapor of various forms in the purge gas is given as

$$P = P_{T_2O} + P_{H_2O} + P'_{H_2O}\ (\mathrm{Pa}), \qquad (4)$$

where HTO is treated as a mixture of $\frac{1}{2}T_2O$ and $\frac{1}{2}H_2O$ in this study following the way taken in consideration of tritium behavior in a catalyst bed or an adsorption bed by the present authors [15].

The mass transfer resistance at transfer of tritium through pores of sintered pebbles and that at transfer through boundary layer formed around the pebble surface are considered to be negligibly small in this study.

The average residence time of tritium at each transfer step is given by division of inventory by the tritium generation rate.

$$\begin{aligned}\theta_{\mathrm{Total}} &= (I_{\mathrm{D}} + I_{\mathrm{ad}} + I_{\mathrm{ab}} + I_{\mathrm{ex}})/G_{\mathrm{T}} \\ &= \theta_{\mathrm{D}} + \theta_{\mathrm{ad}} + \theta_{\mathrm{ab}} + \theta_{\mathrm{ex}}\ (\mathrm{s}). \end{aligned} \tag{5}$$

As can be seen from the above equations, the effects of tritium generation rate, temperature, grain size, partial pressure of H_2, partial pressure of water vapor or flow rate of purge gas on each tritium inventory are different.

3. Experimental

The schematic diagram of experimental apparatus is shown in Fig. 1. At the tritium sorption procedure, tritium is introduced to the sample bed after conversion of HT to HTO using the CuO bed. Then, dry nitrogen gas and humidified nitrogen gas are introduced in turn to purge tritium from the sample bed. The details of the experimental procedure are descried in the previous papers on the system effect of tritium [16,17].

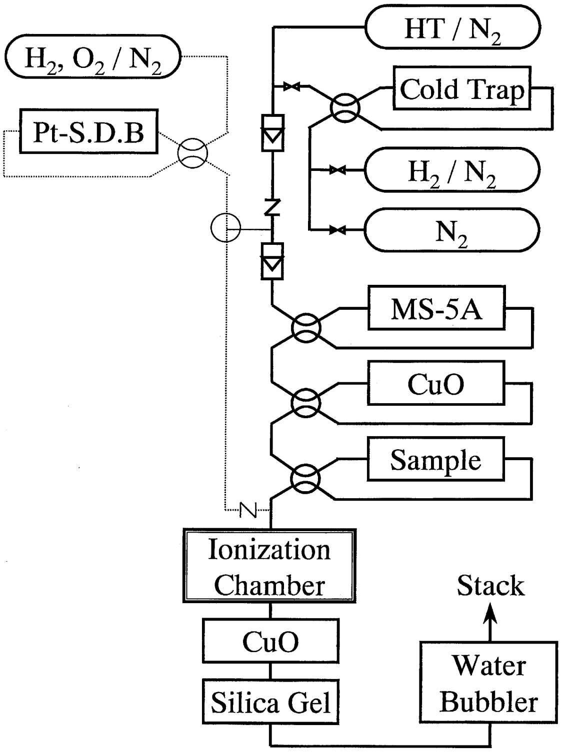

Fig. 1. Schematic diagram of the experimental apparatus.

Fig. 2(a) shows the change of reading of an ionization chamber with time after introduction of HTO/N_2 gas to the system where HTO is formed by oxidation of HT in the hot CuO bed. The first concave of reading in this figure was obtained when the flow of HTO to the Li_2TiO_3 packed bed in the quartz tube was by-passed, that is, this concave corresponds to tritium sorbed onto the surface of pipings. The second concave obtained when the flow of HTO was connected to the Li_2TiO_3 packed bed corresponds to tritium sorbed to Li_2TiO_3, quartz tube and pipings around the packed bed and to tritium remind at void in the quartz tube and pipings.

Fig. 2(b) shows the reading of an ionization chamber obtained when desorption operation is performed using dry N_2 or humidified N_2 as the purge gas after the sorption operation shown in Fig. 2(a) is finished. The first peak in Fig. 2(b) correspond to tritium released by

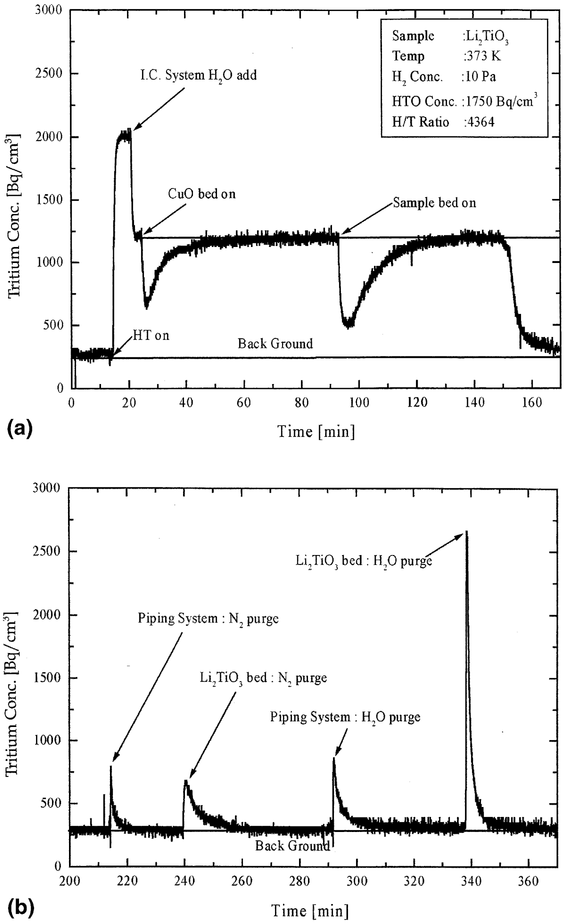

Fig. 2. (a) Example of output curve of the ionization chamber at sorption step and (b) at desorption step.

Table 1
Experimental condition

Sample name	Li_2TiO_3 (NFI)[a]	Li_2TiO_3 (CEA)[b]
Grain size (μm)	1.0	1.0
Density (g/cm^3)	2.57 (75%TD)	2.84 (83.1%TD)
BET surface area (m^2/g)		
Carrier gas	N_2	N_2
Purge gas to release tritium	N_2 with water vapor	N_2 with water vapor
Flow rate (l/min)	0.45	0.45
Concentration of hydrogen isotopes (Pa)	7.5–250 Pa	7.5–250 Pa
H/T ratio	1902–140 000	2100–115 000
Temperature (K)	296–773	473–773
Bed height (mm)	10	10
Bed diameter (mm)	18	18
Tritium concentration	0.05–0.25	0.05–0.25
Pellet size	0.8–1.2 mm	16–32 mesh

The BET surface area in this Table is estimated from the grain size evaluated using SEM pictures.
[a] Nuclear Fuel Industries Ltd. (Japan).
[b] The France Atomic Energy Commission (France).

dry N_2 gas from the surface of pipings. From the second peak, the amount of tritium releasable from Li_2TiO_3 by the purge using dry N_2 gas Q_{N_2} was evaluated. The third peak correspond to tritium released by humidified N_2 gas from the surface of the pipings, and the amount of tritium released from Li_2TiO_3 only by the isotope exchange reaction Q_{H_2O} was evaluated from the forth peak.

The amount of tritium captured to Li_2TiO_3 itself is obtained by subtraction of tritium sorbed to quartz tube and pipings evaluated from the blank test performed at each experiment from the amount of tritium evaluated from the second concave at sorption experiment as shown in Fig. 2(a) or from the second and fourth peaks in desorption experiment as shown in Fig. 2(b).

The total amount of tritium obtained from the first and third peaks, or from the second and forth peaks in Fig. 2(b) agree with the amount obtained from the first concave or second concave within 10% difference, respectively.

Experimental conditions of this study are shown in Table 1. The grain size is evaluated from SEM pictures considering that the shape of the crystal grain is spherical in this study. No closed pores are assumed in calculation of the tritium inventory.

4. Results and discussions

4.1. Tritium sorption capacity

The tritium sorption capacity Q_{N_2} which corresponds to the amount of tritium releasable by the purge operation using dry N_2 gas from Li_2TiO_3 are shown in Fig. 3. It can be seen from this figure that Q_{N_2} obtained for pebbles from NFI agrees with Q_{N_2} for pebbles from CEA.

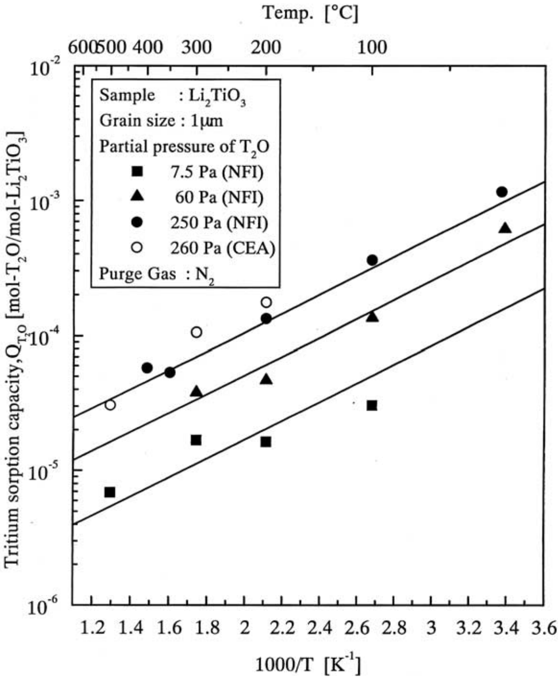

Fig. 3. Adsorption capacity releasable by dry gas purge observed for Li_2TiO_3.

It has been considered by the present authors that some amount of water or OH bases exists on the surface of various materials and they are named as the surface water as a whole [18]. It is also considered that the surface water consists of physically adsorbed water, chemically adsorbed water and structural water, and that the physically adsorbed water changes its amount reversibly with the vapor pressure in the gas phase at the given temperature though the chemically adsorbed water cannot be desorbed by the purging operation with dry gas at the same temperature as it is adsorbed. The structural water represents any chemically adsorbed water, crystal water and some chemical groups which are strongly connected with surface and its amount does not depends on the vapor pressure. Then, $Q_{N_2}(=Q_{ad,1})$ corresponds to the physically adsorbed water and the following equation is obtained for the range of this work.

$$Q_{N_2} = 1.5\times 10^{-7}A_{BET}P^{1/2}\exp(13.4\ [\mathrm{kJ/mol}]/RT) \\ \times (\mathrm{mol\ H_2O/mol\ Li\ compound}), \qquad (6)$$

where A_{BET} $(=6/\rho d_p,\ \rho$: density $[\mathrm{g/m^3}])$ is the BET surface area of the breeder particles where grain is assumed to be spherical.

The tritium sorption capacity Q_{H_2O} which corresponds to the amount of tritium that can be extracted from Li_2TiO_3 by the purge with humidified N_2 gas from Li_2TiO_3 after the purge with dry N_2 gas are shown in Fig. 4 and the following equation is obtained:

$$Q_{H_2O} = \frac{3.7\times 10^{-21}A_{BET}\exp(174.6\ [\mathrm{kJ/mol}]/RT)}{1+1.1\times 10^{-16}\exp(174.6\ [\mathrm{kJ/mol}]/RT)} \\ + 3.2\times 10^{-7}A_{BET}P^{1/2} \\ \times \exp(10.6\ [\mathrm{kJ/mol}]/RT) \\ \times [\mathrm{mol\ H_2O/mol\ Li\ compound}], \\ = Q_{ex} + Q_{ad,2}. \qquad (7)$$

It is considered that Q_{H_2O} includes the isotope exchange capacity Q_{ex}, which corresponds to the structural water, and the adsorption capacity which corresponds to the chemically adsorbed water $Q_{ad,2}$. Then, sum of $Q_{ad,1}$ and $Q_{ad,2}$ gives the adsorption capacity Q_{ad}.

Q_{ex} observed for Li_2TiO_3 in this study is much smaller than Q_{ex} observed for $LiAlO_2$ [1] or Li_4SiO_4 [19] by the present authors.

The estimated values using Eqs. (6) or (7) obtained in this study represent well the observed values as can be seen from Figs. 3 or 4 where estimated values are shown by solid lines.

The total tritium sorption capacity on the grain surface $Q_{s,total}(=Q_{ad}+Q_{ex}=Q_{N_2}+Q_{H_2O})$ obtained for Li_2TiO_3 are compared in Fig. 5, where solid lines show the sum of estimated values from Eqs. (6) and (7). It can be said from this figure that the isotope exchange capacity plays a major part of the tritium inventory of the Li_2TiO_3 blanket when the blanket temperature is around 400–600 K because the total vapor pressure in the blanket purge gas must be lower than several Pa.

It is also observed in the course of this study that Li_2TiO_3 has no detectable absorption capacity.

4.2. Comparison of average residence time

Following the results of the present author [20], the isotope exchange reaction rate between H_2 in the purge gas and tritium on the grain surface, r_{ex1}, is shown by

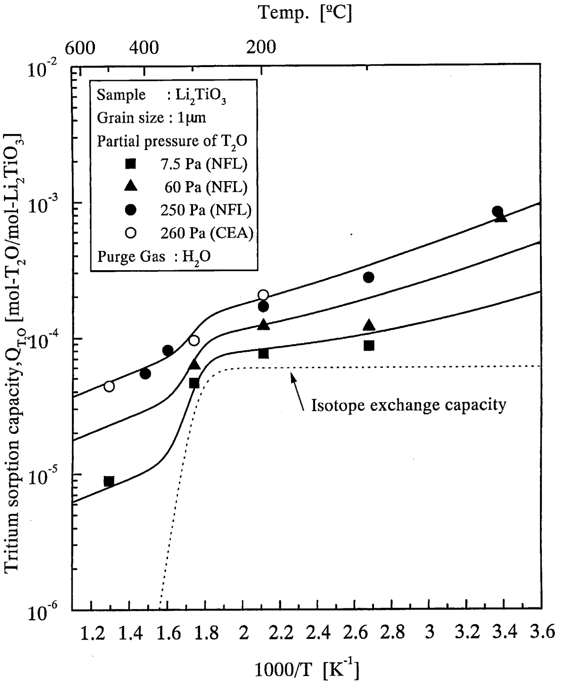

Fig. 4. Tritium sorption capacity Q_{T2O} observed for Li_2TiO_3.

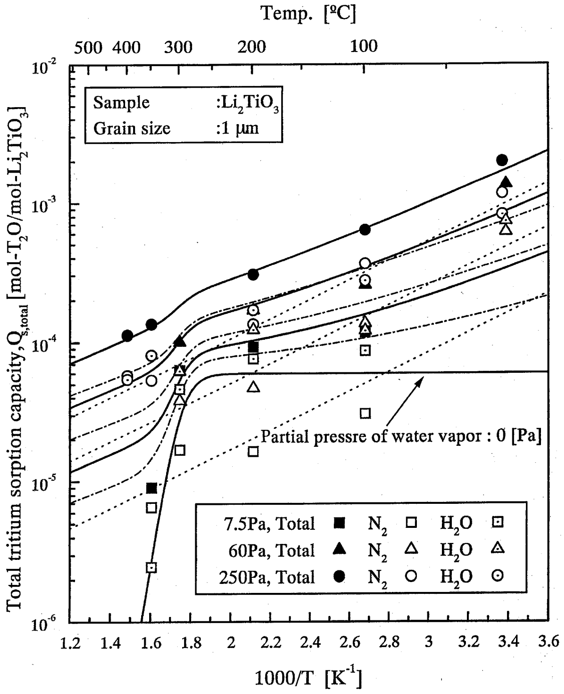

Fig. 5. Total sorption capacity of tritium observed for Li_2TiO_3.

$$r_{\mathrm{ex1}} = k_{\mathrm{ex1}} a_v C_{\mathrm{H_2}} \left\{ 1 - \frac{q_{\mathrm{H_2O}}/(q_{\mathrm{H_2O}} + q_{\mathrm{T_2O}})}{C_{\mathrm{H_2}}/(C_{\mathrm{H_2}} + C_{\mathrm{T_2}})} \right\} \ (\mathrm{mol/m^3\ s}), \tag{8}$$

where k_{ex1} (m/s) is the mass transfer coefficient representing the isotope exchange reaction 1 and a_v (= surface area/volume of packed bed) is the specific surface area of grains in a packed bed. Then, $k_{\mathrm{ex1}} a_v$ is called as the mass transfer capacity coefficient.

In the recent study of the present authors [20] the following equation is obtained for k_{ex1} in the Li_2ZrO_3 bed:

$$k_{\mathrm{ex1}} = 1.6 \times 10^2 \exp(-121\ [\mathrm{kJ/mol}]/RT)\ (\mathrm{m/s}). \tag{9}$$

In this study, k_{ex1} on Li_2ZrO_3 is employed to estimate tritium inventory in the Li_2TiO_3 blanket assuming that k_{ex1} on Li_2TiO_3 is similar to k_{ex1} on Li_2ZrO_3 because k_{ex1} observed for Li_2ZrO_3, $LiAlO_2$, Li_4SiO_4 and Li_2O are similar [21], though this assumption is not empirically assured yet.

The present authors also have presented the following equation for the rate constant of the isotope exchange reaction between H_2O in the purge gas and tritium on the surface of 304SS or copper [22]:

$$k_{\mathrm{ex2}} = 2.20 \times 10^{-4}\ (\mathrm{m/s}). \tag{10}$$

The effectiveness correction factors defined in Eq. (3), $\alpha_{\mathrm{ex}}(= k_{\mathrm{ex2}}/k_{\mathrm{ex1}})$ and $\alpha'_{\mathrm{ex}}(= k_{\mathrm{ex1}}/k_{\mathrm{ex2}})$, are evaluated using above equations in this study, assuming that k_{ex2} on Li_2TiO_3 is similar to k_{ex2} on 304SS as in the case of Li_2ZrO_3 blanket [2], though this assumption is empirically certified only for Li_2ZrO_3.

Then, the surface inventory of tritium in a Li_2TiO_3 blanket can be evaluated for various conditions using Eqs. (2)–(4).

Estimated values of the total residence time of tritium for Li_2TiO_3 blanket purged by 100 kPa He containing 100 Pa H_2 and 1 Pa water vapor are shown in Fig. 6, where operating of a 1 GWe D-T fusion reactor is supposed. Then, the total residence time of 1 h corresponds to the tritium inventory of 16.7 g in the whole blanket.

The effective diffusivity of tritium in the bulk of Li_2TiO_3 is reported by Tanifuji et al. [23] as

$$D_{\mathrm{T}} = 1.00 \times 10^5 \exp(-104\ [\mathrm{kJ/mol}]/RT)\ (\mathrm{m^2/s}). \tag{11}$$

Fig. 6 shows that the residence time of tritium in the Li_2TiO_3 blanket is controlled by the surface inventory even at so low temperature as 573 K even when the grain size is 20 μm, because the effective diffusivity from Eq. (11) is much larger than that of $LiAlO_2$ or Li_2ZrO_3 which is evaluated from data in the EXOTIC-6 experiment. Accordingly, it can be said that the tritium release behavior from the grain surface is important in discussion of Li_2TiO_3 blanket.

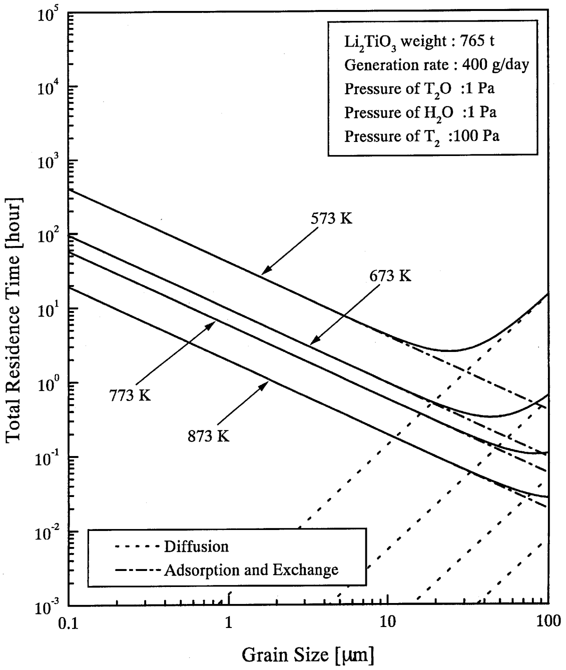

Fig. 6. Effect of grain size and temperature on total residence time of Li_2TiO_3 blanket.

The estimated values of the total residence time of tritium for Li_2TiO_3 blanket in the conditions of the EXOTIC-8 experiment [24] is shown in Fig. 7 assuming some water vapor in the purge gas. This figure shows fair agreement of the estimated values with the observed values in the EXOTIC-8 experiment.

Though it is considered that the effective diffusivity of tritium in the grain changes with the amount of irradiation defects, the effect due to irradiation in Li_2TiO_3 is not observed, yet. It is possible that the diffusivity reported by Tanifuji et al., can be largely different from the effective diffusivity representing the tritium migration through grains in the EXOTIC-8 experiment. However, results in Fig. 7 implies that diffusivity plays a small part in the total residence time even when diffusivity decreases to one-thousandth of the value given by Eq. (11).

The total residence time of tritium estimated for $LiAlO_2$, Li_2ZrO_3 and Li_2TiO_3 blanket are compared in Figs. 8 and 9. In this comparison, Li_4SiO_4 is excluded because the equation to quantify the isotope exchange capacity of Li_4SiO_4 has not been reported yet, though existence of rather large exchange capacity is observed in the preparatory experiments of the present authors [19]. The total residence time for Li_2TiO_3 decreases rather rapidly at the higher temperature than 573 K because the isotope exchange capacity decrease rapidly above this temperature.

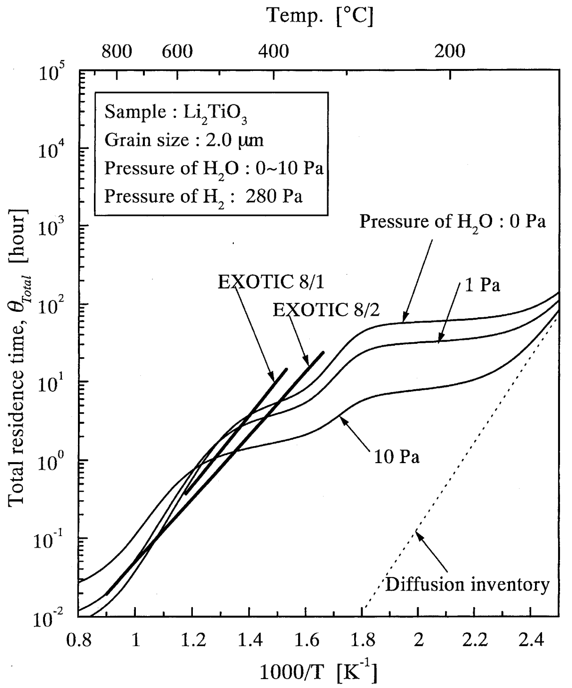

Fig. 7. Total residence time of tritium of Li_2TiO_3 blanket under the EXOTIC-8 condition.

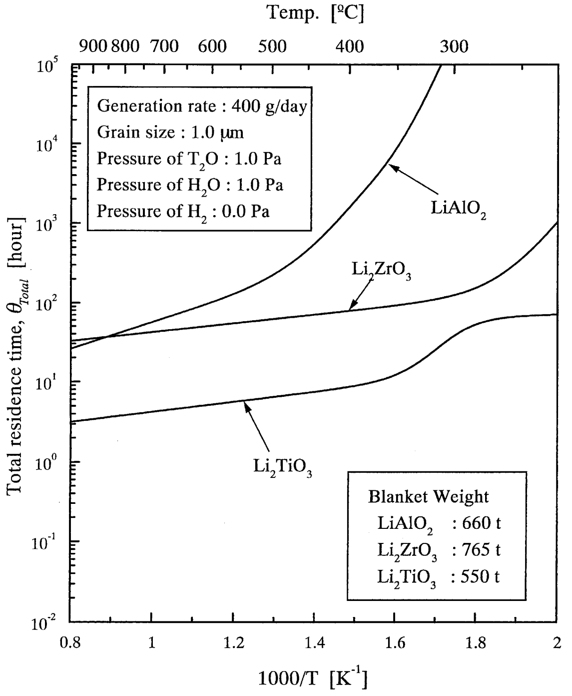

Fig. 8. Comparison of total residence time for $LiAlO_2$, Li_2ZrO_3 and Li_2TiO_3 blankets (He purge).

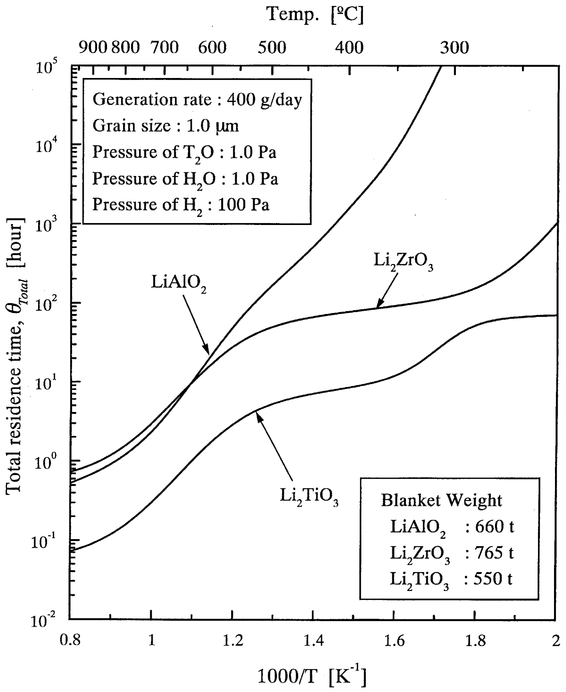

Fig. 9. Comparison of total residence time for $LiAlO_2$, Li_2ZrO_3 and Li_2TiO_3 blankets (He + 100 Pa H_2 purge).

It is said from above comparison that the total residence time for Li_2TiO_3 blanket is smaller than that for $LiAlO_2$ or Li_2ZrO_3 by almost one order if the assumption that k_{ex1} for Li_2TiO_3 is similar to k_{ex1} for other ceramic blanket materials is proper. The total residence time for Li_2TiO_3 blanket is also smaller than the residence time estimated for Li_4SiO_4 blanket excluding the isotope exchange capacity. Accordingly, it can be said that much smaller tritium inventory is expected in a Li_2TiO_3 blanket than the inventory in $LiAlO_2$, Li_2ZrO_3 or Li_4SiO_4 blanket.

5. Conclusions

Adsorption capacity and isotope exchange capacity of Li_2TiO_3 pebbles are experimentally obtained in this study and the following conclusions are obtained.

1. The tritium inventory in the Li_2TiO_3 blanket under the steady-state condition can be estimated using the same method as applied to estimation of inventory for $LiAlO_2$ or Li_2ZrO_3 blanket by the present authors.
2. It can be said that the inventory on the surface occupies the major part of the tritium inventory in the Li_2TiO_3 blanket when the diffusivity by Tanifuji et al., is applicable to the Li_2TiO_3 blanket under irradiation.

3. It is also said that tritium inventory for Li_2TiO_3 blanket is much smaller than that for $LiAlO_2$, Li_2ZrO_3 or Li_4SiO_4 blanket over the wide range of temperature if the assumption that k_{ex1} for Li_2TiO_3 is similar to k_{ex1} for other ceramic blanket materials is proper.

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