

# Deuterium Retention and Desorption Behavior of $\text{Li}_2\text{TiO}_3$ after Deuterium Ion Irradiations with Different Temperatures

Hironobu SHIBATA, Yuji NOBUTA, Yuji YAMAUCHI and Tomoaki HINO

*Laboratory of Plasma Physics and Engineering, Hokkaido University, Kita-13, Nishi-8, Kita-ku, Sapporo, 060-8628 Japan*

Masato AKIBA and Satoshi SUZUKI

*Naka Fusion Institute, Japan Atomic Energy Agency, Naka-shi, Ibaraki-ken, 311-0193 Japan*

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Lithium Titanate ( $\text{Li}_2\text{TiO}_3$ ) pebbles were irradiated by deuterium ion ( $\text{D}^+$ , 1.7 keV) under different irradiation temperatures (RT, 473 K, 573 K, 673 K and 773 K), and the amount of retained deuterium and the desorption behavior were investigated using a technique of thermal desorption spectroscopy (TDS). The amount of retained deuterium was held relatively constant when the irradiation temperature stayed below 473 K, while the amount decreased sharply when irradiation temperature was raised above 473 K. When the temperature was raised to 773 K, the amount of retained deuterium became almost zero. This result suggests that the tritium produced in  $\text{Li}_2\text{TiO}_3$  pebbles in the region with temperature higher than 773 K completely desorbs during the operation, although a small fraction of the tritium remains in the pebbles when the temperature is less than 773 K.

**Keywords:** test blanket module (TBM),  $\text{Li}_2\text{TiO}_3$  pebble, deuterium ion irradiation, thermal desorption spectroscopy tritium inventory

## 1. Introduction

Lithium Titanate ( $\text{Li}_2\text{TiO}_3$ ) pebbles are employed as solid tritium breeding materials in test blanket modules (TBM) of ITER [1]. As the solid breeder material,  $\text{Li}_2\text{TiO}_3$  shows good chemical stability and tritium (T) release by thermal desorption [2,3]. In the fusion blanket, the pebbles are irradiated by fusion neutrons and tritium is produced by nuclear reactions of  $^6\text{Li}$  ( $n, \alpha$ ) T or  $^7\text{Li}$  ( $n, n\alpha$ ) T in the pebbles. The temperature distribution of the solid breeding material in TBM has been estimated with to be in the range from 573 K to 1173 K, taking into account of nuclear heating [4]. Although it is desired for the tritium to be recovered completely in the whole blanket region during ITER operation, a small fraction of the tritium remains in the low temperature region [5]. Therefore, in order to estimate the tritium inventory in  $\text{Li}_2\text{TiO}_3$  pebbles in the blanket of ITER, the retention and desorption behavior of the tritium has to be investigated.

In the present study, the deuterium ion irradiation experiment was conducted in  $\text{Li}_2\text{TiO}_3$  pebbles to investigate the hydrogen isotope retention and desorption behavior. The pebbles were irradiated with deuterium ions with energy of 1.7 keV at various irradiation temperatures (Room Temperature (RT), 473 K, 573 K, 673 K, 773 K). Next, the retention and desorption behavior of retained deuterium was investigated using a technique of thermal desorption spectroscopy (TDS). Based on the obtained results, the tritium inventory in the TBM during ITER operation was estimated.

## 2. Experiments

18  $\text{Li}_2\text{TiO}_3$  pebbles (about 2 mm $\phi$ ) [6] were used for a deuterium ion irradiation experiment. These pebbles were heated at 973 K for an hour in a vacuum chamber to remove impurities such as hydroxides and carbon oxides [6]. After the heating, the pebbles were installed into a sample holder in an electron cyclotron resonance (ECR) ion source (Fig.1) and irradiated by deuterium ions. The sample holder was made of a Ta plate, and Mo mesh was used to fix these pebbles. The pebbles were heated by indirect resistive heating. The irradiation temperature (surface temperature of the pebbles) was changed from RT to 773 K. Before the irradiation experiments, surface temperature and sample holder temperature were simultaneously measured by thermocouples. The temperature measurement was carried out three times for

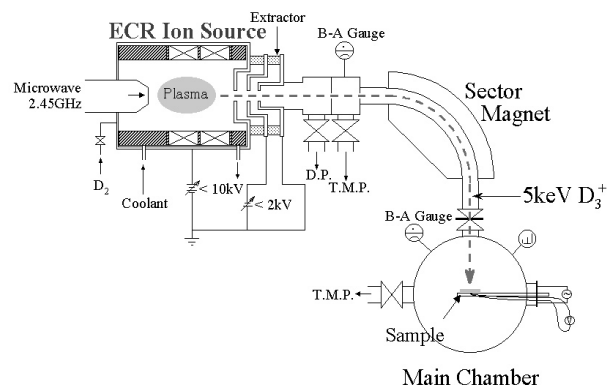


Fig.1 ECR ion source apparatus.

author's e-mail: shibata@queen.qe.eng.hokudai.ac.jp

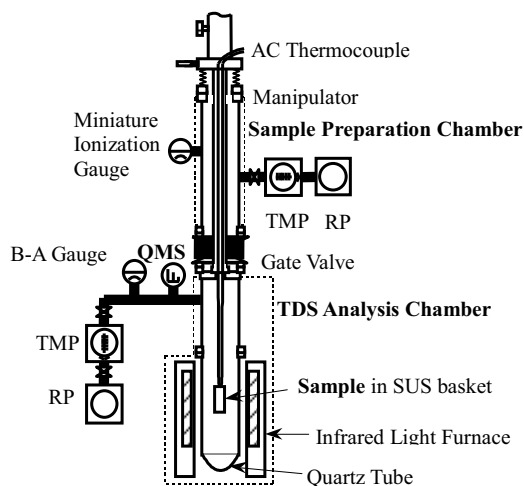


Fig. 2 TDS apparatus.

each temperature, and the error among the measurements was approximately 20 K. During the irradiation, the surface temperature was estimated from the temperature of the sample holder. The deuterium ion energy was 1.7 keV, the fluence was  $5 \times 10^{18}$  D/cm<sup>2</sup> and the flux was approximately  $9 \times 10^{14}$  D/cm<sup>2</sup>·s. After the ion irradiation, the pebbles were transferred to the TDS chamber (Fig.2). The sample was then heated by an infrared light furnace from RT to 973 K with a heating rate of 10 K/min. At 973 K, the heating was conducted for an hour. The ultimate pressure before the TDS analysis was approximately  $10^{-8}$  Pa.

### 3. Results and Discussion

Figure 3 shows the thermal desorption spectra of gases containing the deuterium in the deuterium-irradiated  $\text{Li}_2\text{TiO}_3$  pebbles with the irradiation temperatures of (a)RT, (b)573 K and (c)773 K. Deuterium retained in these pebbles desorbed in forms of HD, D<sub>2</sub>, HDO and D<sub>2</sub>O. In our previous study, the deuterium desorption behavior of the  $\text{Li}_2\text{TiO}_3$  pebbles was similar with that of the Li film, therefore most of the retained deuterium is trapped in forms of Li-D and Li-O-D [5]. Desorption rates of these gases decreased with the increase of irradiation temperature. At the irradiation temperature of 773 K, the retained deuterium completely desorbed during the irradiation. This result suggests that the tritium produced in  $\text{Li}_2\text{TiO}_3$  pebbles at a temperature higher than 773 K completely desorbs during the ITER operation. When the irradiation temperature increased, the deuterium desorption temperature peak (~750 K) shifted to an even higher temperature. This peak shift was also reported by other deuterium ion irradiation experiments [7]. The reason of the shift, however, was not clear, thus further investigation is necessary.

Figure 4 shows the amounts of gases containing the deuterium desorbed from the pebbles as a function of the

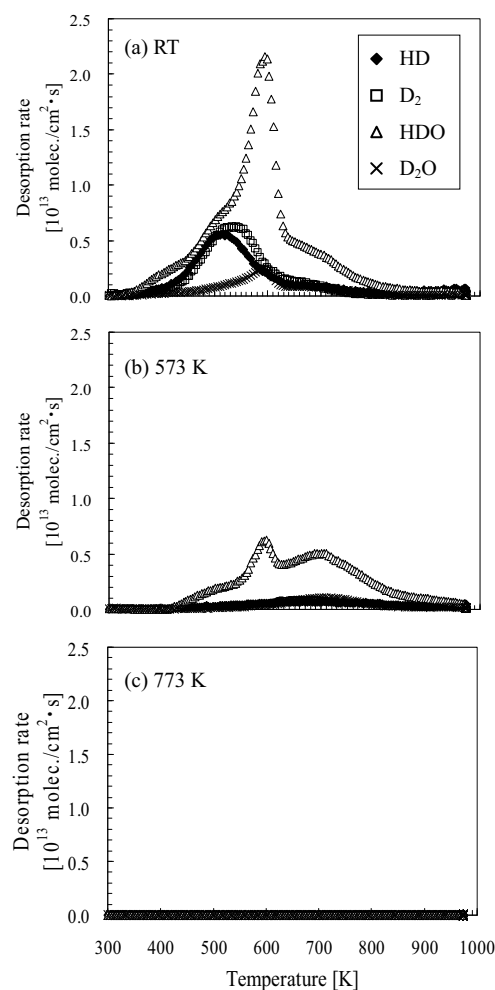


Fig.3 Thermal desorption spectra of gases containing deuterium for deuterium ion irradiated  $\text{Li}_2\text{TiO}_3$  pebbles at the irradiation temperatures of (a)RT, (b)573 K and (c)773 K.

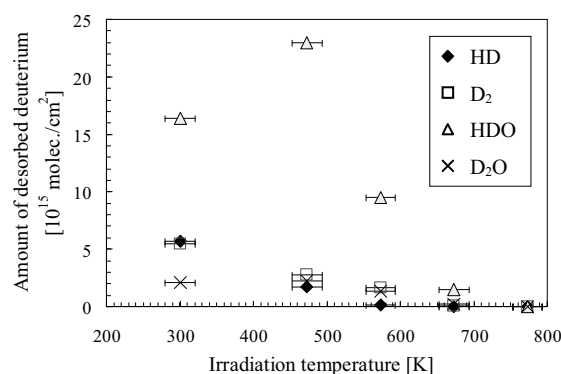


Fig.4 Amounts of gases containing deuterium desorbed from  $\text{Li}_2\text{TiO}_3$  pebbles as a function of irradiation temperature.

irradiation temperature. It is noted that the error bars in Fig.4 and Fig.5 result from the measurement error of

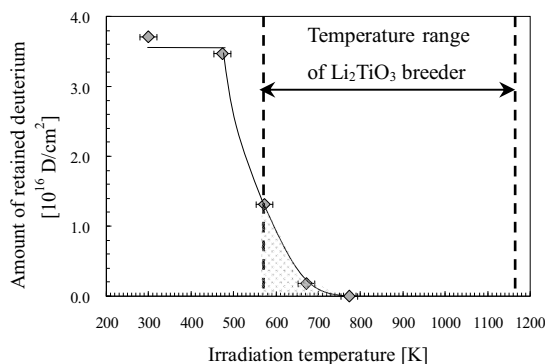


Fig. 5 Amounts of retained deuterium in  $\text{Li}_2\text{TiO}_3$  pebbles as a function of irradiation temperature.

irradiation temperatures by thermocouples. In the cases of HD and  $\text{D}_2$ , the amounts of desorbed gases decreased with the increase of irradiation temperature. On the other hand, in the cases of HDO and  $\text{D}_2\text{O}$ , the amounts of desorbed gases increased when the irradiation temperature was lower than 473 K, and then decreased when the temperature was raised above 473 K. This difference in the dependence of the desorption amount on irradiation temperature occurs due to the different desorption peak temperatures of these gases. The amount of deuterium desorbed in the form of HDO was largest in these gases at every irradiation temperature.

Figure 5 shows the amount of retained deuterium (total amount of retained deuterium atoms). The dashed lines in Fig. 5 show the temperature range of  $\text{Li}_2\text{TiO}_3$  breeder in TBM. The total amount of retained deuterium was held almost constant when the temperature was less than 473 K, while the total amount decreased significantly when the temperature was raised above 473 K. The retained deuterium completely desorbed when the temperature was raised above 773 K, while a small fraction of the deuterium was retained when the temperature was kept lower than 773 K. In the TBM, tritium can exist when the temperature is less than 773 K. The amount of retained tritium in this region determines the tritium inventory. Namely, the tritium is retained in the region with temperature of 573-773 K (shown as dot region in Fig.5).

The amount of retained tritium in the TBM can be estimated using the present result. For  $\text{Li}_2\text{TiO}_3$  pebbles, provided that the deuterium is trapped only by Li, the deuterium concentration in the atomic ratio of D/Li at RT is approximately 0.5 [5]. In the case of TMB, the amount of lithium is approximately 7 kg [8]. Lithium burn-up of  $\text{Li}_2\text{TiO}_3$  pebbles during ITER operation is estimated at about 0.32 % [9], and thus the reduction of lithium in these pebbles by neutron irradiation is negligible. In this case, the maximum tritium inventory is estimated to be 1.5 kg if the temperature in the TBM is RT. Additionally,

the ratio when the temperature is lower than 773 K in the TBM is estimated as  $\sim 0.1$  [8]. From the results obtained in the present study, the amount of tritium retained in the region at 573-773 K can be estimated as approximately 8 % of the amount at RT. Under the assumption that the tritium trapped in the region above 773 K is completely desorbed during the ITER operation, the tritium inventory in the TBM during the operation becomes approximately 12 g. This estimation suggests that the tritium inventory of the TBM is only a few grams. The tritium inventory can be reduced if the region with temperature lower than 773 K is easily reduced by the design.

#### 4. Conclusion

In the present study,  $\text{Li}_2\text{TiO}_3$  pebbles were irradiated by deuterium ions under various deuterium ion irradiation temperatures (RT, 473 K, 573 K, 673 K, 773 K), and the retention and desorption behavior of deuterium was investigated using a technique of thermal desorption spectroscopy. The amount of retained deuterium for  $\text{Li}_2\text{TiO}_3$  pebbles was almost the same when the irradiation temperature range was lower than 473 K, while the amount decreased sharply with the increase of irradiation temperature above 473 K. The amount of retained deuterium became approximately zero at 773 K. The temperature distribution of the solid breeding material in the TBM is in the range of 573 K to 1173 K. The present results suggest that the tritium inventory of the blanket is only a few grams. If the region with temperature lower than 773 K is reduced by the design work, the tritium inventory decreases even more.

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