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# Gas and hydrogen ion gettering properties of lithium

T. Hino [a,*], K. Kanaya [a], I. Takahashi [a], H. Yanagihara [a], M. Hashiba [a], Y. Hirohata [a], K. Mori [b]

[a] *Department of Nuclear Engineering, Hokkaido University, Kita-13, Nishi-8, Kita-ku, Sapporo 060, Japan*

[b] *Gunma Kosen, Toba-cho, Maehashi 371, Japan*

**Abstract**

The gettering properties of lithium films for $O_2$, $CO_2$ and $H_2O$ were examined in a vacuum system with a heater for lithium evaporation. The absorbed amounts of $O_2$, $CO_2$ and $H_2O$ observed were in the ratio, $O_2/Li = 0.2$, $CO_2/Li = 0.01$ and $H_2O/Li = 0.12$. The gettering action for $H_2O$ was saturated at a relatively low lithium evaporation amount. The hydrogen uptake of a lithium oxide film was also investigated by using an ion irradiation apparatus with a thermal desorption spectroscopy. The ratio of retained hydrogen was $H/Li \simeq 0.7$, which was roughly the same as in the case of a fresh lithium film. The helium ion irradiation reduced the hydrogen retention of the lithium oxide film. The retained amount was 50% of that before the helium ion irradiation.

## 1. Introduction

Gettering effects of evaporated lithium have been demonstrated on oxygen, hydrogen atom or ion, carbon monoxide and methane in a small dc glow discharge device with a vacuum evaporation of lithium metal [1–3]. In particular, it is shown that both the hydrogen retention capability of the lithium exposed to the hydrogen plasma and the oxygen gettering capability were large, i.e. the atomic ratio of trapped hydrogen and oxygen are $H/Li \simeq 1$ and $O/Li \simeq 1/2$, respectively. In the case that lithium is coated on a graphite liner, the release of CO produced by a reaction of oxygen impurity with carbon is largely suppressed [3].

On the other hand, lithium wall conditioning using lithium pellet injection has been carried out in TFTR [4], JIPPT-IIU [5] and DIII-D [6]. In these fusion devices, major effects were substantial decreases of levels of oxygen and carbon impurities, and of hydrogen recycling. Therefore, the use of lithium is attractive for the wall conditioning although the melting point, 179°C, is low.

In a fusion device, the major outgassing species are $H_2$, $H_2O$, CO, $CO_2$ and hydrocarbons such as $CH_4$. It is known that $H_2$ gas hardly reacts with the lithium surface but hydrogen atoms or ions in the dc glow discharge are largely absorbed [2]. It is observed that CO and $CH_4$ are slightly absorbed in the lithium [3]. Because the gettering effects of lithium on $H_2O$ and $CO_2$ are not systematically investigated, we measured the absorption amounts of $H_2O$ and $CO_2$ in a lithium evaporation apparatus. For comparison, the gettering effect on $O_2$ was also examined in the same apparatus. These gas gettering experiments were conducted with the wall at room temperature (RT). There may be a question of whether the gettering effect changes with the wall at elevated temperature. The gettering effects on $O_2$ and $CO_2$ with a wall temperature of 100°C were also examined.

In a fusion device, lithium evaporated on the wall may be oxidized if the vacuum chamber is exposed to air. In such a case, the hydrogen uptake behavior might change, compared with the case of fresh lithium film. In order to examine the hydrogen uptake by the lithium oxide, we conducted a hydrogen ion irradiation for the lithium oxide film made by electron beam evaporation (EBE), and measured the amount of retained hydrogen by thermal desorption spectroscopy (TDS).

Helium discharge cleaning has been carried out to reduce the hydrogen retention of plasma facing walls. In order to simulate the amount of ion impact desorption, helium ion irradiation for the film with implanted hydrogens was conducted, and the residual amount of hydrogen was measured.

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[*] Corresponding author. Tel.: +81 11706 7195; fax: +81 11709 6413; e-mail: tomhino@apollo.qe.eng.hokudai.ac.jp.

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## 2. Experimental

To measure gettering of gaseous species by lithium, the vacuum apparatus shown in Fig. 1 was fabricated. This device consists of a gas flow system with a variable leak valve (VLV), a getter chamber with a heater for lithium evaporation, a partial pressure measurement system with a quadrupole mass spectrometer (QMS) and a main pumping system. The ultimate pressure of the getter chamber was $1 \times 10^{-4}$ Pa and the effective pumping speed for $N_2$ gas was $2.7 \times 10^{-3}$ $m^3/s$. For the partial pressure measurement with QMS, it was assumed that the pumping speed for a gaseous species was proportional to the inverse of square of the molecular weight. The total pressure was monitored by a B-A gauge, MIG430.

Lithium metal placed at a Ta heater was evaporated within about 10 min. After the evaporation, the gas such as $O_2$, $CO_2$, $H_2O$ and $CH_4$ was introduced into the getter chamber through VLV. The chamber temperature was kept at RT. The reduction of the total and the partial pressure corresponds to the gas amount absorbed by the lithium film deposited on the wall. For $O_2$ that is well absorbed by lithium, the evaporation amount of lithium was changed, and the relation between the absorption amount of gasous species and the evaporation amount of lithium was examined. For $O_2$ and $CO_2$, a similar experiment was conducted with the getter chamber kept at 100°C.

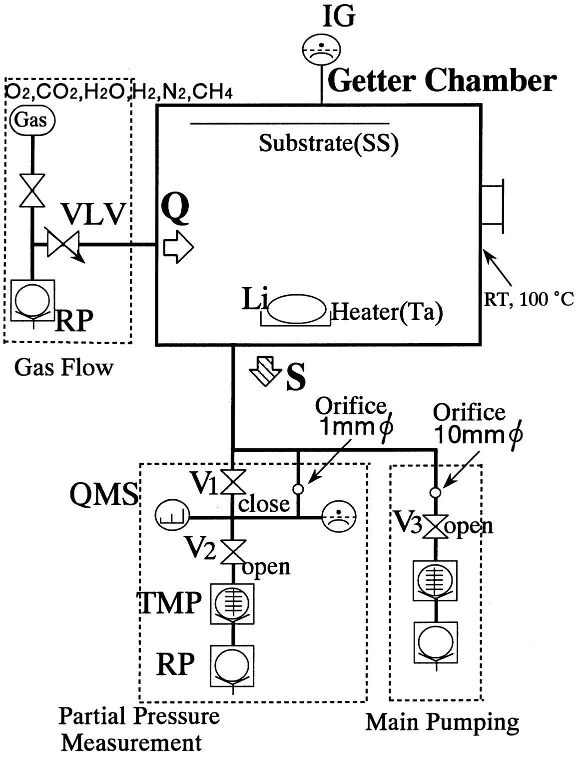

Fig. 1. Apparatus for gas gettering of lithium.

In order to examine the hydrogen uptake of a lithium oxide film, a different experiment was conducted. First, a lithium film with a thickness of about 1 μm was deposited on a stainless steel substrate by using EBE. After this lithium film was exposed to air, an analysis with Auger electron spectroscopy (AES) was performed to examine the atomic composition. The result showed that the oxygen and lithium fractions were ~40 and ~60 at.% over the entire depth region, respectively. Since the atomic ratio, O/Li, was 2/3, it is assumed that $Li_2O$ was produced.

For this lithium oxide film, the hydrogen ion irradiation was conducted in an ECR ion irradiation apparatus shown in Fig. 2. The energy and the fluence of hydrogen ion were 1.7 keV and $(1–10) \times 10^{17}$ $H/cm^2$. After the hydrogen ion irradiation, the amount of retained hydrogen was quantitatively measured by TDS in the same apparatus. The heating rate in the TDS measurement was 50°C/min.

In order to simulate the helium ion impact desorption of retained hydrogen, a helium ion irradiation at 5 keV was carried out after the hydrogen ions were sufficiently implanted in the same apparatus. The fluence of helium ions was $(0–20) \times 10^{17}$ $He/cm^2$. For the measurement of the residual hydrogen amount, the TDS was conducted.

## 3. Results

### 3.1. Gas gettering capability of lithium

For $O_2$ gas, the change of the total pressure after the lithium evaporation followed by the $O_2$ gas flow was measured. The oxygen flow rate was 0.01 standard cubic centimeter per minutes (sccm), which corresponded to the pressure of $8 \times 10^{-3}$ Pa. The evaporation amount of lithium was 0.5, 3, 7 and 11 mg. The absorption amount of $O_2$ plotted against the amount of evaporated lithium is shown in Fig. 3. It is seen that the ratio, $O_2/Li$, is approximately 0.2, indicating the formation of $Li_2O$. In the case of $CO_2$, the $CO_2/Li$ ratio was ~0.01, much smaller than that for $O_2$. The AES analysis showed that the carbon concentration was high only at the surface region after the $CO_2$ gettering, although the oxygen concentration was uniform (~40 at.%) in the entire depth region after the $O_2$ gettering.

For $H_2O$ gas, in addition to the reduction of $H_2O$ partial pressure, the increase of $H_2$ partial pressure was observed. This result suggests that the reaction, $2H_2O(g) + 2Li(s) \rightarrow 2LiOH(s) + H_2(g)$, took place during the gettering of $H_2O$. The relation between absorbed $H_2O$ amount and evaporation amount of lithium is shown in Fig. 4. The absorbed $H_2O$ amount was ob-

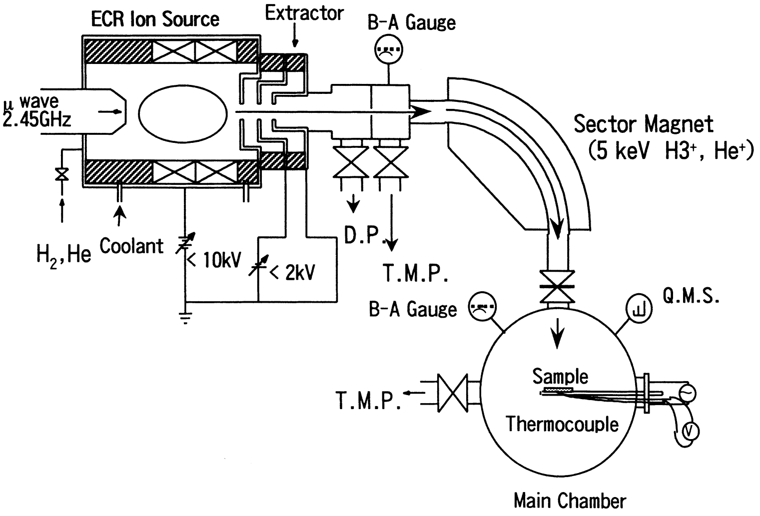

Fig. 2. ECR ion irradiation apparatus.

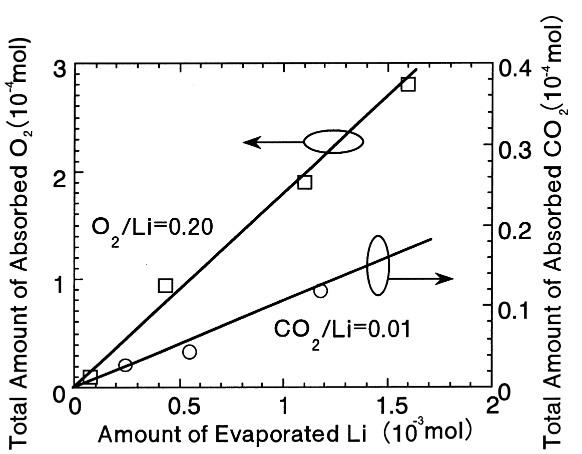

Fig. 3. Amount of absorbed $O_2$ or $CO_2$ versus evaporated Li amount.

served to saturate with increasing amount of the lithium evaporation. It is conceivable that the formation of LiOH is a barrier for $H_2O$ penetration, although this reason is not clearly known. In the region before saturation, the gettering ratio, $H_2O/Li$, was 0.12. For $H_2$, $N_2$ and $CH_4$ gas, the gettering effect was scarcely observed.

The above data were taken when the temperature of the getter chamber was kept at RT. The gettering experiment with $O_2$ and $CO_2$ gas was also carried out with the wall temperature at 100°C. The results are shown in Fig. 5. The absorption amounts in both cases were very similar to those at RT. It can be regarded that the gettering action remains the same at 100°C.

### 3.2. Hydrogen uptake of lithium oxide

After the hydrogen ion irradiation with a fluence of (1–10) × $10^{18}$ H/cm$^2$, the $H_2$ desorption spectra were

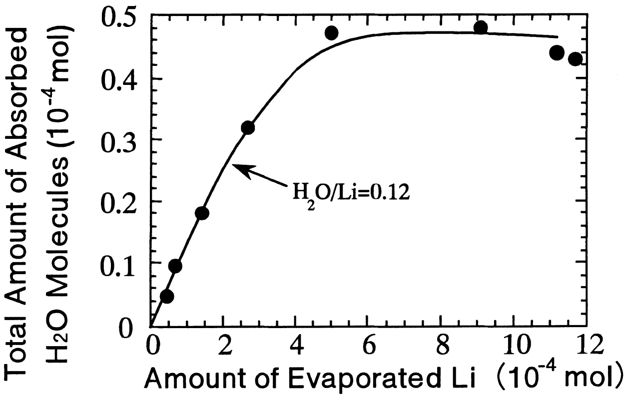

Fig. 4. Amounts of absorbed $H_2O$ vs. evaporated Li amount.

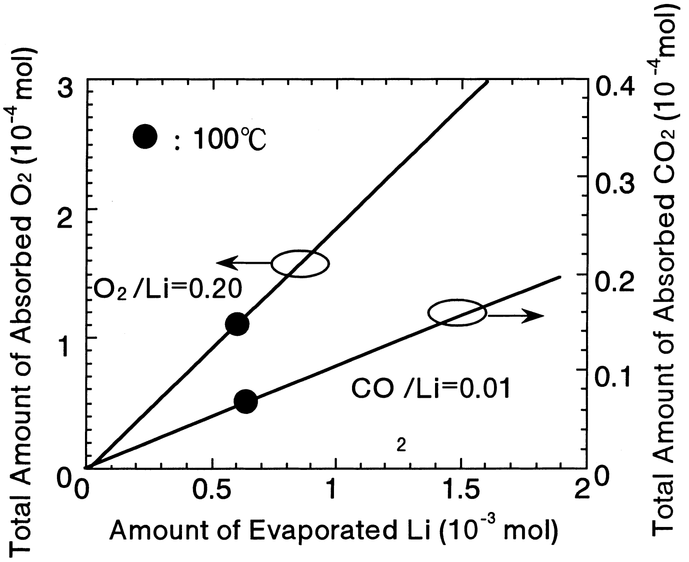

Fig. 5. Amounts of absorbed $O_2$ and $CO_2$ vs. evaporated Li amount for wall temperature of 100°C.

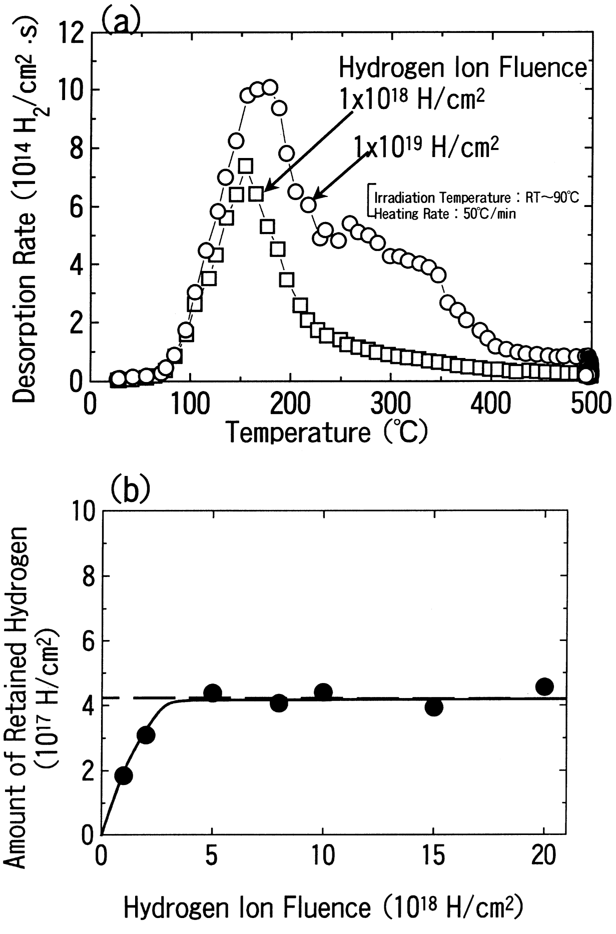

Fig. 6. (a) $H_2$ desorption spectra of lithium oxide film after hydrogen ion irradiation. (b) Hydrogen ion fluence dependence of retained hydrogen amount.

obtained. For example, the spectra observed at the fluences of 1 × $10^{18}$ and 1 × $10^{19}$ H/$cm^2$ are shown in Fig. 6(a). In the case of a lower fluence, the single peak at 200°C was observed. This temperature is close to the melting point of lithium. Thus, the desorption at around 200°C may be due to the dissolved hydrogen in the lithium. In the case of a higher fluence, another peak appeared at about 350°C. This desorption corresponds to the decomposition of lithium hydride [2]. The fluence dependence of retained hydrogen amount is shown in Fig. 6(b). At the fluence of ~5 × $10^{18}$ H/$cm^2$, the retained amount saturated. The ratio of hydrogen to lithium, H/Li, in the implanted depth region was about 0.7. This result is very close to the value obtained for fresh lithium film, H/Li ≃ 1 [2]. Therefore, it is assumed that the present hydrogen retention of the lithium oxide film in the implanted depth is similar to that of fresh lithium film.

For the film with a saturated amount of hydrogen, a helium ion irradiation was conducted. In Fig. 7(a), the $H_2$ desorption spectra before and after helium ion irradiation are shown. The peak at lower temperature was largely reduced by the helium ion bombardment. The helium fluence dependence of the residual hydrogen amount is plotted in Fig. 7(b). The reduction ratio was observed to be as high as 50%, which is considerably large.

## 4. Summary

Gas gettering experiments were conducted in a vacuum apparatus with a heater for lithium evaporation. The atomic ratios of $O_2$, $H_2O$ and $CO_2$ absorbed by lithium were $O_2/Li = 0.2$, $H_2O/Li = 0.12$ and $CO_2/Li = 0.01$, respectively. After the gettering of $O_2$ gas, the oxygen was uniformly trapped in the entire depth region. The carbon, on the other hand, was trapped only at the surface region after the gettering of $CO_2$ gas. In the gettering experiment for $H_2O$ gas, the absorption amount saturated at relatively low lithium evaporation amount. For $H_2$, $N_2$ and $CH_4$, almost no gettering effect was observed.

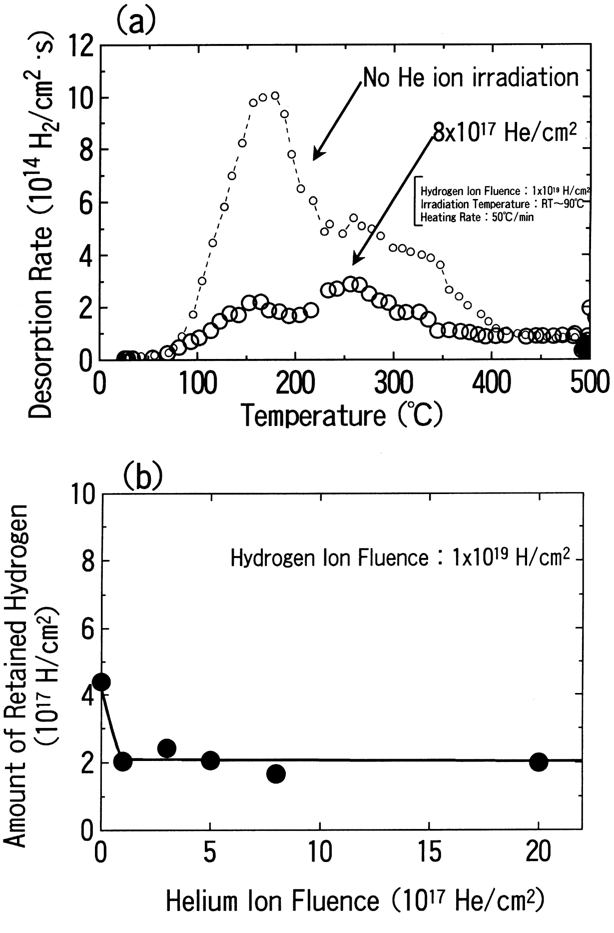

Fig. 7. (a) $H_2$ desorption spectra before and after helium ion irradiation. (b) Helium fluence dependence of residual hydrogen amount.

For the lithium oxide film, the gettering effect of hydrogen ion was examined. The atomic ratio of trapped hydrogen in the implanted depth was H/Li ~0.7. The retained hydrogen desorbed at 200°C and 350°C. The desorption of retained hydrogen was greatly enhanced by the helium ion bombardment. The hydrogen retention was reduced to 50% of that before the helium irradiation.

## Acknowledgements

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