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Detrapping behavior of tritium trapped via hot atom chemical process in neutron-irradiated ternary lithium oxides

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Abstract

Tritium detrapping behavior in neutron-irradiated ternary lithium oxides was investigated by the comparison of the annihilation of irradiation defects with the tritium release. It was revealed that the annihilation of irradiation defects would consist of two processes; namely the fast and the slow ones. The slow annihilation process has correlation with the tritium release, indicating that E′-center or F^+-center could act as tritium trapping site, and from its activation energy of each sample, the annihilation of E′-center and F^+-center could be attributed to the recovery of oxygen via diffusion, triggering the tritium release. Meyer–Neldel plots of these results indicate that the slow annihilation process was governed by the formation entropy of a pair of vacancy and interstitial atom of oxygen. Therefore, the trapped tritium would be detrapped by oxygen recovery to E′-center or F^+-center, and its kinetics would be determined by the population of oxygen vacancy under thermal equilibrium.

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1. Introduction

It is important to establish the tritium recovery system from the viewpoint of the cycle of fueling and tritium safety. Many studies have been carried out over the decades to reveal the tritium release behavior from the tritium breeding materials. In the test blanket modules for ITER, Li_2TiO_3 will be selected as one of the candidates due to its good tritium release, low tritium inventory, good chemical stability, and so on [1–4]. However, there could be still possibilities remaining to use the other tritium breeding materials, such as $LiAlO_2$, Li_4SiO_4, and Li_2ZrO_3, because of their specific properties of the considerable chemical stability, high lithium density and self neutron multipliability, respectively. The elucidation of the comprehensive mechanism of tritium behavior in such ternary lithium oxides could lead to the determination of the most proper tritium breeding material. It can be suggested that there are six following elementary processes from tritium generation to its release: (i) tritium generation via the nuclear reaction mainly of $^6Li(n,\alpha)T$, (ii) thermalization of energetic tritium, (iii) tritium trapping at trapping sites, (iv) tritium detrapping from trapping sites, (v) tritium diffusion in bulk, and (vi) tritium release by recombination or isotope exchange reaction on the surface. Particularly, the processes (v) and (vi) have been well investigated [4–6]. However, the process (iv) is also thought to be one of the key processes for understanding tritium recovery processes, although the processes (iii) and (iv) have little been investigated so far. To elucidate this process, it is important to take the viewpoints of hot atom chemical reactions into consideration, as the trapping of energetic tritium would be governed by hot atom chemistry. It was suggested from

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the report made by Okuno et al. [7] that oxygen vacancies of F^+-centers, which were produced in neutron-irradiated Li_2O, would play an important role on the tritium trapping. Asaoka et al. and Moritani et al. have also reported the production behavior of F type centers (F^0 and F^+-center) in several lithium oxides under H^+ or He^+ implantation and its implantation temperature dependence [8–10]. Ab initio calculations for the trapping of hydrogen isotopes in Li_2O reported by Tanigawa et al. have indicated that F^0-center (oxygen vacancy occupied by a pair of electrons) could strongly interact with hydrogen isotopes [11,12]. In our previous studies, it has been reported for neutron-irradiated Li_4SiO_4, Li_2TiO_3, and Li_2ZrO_3 [13–20] that the tritium was trapped by E′-center and its annihilation due to the oxygen recovery to the E′-center would trigger the tritium release. Although the tritium trapping sites and the mechanism of detrapping are comparable among the samples, physicochemical constants, such as rate constants and activation energies of the annihilation process of E′-center were considerably different among the samples.

In the present study, therefore, the experimental results of the comparisons between annihilation behavior of irradiation defects and tritium release behavior for $LiAlO_2$ and Li_2SiO_3 were shown, although the experimental results for the comparison between annihilation behavior of F^+-center and tritium release behavior for $LiAlO_2$ and annihilation behavior of irradiation defects in Li_2SiO_3 can be found in the Refs. [17,20], respectively, and thereafter, the comprehensive mechanism of the tritium detrapping in all the neutron-irradiated ternary lithium oxides was discussed to clarify the key process of tritium detrapping.

2. Experimental

Pebbles of lithium aluminate ($LiAlO_2$) were purchased from Kawasaki Heavy Industries Ltd., and powder of lithium meta-silicate (Li_2SiO_3) purchased from Aldrich Co., both of which were the same as previously used sample in Refs. [17,20], were sintered at 1173 K for 3 h using conventional electric furnace. Catalytic metal of Pd was deposited on Li_2SiO_3 for tritium release experiments to enhance the surface reaction to obtain the bulk information. The details for catalytic metal deposition on tritium breeding materials were reported in Refs. [21,22]. All the samples were preheated from room temperature up to 673 K for 8 h and then kept for 4 h under a reduced pressure of He atmosphere in quartz tube in order to remove the moisture and hydroxides on the surface. Thereafter, these samples were sealed by fusing the quartz tubes.

The samples were in-pile-irradiated with the thermal neutron fluence of $3 \times 10^{15}\,\mathrm{cm}^{-2}$ for Li_2SiO_3 and $1 \times 10^{16}\,\mathrm{cm}^{-2}$ for $LiAlO_2$ at ambient temperature in Pneumatic tube 2 of Research Reactor Institute, Kyoto University (KURRI).

ESR measurements (JES-TE200, produced by JEOL) were carried out to observe the irradiation defects and to reveal their annihilation behavior in the samples during the isothermal annealing experiments.

Isothermal annealing experiments for neutron-irradiated $LiAlO_2$ were performed to establish the kinetics of the annihilation processes of irradiation defects. The temperature region for the isothermal annealing experiments was selected using the result of tritium release experiment reported in Ref. [17]. After each heating, the sample was immediately cooled down to liquid nitrogen temperature (77 K) and then ESR measurement was performed. Each isothermal annealing experiment was carried out until the ESR signals intensities became less than detection limit.

The tritium release experiments for neutron-irradiated Li_2SiO_3 with Pd deposition were carried out to compare with the annihilation behavior of irradiation defects previously reported in Ref. [20], using the tritium release set-up as described in Ref. [21]. The sample was heated from room temperature up to 1073 K with a heating rate of 5 K min^{-1}. The released tritium was swept by 1000 ppm H_2 diluted by N_2, and measured with an ion chamber.

3. Results

Fig. 1 shows typical ESR spectra for $LiAlO_2$ before and after neutron irradiation. From these spectra, it is clearly indicated that some sorts of irradiation defects were induced by neutron irradiation, and also indicated that the induced irradiation defects could be identified as F^+-center, peroxide radical (POR), non-bridging oxygen hole center (NBOHC) and hole-trapped oxygen from the comparison with the previous studies and Refs. [13–20,23–28], in analogy with the previous studies for Li_2SiO_3, Li_4SiO_4, Li_2TiO_3 and Li_2ZrO_3, except for hole-trapped oxygen. Fig. 2 shows a typical result of isothermal annealing experiments at 673 K for neutron-irradiated $LiAlO_2$. A combination of two first-order reactions was applied to fit and simulate the annihilation behavior in analogy with the

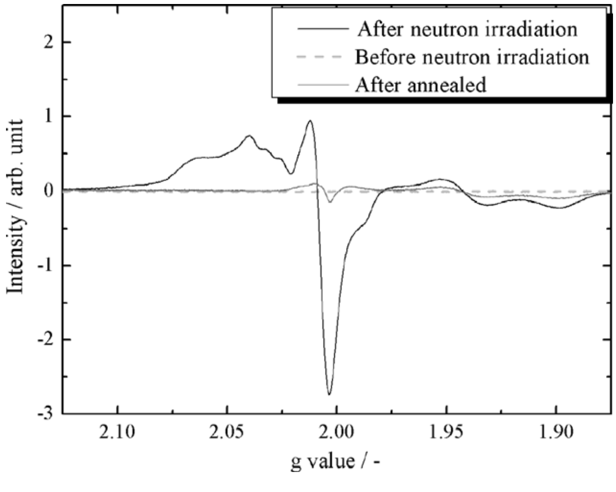

Fig. 1. ESR spectra for $LiAlO_2$ before and after neutron irradiation, and that with neutron irradiation after annealed at 673 K for 1800 s.

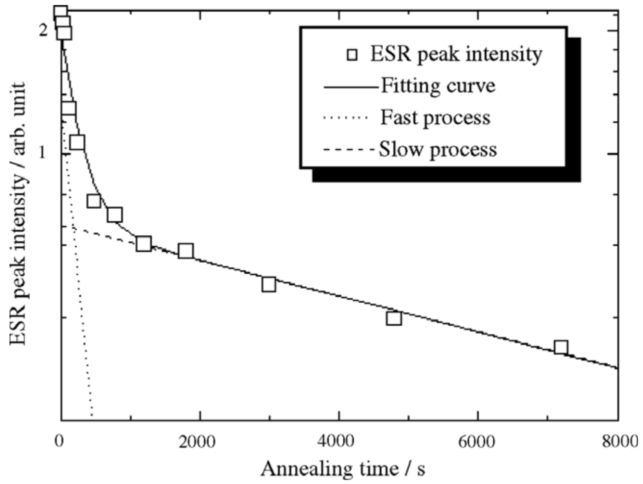

Fig. 2. Changes of ESR intensity during isothermal annealing experiment at 673 K for neutron-irradiated $LiAlO_2$.

other ternary lithium oxides [13–20], described as followings:

$$[N] = [F] + [S], \tag{1}$$

$$[F] = [F_0]\exp(-k_f t), \tag{2}$$

$$[S] = [S_0]\exp(-k_s t), \tag{3}$$

where $[N]$ is the amount of total irradiation defects induced by neutron irradiation and, $[F]$ and $[S]$ are those of annihilated ones in the fast and the slow processes, respectively. The subscript of '0' means their initial value. k_f and k_s are the rate constants of the fast and the slow processes, respectively, and t indicates the annealing time. It should be noted that the major defects annihilated in the slow process for neutron-irradiated $LiAlO_2$ were F^+-center and hole-trapped oxygen as indicated from ESR spectrum for neutron-irradiated $LiAlO_2$ after annealed at 673 K for 1800 s. Using the rate constants of k_f and k_s, the Arrhenius plots were applied to obtain the activation energies and pre-exponential factors for the processes as shown in Fig. 3. From this figure, the activation energies for the fast and the slow processes were estimated to be 0.52 ± 0.03 and 1.4 ± 0.1 eV, respectively. For the activation energy for the slow process, that of oxygen diffusion of 1.5 eV [27] could be comparable, so that the slow process could be dominated by oxygen diffusion as well as that in Li_2SiO_3, Li_4SiO_4, Li_2TiO_3 and Li_2ZrO_3 [13–20].

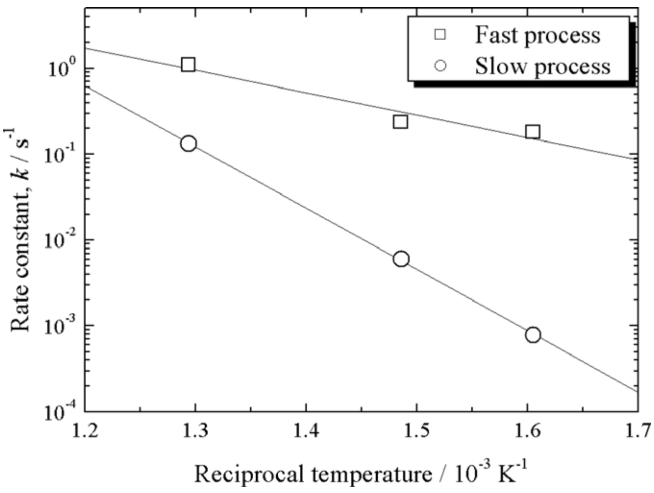

Fig. 3. Arrhenius plots of the fast and the slow annihilation processes of irradiation defects in neutron-irradiated $LiAlO_2$.

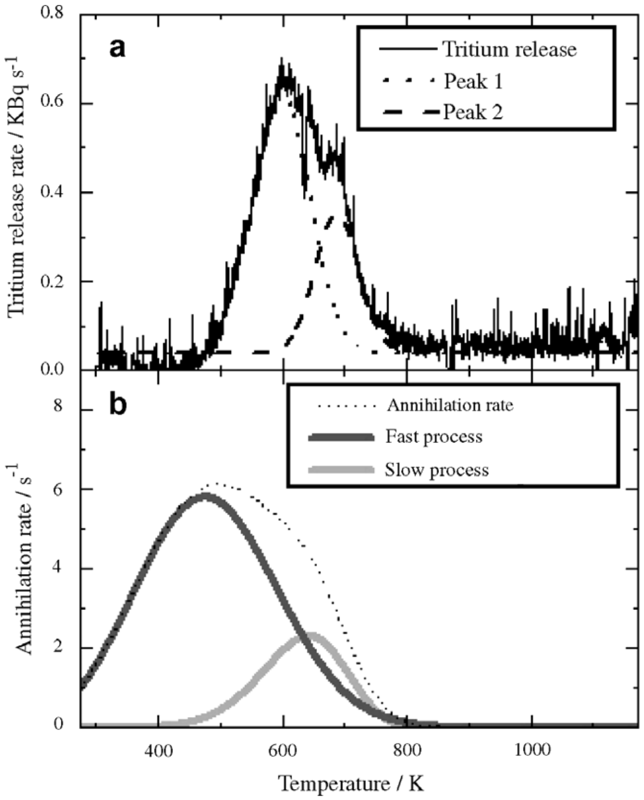

Fig. 4. Tritium release (a) and numerically calculated annihilation of irradiation defects (b) for neutron-irradiated Li_2SiO_3.

The tritium release spectrum for the neutron-irradiated Li_2SiO_3 with Pd deposition is shown in Fig. 4(a) with peak analysis by the Gaussian distribution function. It was found to consist of two desorption stages: namely 600 K and 700 K.

4. Discussion

4.1. Comparison between the annihilation of irradiation defect and the tritium release

Using the pre-exponential factors and activation energies obtained in the previous [13–20] and present studies as summarized in Table 1, numerical simulations of the annihilation of irradiation defects was performed to make it comparable to the tritium release behavior as followings:

$$[F_n] = [F_{n-1}]\exp\left\{-\Delta t A_0^{\mathrm{f}} \exp\left(-\frac{E_{\mathrm{a}}^{\mathrm{f}}}{RT_n}\right)\right\}, \tag{4}$$

Table 1
Temperature region for annihilation of defects, pre-exponential factors, and activation energies for the fast and the slow processes [16,18–20]

Sample	Annihilation temperature/K	A_0/s^{-1}		E_a/eV	
		Fast	Slow	Fast	Slow
$LiAlO_2$	600–800	2.4×10^3	2.4×10^8	0.52	1.4
Li_2SiO_3	400–700	7.2×10^{-2}	1.2×10^2	0.12	0.63
Li_4SiO_4	500–800	6.4×10^{-2}	37	0.12	0.56
Li_2TiO_3	400–700	56	2.6	0.43	0.41
Li_2ZrO_3	500–700	5.3	2.3×10^3	0.32	0.80

$$[S_n] = [S_{n-1}] \exp\left\{-\Delta t A_0^{s} \exp\left(-\frac{E_a^{s}}{RT_n}\right)\right\}, \tag{5}$$

$$T_n = T_{n-1} + \beta\Delta t, \tag{6}$$

where Δt is the minimal time, β is the heating rate, superscript of ‘f’ and ‘s’ mean the fast and the slow processes, respectively, R is gas constant, and T is the absolute temperature. Figs. 4 and 5 show the comparison of the annihilation behavior of irradiation defects with the tritium release behavior for Li_2SiO_3 and $LiAlO_2$, respectively, indicating that the slow annihilation proceed simultaneously with the tritium release of Peak 1. These results were consistent with our previous results [18–20]. Taking this and the previous report for neutron-irradiated Li_2O [7], in which indicated that there must be certain correlation between F^+-center and tritium, into consideration, it was indicated that the one of the tritium trapping sites could be attributed to E′-center and F^+-center in neutron-irradiated Li_2SiO_3 and $LiAlO_2$, respectively, and their annihilation could be dominated by oxygen diffusion.

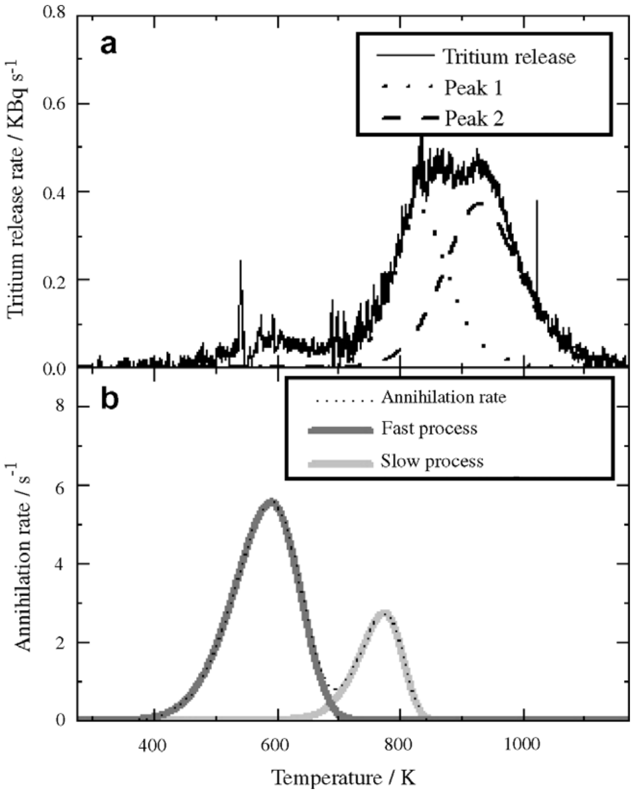

Fig. 5. Tritium release (a) and numerically calculated annihilation of irradiation defects (b) for neutron-irradiated $LiAlO_2$.

4.2. Application of Meyer–Neldel rule

From these experimental results, there indicated to be the following three elementary processes from annihilation of irradiation defects to tritium release: (A) the fast annihilation process with no correlation to the tritium release, (B) the slow annihilation process with correlation with tritium release of Peak 1 and (C) tritium release of Peak 2 with no correlation with the annihilation of irradiation defects. Additionally, it was found that the activation energy of the slow annihilation process in each sample was comparable to that of oxygen diffusivity in each constituent oxide [27,29–31], indicating that the annihilation of E′-center or F^+-center could be attributed to the oxygen recovery via the oxygen diffusion. However, the activation energies and pre-exponential factors are quite different among the samples as shown in Table 1. Therefore, an empirical method was applied to clarify whether kinetics of the annihilation of irradiation defects could be commonly described as one theory among the samples and to understand the key mechanism for these processes by plotting the logarithm of the pre-exponential factor, $\ln(A_0)$, as a function of the activation energy, E_a. Such treatment has been often performed for some experimental results which were expected to have a same origin. This is known as Meyer–Neldel rule (the compensation law or isokinetic effect) [31–37] and is expressed as

$$\ln A_0 = \alpha E_a + \beta, \tag{7}$$

where α and β are constants. A number of thermally activated processes obey this rule such as conductivity in semiconductors [32], heterogeneously catalyzed reactions [35], diffusion in solids and glasses [31,33,36], and so on. Fig. 6 shows the good agreement with the Meyer–Neldel rule for each process, indicating that each process commonly proceeded among the sample. For the fast process, it might be attributed to diffusion of trapped electrons which act as radical scavengers, because of low activation energies. On the other hand, the slow process would be attributed to oxygen recovery to E′-center or F^+-center via the oxygen diffusion [34–37].

On the other hand, the Arrhenius equation was described as

$$k = A_0 \exp\left(-\frac{E_a}{k_B T}\right), \tag{8}$$

where k_B is Boltzmann constant. Eq. (5) could be transcribed as following:

$$A_0 = k_0 \exp\left(\frac{E_a}{k_B T_0}\right), \tag{9}$$

where T_0 and k_0 are constants, namely Meyer–Neldel temperature and Meyer–Neldel rate constant, and $\alpha = (k_B T_0)^{-1}$

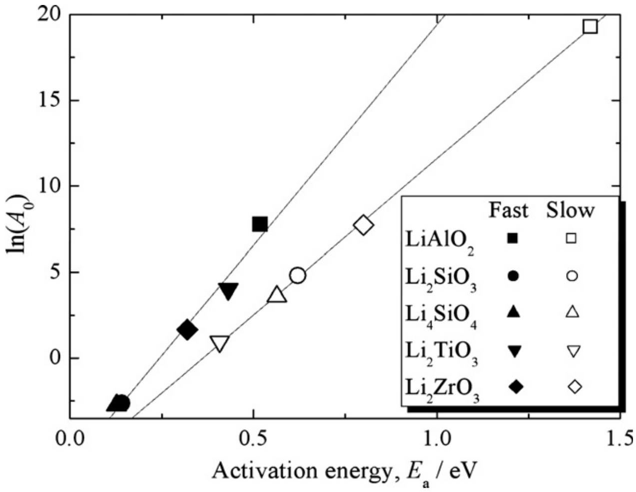

Fig. 6. Meyer–Neldel plots of the fast and the slow annihilation processes.

and $\beta = \ln k_0$. From Eqs. (5) and (6), following equation can be derived:

$$k = k_0 \exp\left(\frac{E_a}{k_B T_0}\right) \exp\left(-\frac{E_a}{k_B T}\right). \tag{10}$$

From these results, since it was indicated that the mechanism of oxygen diffusion would be the same among the sample and that in Li doped Al_2O_3, Ti_2O_3/TiO_2 and yttria stabilized zirconia, which could be used as oxygen ionic conductor [27,30], were dominated by vacancy diffusion mechanism, a simplified vacancy diffusion model is proposed as shown in Fig. 7. In this model, E′-center and F^+-center were indicated as V_O^+. Therefore, the reaction formulae of the annihilation of V_O^+ via the vacancy diffusion model were suggested as followings:

$$O_{lp} \rightleftarrows O_{is}^{2-} + V_O^{2+}, \tag{11}$$

$$O_{is}^{2-} + V_O^+ \rightarrow O_{lp} + e^- \uparrow, \tag{12}$$

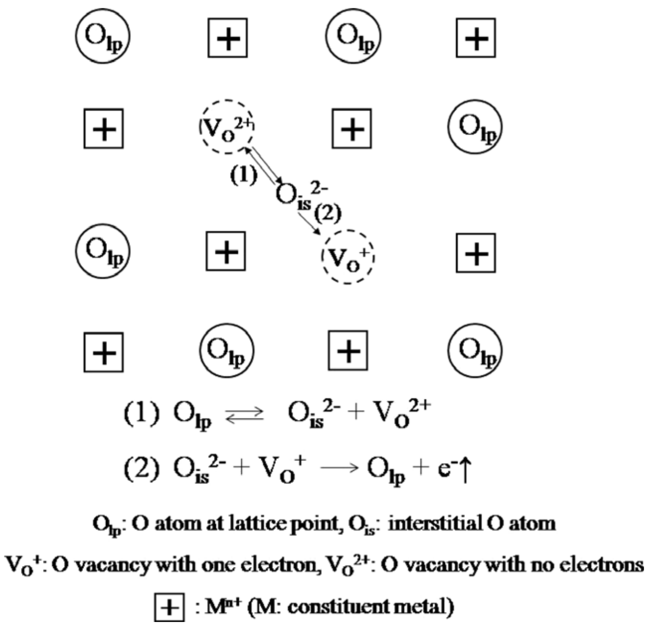

Fig. 7. Tritium detrapping model for neutron-irradiated ternary lithium oxides.

where O_{lp} and O_{is} mean oxygen atoms at lattice point and that at interstice, respectively, and up-arrow means detrapping. The detrapped electron is expected to diffuse immediately, because the fast annihilation process would be due to the diffusion of trapped electrons and its rate is much faster than that of the slow process. From Eqs. (7) and (8), the jumping frequency of oxygen vacancy, ν, could be described as following:

$$\nu = \nu_0 \exp\left(\frac{S_j}{k_B}\right) \exp\left(\frac{E_j}{k_B T}\right), \tag{13}$$

where ν_0, S_j and E_j are Debye frequency, activation entropy and activation energy for jumping of oxygen vacancy, respectively.

Comparing Eqs. (7) and (10), the terms of E_a/T_0 and E_a could be comparable to S_j and E_j, respectively. It was also reported that in a system which obeys the Meyer–Neldel rule, the following relational expression can be approved as reported in Ref. [30]:

$$S_j = \frac{E_a}{T_0}. \tag{14}$$

Therefore, the entropy for oxygen vacancy formation in the samples can be estimated and summarized in Table 2. In addition, population of oxygen vacancy of $[V_O]$ under thermal equilibrium can be described as

$$\frac{[V_O]}{[O_{lp}]} = \exp\left(\frac{S_f}{k_B}\right) \exp\left(-\frac{E_f}{k_B T}\right), \tag{15}$$

where $[O_{lp}]$ means the population of oxygen atom at lattice point, and S_f and E_f mean the entropy and energy to form the vacancy, respectively. In simplified model described in Fig. 7, S_f and E_f could be approximated to S_j and E_j, respectively. Therefore, it can be said that the pair of S_f and E_j would be determined by the population of oxygen vacancy under thermal equilibrium.

From above results, it can be concluded that the elemental processes of (ii), (iii) and (iv) can be explained as follows: (ii) thermalization of energetic tritium with inducing several kinds of irradiation defects including E′-center or F^+-center, (iii) tritium trapping in E′-center or F^+-center,

Table 2
Entropy of oxygen jumping of S_j in each sample

Sample	S_j/meV K^{-1}
$LiAlO_2$	1.7
Li_2SiO_3	0.74
Li_4SiO_4	0.67
Li_2TiO_3	0.49
Li_2ZrO_3	0.96

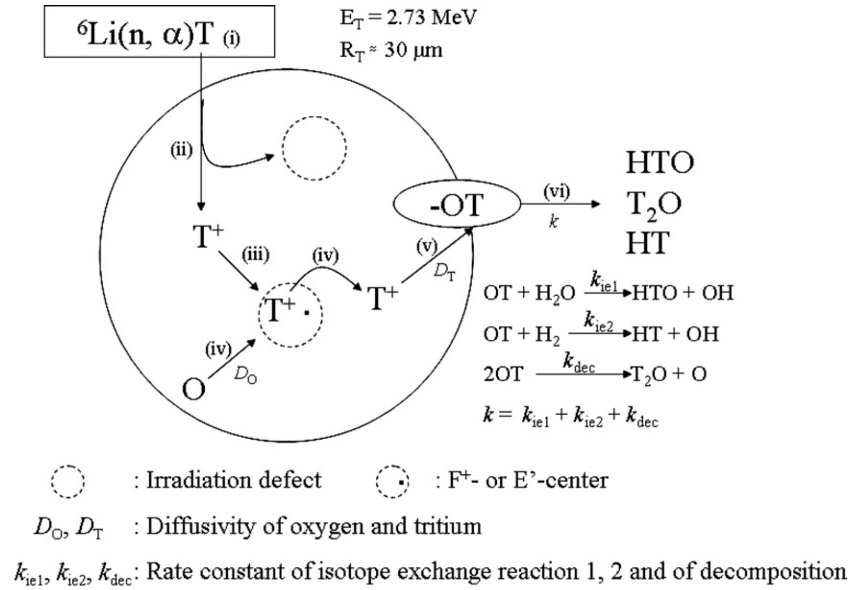

Fig. 8. A suggested model for tritium behavior from generation to release as: (i) generation, (ii) thermalization with inducing irradiation defects, (iii) trapping in E′-center or F^+-center, (iv) detrapping by the recovery of trapping sites via oxygen diffusion, (v) diffusion and (vi) release by surface reactions.

and (iv) tritium detrapping triggered by the oxygen recovery to E′-center or F^+-center via vacancy diffusion mechanism. A comprehensive model for tritium behavior in neutron-irradiated ternary lithium oxides could be suggested in Fig. 8, and it can be concluded that kinetics of tritium detrapping is governed by the population of oxygen vacancy under thermal equilibrium.

5. Conclusion

The tritium detrapping behavior in several neutron-irradiated ternary lithium oxides was investigated by the comparison of the annihilation of irradiation defects with the tritium release, and comprehensive mechanism for tritium detrapping was proposed. Comparison of the annihilation of irradiation defects with tritium release revealed that the main tritium release had a correlation with the slow process. This indicates that E′-center or F^+-center would be act as tritium trapping site, and its oxygen recovery trigger the release of trapped tritium. From Meyer–Neldel plot, it was indicated that the annihilation of irradiation defects could uniformly proceed among the samples: In the fast process, trapped electrons would diffuse with scavenging various unpaired electron, and in the slow process, oxygen would diffuse and annihilate E′-center or F^+-center. It was also suggested from the comparison with vacancy diffusion model that kinetics of the annihilation of E′-center and F^+-center was dominated by the population of oxygen vacancy under thermal equilibrium. Therefore, the tritium detrapping would be attributed to the oxygen recovery to E′-center and F^+-center and its kinetics could be determined by the activation entropy of oxygen diffusion, that is, determined by the population of oxygen vacancy under thermal equilibrium.

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