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Irradiation effects in ceramic breeder materials

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Abstract

For understanding of the irradiation effects in solid breeder materials, extensive studies have been performed with lithium-containing ceramics such as Li_2O, $LiAlO_2$, Li_4SiO_4 and Li_2ZrO_3. In parallel with the studies of ordinary irradiation performance testing, a number of the studies have been performed focussed on the fundamental aspects of irradiation-induced defects and their effects in lithium ceramics. In the present paper, the fundamental studies are reviewed and discussed to improve our knowledge on the irradiation effects in lithium ceramics. Their production behaviors, reaction kinetics, and effects on the tritium release performance and microstructural changes of lithium ceramics are described.

1. Introduction

The breeding blanket is a key component of the fusion reactor since it involves tritium breeding and energy extraction, both of which are critically important for the development of fusion power. Particularly because of their safety advantages, lithium-containing ceramics such as Li_2O, $LiAlO_2$, Li_4SiO_4 and Li_2ZrO_3 have been recognized as promising tritium breeding materials, and extensive studies have been performed on these ceramics [1–3]. According to the current database, these ceramics exhibit acceptable thermal, mechanical, chemical and irradiation behavior. However, the materials behavior at high burn-up levels is a critical issue since the database is still limited to rather low burn-up levels.

In a fusion reactor solid blanket system, tritium breeding lithium ceramics are attacked by high energy neutrons and energetic particles from nuclear reactions, and severe irradiation damage may be expected. The irradiation behavior of lithium ceramics is thus important for the performance assessment of fusion reactor blanket systems. Considering the two functions of blanket systems, which are tritium breeding and energy extraction, the effects of long-term irradiation on tritium release behavior and microstructural changes of lithium ceramics are of special concern for the design of blanket systems. Thus experimental efforts have been devoted to the irradiation performance testing at higher burn-up levels as seen in the BEATRIX-II experiments [4]. Also, fundamental studies on the irradiation behavior of lithium ceramics have been performed in order to understand and control the phenomena [5].

In the present paper, the studies on the fundamental aspects of the irradiation behavior of lithium ceramics are reviewed and discussed to improve our knowledge on the irradiation effects in lithium ceramics. Some details of their production behaviors, reaction kinetics, and effects on the tritium release performance and microstructural changes of lithium ceramics are described.

2. Observation of irradiation-induced defects

Irradiation-induced defects of F-type centers, colloidal metals and some other decomposition products have been observed to be formed in lithium ceramics such as Li_2O. Noda et al. [5–11] have reported the results of electron spin resonance (ESR) and optical-absorption measurements of irradiation-induced defects formed in Li_2O. The formation of the F^+ center, which is an

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oxygen vacancy trapping an electron and thus has a positive charge, was observed in both Li_2O single crystals and sintered pellets irradiated by thermal neutrons to a fluence of 10^{20}–10^{23} m^{-2}. The formation of colloidal lithium metals was also observed in the sintered pellets irradiated to 10^{23} m^{-2} by thermal neutrons, in the single crystals irradiated to 3×10^{20} m^{-2} by 100 MeV oxygen ions [11] and in the Li_2O samples of the BEATRIX-II experiments [12] which were irradiated to 3.9×10^{26} m^{-2} in FFTR for 300 effective full power days at 375°C.

Thermal recovery behavior of the F^+ centers and colloidal lithium metals was studied by isochronal and isothermal annealing experiments [6–8]. It has been found that the number of the F^+ centers decreases in the range of 420–570 K and almost all disappears above 630 K. The activation energy for the recovery of the F^+ centers has been determined to be 135 kJ mol^{-1}. On the other hand, the number of colloidal lithium metals increases in the range of 400–600 K and begins to decrease at 700 K, disappearing completely at 870 K. Comparing the annealing behavior of both species, it may be suggested that the F^+ centers are partly transformed into the colloidal species (for example, through the reactions of $2(F^+ \cdot O^-) \rightarrow 2F^0 + O_2 + 2h\nu'$ and $nF^0 \rightarrow F_n \rightarrow Li_n$).

More recently, Vajda and Beuneu have measured the EPR spectra of poly- and monocrystalline samples of Li_2O irradiated with electrons at different temperatures [13–15]. Together with the typical hyperfine structure lines of the F^+ centers, they have observed a much narrower line in the spectra, which is assigned to colloidal lithium metals as confirmed by NMR and dielectric-constant measurements. They have suggested a different temperature dependence of colloidal species production from that of Noda et al. [7].

The in situ luminescence measurement of the irradiation-induced defects in Li_2O were performed under ion beam [16,17], gamma-ray [18] and reactor [19,20] irradiations. The post irradiation research on the defects after the exposure of Li_2O to gamma-rays [21] and to accelerated electrons (5 MeV) [22] was also conducted by using the methods of chemical scavenger, thermoluminescence and others.

In the in situ luminescence measurement of Li_2O under various irradiations [16–20], it has been found that the luminescence bands of F^+, F^0 and F_2 centers are common for all irradiation types. The irradiation types influenced only the fractional intensity of each luminescence band in the spectrum. This effect is due to the differences in specific ionization among ion beam, gamma-ray and reactor irradiations. In the case of reactor irradiation, the two main bands of the F^+ and F^0 centers have been observed in the temperature range from 673 to 873 K [20]. The tritium breeding blanket is operated in a similar temperature range and, hence, the effect of such *F*-type centers on tritium release behavior can be expected.

In the in situ luminescence measurement under ion beam irradiation was determined the conditions for the formation of various kinds of defects in Li_2O [17]. It has been shown that the absorbed dose of irradiation plays a vital role in the processes of the luminescence emission from Li_2O. With increasing absorbed dose, some of the luminescence bands have been observed to decrease in their intensity, and the others to increase, as shown in Fig. 1. The efficient accumulation of the F-center aggregates in Li_2O under ion beam irradiation has been observed at temperatures below 423 K but hardly occurs at temperatures higher than 573 K. On the other hand, the production of F_2 and F^+ centers efficiently occurs in the temperature region from 423 to 523 K.

The formation of F-centers in polycrystalline Li_2O irradiated by gamma-rays was studied by the methods of thermo-luminescence and chemical scavenger [21], and the production of these defects by gamma-ray irradiation was confirmed. The radiolysis of Li_2O ceramics under the electron beam irradiation (energy of 5 MeV) at 373 K has also been performed in the absorbed dose

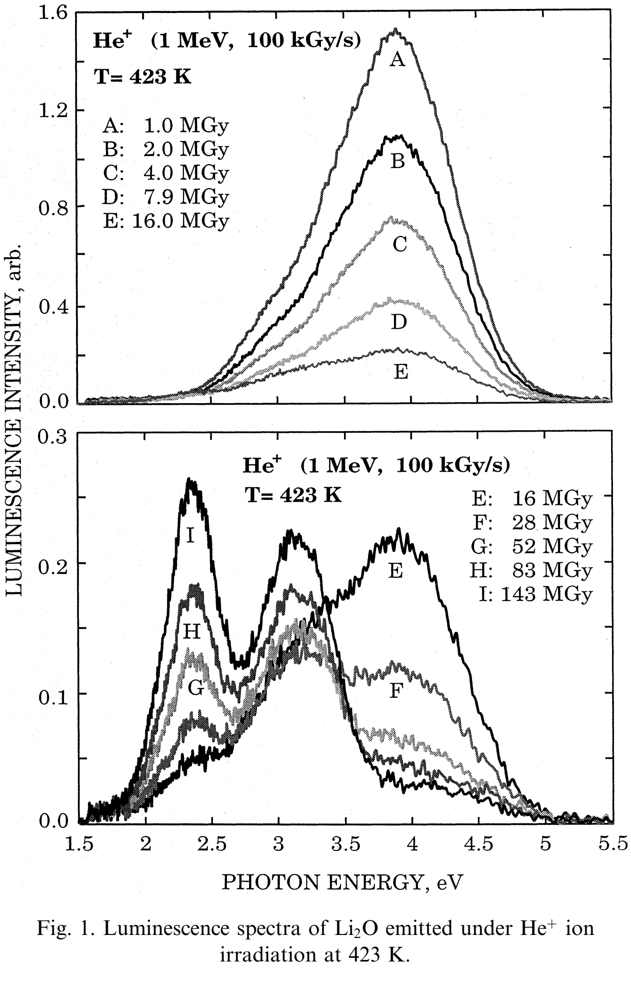

Fig. 1. Luminescence spectra of Li_2O emitted under He^+ ion irradiation at 423 K.

up to 250 MGy [22]. In this case, irradiation-induced defects and radiolysis products have been analyzed by chemical scavenger, thermo-luminescence and ESR methods. As shown in Fig. 2, unusual dose dependence has been observed: the rapid growth of the concentration of irradiation-induced defects and radiolysis products in the low dose region below 50 MGy is followed by the radiation-induced annealing. The explanation was based on two different mechanisms: (1) formation near the intrinsic defects and (2) via the radiolysis of matrix.

As summarized in Table 1, the formation of the irradiation-induced defects has been observed in various lithium ceramics [7,8,11,13–19,23–28]. Although intensive studies have been performed, the knowledge on the formation conditions of temperature, dose rate and absorbed dose for each species is still insufficient and needs further studies. The colloidal Li will be trapping tritium into Li–T bond, which is tolerant to high temperature, and may affect the tritium recovery from the lithium ceramics leading to the increase in tritium inventory.

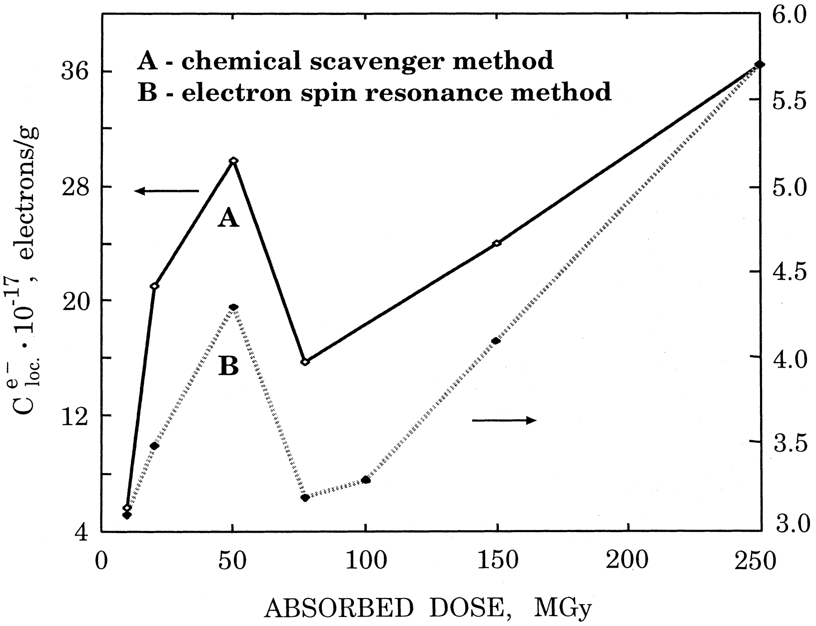

Fig. 2. Dose dependence of the concentration of electron radiation defects in Li_2O ceramics after irradiation by 5 MeV electrons at 373 K.

Table 1
Irradiation-induced defects observed in lithium ceramics

Lithium ceramics	Defects	Methods
Li_2O	F^+	ESR/absorption [8,13–15]
	F^0	Luminescence [16–19]
	F_2	Luminescence [17–19]
	F-aggregates	Luminescence [13–15,17–19]
	Colloidal Li	ESR [7,11,13–15]
$LiAlO_2$	F^+	ESR/absorption [23,24]
		Luminescence [25]
	F^0	Luminescence [25]
Li_4SiO_4,Li_2SiO_3	E′	Luminescence [26,27]
	NBOHC [a]	Luminescence [26,27]
	peroxy species	Luminescence [26,27]
Li_2TiO_3,Li_2ZrO_3,Li_2SnO_3	F^+	Luminescence [28]
	F^0	Luminescence [28]

[a] Non-bridging oxygen hole centers.

3. Production behavior of irradiation-induced defects

For understanding the kinetic aspects of the production behavior of irradiation-induced defects, the in-situ luminescence measurement technique is very useful and has been applied to some solid breeder materials [29,30]. Knowledge of the irradiation defect production is much improved and is extended to understand the tritium behavior under irradiation.

As shown in Fig. 3 [29], the luminescence band of the F^+ centers is predominantly observed in Li_2O at lower temperatures, while the band of the F^0 centers is observed at higher temperatures. This indicates the importance of the F^0 centers in the reactions of tritium at the temperatures of actual blanket conditions. In order to discuss the reaction kinetics, the temperature transient behavior of irradiation defect production has been measured in Li_2O. For a temperature increase, the luminescence intensity of the F^+ centers rapidly decreases as expected from the temperature dependence of the equilibrium luminescence intensity. On the other hand, an abnormal temperature transient behavior has been observed for the F^0 centers; an excess luminescence is observed for the temperature increase, as shown in Fig. 4 [29]. Also, the excess luminescence has been found to depend on the irradiation history and to increase with increasing absorbed dose before the temperature change. By considering the observations, the following reaction scheme has been given for the production mechanism of the F^+ and F^0 centers:

$$Li_2O \rightarrow Li_2O^*, \qquad (1)$$

$$Li_2O^* \rightarrow (F^+ \cdot O^-) + h\nu, \qquad (2)$$

$$2Li_2O^* \rightarrow 2F^0 + O_2 + 2h\nu', \qquad (3)$$

$$(F^+ \cdot O^-) + Li_2O \rightarrow 2F^0 + O_2 + 2h\nu'. \qquad (4)$$

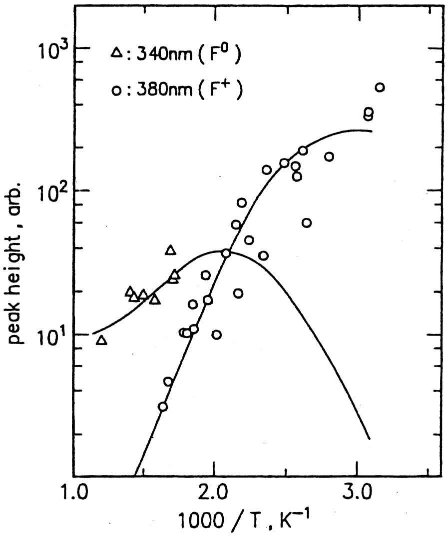

Fig. 3. Arrhenius plots of luminescence intensities at 340 and 380 nm [29]. Marks are experimental and curves are calculated.

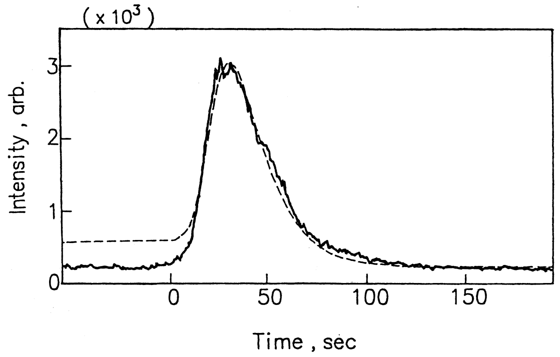

Fig. 4. Temperature-transient behavior of the 340 nm luminescence for a change from 528 to 723 K [29]. Irradiation time before a temperature increase was 10 min and irradiation current was 40 nA. Solid curve is experimental and dashed is calculated.

Reaction (1) represents the production of an excited Li_2O, Li_2O^*, by ion beam irradiation. The F^+ center is considered to be associated with an O^- interstitial under Coulomb interactions as ($F^+ \cdot O^-$), and is produced with the luminescence of hν by reaction (2), which dominates at lower temperatures. At higher temperatures, however, reactions (3) and (4) will take place and the F^0 centers and their partners, O_2 molecules, are produced with the luminescence of $h\nu'$. These second-order reactions for the formation of F^0 centers require some thermal activation, and will take place at higher temperatures. This is the reason why the luminescence of the F^0 centers is observed especially at higher temperatures. Based on this reaction scheme, some calculations have been performed for the luminescence behavior. Agreements between the calculations and observations are satisfactorily good as seen in Figs. 3 and 4.

Recently, the same technique has been applied to the ternary lithium ceramics of Li_2TiO_3, Li_2ZrO_3 and Li_2SnO_3 [30]. In this case, however, it has been shown that the transient behavior of the luminescence for a temperature change is little dependent on the irradiation history and that the equilibrium condition is attained shortly. This may suggest little participation of reaction (4) in Li_2TiO_3, Li_2ZrO_3 and Li_2SnO_3, although the reaction scheme will be similar to that of Li_2O.

As shown here, the luminescence measurement technique is very useful for studying the kinetic aspects of the production behavior of irradiation-induced defects. However, this technique hardly provides any quantitative information on their concentrations. In order to overcome this problem, it is suggested to combine this technique with some other methods such as ionic conductivity measurement technique [31].

4. Irradiation effects on tritium release performance

Tritium recovery from solid breeder materials has been investigated extensively, and a number of factors, namely dissolution, diffusion, surface reaction and desorption are recognized to participate in the tritium recovery process [1–3]. In addition to these, several investigations have suggested a definite role of irradiation defects. In fact, apparent effects of irradiation have been observed on the tritium behavior in lithium ceramics [32–41].

A typical result has been obtained in the radiochemical measurement of tritium species produced in Li_2O by 14 MeV neutron irradiation [39]. The chemical state distribution has been found to be dependent on the neutron fluence; the T^- fraction increases and the T^+ fraction decreases with increasing neutron fluence. This indicates the presence of the interactions of tritium with irradiation defects. The neutron fluence dependence is also observed in the tritium release behavior. A fraction of tritium is recovered as non-condensable form (HT or T_2) with a hydrogen containing sweep gas and the HT fraction increases with increasing neutron fluence.

It is very important to determine the interactions of tritium with irradiation defects for predicting the effects of irradiation under a high neutron flux in actual blanket conditions. An isothermal annealing experiment of neutron-irradiated Li_2O has thus been performed in order to determine some details of the mechanisms and kinetics [40]. The tritium species of T^+, T^- and T^0(HT, T_2) remaining in the annealed sample have been analyzed by a radiochemical method, and the chemical state distribution of tritium has been observed to change with the annealing temperature and time duration. As well as the F^+ centers, the F^0 centers are considered to act as the reducing agents and to interact with tritium species. It is interesting to note that similar effects of irradiation have been observed in lithium salts [38] and lithium alloys [42].

For the assessment of tritium release behavior of Li_2O under irradiation, a model reaction scheme has been presented by taking into account the interactions of irradiation defects with tritium [43]. Tritium is produced in Li_2O grains by nuclear reactions and stabilized in the chemical states of T^+(LiOT) or T^-(LiT). The tritium species interact with the F^0 centers and O_2 molecules, which are predominantly produced at higher temperatures, before their release from the grain surface.

$$\mathrm{LiOT} + F^0 \rightarrow \mathrm{LiT} + \mathrm{Li_2O}, \tag{5}$$

$$\mathrm{LiT} + 1/2\mathrm{O_2} \rightarrow \mathrm{LiOT}. \tag{6}$$

After a number of conversions between LiOT and LiT, tritium diffuses to the grain surface where the following surface reactions take place.

$$\mathrm{LiT} + \mathrm{LiT} \rightarrow 2\mathrm{Li} + \mathrm{T_2} \uparrow, \tag{7}$$

$$\mathrm{LiT} + \mathrm{LiOT} \rightarrow \mathrm{Li_2O} + \mathrm{T_2} \uparrow, \tag{8}$$

$$\mathrm{LiOT} + \mathrm{LiOT} \rightarrow \mathrm{Li_2O} + \mathrm{T_2O} \uparrow . \tag{9}$$

Tritium is thus released in the form of T_2 or T_2O to the sweep gas. By taking this reaction scheme, some predictions have been made and compared with the observations in the BEATRIX-II experiment [4,44–47]. The observed tritium release behavior has successfully been interpreted. It may be noted that tritium chemistry is sensitive to impurity and oxygen potential (sweepgas composition). In practice, there is often protium (H) in these experimental systems so that the species released are HT and HTO. Also, the effect of H_2 addition to the sweep gas is quite important under irradiation [43].

It is important that irradiation defects participate in the reaction mechanisms even at such high temperatures, because material stability will be affected as well as tritium release kinetics. In the case of H_2-containing sweep gas, F^0 and O_2 are considered to react with H_2 at the grain surface as:

$$F^0 + \mathrm{H_2} \rightarrow 2\mathrm{LiH}, \tag{10}$$

$$\mathrm{O_2} + 2\mathrm{H_2} \rightarrow 2\mathrm{H_2O} \uparrow . \tag{11}$$

Due to reaction (11), the moisture concentration of H_2O will increase with the increasing H_2 concentration in the sweep gas, as observed in the BEATRIX-II, and then the material degradation will be accelerated in the presence of H_2. Further studies are required for its importance.

For the further development of the model, the equilibrium constant and rate constant of each reaction are to be determined. Such data are still lacking at the present time, and are to be determined by further studies. As well as the steady state chemistry, the dynamic behavior of tritium release may be studied for the determination of these constants.

5. Irradiation effects on structural changes

A number of the studies have been performed for demonstrating the materials stability and performance under irradiation. For example, post-irradiation examination focused on the dimensional stability, physical integrity, and microstructural and mechanical testing with the samples irradiated up to the lithium burn-up of 3% [47,48]. Fragmentation, which was assigned to result mostly from thermal gradients and thermal shocks, was observed in some cases. In the FUBR-1A test series, the materials stability was studied by using the EBR-II [49], and Li_2O was found to exhibit high swelling though the ternary ceramics showed relatively small swelling rates.

Thermal conductivity is a property of major concern for materials acting as heat transfer agents. However, the values of the lithium ceramics are rather low. Generally the values decrease with increasing porosity and increasing temperature and are much sensitive to impurities and defects. It is thus important to demonstrate the thermal conductivity performance of the lithium ceramics under irradition. In the FUBR-1A test [50], a significant reduction was observed in the thermal conductivity of Li_2O and $LiAlO_2$ at lower temperatures up to 400°C. At higher temperatures, however, the thermal conductivity values were unchanged. The results are consistent with those of the isochronal annealing experiment of the fractional volume changes of the irradiated Li_2O [51]. The fractional volume change decreased with increasing annealing temperature and might be interpreted by considering the recovery behavior of irradiation-induced defects in Li_2O.

It is thus needed to take into account the role of the irradiation-induced defects for fully understanding and controlling the materials performance under irradiation. In addition to the above mentioned measurements of the irradiation-induced defects, the studies on the nucleation and growth process of defect clusters are also needed by ordinary transmission electron microscopy [52] and other new techniques. An example for such new techniques is given below.

Structural change of Li_4SiO_4 due to irradiation, which led to formation of the other compounds, was investigated by FT-IR PAS (Fourier Transform Infrared Photoacoustic spectroscopy) and the semi-empirical molecular orbital calculation [53]. The FT-IR PAS spectra of Li_4SiO_4 irradiated to the fluence of 8.7×10^{18}, 5.0×10^{19} and 1.7×10^{20} ions m^{-2} with 120 MeV oxygen ions are shown in Fig. 5. For Li_4SiO_4 irradiated to 8.7×10^{18} ions m^{-2}, many absorptions due to the irradiation appeared in the range 1100–800 cm^{-1}, as shown in the spectrum (B). The absorptions, especially the absorptions at 1042 and 950 cm^{-1} grew with fluence (see the spectra (C) and (D)). It is also recognized that the absorptions near 800 cm^{-1} grew slightly with fluence. The absorptions near 800 cm^{-1} seem to be formed with some small absorptions.

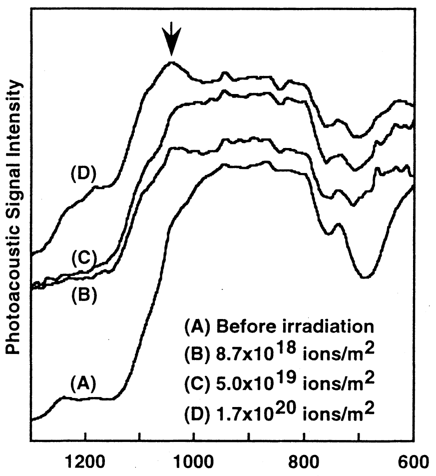

Fig. 5. FT-IR PAS spectra of Li_4SiO_4 irradiated with 120 MeV oxygen ions [53].

The calculation of theoretical IR spectra was carried out with the semi-empirical molecular orbital method, MNDO, with the program package MOPAC 93 [54], in order to identify structural changes causing the observed change of FT-IR PAS spectra due to irradiation. The IR spectra were calculated for the Li_4SiO_4, $Li_6Si_2O_7$, $Li_8Si_3O_{10}$, $Li_{10}Si_4O_{13}$ and $Li_{12}Si_5O_{16}$ clusters. The strong absorption associated with the stretching vibration of the SiO_4 unit and the weak absorption due to the deformation vibration of SiO_4 unit appeared in the theoretical spectrum of Li_4SiO_4. On the other hand, Si–OLi stretching, Si–O stretching, Si–O–Si stretching, Si–O–Si bending and O–Si–O asymmetric vibrations etc. were observed in the theoretical spectra of $Li_6Si_2O_7$, $Li_8Si_3O_{10}$, $Li_{10}Si_4O_{13}$ and $Li_{12}Si_5O_{16}$ clusters with one to four bridging oxygens.

Theoretical line spectra of $Li_{10}Si_4O_{13}$ (sheet structure) and $Li_{12}Si_5O_{16}$ (three-dimensional-framework structure) are shown in Fig. 6 along with the experi-

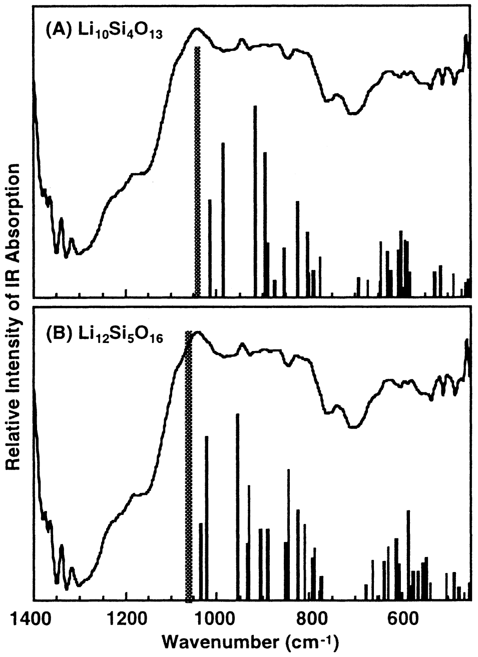

Fig. 6. Experimental FT-IR PAS spectrum of Li_4SiO_4 irradiated to 1.7×10^{20} ions m^{-2} with 120 MeV oxygen ions and the theoretical IR line spectra of $Li_{10}Si_4O_{13}$ and $Li_{12}Si_5O_{16}$ [53].

mental FT-IR PAS spectrum of Li_4SiO_4 irradiated to the fluence of 1.7×10^{20} ions m^{-2}. The strong absorptions at 1060 and 1040 cm^{-1}, which were calculated for $Li_{12}Si_5O_{16}$ and $Li_{10}Si_4O_{13}$ clusters, respectively, can be found in the experimental FT-IR PAS spectrum of Li_4SiO_4 irradiated. Such strong absorptions near 1040 cm^{-1} are not in the theoretical IR spectra of the other clusters. The prominent absorptions near 1040 cm^{-1} are considered to be due to the slightly deformed Si–O–Si stretching vibration in the sheet and three-dimensional-framework structures. Many weak absorptions also appeared at 950–800 cm^{-1} correspond to O–Si–O asymmetric vibrations in the end of SiO_4 units of the clusters.

Recovery behavior of the structural change was preliminarily investigated [55]. The absorptions near 1040 cm^{-1}, i.e., $Li_{10}Si_4O_{13}$ (sheet structure) and $Li_{12}Si_5O_{16}$ (three-dimensional-framework structure), were not recovered by annealing at 770 K for 30 min.

6. Conclusions

(1) The production of irradiation-induced defects was observed in lithium ceramics irradiated by electrons, neutrons and ion beams, and the F-type centers and colloidal metals have been identified by usual ESR and optical absorption measurements.

(2) Kinetic aspect of the production behavior of the irradiation-induced defects was also studied in some detail by an in situ luminescence measurement under ion beam irradiation, and some of the defect species were found to be produced even at higher temperatures of blanket conditions.

(3) Irradiation effects on tritium recovery performance were observed in many experiments, and were explained by taking into account the interactions of tritium with the defect species. However, the equilibrium constants and reaction rate constants are still lacking for a complete evaluation of tritium recovery performance at actual blanket conditions.

(4) The defect species will participate in the microstructural changes of lithium ceramics and then the materials performance such as the thermal conductivity under irradiation. In order to control the phenomena, further experimental efforts are needed to give more attention not only to integrated experiments but also to fundamental aspects of the phenomena.

(5) For the design of blanket systems, long-term effects on tritium release behavior and microstructural changes of lithium ceramics are critically important. The studies at high burn-up levels are needed since the database is still limited to rather low burn-up levels.

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