

Influence of various gases and water vapors on the processes of tritium release from two-phase lithium ceramics

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ARTICLE INFO

Keywords:

Two-phase lithium ceramics

Tritium release

Hydrogen isotopes

Water vapor

Neutron irradiation

ABSTRACT

The processes of tritium generation and release in lithium-ceramic breeder blankets are affected by many factors, for example, the presence of impurities in the purge gas. As was shown earlier, oxygen, water vapor and hydrogen have the greatest influence on gas release from lithium ceramics. Hydrogen is planned to be specifically added to the purge gas mixture in breeder blanket to facilitate tritium release.

The paper presents data of reactor experiments on irradiation of two-phase lithium ceramics (65 mol% Li_4SiO_4 / 35 mol% Li_2TiO_3) under conditions of supplying various gases and water vapor into the chamber with samples.

We analyzed the sections of experiments in which sequential, step-by-step injection of water, oxygen, hydrogen and deuterium vapors into a continuously pumped chamber with samples at steady-state quasi-equilibrium release of tritium from ceramics was carried out. Using a mass spectrometer, the gas composition in the chamber and, in particular, the release features of tritium-containing molecules, were recorded. Based on the obtained data, the mechanisms of tritium interaction with the investigated gases and water vapor on the ceramic surface were determined, as well as their influence on the processes of tritium release from ceramics.

1. Introduction

The choice of materials for fusion reactors (FR) and, in particular, for blanket systems is one of the most important tasks determining the development of fusion power engineering [1,2]. Two-phase lithium ceramics (65 mol% Li_4SiO_4 / 35 mol% Li_2TiO_3) is considered as a promising candidate material for solid-state breeder blankets of future fusion reactors because of their low activation and chemical activity, as well as the fact that they release tritium well [3–6].

To date, an accurate understanding of the processes of generation and release of tritium and helium occurring in breeder blanket materials under the neutron irradiation can be obtained only on the basis of the results of reactor experiments. During reactor irradiation of lithium ceramics, tritium and helium are produced in the samples according to the nuclear reaction ${}^6\text{Li}(n,\alpha){}^3\text{H}$. The in-reactor experiments make it possible to evaluate the kinetics of the change in the gas release levels

from the samples as a function of changes in temperature and accumulated fluence.

The following experiments on the study of tritium release from lithium ceramics under reactor irradiation conditions were carried out: LISA series experiments [7,8], BEATRIX (Breeder Materials Experimental Matrix) program [9,10], EXOTIC (EXtraction of Tritium In Ceramics) program [11,12], JMTR reactor [13,14], CORELLI-2 experiment [15], IVV2-M reactor [16], SPRR-300 reactor [17], WWR-K [18–21] and IVG.1 M research reactors [22–24].

The processes of tritium generation and release in lithium-ceramic breeder blankets depends are affected by many factors, for example, the presence of impurities in the purge gas. As shown earlier, oxygen, water vapor and hydrogen are the most influential gas impurities.

Therefore, in the present paper the influence of the presence of gases such as hydrogen, deuterium, oxygen and water vapor in a chamber with samples of two-phase lithium ceramics on the processes of tritium

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release was studied in the mode of in-situ reactor experiments.

By varying the pressure level of the gases injected into the evacuated chamber, it was possible to obtain the dependence of the change in the composition of the released tritium-containing molecules. It is worth noting here that under actual operating conditions of a fusion reactor, lithium ceramics will be purged with helium, and in the presented reactor experiments, the samples are under conditions of continuous pumping (the pressure level in the chamber with the samples is about 10^{-6} torr without gas injection and up to 10^{-4} torr when releasing gases).

This mode of conducting experiments allows us to obtain additional information about the parameters of the separation processes, since it allows us to record the entire composition of gases in the chamber. In this mode, it is easier to identify the main limiting stage of tritium release from ceramics based on the kinetics of changes in partial pressures; understand how redistribution occurs in the composition of released tritium molecules.

The data obtained can be used to analyze and determine the mechanisms of tritium release from ceramics under real operating conditions of fusion reactors.

This article is a continuation of a series of works by the authors, the main results of which were published in [25–29]. In particular, in [25] similar reactor studies with lithium ceramics (75 mol% Li_4SiO_4 / 25 mol% Li_2TiO_3) were carried out.

The present work presents data of reactor experiments on irradiation of another two-phase lithium ceramics (65 mol% Li_4SiO_4 / 35 mol% Li_2TiO_3) under conditions of supplying various gases and water vapor to the chamber with samples.

2. Materials and methods

The samples of two-phase lithium ceramics (65 mol% Li_4SiO_4 / 35 mol% Li_2TiO_3) in the form of pebbles with sizes from 250 to 1250 μm and total mass ~ 5 g, were manufactured according to the KALOS process [30] and provided by the Karlsruhe Institute of Technology (KIT). Reactor experiments with two-phase lithium ceramics were performed at the CIRRA experimental facility for the study of gas release from materials of fission and fusion reactors based on the WWR-K research reactor [31]. The test samples of lithium ceramics were placed inside the experimental cell located in a pumped experimental ampoule, which was connected to the CIRRA facility. The ampoule was equipped with a heater to provide the required temperature conditions. The ampoule temperature was controlled by means of thermoelectric converters of CrAl type (chromel-alumel thermocouples) installed on the walls and on the bottom of the ampoule. At the presented sections of the reactor experiments, the ceramic temperature was ~ 930 K.

The experimental equipment, the design of the ampoule device with the investigated sample, and the methodology of reactor experiments are described in [25,26,31]. The block diagram of in-reactor experiments with the ampoule device and a sample of two-phase lithium ceramics connected to the gas-vacuum system of the CIRRA unit are presented in Fig. 1. It should be mentioned that all installation paths and ampoule are made of stainless steel. Due to external heaters, it is possible to heat up the installation (with the exception of the mass spectrometer and turbomolecular pump) to a temperature of 423 K during experiments. Such heating was carried out periodically to estimate the amount of tritiated water sorbed by the walls.

Reactor experiments were performed by vacuum extraction method: two-phase lithium ceramics were irradiated under continuous pumping conditions, and a quadrupole mass spectrometer (RGA-100) recorded the release of tritium-containing molecules.

The irradiation period was 21 days with the reactor thermal power equal to 6 MW and thermal neutron flux in the irradiation area $\sim 5 \cdot 10^{13}$ $\text{n}/\text{cm}^2 \cdot \text{s}$. Experiments with various gas impurities and water vapor were performed on the 8th and 15th days of samples irradiation. The accumulated fluence was $3.4 \cdot 10^{19}$ n/cm^2 and

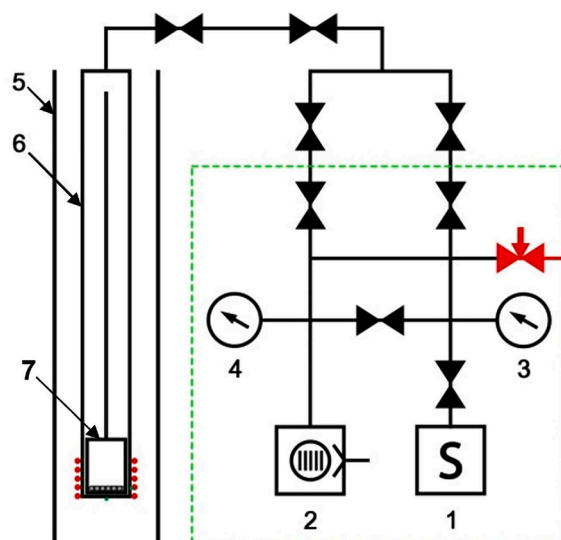


Fig. 1. Block diagram of in-reactor experiments with the investigated sample of two-phase lithium ceramics: 1 – mass-spectrometer, 2 – turbomolecular pump, 3,4 – pressure sensors, 5 – irradiation channel, 6 – ampoule device, 7 – capsule with test sample, allocated in red – leak-valve for inlet of investigated gases.

$6.4 \cdot 10^{19}$ n/cm^2 , respectively.

The methodology of the experiment was as follows. Working gas (hydrogen, deuterium, water vapor, and oxygen) was injected through a special gas-vacuum system of the CIRRA unit during irradiation of lithium ceramics. The pressure of residual gases in the chamber was $\sim 10^{-6}$ Torr. The gas was injected in steps, with duration of about 10 min at each step at a constant flow into the installation chamber. Typically, the gas flow supplied to the chamber at each inlet stage was successively increased up to the level of gas pressure in the chamber $\sim 10^{-4}$ Torr, then stepwise decreased until the gas supply system was completely closed. Water vapor was injected from a special heated capsule in the mode of supplying vapor into the chamber to a pressure level of $8 \cdot 10^{-5}$ Torr, while the entire installation was heated to 353 K (with the exception of the mass spectrometer and turbomolecular pump). During the experiment, changes in the partial pressures of residual gases with mass numbers from M2 to M46 in the installation chamber were recorded until the quasi-equilibrium state was reached. The registration was carried out in the form of an expanded mass spectrum from 2 to 46 a.u.m. continuously with an interval of 21 s between scans. The dependences of pressures changes of molecules HT, T_2 , H_2O , etc., were made on the basis of calculations from calibration experiments data and analytical estimates, which are partially given in a number of papers on reactor experiments with two-phase lithium ceramics [25–29].

Calibration of the mass spectrometer readings was carried out in pre-reactor experiments for gases: hydrogen, deuterium, helium, nitrogen (for an installation configuration similar to reactor experiments).

In the reactor experiments for hydrogen, HT, DT, deuterium and tritium molecules, the calibration coefficient obtained in the pre-reactor experiments for hydrogen and deuterium were used (since the difference in the calibration coefficient for hydrogen and deuterium was less than 10 %).

In experiments with deuterium infusion, tritium calculations were carried out taking into account the formation of the D_3^+ complex, as in previous experiments [link to article with infusions]

3. Results and discussion

Below are the results of experiments when different gases and water vapor are injected into the chamber with lithium ceramics samples. The experiments presented are small sections of long-term experiments on

irradiation of two-phase lithium ceramics under conditions of continuous pumping, which were carried out at an equilibrium state of tritium release from ceramics at a level of $1.25 \cdot 10^{-7}$ mol/(kg·s). A description of the complete experiments is given in [29].

Fig. 2 shows the data on the change of partial pressures of different gases in the experimental chamber with a sample of two-phase lithium ceramics when hydrogen is injected into the ampoule.

As can be seen from the figure, with increasing hydrogen pressure in the installation chamber, a decrease in the partial pressure of tritium T_2 and an increase in the HT gas pressure are observed. The values of partial pressures of tritiated water for experiments with hydrogen injection were significantly lower than the total release of tritium and had virtually no effect on its balance. The observed change is due to the fact that tritium, generated in ceramics under irradiation, comes to the surface of the sample as a result of transfer processes from the ceramic volume, while hydrogen is sorbed by the ceramic surface from the gas phase and further surface reactions occur, resulting in desorption of T_2 and HT molecules. With increasing hydrogen pressure in the installation chamber, the amount of hydrogen on the ceramic surface increases, this leads to the observed effects.

In deuterium injection experiments, deuterium sorption onto the ceramic surface and subsequent isotope exchange reactions and desorption of associated tritium and deuterium atoms similarly occur, with deuterium and tritium forming a DT molecule (Fig. 3).

This becomes an additional channel for tritium release from lithium ceramics. When the pressure of deuterium in the gas phase increases, there is a preferential increase in the release of tritium-containing molecules through this channel (in the form of DT). That in turn leads to a decrease in the release of tritium in the form of HT and T_2 molecules to almost complete termination.

We would like to note that in Fig. 3 the data on measurement of T_2 molecule release in the pressure region below $3 \cdot 10^{-8}$ Torr are limited due to the peculiarities of calculation of the T_2 gas pressure associated with the D_2D^+ complex formed in the ion source of the RGA-100 mass spectrometer at deuterium injection. This effect is described in detail in [25].

In general, there is a very good qualitative coincidence of the results of these experiments with the results of reactor experiments that were carried out earlier with lithium ceramics 75 mol% Li_4SiO_4 / 25 mol% Li_2TiO_3 [25]. For example, comparison of the dependence of DT molecule release on the deuterium pressure in the installation chamber for different types of ceramics showed that they coincide quite well (Fig. 4).

The peculiarity of the experiments with water vapor injection into the installation chamber was that a single injection pressure was

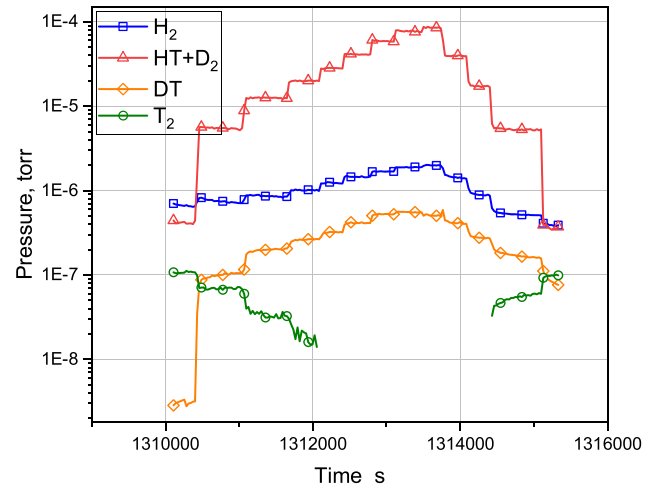


Fig. 3. Dependence of DT and T_2 pressure change on change of deuterium pressure in installation chamber.

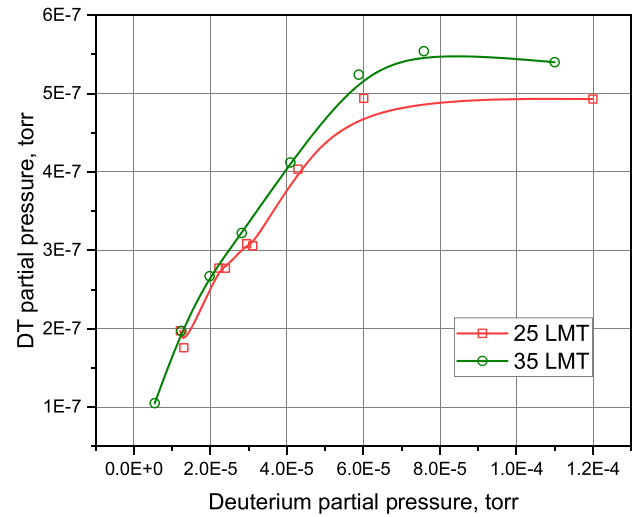


Fig. 4. Dependence of DT molecule pressure on deuterium pressure in the installation chamber for different types of ceramics.

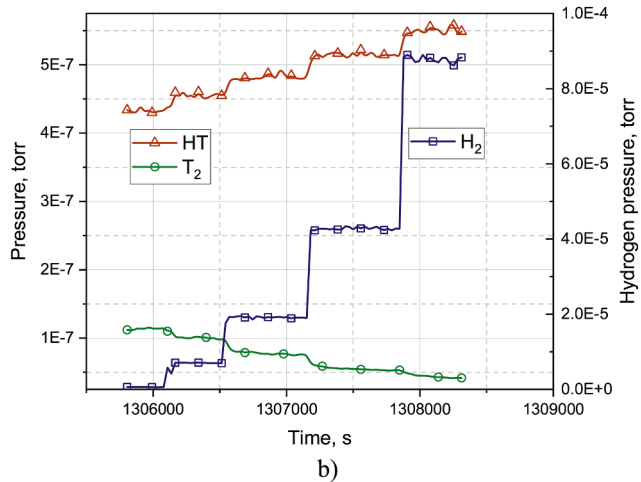
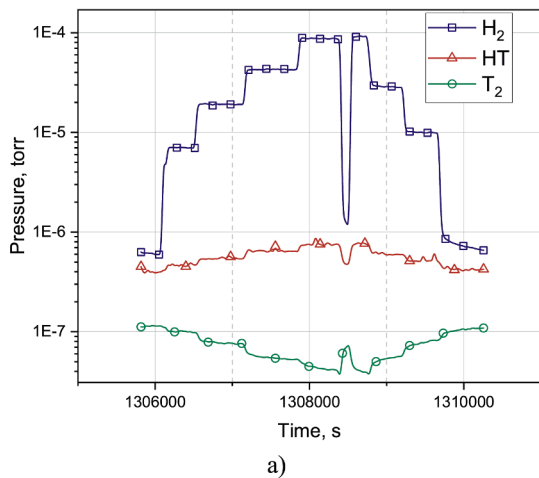


Fig. 2. Dependence of HT and T_2 pressure change on hydrogen pressure in installation chamber: a) whole diagram of experiment in log scale; b) enlarged graph of hydrogen supply section (linear scale for HT and T_2 pressures).

investigated, since it was difficult to organize a stepwise supply of water vapor into the chamber. When water vapor was injected into the installation chamber (Fig. 5), up to pressures of about

10^{-4} Torr, a sharp decrease in the release of T_2 molecules (practically to 0) and HT molecules (more than 5 times) was observed.

Fig. 6 illustrates the ratio of released tritium-containing molecules (HTO, T_2 and HT) when water vapor is injected into the installation chamber. The main release channel for tritium here becomes its release in the form of tritiated water HTO.

Fig. 7 shows the ratio of released tritium-containing molecules (HTO, T_2 and HT) for different pressures of oxygen injected into the installation chamber. As can be seen, the oxygen inlet does not lead to such significant changes as the water vapor inlet: there is a slight decrease in the release of tritium in the form of HT and T_2 molecules and a slight increase in the release of tritium in the form of tritiated water HTO.

Preliminarily, we discuss the mechanism of tritium molecules release from the surface of lithium ceramics under irradiation conditions without gas injection. We observe that in the equilibrium state tritium is mainly released in the form of HT and T_2 molecules, and with time the flux of HT molecules decreases and the release of T_2 molecules increases (here we observe a dependence on the hydrogen pressure in the installation chamber, which also decreases with time as the heated elements of the ampoule are degassed). Hence, it can be assumed that on the ceramic surface, the tritium atom binds to the hydrogen (tritium) atom and desorbs as HT or T_2 molecule.

As was shown in [25] for lithium ceramics 75 mol% Li_4SiO_4 / 25 mol % Li_2TiO_3 , the near-surface bulk concentration of hydrogen atoms is proportional to the hydrogen pressure to the extent of 0.5, which is close to Sieverts' law, and indicates that tritium release occurs due to interaction with hydrogen isotopes that dissociate to atoms on the ceramic surface after their sorption from the gas phase (Fig. 9). This mechanism is better described than, for example, the exchange of tritium and hydrogen atoms with a physically adsorbed hydrogen molecule (H_2) on the ceramic surface.

Since, in the mode of experiment without gas injection practically no tritiated water vapor emission is observed, it can be assumed that the number of O-T and O-H complexes on the ceramic surface is insignificant and tritium release by reactions $O-H+T \rightarrow HTO$ and $O-T+H \rightarrow HTO$ is very small.

When the pressure of hydrogen (deuterium) into the installation chamber is significantly higher than the background value, the concentration of hydrogen (deuterium) atoms on the ceramic surface increases, respectively, the emission of HT molecule (DT) increases, and the emission of tritium molecules T_2 decreases, while the total amount of

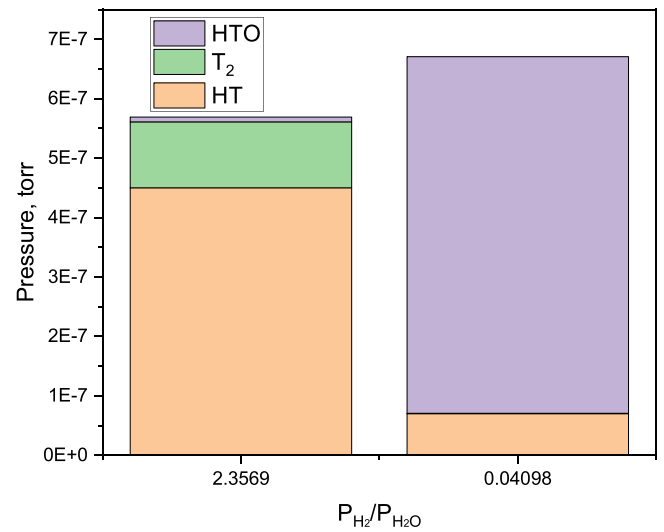


Fig. 6. Ratio of emitted tritium-containing molecules when water vapor is injected into the installation chamber.

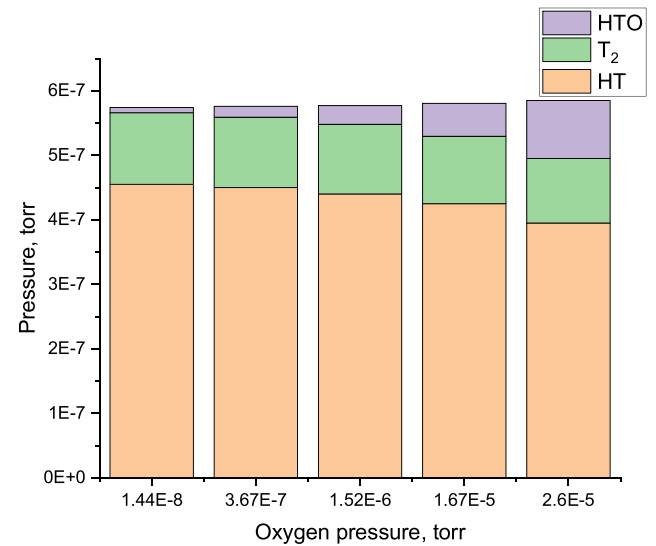


Fig. 7. Ratio of released tritium-containing molecules for different pressures of oxygen supplied to the installation chamber.

emitted tritium ($HT+2 \cdot T_2$) does not change.

Analyzing the change in gas composition when significant pressures of water vapor are injected into the chamber, it can be assumed that the following mechanism of tritium release is observed (Fig. 9):

- part of water molecules reaching the surface of ceramics dissociates into O-H complex and hydrogen atom;
- further, the concentration of O-H complexes increases significantly on the ceramic surface, which are desorbed from the ceramic surface through the channels $O-H+T \rightarrow HTO$ and $O-H+H \rightarrow H_2O$ with less probability than hydrogen atoms through the channels $H+T \rightarrow HT$ and $H+H \rightarrow H_2$;
- in a relatively short time after water vapor injection, the equilibrium state on the ceramic surface is as follows: tritium steadily enters the ceramic surface, where there is a significant amount of O-H complexes and some hydrogen atoms, after which it is desorbed from the surface by interacting either with the O-H complex or with a hydrogen atom.

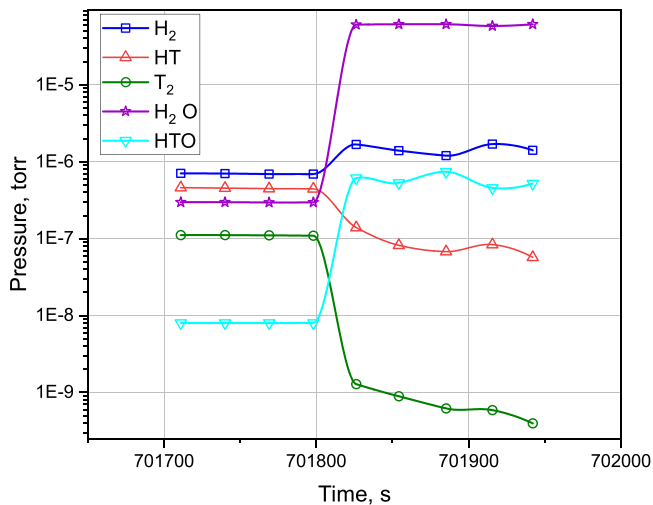


Fig. 5. Dependence of change in pressure of tritium-containing molecules on water vapor pressure in the installation chamber.

We can qualitatively estimate the ratio of hydrogen concentrations on the ceramic surface for hydrogen and water injection (at approximately the same pressures). Assuming that the concentration of tritium on the surface is the same (this follows from the fact that the total amount of tritium released from the ceramic does not change significantly), then from the expressions for the flux of HT molecule release written as in Eqs. (1) and (2) we can obtain Eq. (3) for the ratio of these fluxes at H₂ and H₂O injections). It was found to be equal to ~ 6.8. This value of 6.8 was obtained by dividing the quasi-equilibrium maximum pressure (which is proportional to the flow) of the HT molecule (for H₂ infusion experiments) by the quasi-equilibrium pressure of the HT molecule (for H₂O infusion experiments). This means that for the experiments with water vapor the effective area on which hydrogen atoms are located is 15 % of the value of the effective area in the experiments with hydrogen. Further also on a qualitative level it is possible to estimate the ratio of desorption rates from the ceramic surface of HT and HTO molecules. Assuming that the effective area of the sample in the experiments with H₂ and H₂O injection is the same, and using Eq. (4) we can obtain the ratio of desorption rate constants for HT and HTO molecules (Eq. (5)).

Further, if we assume that the total number of sites for OH complexes and hydrogen atoms on the surface of the samples is constant, then we can obtain the ratio $\frac{C_{HTO}^{H_2O}}{C_{HTO}^{H_2}} = \frac{0.15}{0.85} = 0.18$.

Having determined the value $\frac{\varphi(HT)_{H_2O}}{\varphi(HTO)_{H_2O}} = 0.11$ from the graph in Fig. 6, we obtain the ratio for $\frac{b_1}{b_2} \sim 0.65$.

$$\varphi(HT)_{H_2} \sim b_1 \cdot C_H^{H_2} \cdot C_T^{H_2}, \quad (1)$$

$$\varphi(HT)_{H_2O} \sim b_1 \cdot C_H^{H_2O} \cdot C_T^{H_2O}, \quad (2)$$

$$\frac{\varphi(HT)_{H_2}}{\varphi(HT)_{H_2O}} = \frac{C_H^{H_2}}{C_H^{H_2O}} \quad (3)$$

$$\varphi(HTO)_{H_2O} \sim b_2 \cdot C_{HO}^{H_2O} \cdot C_T^{H_2O} \quad (4)$$

$$\frac{\varphi(HT)_{H_2O}}{\varphi(HTO)_{H_2O}} = b_1 \left/ b_2 \cdot C_H^{H_2O} \right/ C_{HO}^{H_2O} \quad (5)$$

where $C_H^{H_2}$, $C_H^{H_2O}$, $C_T^{H_2}$ and $C_T^{H_2O}$ – relative concentrations of H and T atoms on the surface of ceramics (at H₂ and H₂O injections, respectively), $C_{HO}^{H_2O}$ – relative concentration of OH complexes on the ceramic surface (at H₂O injection), b_1 – desorption rate constant for reaction H+T→HT, b_2 – desorption rate constant for the reaction O+H+T→HTO. As for the release of tritium at oxygen injection, the mechanism is evidently the following (Fig. 8):

- initially an oxygen molecule adsorbs onto the surface of the ceramic and dissociates into oxygen atoms;
- then, the oxygen atom interacts with the hydrogen atom, forming O—H, or O—T complexes on the surface, which is desorbed as tritiated water (mainly HTO).

In general, since the process of tritium release in the form of tritiated water is two-stage, it has less influence on the composition of the released tritium-containing molecules, unlike water vapor.

4. Conclusions

This paper presents the results of reactor experiments to investigate the influence of gas phase composition on the processes of tritium release from two-phase lithium ceramics 65 mol% Li₄SiO₄ / 35 mol% Li₂TiO₃, which are a continuation of earlier similar works with ceramics 75 mol% Li₄SiO₄ / 25 mol% Li₂TiO₃ [25]. The experiments allowed us to

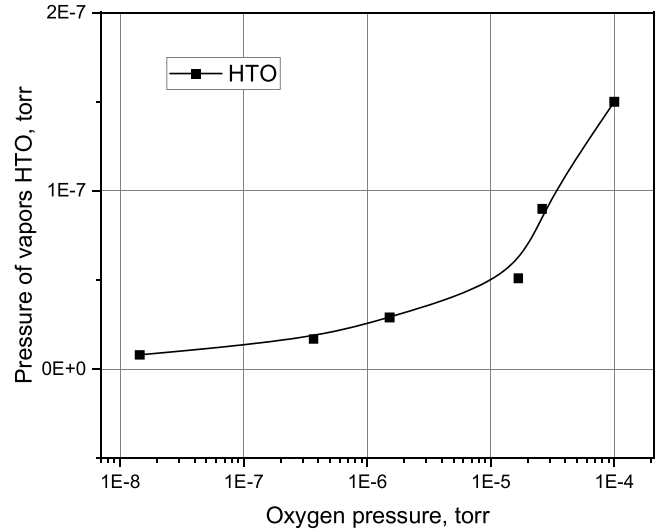


Fig. 8. Dependence of HTO vapor release on oxygen pressure.

record the influence of hydrogen, deuterium, water vapor and oxygen (separately) on the changes in the composition of the released tritium-containing molecules.

As for the experiments with hydrogen and deuterium injection, they in general well repeated (one can even say coincided) the previously obtained effects in the experiments with 75 mol% Li₄SiO₄ / 25 mol% Li₂TiO₃ ceramics.

When hydrogen was injected, a decrease in the partial pressure of tritium T₂ and an increase in the HT pressure were observed, which is associated with the interaction of hydrogen with tritium on the surface of lithium ceramics. Here was confirmed the mechanism associated with the fact that tritium, produced in ceramics under irradiation, comes to the surface of the sample as a result of transfer processes from the ceramics bulk, and hydrogen is sorbed by the surface of ceramics from the gas phase and then there are surface reactions, as a result of which molecules T₂ and HT are desorbed. For experiments with deuterium injection, a similar picture was observed with the difference that deuterium and tritium formed desorbed DT molecules.

In experiments with water vapor injections, a significant decrease in the amount of tritium released in the form of T₂ and HT molecules and a pronounced increase in the release of tritium in the form of tritiated water HTO were obtained. The mechanism of such tritium release was proposed, which is associated with the formation of OH complexes on the ceramic surface during its interaction with water vapor, and a number of parameters of desorption processes were estimated, in particular, the ratio of tritium desorption rates through the H+T and OH+T channels.

CRediT authorship contribution statement

Timur Kulsartov: Writing – review & editing, Writing – original draft, Investigation, Conceptualization. **Inesh Kenzhina:** Writing – original draft, Resources, Investigation. **Regina Knitter:** Supervision, Project administration. **Julia Leys:** Supervision, Resources. **Zhanna Zaurbekova:** Writing – review & editing, Investigation. **Asset Shaimerdenov:** Supervision, Resources, Methodology, Investigation. **Saulet Askerbekov:** Methodology, Investigation. **Magzhan Aitkulov:** Methodology, Investigation. **Alexandr Yelishenkov:** Validation, Investigation. **Anastasiya Yevdakova:** Investigation. **Timur Zholdybayev:** Supervision, Project administration, Funding acquisition, Formal analysis.

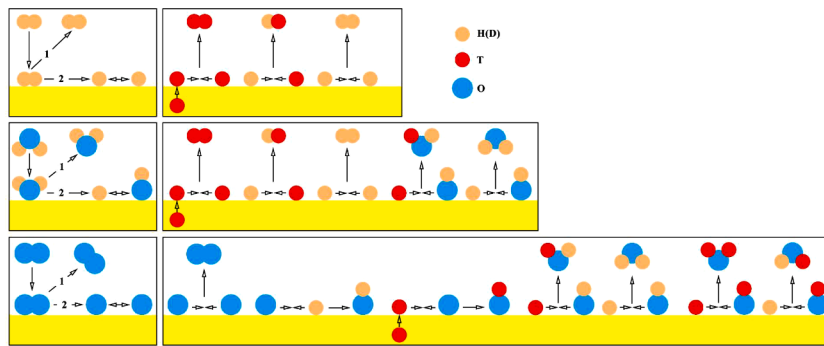


Fig. 9. Scheme of gas interaction on the ceramic surface.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

Acknowledgments

This research has been funded by the Committee of Science of the Ministry of Science and Higher Education of the Republic of Kazakhstan with Program No. BR10965174.

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