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Investigation of hydrogen and deuterium impact on the release of tritium from two-phase lithium ceramics under reactor irradiation

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ABSTRACT

In the development of fusion energy, an important task is the study and improvement of tritium production technologies. In this case, one of the most promising materials for tritium generation is lithium ceramics. Considering the importance of the task, numerous studies are aimed at solving the problem of determining the parameters and mechanisms of tritium release in lithium-containing materials. This paper presents the results of a study of tritium release processes from two-phase lithium ceramics of Li_4SiO_4/Li_2TiO_3 during reactor irradiation when hydrogen and deuterium are injected into the chamber with irradiated samples. The mechanisms regularities of the tritium yield process in the presence of these isotopes were established. The experiments were carried out in the WWR-K research reactor at a neutron flux density of $5 \cdot 10^{13} n/cm^2 \cdot s$ and sample temperatures from 650 to 700 °C.

1. Introduction

Tritium is the main fuel of future fusion reactors [1,2]. Since it is absent in nature, it is important to improve the technology for tritium generation using nuclear reactions. Therefore, future designs of fusion plants will include a breeder blanket for tritium generation [3]. The choice of the optimal functional material for a blanket is a key task in solving the issues of closing the fuel cycle and self-supply of fuel for fusion reactors. In the concept of solid breeder blanket lithium-containing ceramics are used to produce tritium. And the most promising candidates for this purpose are lithium orthosilicate Li_4SiO_4 (LOS) and lithium metatitanate Li_2TiO_3 (LMT) [4,5]. Considering the importance of the problem, numerous studies are aimed at solving the problem of determining the parameters and mechanisms of tritium release in lithium ceramics. As shown in [6–10], the release of produced tritium significantly depends on the conditions under which the lithium-containing material is irradiated: for example, the addition of a small amount of hydrogen to the chamber with the sample significantly increases the rate of tritium release. Therefore, it is important to know the mechanisms of processes occurring in the volume with samples of lithium ceramics at the injection of various gases in the gas phase. This study is aimed at determining the influence of hydrogen and deuterium on the processes of tritium release from two-phase lithium ceramic Li_4SiO_4/Li_2TiO_3 during reactor irradiation.

2. Materials and method

2.1. Object of the study

The object of the study is a natural enriched, two-phase lithium ceramic containing 25 mol% of Li_2TiO_3 (LMT) and 75 mol% of Li_4SiO_4 (LOS) in the form of pebbles with a size range of 250 to 1250 μm (Fig. 1).

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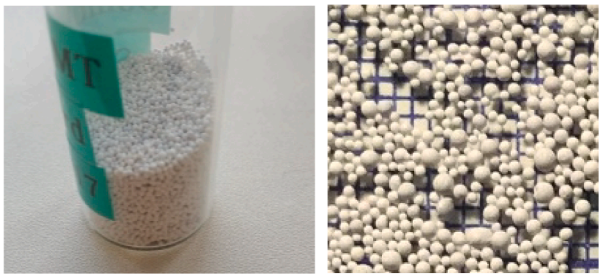

Fig. 1. Samples of two-phase lithium ceramic in the form of pebbles.

The pebbles were made using KALOS method [5] and provided by KIT (Karlsruhe Institute of Technology). More details on the material is given in [11].

The samples were fabricated in March 2016 and then were stored under dry nitrogen. In June 2017 the samples were heated up to 300 °C and filled into small glass bottle (under air) and are subsequently packed in plastic bags that were then evacuated and sealed. In December 2020 the bottle was opened just before irradiation experiments.

2.2. Experimental method

Prior to the experiments with hydrogen isotopes injected, ceramic samples were irradiated under conditions of continuous evacuation (gas pressure ~ 10^{-6} torr; mainly hydrogen and water vapor) at a temperature of ~ 680 ℃ for ~ 2.5 days. The flux of thermal neutrons in the experimental channel of the reactor was about $5{\cdot}10^{13}$n/(cm^2·s). Neutron spectrum of the WWR-K reactor is given in Fig. 2. At the beginning of irradiation and reaching the steady-state power level of the reactor, significant gas evolution was observed with mass numbers M2 (H_2), M18 (H_2O), M28 (CO) and M44 (CO_2) (to pressures of ~ $3{\cdot}10^{-5}$ torr, which then rapidly decreased (and continued to decline throughout the irradiation).

Experiments with hydrogen/deuterium supply were carried out at the CIRRA (Complex of In-Reactor gas Release Analysis) facility [12,13], located at the WWR-K research reactor in Almaty, Kazakhstan (Fig. 3). Experimental ampoule device with an investigated sample of two-phase lithium ceramic Li_4SiO_4/Li_2TiO_3 is shown in Fig. 4.

The temperature of the ampoule device was measured using 2 thermocouples of the Ch-A type (chromel–alumel) fixed on the bottom and wall of the ampoule device.

After the establishment of a quasi-equilibrium state of the residual gas composition in the permanently pumped ampoule, the working gas (hydrogen or deuterium) was injected through a special system of the

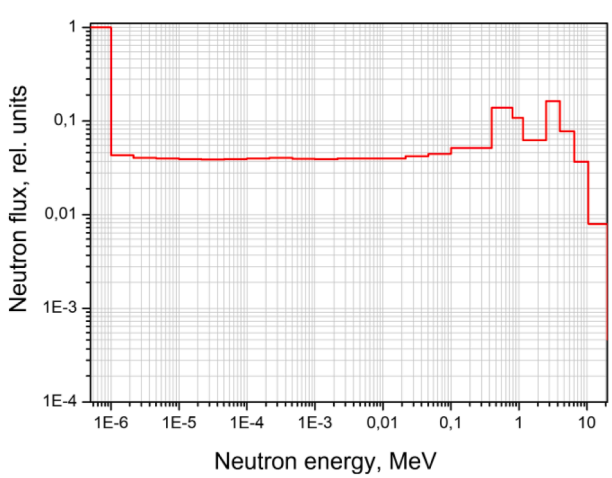

Fig. 2. Neutron spectrum of the WWR-K reactor.

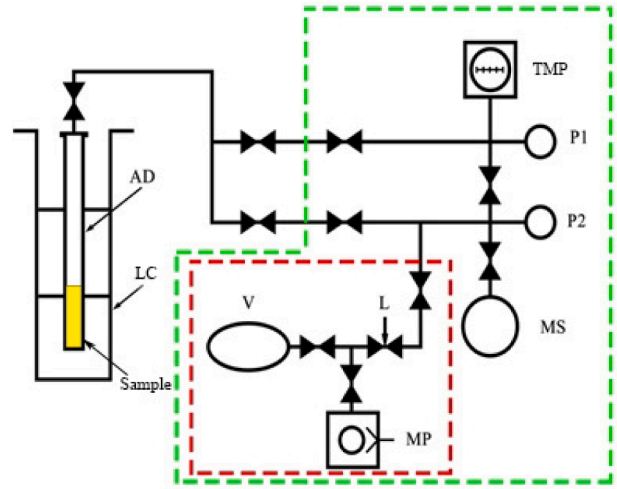

Fig. 3. The CIRRA facility for vacuum extraction experiments [12]: AD – ampoule device, LC – loading channel, MS – mass-spectrometer, TMP – turbomolecular pump, P – pressure sensor, L – leak valve, V – tank with gas, MP – mechanical pump.

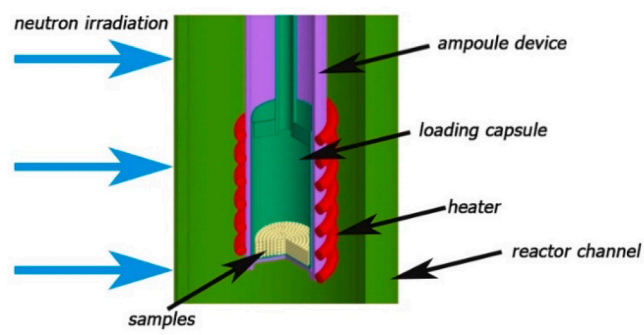

Fig. 4. Experimental ampoule device with an investigated sample (pebbles filling) [12]

CIRRA facility during the irradiation of lithium ceramics. Gas injection was carried out stepwise, with durations of 10 min at each step at a constant flow rate. The flow rate of the gas fed into the chamber at each stage of the inlet was sequentially increased to a gas pressure level of ~ 10^{-4} torr in the chamber, then it was also decreased in steps until the gas supply system was completely closed. In the course of the experiment, changes in partial pressures of residual gases in the chamber of the facility were recorded until the quasi-equilibrium state was reached.

Changes in the sample temperature during irradiation are shown in Fig. 5. As can be seen from the graph, it practically did not change during the experiment.

3. Results

3.1. Results of experiments with hydrogen injection

Figs. 6-7 show data on changes in the partial pressures of various gases in the experimental chamber at different levels of hydrogen flows into the ampoule. First, consider the experimental data on changes in the partial pressures of gases that do not contain a tritium atom. As can be seen from Fig. 6, when hydrogen is injected into the ampoule:

- the pressure of hydrogen (M2) in the chamber is increased, and this value exceeds the pressure level of other gases by more than two orders of magnitude and reaches ~ 10^{-4} torr;

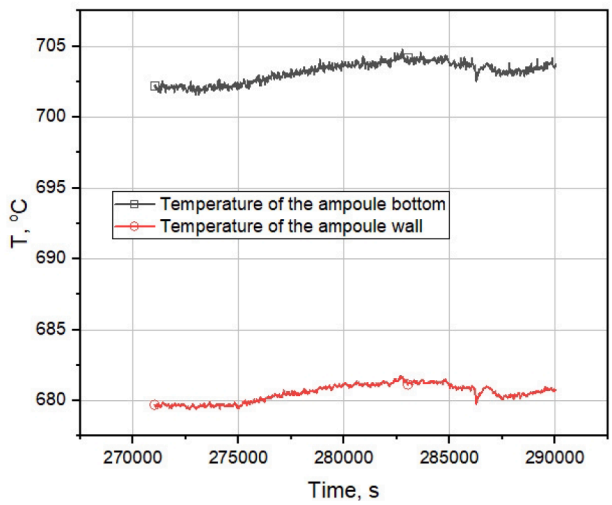

Fig. 5. Temperature of the ampoule with samples during the experiments.

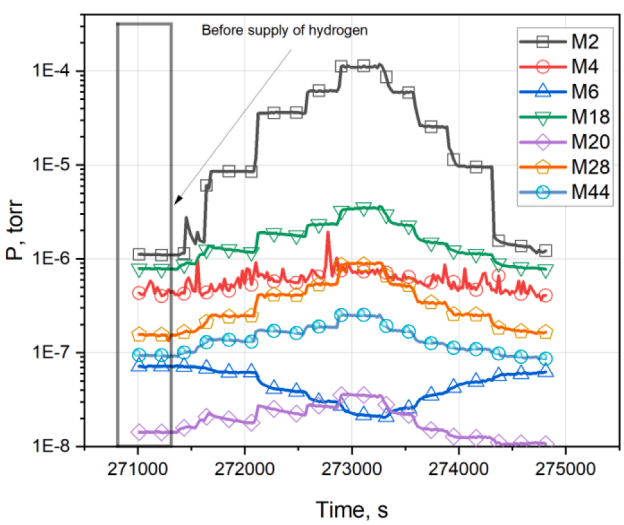

Fig. 6. Change of partial pressures of gases in the chamber of the facility at different hydrogen flows into the ampoule during irradiation time.

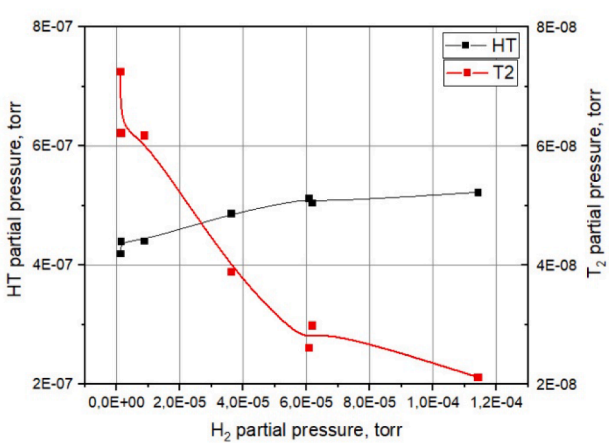

Fig. 7. Change in partial pressures of tritium-containing gases in the chamber of the facility at different hydrogen flows into the ampoule.

- a slight increase in the water vapor pressure (M18) occurs in the chamber with the sample, which is apparently due to the interaction of the injected hydrogen with surface oxides and oxides of the ampoule materials and samples according to reaction:

$$\boldsymbol{AO_x + H_2 \rightarrow AO_{x-1} + H_2O}; \tag{1}$$

- a relatively small increase in the pressures of nitrogen (M28) and carbon dioxide (M44) in the chamber is observed. This is caused by the fact that there is a small amount of atmosphere gases (less than 0.1 % of the amount of hydrogen) in the gas composition of the inlet system. In this case, oxygen, entering the chamber, effectively interacts with carbon-containing molecules on the surface of the internal ampoule elements (heated to high temperatures) and forms carbon dioxide molecules.

The pressures of gases with tritium-containing molecules change in experiments with the supply of hydrogen to the chamber:

- there is a decrease in the T_2 (M6) gas pressure and an increase in the HT (M4) gas pressure. It is connected with interaction of hydrogen with tritium on a ceramic surface, and it can be both, the exchange reaction

$$T + H_2 \rightarrow H + HT \tag{2}$$

and the desorption of the associated atoms of tritium and hydrogen atoms on a ceramic surface. In the latter case, the tritium produced under irradiation in the ceramic enters the sample surface as a result of transfer processes from the ceramic volume, while the hydrogen is sorbed by the ceramic surface from the gas phase;

- there is a slight increase in the pressure of gas with mass number 20, which may be tritiated water vapor HTO or one of the peaks of water H_2O (according to [14], about 0.2 % of the total peak of water is the peak with the mass number M20 (H_2O^{18})).The formation of HTO is apparently caused by the exchange reaction of normal water (H_2O) with tritium on the ceramic surface (when hydrogen is fed into the chamber there is an increase of pressure of normal water).

Determination the quasi-equilibrium release levels of gases with mass number 4 (HT and helium molecules) cased some difficulties. Due to the peak nature of the helium release and the small relative increase in the HT pressure over the time when hydrogen is supplied to the ampoule, the pressure increase of gases with mass number M4 has a sawtooth appearance and is determined with a significant error. For further analysis, the graph in Fig. 7 was constructed based on the following considerations: the total quasi-equilibrium tritium release remained unchanged when hydrogen was introduced into the chamber, that is, the sum of the pressures (HTO contribution was estimated to be less than 2% and was not taken into account):

$$2P_{T_2} + P_{HT} = K(constant) \tag{3}$$

and it is determined by the total tritium release before hydrogen was introduced. Then the HT gas pressure can be obtained as:

$$P_{HT} = K(constant) - 2P_{T_2}. \tag{4}$$

The flow of HT and T_2 molecules (proportional to their pressure) from the samples surface can be determined by the equations:

$$\varphi_{HT} = 2\hat{A}\cdot K_{HT}\hat{A}\cdot C_H\hat{A}\cdot\frac{C_T}{(C_H + C_T + C_{H_2})^2} + K_{1_{HT}}\bullet\frac{C_{H_2}\hat{A}\cdot C_T}{(C_H + C_T + C_{H_2})^2},$$

$$\boldsymbol{\varphi_{T_2} = K_{T_2}\hat{A}\cdot C_T^2/(C_H + C_T + C_{H_2})^2}, \tag{5}$$

where φ_{HT} and φ_{T_2} – flows of HT and T_2 gases into the chamber of the facility from the ceramic surface (mol/s); K_{HT} and K_{T_2} – desorption

constants of HT and T_2 molecules from the ceramic surface (mol/s); C_H, C_T and C_{H_2}– concentrations of hydrogen and tritium atoms, and H_2 molecule on the ceramic surface (mol/m^2).

Further, some estimates are made on the assumption that C_H, C_T. ≫ C_{H_2}. Then:

$$\varphi_{HT} = 2\hat{A}\cdot K_{HT}\hat{A}\cdot C_H\hat{A}\cdot\frac{C_T}{(C_H + C_T)^2},$$

$$\varphi_{T_2} = K_{T_2}\hat{A}\cdot\frac{C_T^2}{(C_H + C_T)^2}. \quad (6)$$

It is assumed that in the quasi-equilibrium state, the concentration of tritium atoms on the ceramic surface is constant and identical, and the hydrogen concentration depends on the hydrogen pressure level in the chamber. Then the following equation for the concentration of hydrogen atoms on the surface can be written:

$$\left(\frac{\varphi_{HT}}{\varphi_{T_2}}\right)_i = (C_H)_i\bullet\left(2\bullet\frac{K_{HT}}{K_{T_2}}\bullet C_T\right) \quad (7)$$

It should be mentioned, that the expression $\left(2\bullet\frac{K_{HT}}{K_{T_2}}\bullet C_T\right)$ is a constant. Then the dependence of hydrogen concentration at the surface on the hydrogen pressure in the facility chamber (see Fig. 8) was plotted.

According to calculations, the concentration of hydrogen atoms on the surface of ceramics (C_S (1/m^2)) depends on pressure to the power of 0.37 ($P_{H2}^{0.37}$), and the surface concentration of hydrogen atoms (C_V (1/m^3)) is proportional to pressure to the power of 0.56.

3.1. Results of experiments with deuterium injection

Experiments with deuterium injection were carried out according to the same scheme as the experiments with hydrogen injection. In total, two series of experiments with deuterium injection were carried out, the results of which were similar in the main patterns. A peculiarity of the experiments was that, due to the high deuterium pressures in the facility chamber, and the problem of insufficient resolution of the mass spectrometer, a more complex analysis of the mass spectra was necessary. First of all, we note that due to the low resolution of the peaks for gases with a mass number ~ 4 in the experiments we did not obtain information about changes in the partial pressure of HT gas: the pressure levels of deuterium fed into the chamber (D_2) were significantly higher than the HT gas pressure, and therefore the HT share was negligibly small and was not considered, and the partial pressure of gas with a mass number M4 was taken as the deuterium pressure.

As for the estimation of the tritium pressure (T_2) in the chamber of the unit, it was necessary to take into account the contributions to the peak with the mass number M6 both tritium pressure and pressure of the additional D_3^+ complex formed in the ion source of the RGA-100 mass spectrometer. As follows from [15] in the registration of deuterium using mass spectrometer, in addition to the main peak with a mass number of 4, a peak with a mass number of 6 is observed. This is associated with the fact that in the mass spectrometer ion source there are reactions of the second order, which yield D_3^+ ions. The source of D_3^+ is mainly the reaction:

$$D_2^+ + D_2 \rightarrow D_3^+ + D^0 \quad (8)$$

which has the largest cross section at the electron energy levels typical of the ionizing electron beam in the mass spectrometer. Assuming a second-order reaction, the signal from the mass number M6 (D_3^+) should follow a quadratic dependence on deuterium pressure. The experimental data obtained in [15] agree well with this theoretical law: the fitted curve gives a dependence of the form Pn at n = 1.9.

In the experiments, the dependence of the D_3^+ peak magnitude on the deuterium pressure was obtained on the basis of methodical experiments and is shown in Fig. 9. The function plotted from the experimental points had the form of (9):

$$P_{D_3} = (41.9 \pm 6.1)\bullet P_{D_2}^{(1.82\pm0.01)} \quad (9)$$

where P_{D_3} – value of M6 peak, caused by the formation of D_3^+ (torr); P_{D_2} – deuterium pressure in the chamber of the facility (torr).

As can be seen from the Fig. 9, the exponent of the power function is close to the quadratic dependence, as in [15].

Using equation (9) tritium pressures in the experiment were calculated according to equation:

$$P_{T_2}\ P_{M_6} - (41.9 \pm 6.1)\bullet P_{D_2}^{(1.82\pm0.01)} \quad (10)$$

where P_{T_2} – tritium (T_2) pressure in the facility chamber (torr); P_{M_6} – value of M6 peak (torr).

Data on changes in the values of partial pressures of gases in the chamber of the unit at various deuterium flows into the ampoule (including calculated data for tritium with the subtraction of the D_3^+ peak) are shown in Fig. 10.

As can be seen from the above data, when deuterium is fed into the ampoule:

a significant increase in the pressure of gases with a mass number of M4 is observed due to the growth of deuterium (D_2) pressure;

a decrease in the partial pressure of gas T_2 and an increase in the gas pressure DT take place. This is due to the interaction of deuterium with tritium on the surface of the lithium ceramic; moreover, it can be both,

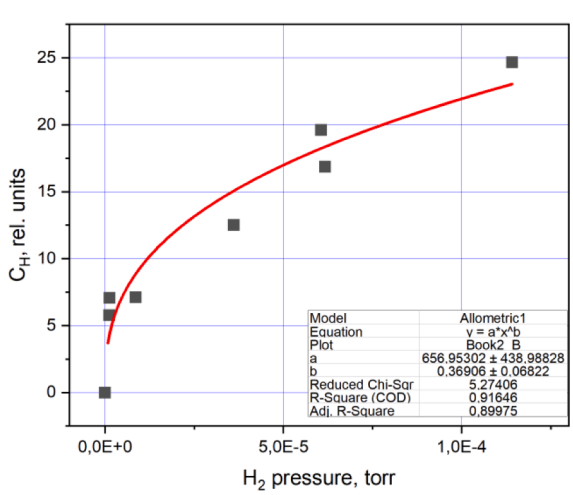

Fig. 8. Dependence of hydrogen atom concentration on the ceramic surface on hydrogen pressure in the chamber of the facility.

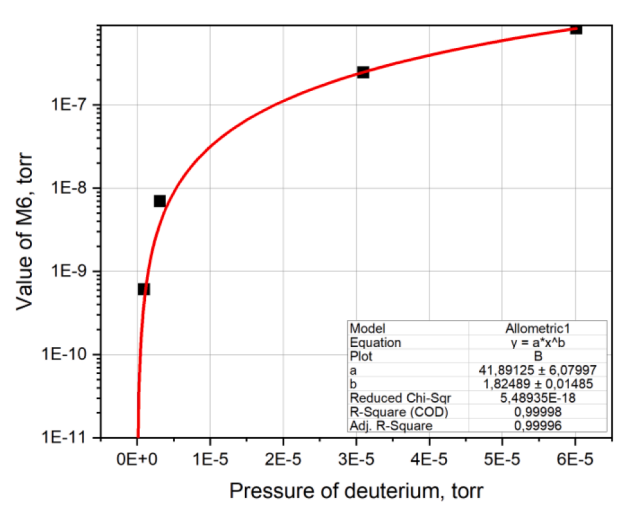

Fig. 9. Dependence of the D_3^+ peak value on deuterium pressure.

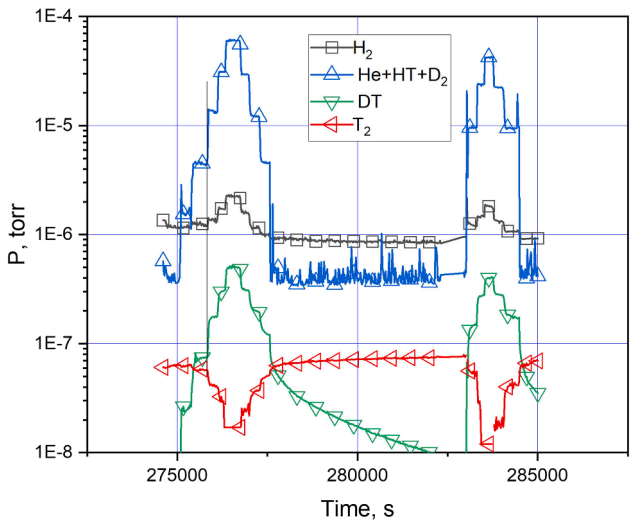

Fig. 10. Change of partial pressures of gases in the facility chamber at different deuterium flows into the ampoule.

the exchange reaction

$$T + D_2 \rightarrow D + DT \tag{11}$$

and the reaction of desorption of associated tritium and deuterium atoms on the ceramic surface. In this case, the tritium produced in the ceramic under irradiation enters the surface of the sample as a result of transfer processes from the volume of the ceramics, while the deuterium is sorbed by the ceramic surface from the gas phase. It is assumed that the processes on the sample surface during deuterium injection are similar to the processes occurring in experiments with hydrogen injection (see above);

the HT gas pressure, which is reliably registered before the injection of deuterium, cannot be determined, since the level of pressure of deuterium fed into the chamber is very high and the resolution of the mass spectrometer does not allow to register the HT and D_2 gas peaks separately;

the necessity to use a calibration curve to account the D_3^+ complex contribution to the mass M6 peak leads to significant errors in determining the T_2 pressure at high deuterium pressures in the facility chamber.

4. Discussion

After a nuclear reaction between a neutron and a lithium nucleus, high-energy tritium and helium atoms are formed in the sample, which begin to migrate in the sample through different channels. Due to a small diffusion coefficient helium atoms begin to accumulate in free microvolumes inside the ceramic, moving along cracks and are ejected at opening "pores" into the ampoule volume. So in the graphs we observe saw-tooth shapes for the curve of mass number M4. Tritium atoms, given their large diffusion coefficient at high temperatures, can easily penetrate along the grain boundaries to the open surface and begin to participate in reactions on the sample surface. At the surface they associate either with other tritium atoms or with hydrogen atoms and are desorbed in the form of T_2 or HT, or, in the presence of deuterium, in the form of DT.

As was shown above, the near-surface bulk concentration of hydrogen atoms on the ceramic surface is proportional to the pressure in the power of 0.5, which is close to Sievers' law, and indicates that the release of tritium occurs through interaction with hydrogen isotopes that dissociate to atoms on the ceramic surface after their sorption from the gas phase.

The release levels of HT and DT molecules at pressures larger than 10^{-5} torr were evaluated. At such pressures, the release occurs mainly as HT molecules in the presence of hydrogen, or DT molecules in the presence of deuterium.

Fig. 11 shows that the quasi-equilibrium partial pressure of DT molecule is higher than HT by 20 %, indicating a more intense interaction of deuterium with tritium on the ceramic surface than hydrogen with tritium. Therefore, in the processes of tritium yield from lithium ceramics, there is a significant isotope effect in the interaction with hydrogen and deuterium.

5. Conclusions

This paper presents the results of experiments with hydrogen and deuterium injection into an ampoule with lithium ceramic samples under reactor irradiation. The research methodology is described and the processing and analysis of the experimental data are carried out.

At hydrogen injection a decrease of the partial pressure of tritium T_2, and a growth of the HT pressure is observed, that is apparently connected with the interaction of hydrogen with tritium at the ceramic surface. It is assumed that the tritium, which is formed in the ceramic under irradiation, enters the surface of the sample as a result of transfer processes from the volume of the ceramic. Hydrogen is sorbed at the surface from the gas phase and subsequently T_2 and HT are desorbed as a result of surface reactions.

At the injection of deuterium similarly its sorption on the ceramic surface and the subsequent reactions of isotope exchange occur, as well as desorption of associated tritium atoms and deuterium atoms, with the deuterium and tritium forming a DT molecule. This becomes an additional channel for the release of tritium from lithium ceramic. When the pressure of deuterium in the gas phase increases, there is a preferential increase in the release of molecules containing tritium through this channel (as DT), which in turn leads to a decrease in the tritium release in the form of HT and T_2 molecules to almost complete termination.

According to the presented dependences of the release of DT (at deuterium injection) and HT molecules (at hydrogen injection), it is clear that the quasi-equilibrium partial pressure of DT molecule is higher than that of HT by about 20 %, indicating that the intensity of deuterium interaction with tritium on the ceramic surface is higher than that of hydrogen with tritium. Consequently, there is a significant isotopic effect in the processes of tritium release from lithium ceramic during the interaction with hydrogen and deuterium.

To understand the mechanisms of these processes, further analysis and additional calculations are needed to confirm the above assumptions.

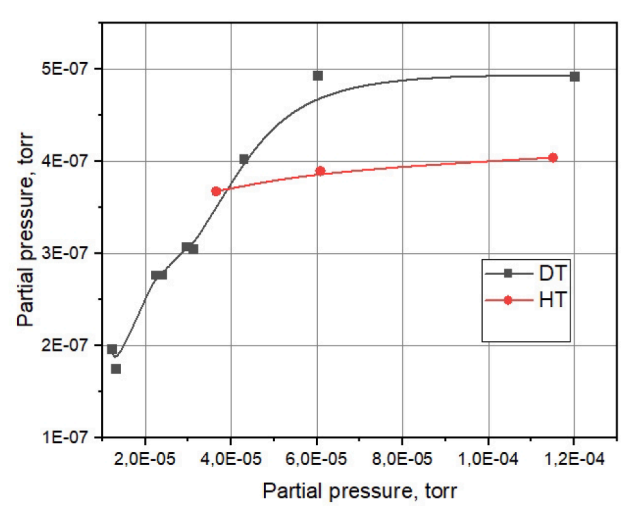

Fig. 11. Dependences of the release of molecules DT (at deuterium injection) and HT (at hydrogen injection).

CRediT authorship contribution statement

Timur Kulsartov: Conceptualization, Methodology, Validation. **Yergazy Kenzhin:** Writing – original draft, Investigation. **Regina Knitter:** Supervision. **Gunta Kizane:** Supervision. **Yevgen Chikhray:** Investigation, Software. **Asset Shaimerdenov:** Investigation. **Saulet Askerbekov:** Investigation. **Assyl Akhanov:** Investigation. **Inesh Kenzhina:** Investigation. **Zhanna Zaurbekova:** Writing – review & editing. **Arturs Zarins:** Data curation. **Darkhan Sairanbayev:** Investigation. **Yuriy Gordienko:** Investigation, Data curation. **Yuriy Ponkratov:** Investigation, Data curation.

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.

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