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Analysis of the reactor experiments results on the study of gas evolution from two-phase Li_2TiO_3-Li_4SiO_4 lithium ceramics

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ABSTRACT

This paper analyzes part of the reactor experiments on the study of tritium and helium release from promising two-phase lithium ceramic (Li_2TiO_3 and Li_4SiO_4) of natural lithium enrichment conducted by vacuum extraction.

The basis for such an analysis was a more careful study of the time trend of pressure changes of gases in the chamber with the test samples. In a particular case, it was clearly shown that the pressure fluctuations observed during irradiation for gases with mass number M4 (to which both HT and He molecules correspond) are determined only by He, which leaves the intergranular regions of the ceramic through open channels or cracks. The kinetics of changes in the amount of helium that is released during irradiation was traced and both the rate of helium release and the frequency of emissions were determined. It was assumed that the observed emissions correspond to a certain "formation of free paths" from the internal cavities of the irradiated ceramics into the chamber of the facility. The data obtained for the helium emissions were compared with the release of tritium-containing molecules from the ceramics. The quasi-equilibrium levels of the release of tritium-containing molecules and their dependence on the reactor power were estimated. The release of helium and tritium was compared with the calculated values of the tritium generation rate in the test sample.

1. Introduction

During the operation of a D-T fusion reactor, energetic tritium is generated by $^6Li(n, \alpha)T$ and $^7Li(n, n \alpha)T$ reactions in tritium-producing materials and stored in their crystalline grains. Lithium ceramic-based pebbles are the main functional material of solid blankets.

For high tritium production values, these ceramic materials must contain large amounts of lithium-6. Moreover, the pebbles must release the generated tritium quickly enough and must not degrade significantly during operation. Since the pebbles will experience significant mechanical stresses (that arise during thermal expansion and cycling), their strength is an important parameter. In addition, factors such as sufficient lithium content and low activation properties of the ceramic material are important.

Numerous lithium ceramics have been proposed and studied as blanket breeder material [1,2]. The properties shown by the candidate materials are the subject of intense research, since they are crucial in the realization of conceptual designs of solid breeder blankets.

Lithium metatitanate (Li_2TiO_3) and lithium orthosilicate (Li_4SiO_4) are considered as candidates for solid breeder blankets of future fusion reactors [3]. These compounds exhibit low activation compared to lithium zirconate (Li_2ZrO_3) and lithium aluminate ($LiAlO_2$) and show satisfying thermomechanical and thermochemical properties [4–7].

Generation of tritium in Li_4SiO_4 is higher than in Li_2TiO_3 because of the higher density of lithium atoms, but data on tritium release for Li_4SiO_4 are very different [8–10]. The concept of a two-phase mixture of

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Li_2TiO_3 and Li_4SiO_4 was proposed several years ago in order to realize the advantageous complementarity of materials [11–13].

Most experiments on tritium release from ceramics are conducted according to the following scheme: irradiation of materials in nuclear reactors is organized and then the samples are studied separately in post-irradiation examinations (PIE). Such experiments are relatively simple, but their disadvantage is the complicated interpretation and not sufficiently accurate and qualitative prediction concerning the real parameters of tritium release. This is because the parameters of tritium release are influenced by the irradiation conditions. The mechanisms of tritium migration in solid tritium-reproducing materials are complex: tritium must undergo several elementary stages, such as diffusion in a crystal grain, capture/release in radiation traps, association/dissociation with compound atoms, and adsorption/desorption on the material surface.

Many studies have been conducted to estimate the rate that determines the overall process of tritium release from solid materials [14–16]. These studies have shown that diffusion is the process that generally determines the tritium release rate from solid breeder materials [17,18]. It turned out that the tritium diffusion coefficients differ greatly [19,20], which may be due to differences in samples size and state , as well as the conditions of the tritium extraction experiments. The latter include capture of tritium by radiation traps and isotope exchange reactions on the surface.

Since the generated tritium and helium atoms have high energies (about 2–3 MeV), radiation damage will be created in tritium-producing materials by recoil particles and act as tritium capture centers. In [21] the dependence of tritium diffusion mobility in Li_4SiO_4 on the neutron fluence was obtained, which showed that tritium mobility decreases with increasing fluence. In [22] the assumption was made that the tritium capture centers are oxygen vacancies and free oxygen atoms in Li_2TiO_3. Consequently, the effects of tritium capture by radiation traps complicate the interpretation of experimental results.

Therefore, to obtain more complete data, it is necessary to conduct studies of tritium release directly in the process of neutron irradiation. Such so-called in-situ experiments are more complicated methodologically and therefore their number is not very large. It should be said that in-situ experiments have been conducted for all of the candidate fusion blanket materials. At present, there are data on almost all single-phase lithium ceramics obtained during in-situ experiments.

As for the new promising two-phase ceramic (Li_2TiO_3-Li_4SiO_4), it has not yet been studied under irradiation conditions. There is only one work [23] in which samples of two-phase lithium ceramics were irradiated in a nuclear research reactor and thermodesorption experiments were conducted after the irradiation. Since this experiment was not performed directly under irradiation conditions, such a study provides only qualitative information about the processes of tritium generation and release. For example, such an experiment does not provide key information on the parameters of changes in tritium release during irradiation. Besides, the irradiation was carried out at insignificant neutron fluxes ($2.75 \cdot 10^{13}$ n/(cm^2•s)) and total fluence less than $1 \cdot 10^{17}$ n/(cm^2•s), which is not comparable with the actually expected fluences during operation of such ceramics.

2. Materials and experimental method

In the described experiments, the samples of two-phase lithium ceramics of natural lithium enrichment were investigated. The samples were pebbles with a diameter of 250–1250 μm with a total weight of 5 g, consisting of 75 mol% Li_4SiO_4 and 25 mol% of Li_2TiO_3. The density of the ceramic is 95 % with about one half of the porosity being open and closed, respectively.

The experiments were conducted at the WWR-K reactor located at the Institute of Nuclear Physics in Almaty, Kazakhstan. The CIRRA (Complex of In-Reactor Release Analysis) (Fig. 1), which is a facility of

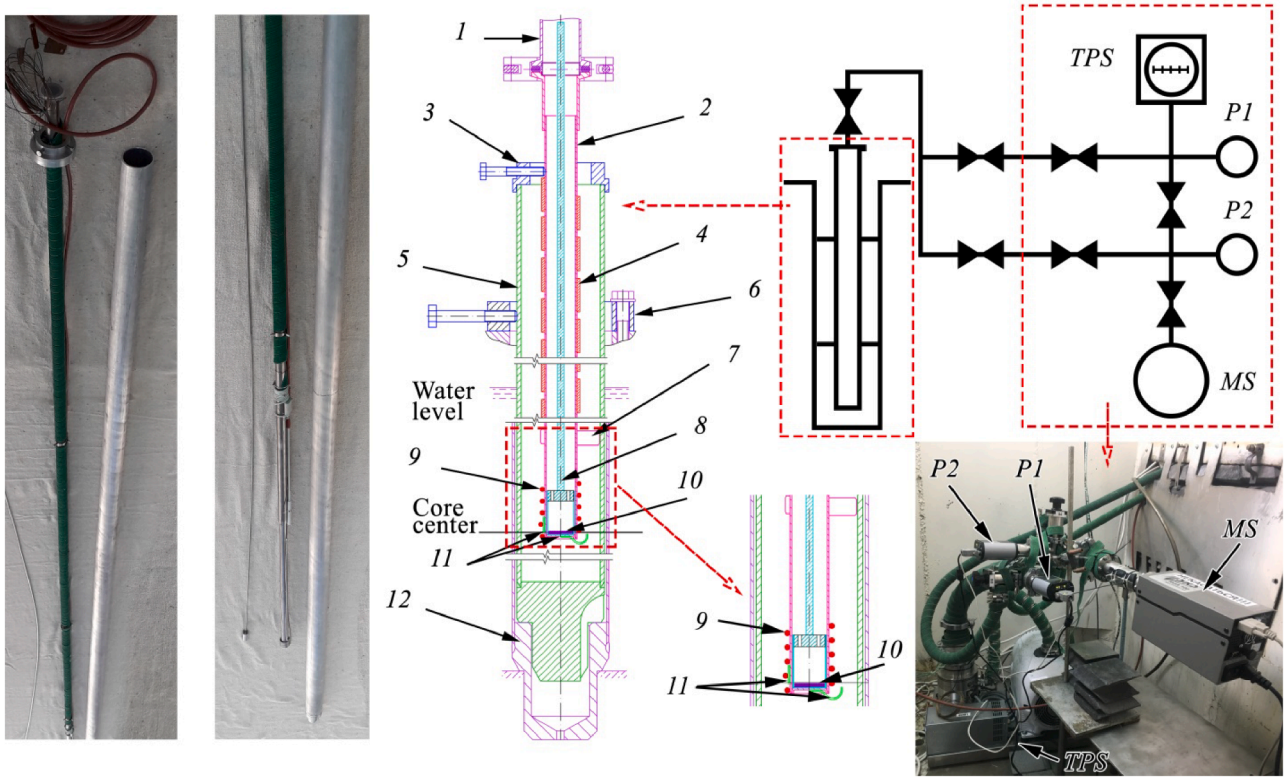


Fig. 1. The experimental facility and ampoule device with samples [24]: 1 – connection to the working unit; 2 – ampoule device; 3 – upper centring flange; 4 – tape heater; 5 – reactor channel; 6 – support; 7 – positioning holder; 8 – capsule; 9 – thermocouple heater; 10 – samples; 11 – thermocouples; 12 – reactor channel displacer; TPS – pumping system; P1, P2 – pressure sensors; MS – mass-spectrometer.

gas-vacuum paths connecting an experimental device located in one of the cells of the WWR-K reactor core to the analytical equipment, was used for the studies. Vacuum extraction method is implemented at the facility. Constant evacuation of the experimental device is carried out by a pumping station with a turbomolecular pump. Gas analysis is performed by mass spectrometric method using RGA-100 residual gas analyzer.

The experimental device consisted of an outer casing made of aluminum alloy and an inner ampoule made of stainless steel. A container with samples was immersed in the ampoule, which was positioned so that the center of the backfill corresponded to the middle of the core height, and then it was connected to the vacuum paths of the unit. A description of the facility is given in [24–27].

The main parameters of the irradiation experiment are given below:
Neutron flux in the irradiation cell ~ $5 \cdot 10^{13}$ n/($cm^2 \cdot s$);
Thermal neutron fluence ~ $1.8 \cdot 10^{19}$ n/cm^2;
Temperature of samples under irradiation ~ 680 °C;
Pressure of residual gases in the irradiation cell ~ $7.5 \cdot 10^{-2}$ Torr;
Irradiation time ~ 4.3 days.

Relative error in the determination of partial pressures using an RGA-100 mass spectrometer is ≤ 10 %.

Neutron-physical calculations were carried out and tritium generation rates in ceramics were estimated. The contributions to the rate of tritium generation of the reactions (1) and (2)

$$6Li + n \rightarrow T + {}^4He + 4.78\,MeV \quad (1)$$

$$7Li + n \rightarrow T + 4He + n' - 2.47\,MeV \quad (2)$$

were also evaluated. The amount of tritium generated during the entire experiment is 1.86 Curies from the reaction (1) and $2.67 \cdot 10^{-4}$ Curies from the reaction (2). The expected tritium generation rate at 6 MW reactor power is ~ $3 \cdot 10^{13}$ $s^{-1} \cdot m^{-3}$.

In the reactor experiment both the release of tritium and helium from the samples under constant experimental conditions (temperature, gas environment), and under changing experimental conditions (changes in the temperature or hydrogen and deuterium supply into the chamber) were studied. In the present work, the sections where no additional effects on the samples were performed other than irradiation will be investigated and analyzed.

3. Experimental results

The results of the reactor experiments are shown in Fig. 2. The shaded sections were not analyzed in the paper and will be presented in separate papers [28,29]. At the beginning, it should be noted that the RGA-100 mass spectrometer used in the experiment did not allow determining the pressure of the HT (P_{HT}) and He (P_{He}) molecules separately (due to low resolution), but only their total pressure ($P_{He} + P_{HT}$).

When interpreting the obtained results of gas composition changes in the chamber during irradiation, it was suggested that the release of the M4 peak can be decomposed into two components. One, which is responsible for the release of the HT molecule, is the lower level of the M4 release curve. And the second one, which is responsible for the variable component, refers to the abrupt release of helium from the inner pores of the ceramic (when the free escape pathways to the pebble surface are formed) as shown in Fig. 2. These helium release pulses have the same shape (Fig. 3): a sharp leading edge (1–2 s long), typical for the rapid process of bursting the accumulated helium from the inner cavity of the sample, and an exponentially decaying back edge, typical for the pumping out of the bursting helium by a vacuum pump. The pulse amplitude is determined both by the volume of the internal cavity, in which the gas accumulated, and by its limiting pressure, which provided the breakthrough. Most pulses of low amplitude apparently correspond to the gas release from semi-open pores and internal voids of the sample, the exit from which does not require much effort. Nevertheless, peaks with an amplitude almost three orders of magnitude higher than the average are also recorded, but they are literally singular.

Here it is also worth noting that the peak releases are characteristic only for the gas with mass M4, and are not observed for the other masses. The possibility of peak values for the gas emission curves with a mass number M4 as a result of mass spectrometer unstable operation was excluded in methodical experiments on irradiation of ceramics using two mass spectrometers of different types. In addition, the analysis of the section of the experiment with reactor power decrease showed that the dependence of the total tritium release under this assumption is proportional to the reactor power.

As can be seen from Fig. 2, the release of tritium-containing molecules is uniform, except for the sections where the irradiation conditions changed markedly. Despite the peculiarities associated with marked changes in the tritium fluxes into the experimental chamber at the sections with changes in the irradiation conditions, when returning to the normal irradiation conditions, the fluxes of the emitted gases also return to some trend values.

The main amount of tritium is released as HT molecule during the entire irradiation, but the flux of HT decreases with time due to the fact that the release of the T_2 molecule increases. The observed decrease in the release of the HT molecule during the experiment seems to be related

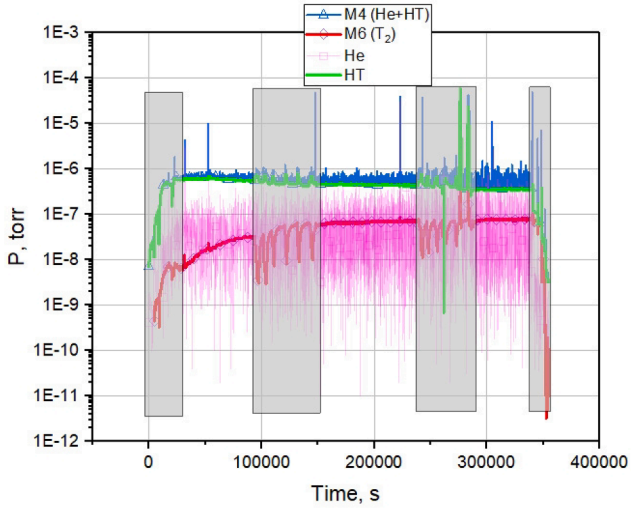


Fig. 2. Gas release of He and T containing species in irradiation experiment.

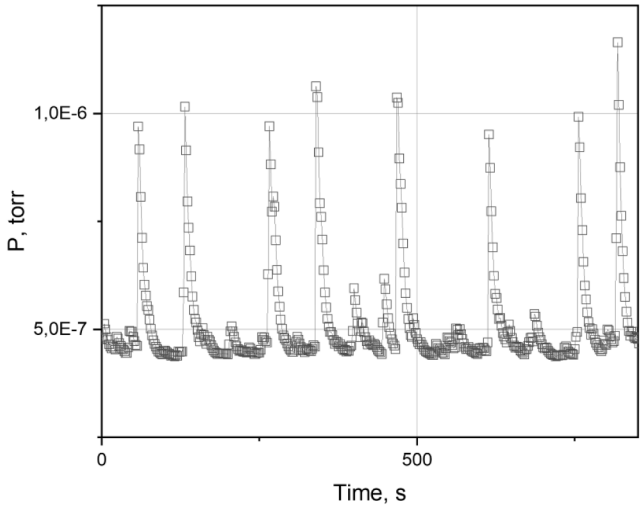


Fig. 3. Pulses of gas release peaks with mass number M4.

to a decrease in the hydrogen concentration in the test sample and a decrease in the hydrogen pressure in the facility chamber due to its gradual annealing. Authors believe that the HT molecule is formed as a result of the association of hydrogen and tritium atoms on the ceramic surface (with its subsequent desorption into the experimental chamber).

It should be noted that the release of tritium in the form of tritiated water is insignificant (less than 1% of the total tritium release) and may not be taken into account.

4. Discussion

Helium and tritium are produced in ceramics as a result of the lithium-6 atom interaction with a neutron. Then tritium diffuses in the volume until it reaches the region communicating with the ceramic surface. After that, it is associated either with a tritium atom or with a hydrogen atom on the ceramic surface and desorbed into the evacuated chamber of the facility. Tritium can most likely interact reversibly with traps. It is assumed, that there is no noticeable accumulation of tritium-containing gases in the inner pores of the ceramics, since tritium, unlike helium, has the ability to dissolve back into the bulk of the ceramics and leave it.

As for helium, it practically does not diffuse in the bulk of the ceramics; however, it can enter the inner pores of the ceramics during the reaction of lithium-6 with a neutron in the regions close to these pores.

Fig. 4a shows a SEM image of the ceramic spherical particles. A detailed analysis of the ceramic surface morphology (Fig. 4b) revealed that the ceramic structure is an ultrafine-grained structure of sintered grains, the size of which is no more than 5 nm, forming large clusters of rhomboid or dendritic shape. In this case, the structure clearly shows tiny pores, microcracks, the size of which does not exceed 30–50 nm, and macrocracks with a branched structure, the size of which exceeds 500 nm. Such defects indicate the presence of a porous structure, which can play an essential role in the accumulation and subsequent migration of helium, which forms in the course of nuclear reactions during the interaction of neutrons with lithium. It is also worth noting that such refined grains that make up the ceramics structure lead to a high density of dislocations (10^{11}-10^{12} dislocations/cm^2), which also plays an essential role in the migration of helium.

Fig. 4c also shows a side chip of the test sample, according to which the internal structure can be assessed. As can be seen from the presented data, there are cavities in the ceramic structure due to the technological process of production, and the grains themselves have a fairly strong bond with each other. In this case, the presence of impurities in the structure was not detected.

Several factors can cause the non-equilibrium mechanism of helium yield during irradiation. First, the presence of a high density of dislocations in ceramics, in contrast to steels and alloys, leads to the formation of additional barriers for accumulating the formed helium and its subsequent migration. In this case, helium can accumulate near the grain boundaries, occupying cavities and pores, while the rate of its migration is much lower due to a large number of defects and dislocations. Secondly, visible microcracks and pores can explain how the accumulated helium can escape through these structural defects. This process does not occur constantly but periodically, associated with the morphological features of the ceramics structure. As a result of irradiation, the ceramics accumulate point defects, which arise due to irradiation and nuclear reactions, producing helium. In the case of high-temperature irradiation, some of these defects are known to be annealed. Accumulation of structural deformations and distortions dominates defects annealing with irradiation time, leading to ceramics disordering and partial swelling. An increase in the irradiation time, and, consequently, the degree of structure disordering and deformation, can lead to the appearance of additional cavities, which could be filled with the formed helium. At the same time, an increase in the degree of distortion and deformation of the structure due to the accumulation of radiation damages during prolonged irradiation leads to the appearance of non-equilibrium regions with a highly disordered structure. Those regions can form pores or cracks, contributing to a sharp increase in the yield of helium located nearby around (diagram in Fig. 5). In this case, an increase in helium concentration in the cavities increases the stress and pressure in it, which, together with the non-equilibrium structure of the defect regions, can lead to a quick ejection of helium from the cavities. So, more cavities undergo cracking, contributing to helium yield as the degree of non-equilibrium of the structure becomes higher due to prolonged irradiation.

It should be noted that the amplitude of the helium released pulses is determined both by the volume of the internal cavity with accumulated helium and its limiting pressure, which provided the breakthrough. Most of the pulses observed in the experiment correspond to the exits of gas from the half-open pores and internal voids of the sample, which does not require significant effort.

The proposed qualitative description of the helium release processes should be considered preliminary and require a thorough study and justification.

Since ceramic consists of two different phases - Li_4SiO_4 and Li_2TiO_3 the processes of tritium and helium release in each crystalline phase at first should be evaluated and described separately.

In the future, it is planned to create computational models for the helium release based on a series of reactor experiments and Post Irradiation Experiments (PIE). Another mechanism of the observed helium release can be the formation (and subsequent opening) of closed cavities between pebbles formed as a result of their sintering during irradiation.

Fig. 6 shows the analyzed helium release segments from the ceramics. As can be seen and deduced from the estimated averaged helium fluxes from the sample, for each of the segments, the magnitude of the peak emissions increases with the time of irradiation. In addition, the frequency of extremely large helium emission peaks increases with irradiation time. The contribution of peaks of different sizes to the total gas emission is shown in Fig. 7

It is assumed that the sections of the experiment in which the

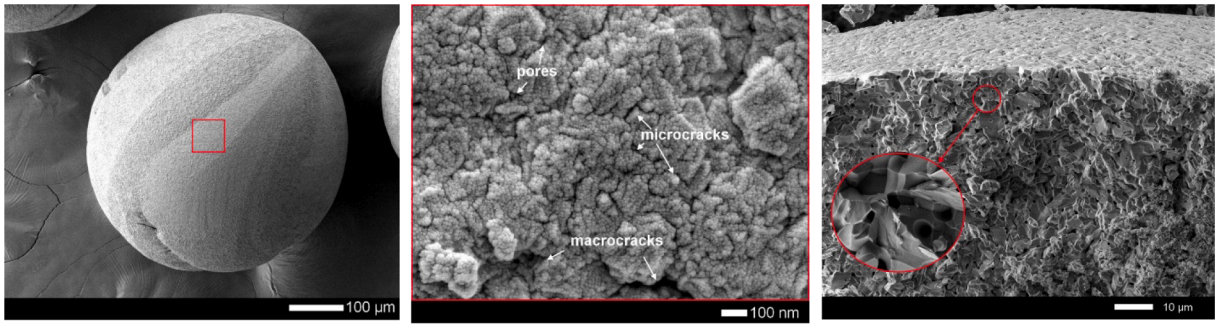


Fig. 4. Results of SEM: a) appearance of the ceramic pebble; b) detail of pebble's surface morphology; c) site chip of the ceramic pebble.

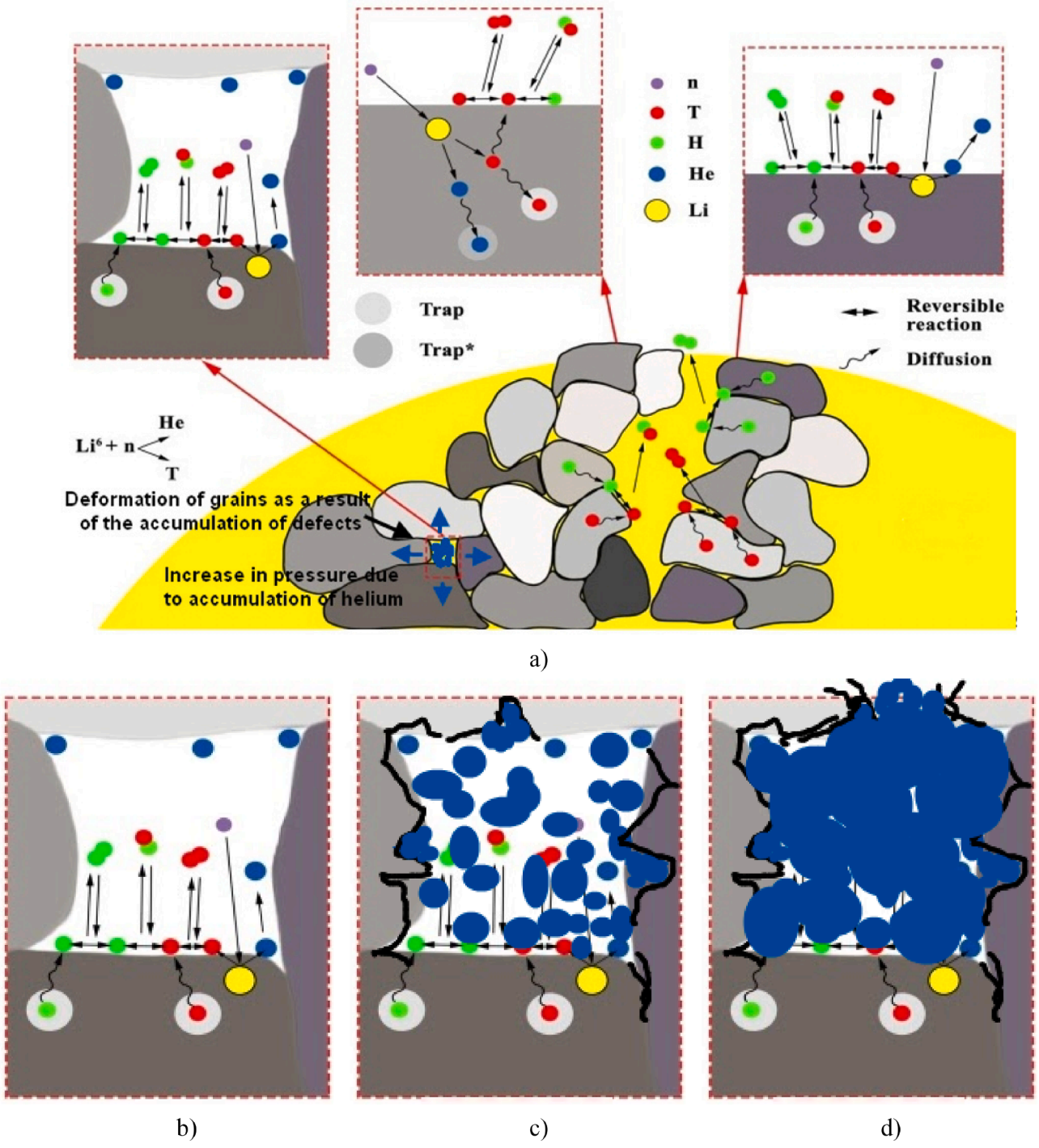


Fig. 5. a) Schematic representation of the mechanisms of formation of helium filled pores; b) The beginning of helium accumulation in the pore; c) Deformation of the pore as a result of an increase in the concentration of helium, as well as defects in the structure; d) Pore rupture due to increased pressure and distortion in the structure.

temperature and composition of the gas phase in the chamber with ceramics changed did not lead to significant qualitative microstructural changes. This is confirmed by the analysis of the dependences of gas evolution shown in Fig. 2. It can be seen that, when active experiments are stopped, they return to some general tracing values.

The rate of the process of free paths formation was estimated (Fig. 8). It was assumed that at a constant rate of opening of initially closed pores into the area of open porosity, the total release of helium should be linearly proportional to the irradiation time. However, despite the increase in helium gas evolution with the exposure time, the expected "proportional increase" is not observed: the expected helium evolution turns out to be less. Moreover, with the time of exposure, such a deviation from the linear dependence becomes stronger. The reasons for this behavior apparently lie in the change in the mechanism of the formation of free paths for the release of helium from the internal volumes of the ceramic to its surface. For example, this may be due to a decrease in the number of such volumes in areas near open pores.

The relative rate of the formation of free paths was estimated as follows:

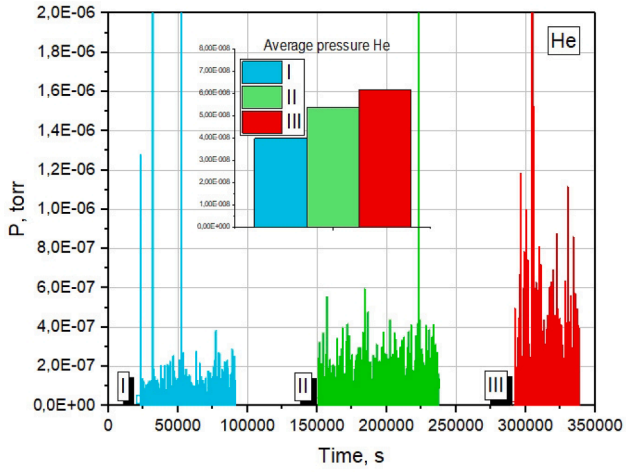


Fig. 6. Dependences of helium release from ceramics in the sections of the reactor experiment without changes in irradiation conditions.

$$v_i = k\hat{A}\cdot S_i/t_i$$

where v_i – the relative rate of the process of free paths formation in the section i (i = I,II,III), s^{-1}; k – normalization factor, which was selected for section I, so that $v_I = 1(mol^{-1})$;S_i – the average amount of helium released in the section i, mol; t_i – average time of section i, s.

Next, the section of the experiment was analyzed with a stepwise decrease in the reactor power (see Fig. 9). It turned out that the total release of tritium is proportional to the power (see Fig. 10). Moreover, a noticeable change in the composition of released tritium-containing molecules is observed: the share in the release of tritium in the form of T_2 molecules increases with a decrease in power and becomes larger than the share of HT molecules at a power of 2 MW. This effect is caused by a decrease in the hydrogen pressure in the sample chamber with a decrease in power.

The construction of such dependences for the assumption that there is a helium contribution to the lower level of the M4 curve (more than 10%) does not lead to a linear dependence; moreover, it becomes difficult to explain the ratio of M6 and M4 partial pressures with a drop in power.

Comparison of experimental data on the release of tritium (taking into account the release of tritium in the form of a T_2 and HT molecules, where HT is the lower level of the M4 curve) showed that the level of tritium release coincides with the neutron-physical calculation within 30 % (in the approximation of a homogeneous medium and without self-shielding effect).

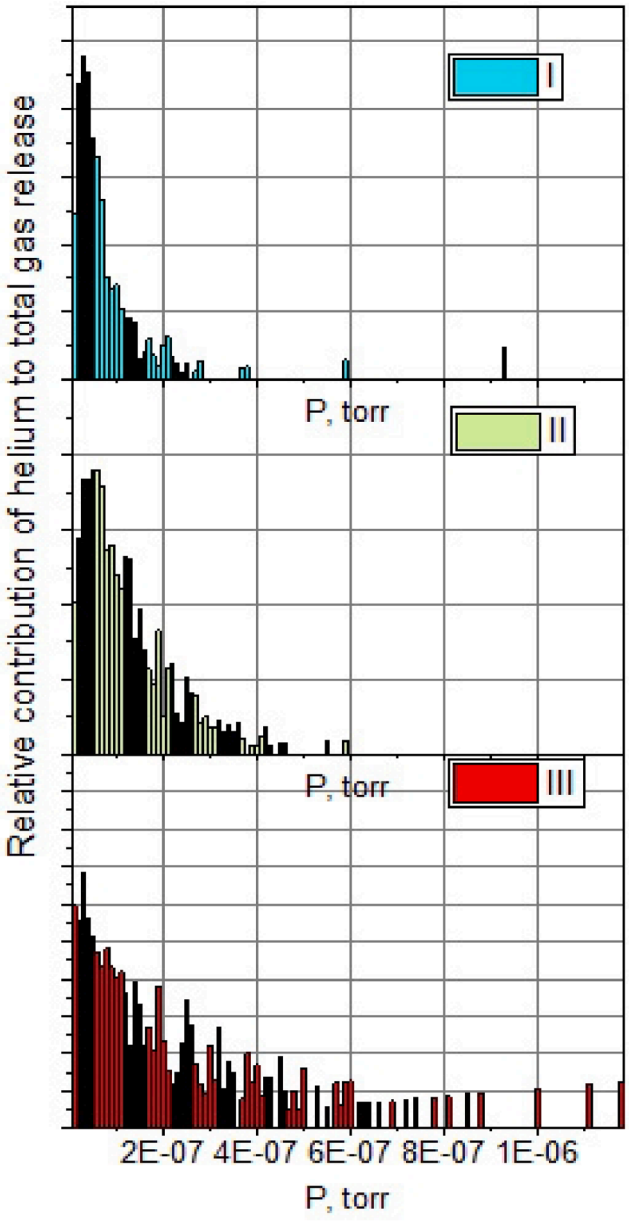


Fig. 7. Dependence of the relative contribution of the helium release on the value of peak.

5. Conclusion

Analysis of the quasi-equilibrium sections of the reactor experiment to study the release of tritium and helium from two-phase lithium ceramics showed that:

1) The release of tritium occurs mainly in the form of molecules HT and T_2 (and is determined by the sum of HT + 2 T_2). This value practically does not change during the investigated sections of the experiments and, in general, coincides with the calculated values on tritium release in irradiated ceramics.
2) The release of helium has a peak character and is apparently associated with the processes of its accumulation in the "pores" of the ceramics and the subsequent appearance of free escape routes from these "pores" to the ceramics surface.
3) The total release of helium increases with the exposure time due to an increase in the amplitude of the helium release peaks, and there is no noticeable increase in the frequency of the peak release with the time of exposure.
4) Sometimes single helium peaks exceeding the average values of the peaks by hundreds of times are observed.
5) Qualitative analysis indicates that with time of irradiation there is a slowing down of the process of formation of free paths from the "pores" of ceramics to its surface.

The process of formation of free pathways from the "pores" of the ceramics to its surface, can significantly affect the tritium release rate (for example, at lower temperatures) and is generally a positive and essential factor arising from neutron irradiation of the ceramics.

CRediT authorship contribution statement

Inesh Kenzhina: Investigation. **Timur Kulsartov:** Writing – original draft, Conceptualization, Methodology. **Regina Knitter:** Supervision. **Yevgen Chikhray:** Investigation, Software. **Yergazy Kenzhin:**

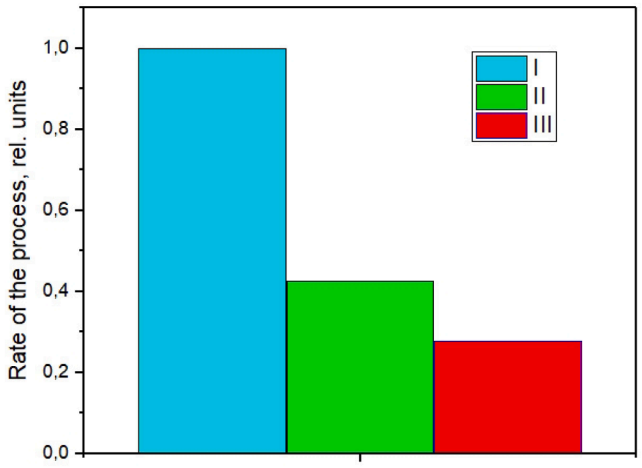


Fig. 8. Rate of formation of free paths from pores to the ceramic surface.

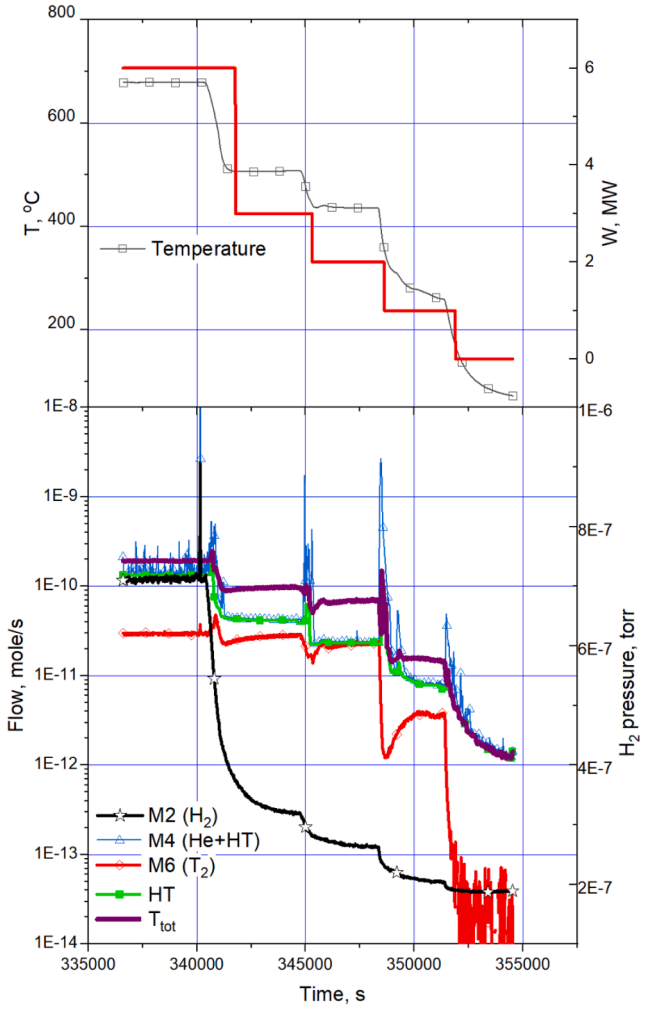


Fig. 9. Results of experiments at the end of irradiation with a stepwise decrease of the reactor power.

Supervision. **Zhanna Zaurbekova:** Validation, Investigation. **Asset Shaimerdenov:** Investigation. **Gunta Kizane:** Supervision. **Arturs Zarins:** Investigation. **Artem Kozlovskiy:** Investigation. **Maratbek Gabdullin:** Investigation, Data curation. **Aktolkyn Tolenova:** Investigation, Data curation. **Evgeny Nesterov:** Investigation.

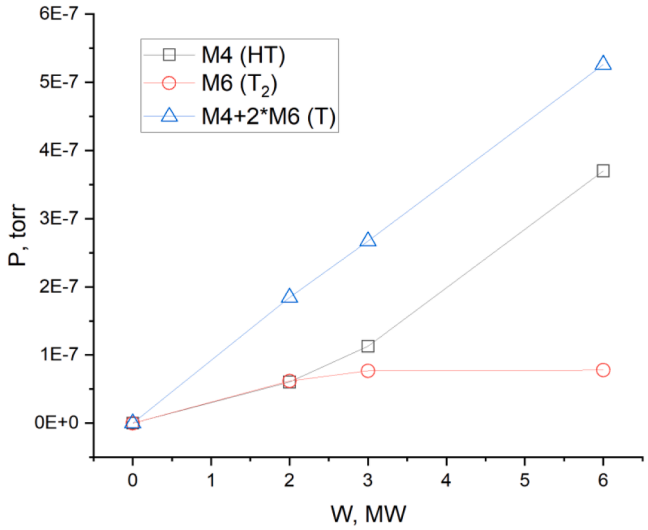


Fig. 10. Tritium release from the sample at different reactor power levels (assuming HT is the lower level of the M4 curve).

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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