

Thermal desorption of tritium and helium from lithium ceramics $\text{Li}_2\text{TiO}_3 + 5\text{mol}\% \text{TiO}_2$ after neutron irradiation

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ABSTRACT

Lithium-based ceramics exhibit good thermophysical properties, have low activation and chemical activity, and also release tritium well. These characteristics make lithium ceramics the best candidates for use as a functional material for a solid breeder blanket. To date, the most accurate understanding of the processes of tritium and helium production and release occurring in the breeder blanket materials can only be obtained from the results of reactor experiments. On the other hand, many important parameters can only be estimated in post-irradiation experiments (PIE). This paper describes studies of tritium and helium release in post-irradiation experiments (PIE) on the thermal desorption from ceramic pebbles $\text{Li}_2\text{TiO}_3 + 5\text{mol}\% \text{TiO}_2$ (with 96% enrichment on lithium-6 isotope), irradiated in the WWR-K reactor for 223 days. In PIE a peak of helium release was recorded for each sample in the temperature range of 1300–1500 K. An assumption was made in the paper to explain the nature of this helium release peaks from lithium ceramic pebbles.

1. Introduction

Breeder blanket is a key element of a future fusion reactor, as it breeds the fusion fuel tritium and extracts the heat from the reactor for power generation. The development of breeder blanket design requires a constant search for improved materials that can withstand heavy loads. Research on the breeder blanket materials is being actively carried out around the world, and it is planned that several concepts of breeder blankets will be tested as test modules at the ITER reactor [1].

Lithium-based ceramics exhibit good thermophysical properties, have low activation and chemical activity [2], and also release tritium well [3–5]. These characteristics make lithium ceramics the best candidates for use as a functional material for a solid breeder blanket [6–10]. The most promising form of manufacturing ceramic elements of a solid breeder blanket are pebbles - granules with a size of up to several millimeters, which provides resistance to mechanical and thermal stress.

The spherical geometry also ensures efficient heat removal from the pebble surface and removal of tritium by the carrier gas. The small size of the pebbles allows to easily fill the free space intended for the breeder material in the blanket, and also facilitates handling procedures.

To date, the most accurate understanding of the processes of tritium and helium production and release occurring in the breeder blanket materials can only be obtained from the results of reactor experiments.

The process of tritium generation and release is complex, which is determined both by the parameters of its diffusion in the material, and by the processes of defect formation in ceramics during irradiation, changes in the structure of the material as a result of irradiation, etc. [11–14].

Previously lithium ceramics were irradiated both in the in-situ mode with simultaneous measurement of the levels of evolved gases [15–19] and in the passive mode. In-pile reactor experiments make it possible to evaluate changes in the levels of gas release from samples depending on

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temperature.

On the other hand, many important parameters, for example, the end-of-irradiation tritium inventory formed in ceramics as a result of neutron irradiation, can only be estimated in post-irradiation experiments (PIE). Thus, the authors of [20–23] studied how the accumulated tritium comes out of ceramics by the TDS method and evaluated the effect of various impurities in the carrier gas on tritium release. A feature of these studies is that TDS was carried out under conditions of helium blowing, thereby missing the opportunity to analyze the accumulation and release of this gas in ceramics. Also in the experiments, ionization chambers are used, which can register only the released tritium.

This paper describes studies of tritium and helium release in post-irradiation experiments (PIE) on the thermal desorption from ceramic pebbles $\text{Li}_2\text{TiO}_3 + 5\text{mol}\% \text{TiO}_2$ (with 96% enrichment on lithium-6 isotope), irradiated in the WWR-K reactor for 223 days. In PIE a peak of helium release was recorded for each sample in the temperature range of 1300–1500 K. An assumption was made in the paper to explain the nature of this helium release peaks from lithium ceramic pebbles.

2. Materials and method

Lithium metatitanate pebbles enriched up to 96% in the ^6Li isotope with 5% addition of TiO_2 were used as the test sample. The enrichment of Li_2TiO_3 with respect to ^6Li was carried out in order to increase the efficiency of tritium generation. Pebbles were obtained by the sol-gel technology [24].

The experiment on the irradiation was carried out at the WWR-K research reactor of the Institute of Nuclear Physics for 223 days (5350 h) at a power level of 6 MW. In this case, a very high (up to 22%) burnup of the ^6Li isotope was achieved. A total of 15 campaigns were conducted, the duration of each campaign was from 9 to 23 days [25, 26]. Ampoules with ceramic samples were constantly located in the reactor core, including the time between campaigns. The energy spectrum of neutrons in the reactor core when ampoules with ceramic samples are placed in the experimental channel is shown in Fig. 1.

Two types of ampoule devices were irradiated in the experiments: active and passive, respectively. In the active ampoule, the release of tritium from the samples was measured in the “in situ” mode. The results of experiments with the active ampoule have previously been published in [27]. The passive ampoule with samples was sealed during irradiation.

In this paper, the authors analyze the release of tritium and helium from lithium ceramic samples irradiated in the passive ampoule device

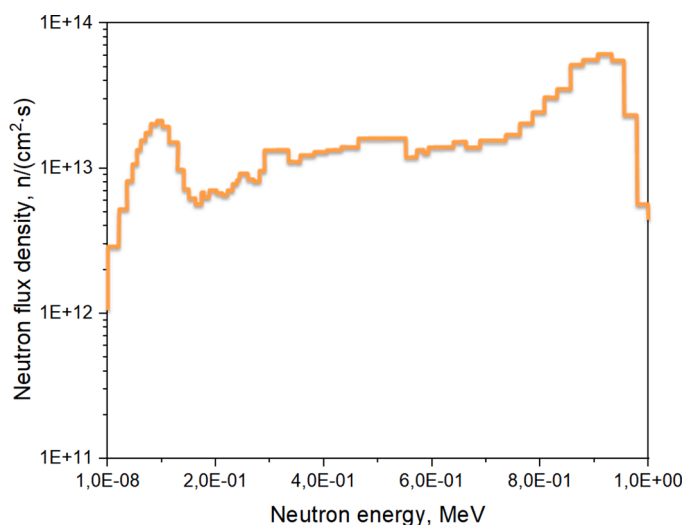


Fig. 1. Neutron energy spectrum in the WWR-K reactor core (taking into account the irradiation ampoule devices with lithium ceramics).

(P2 in Fig. 2). The temperature of the samples during irradiation was ~ 870 K. In total, gas evolution from 7 irradiated pebbles was studied (some of which are shown in Fig. 3). The diameter of the studied pebbles was about 1 mm; the mass was in the range from 0.0014 to 0.0017 g.

The study of tritium and helium release from ceramic samples was performed by the method of thermal desorption spectrometry (TDS). The experimental procedure was as follows: the test sample was loaded on a tantalum crucible into the vacuum chamber (Fig. 4) of the facility (Fig. 5) [28], and then it was evacuated to a vacuum of about 10^{-7} Pa. After that the vacuum chamber was degassed at a temperature of 373 K for 3 h. During TDS experiment the sample was linearly (at a given rate) heated up to 1873 K. Simultaneously, changes in the partial pressures of hydrogen isotopes in a continuously evacuated chamber were recorded. The heating rate of the samples was from 3 to 6 K/min; the residual pressure in the measuring path of the facility was no more than 10^{-5} Pa. The calculated estimation of the pebble temperature gradient under conditions of linear heating in vacuum was up to 20 K at temperatures above 1373 K (at a heating rate of 3 K/min). Moreover, the deviation from the linear rate of change of the pebble average temperature was less than 5%.

3. Results and discussion

The TDS dependences of tritium and helium release from seven samples of irradiated lithium ceramics were taken. Typical TDS curves are shown in Fig. 6.

It should be noted that the TDS curves for each sample were similar and had minor peaks for gasses with mass numbers M4 and M6 observed in the region of 700–1000 K. Further, in the region of 1300–1500 K, a noticeable peak of the mass M4 was registered, and in the region of samples melting (>1900 K), a rapid increase in the peaks for gasses with mass numbers M4 and M6 was observed.

The obtained during the experiment TDS dependences were compared with the current change in the magnetic discharge pump. Since the ionization of helium in the pump is 100 times less efficient than that of hydrogen isotope molecules it was possible to distinguish the ranges when the release of gasses (He or HT) occurs.

The results of helium and tritium release analysis are presented in Figs. 7 and 8. In this case, the reduced number of tritium atoms was calculated (the sum of HT molecules and twice the number of tritium molecules). As for the release of tritium in the form of tritium water molecules HTO and T_2O , it was less than 5% of the total tritium release. For a qualitative description of the observed dependences, the following mechanism for the release of tritium and helium was proposed, which is illustrated in Fig. 9.

The mechanism is as follows: Lithium ceramic pebble has an internal

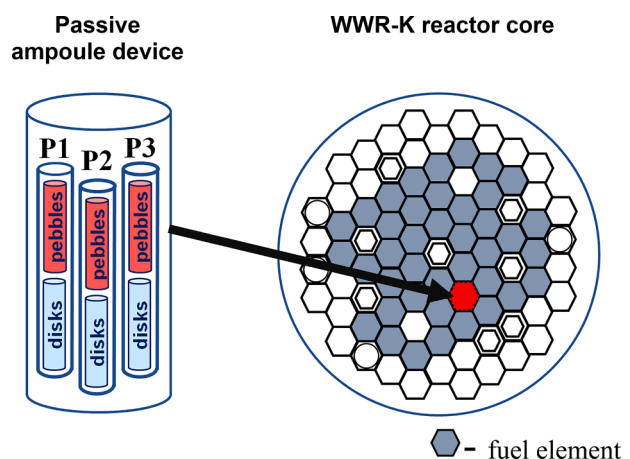


Fig. 2. Layout and location of the passive ampoule device with ceramic samples.

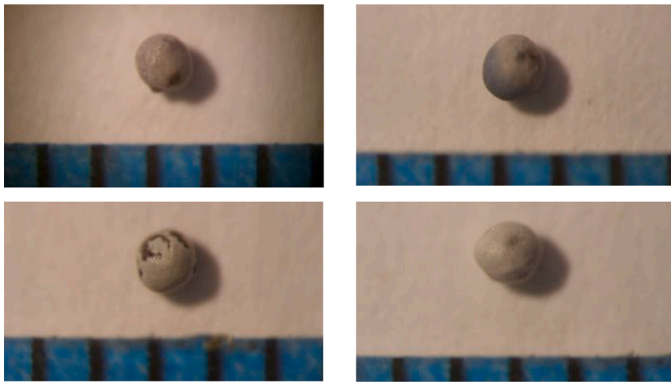


Fig. 3. Irradiated lithium ceramic samples.

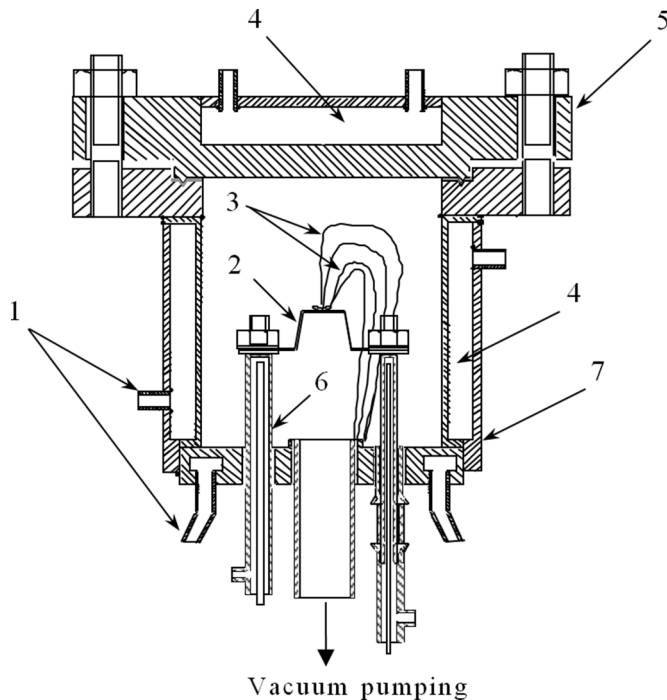


Fig. 4. Vacuum chamber of the TDS facility: 1 – water cooling inlet; 2 – tantalum heater with crucible; 3 – thermocouples; 4 – water cooling path; 5 – vacuum chamber cap; 6 – current feedthrough; 7 – vacuum chamber's body [28].

porosity, that is, it contains closed cavities where gas can be accumulated. Apparently, pebble has a system of pores distributed throughout its volume and communicating with its outer surface. That is, "fast" gas release can occur along such paths after gas enters the pore space. In essence, a pebble is a series of solid areas of ceramics (with different sizes), from which tritium is released into a space that communicates with the outer surface. A similar description for ceramic pebbles was given earlier in [29] and for beryllium pebbles in [30,31].

Tritium atoms can exist in lithium ceramics in various states: a significant number of them are bounded with defects in the ceramic solid areas and are released from such traps only upon melting. However, some of the tritium atoms (that are in a less bound state) can be released by the diffusion mechanism from the ceramic solid areas into the pore space communicating with the outer surface. During linear heating, the release of tritium is due to diffusion, and since the areas of solid ceramics have different sizes, "broad peaks" are observed on the TDS curve, which characterize an increase in tritium release in the temperature range of 700–1000 K.

As for helium atoms, they are located both in the volume of ceramics solid areas and in closed pores (into which helium enters from the surface layers of ceramics at the pore boundary). Here it is worth discussing separately the qualitative mechanism of helium getting into closed pores. The authors suggest that helium mainly got into them during long-term high-temperature reactor irradiation. As a result of a nuclear reaction, a helium atom with an energy of about 2 MeV destroyed the near-surface region of the pore, after which, as a result of facilitated diffusion along a damaged trajectory, it entered the closed pore.

Perhaps, it would be necessary to take into account the ingress of helium into closed pores directly during heating in TDS experiments, but this mechanism was not considered in the proposed model.

It is also worth noting that a significant amount of helium was released immediately before the melting of the sample (however, it was not possible to correctly register such a peak release). The amount of helium released in the region of the melting temperature was at least 50 times greater than during the previous heating of the sample.

From the solid ceramic area helium is released only during melting (due to its very weak diffusion mobility).

From closed pores helium is released at temperatures of 1300–1500 K, what, apparently, is associated with sample cracking and the appearance of "free exit paths" to the surface from the closed pores. This fact is also evidenced by the shape of the helium release peak: namely, at first, a sharp increase is observed, and then an exponential decrease in the release peak).

A similar helium release was observed in [29] during reactor experiments with lithium ceramics, where the shape of the helium release peak was also explained by the formation of free paths for the release of helium from the closed pores of the ceramic during its cracking.

Any modeling of tritium release from the test samples within the framework of the hypotheses stated above has a number of significant assumptions: it is necessary to correctly determine the distribution of solid (non-porous) areas in ceramics by average size, to estimate the uniformity of the tritium concentration over such areas, etc.

The applied simulation of tritium release in the work [32] of the present paper authors made it possible to obtain effective diffusion parameters under the assumption that the pebble has a completely homogeneous ceramic structure and that the tritium concentration is uniform over the pebble. Simulation of tritium release from a pebble divided into several thousand regions of solid ceramics was also considered. One way or another, the obtained data strongly depended on the choice of modeling conditions.

It should be noted that the simulation of tritium release from the pebble, performed in the present work and illustrated in Fig. 10, gave similar results (assuming that the pebble is completely homogeneous, and the initial distribution of the tritium concentration over the pebble is uniform).

It turned out that the diffusion coefficient of tritium has the following temperature dependence:

$$D = 2 \cdot 10^{-7} \left(\frac{m^2}{s} \right) \cdot \exp \left(-\frac{125 \left(\frac{kJ}{mole} \right)}{RT} \right) \quad (1)$$

Whereas

$$D = 1.3 \cdot 10^{-7} \left(\frac{m^2}{s} \right) \cdot \exp \left(-\frac{115 \left(\frac{kJ}{mole} \right)}{RT} \right) \quad (2)$$

is the diffusion coefficient of tritium in the same Li_2TiO_3 ceramic, obtained in [32]. It is worth emphasizing here that the obtained parameters have the meaning of the effective parameters of the Arrhenius dependence of the diffusion coefficients and should be considered as a rough, efficient description useful for this material, as well as the structure and size of the pebbles used in this experiment.

Another model was applied for simulation of helium release. In fact, the release of helium in this case is the effusion of gas from closed pores into a vacuum (continuously evacuated chamber of the installation)

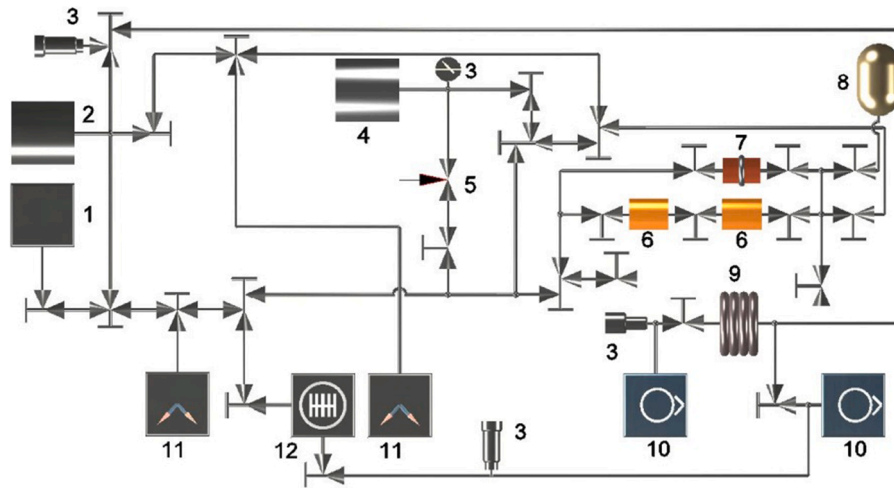


Fig. 5. Vacuum system of the TDS facility: 1 – mass-spectrometer; 2 – vacuum chamber; 3 – pressure sensor; 4 – additional changeable chamber; 5 – leak valve; 6 – gas purification system; 7 – filter; 8 – tank; 9 – nitrogen trap; 10 – fore-vacuum pump; 11 – Penning-type pump; 12 – turbomolecular pump.

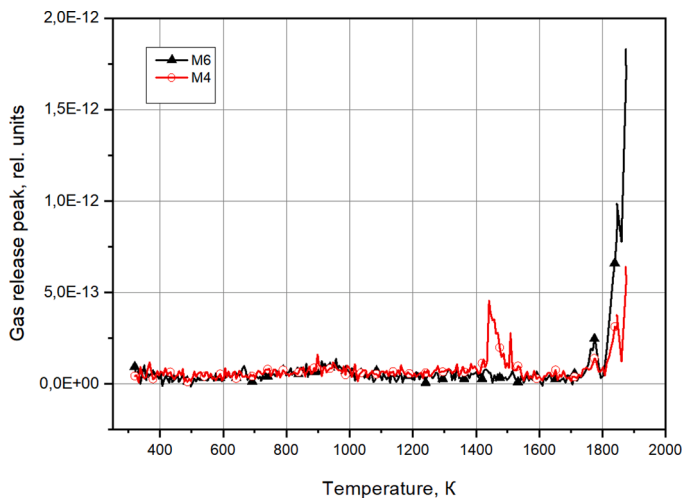


Fig. 6. Release peaks of M6 and M4 masses during linear heating (3 K/min) of irradiated lithium ceramics pebble (0.0014 g).

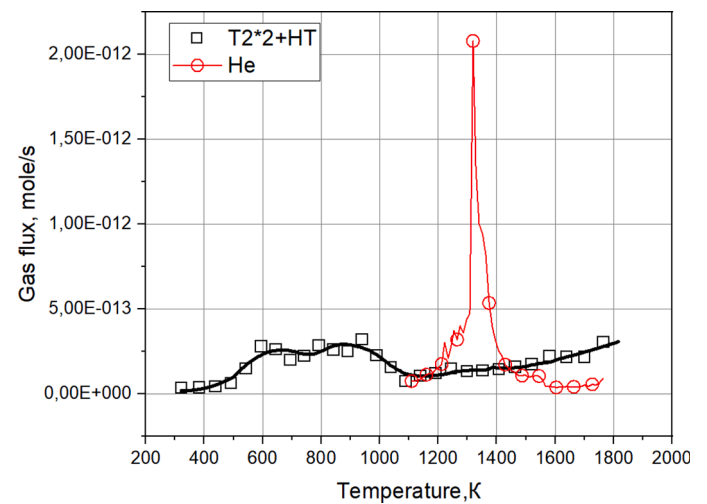


Fig. 8. Helium and tritium flux from a sample of lithium ceramic pebble (6 K/min; $m = 0.0017$ g).

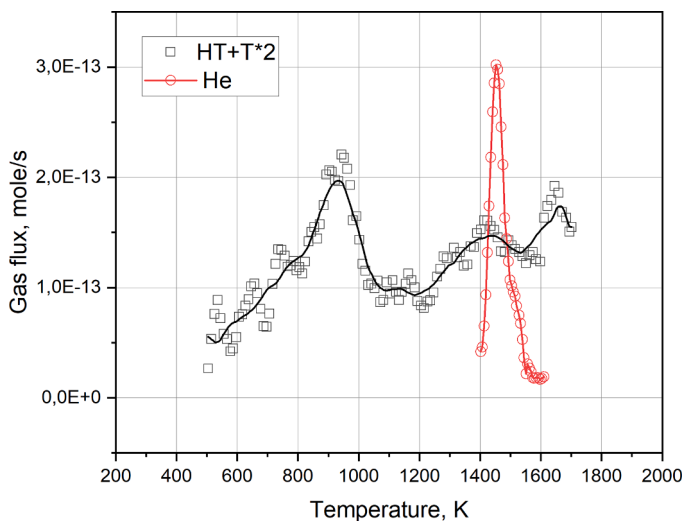


Fig. 7. Helium and tritium flux from a sample of lithium ceramic pebble (3 K/min; $m = 0.0014$ g).

when small holes appear.

With this approach, it can be assumed that when “free exit paths” appear from the region of closed pores, the probability of such an exit for an individual helium atom (per unit time) is equal to some β . Then the temporal change in the number of helium atoms in a pore can be represented as:

$$N(t) = N_0 \cdot e^{-\beta t}, \quad (3)$$

where N_0 – initial number of helium atoms in closed pores.

Hence, the expression for the time dependence of the helium flux from closed pores will have the following form:

$$\frac{dN(t)}{dt} = N_0 \cdot \beta \cdot e^{-\beta t}. \quad (4)$$

Having determined N_0 from the graph as the total number of released helium molecules, you can find β by varying it and achieving the coincidence of the model and real curves (Fig. 11).

The obtained values of β lie in the range from 0.0005 to 0.001.

The obtained values can be used to estimate the cross section of “free paths” from the closed pores of ceramics. For example, if we assume that closed pores make up about 5% of the ceramics volume and their size is

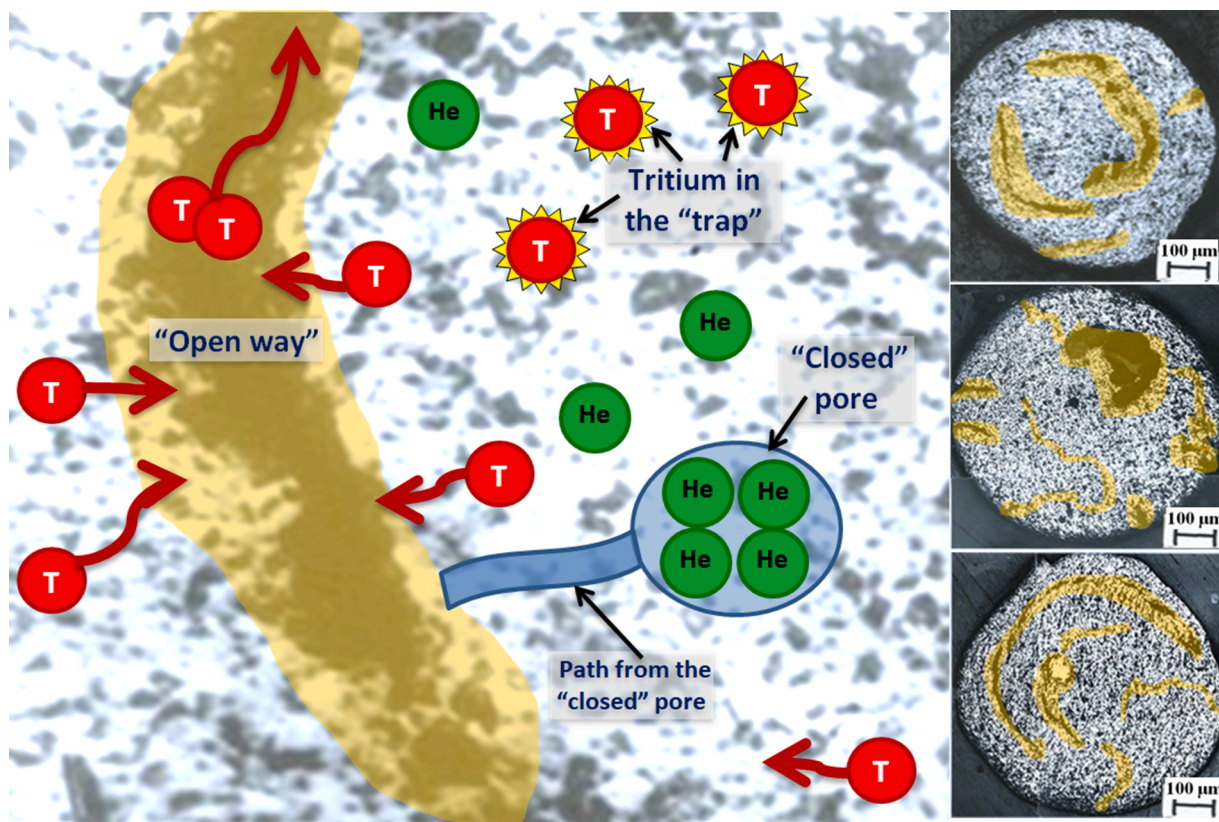


Fig. 9. Mechanism of tritium and helium release from lithium ceramic pebble.

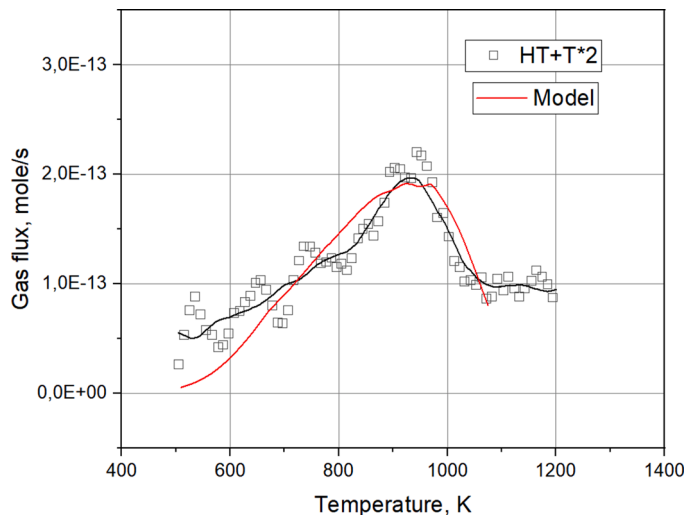


Fig. 10. An example of modeling the tritium diffusion release from a pebble during linear heating (assuming that the pebble has a continuous volume, and the initial distribution of the tritium concentration over the pebble is uniform).

about 50 μm , then the average size of a hole in such pores forming a “free” exit from them will be about 0.02 μm .

4. Conclusions

The TDS studies of irradiated lithium ceramics Li_2TiO_3 (enriched up to 96% in lithium-6 isotope) made it possible to qualitatively describe the release processes of tritium and helium and suggest a substantiated mechanism for this release.

Experiments indicate that the main amount of tritium in ceramics is

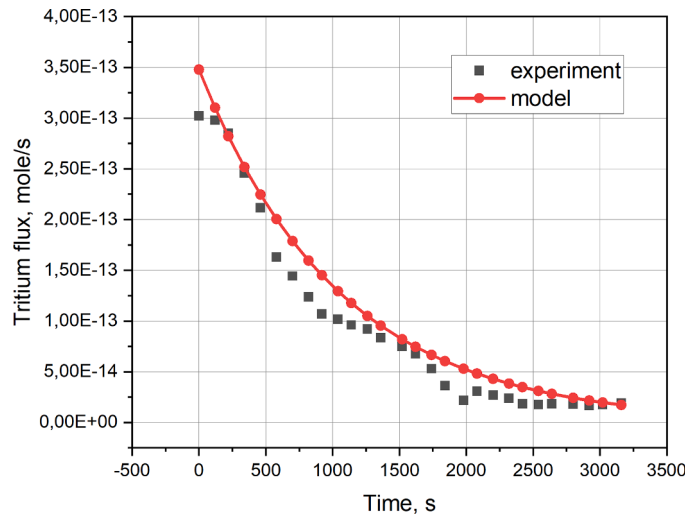


Fig. 11. Simulation of helium release from lithium ceramic pebble ($\beta=0.00095$).

in a bound state and is released at temperatures close to the melting point of ceramics. For tritium not bound in traps, the release is determined by its diffusion in solid pebble regions to the boundaries communicating with the outer surface of the ceramic. Accordingly, the kinetics of release of unbound tritium depends on the number and size of such regions in the pebble. The simulation made it possible to determine the temperature dependence of the effective diffusion coefficient of tritium in irradiated ceramics, which coincided in order of magnitude with the data of similar simulation performed under the assumptions that the pebble volume is homogeneous and the initial distribution of tritium over the pebble is uniform.

As for helium, it was shown that the main amount of helium is in the volume of ceramics and is released in the region close to the melting point, and a small part of it, located in the closed pores of ceramics, is released from them when heated due to the formation of “free exit paths” (for example, pebble cracking) and is well described in terms of gas effusion. The obtained parameters of such precipitation made it possible to estimate the dimensions of the exit paths formed from the closed pores of the ceramic.

CRedit authorship contribution statement

Timur Kulsartov: Conceptualization, Methodology, Validation, Writing – original draft, Writing – review & editing. **Yuriy Ponkratov:** Investigation, Data curation. **Zhanna Zaurbekova:** Writing – original draft, Investigation, Writing – review & editing. **Yuriy Gordienko:** Investigation, Data curation. **Irina Tazhibayeva:** Supervision. **Inesh Kenzhina:** Investigation. **Kuanysh Samarkhanov:** Investigation. **Yevgeniy Tulubayev:** Investigation. **Asset Shaimerdenov:** Investigation. **Sergey Udartsev:** Investigation.

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

Data will be made available on request.

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