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Estimation of tritium transfer parameters in biphasic lithium ceramic based on the results of reactor irradiation experiments

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ABSTRACT

It is expected that the development of thermonuclear fusion will be able to easily satisfy the energy needs of humanity. There are enough fuel reserves for the fusion reaction: tritium can be bred via a $^{6}Li(n,\alpha)^{3}H$ reaction from lithium, which is common in nature, and deuterium is quite easily extracted from seawater. One of the problems on the way to implement thermonuclear fusion today is the search for and development of breeder materials that can withstand the high radiation and energy loads that are expected in a long-pulsed fusion devices.

Various types of lithium-containing ceramic materials are currently being actively studied for use as a breeder material in helium-cooled breeder blanket concepts.

This study focuses on the methodology and results of calculations of tritium residence time in the samples of biphasic lithium ceramic pebble bed containing nominal 65 mol % lithium orthosilicate and 35 mol % lithium metatitanate (pebble diameter 0.25 -1.250 mm).

The values of the tritium residence time obtained as a result of modeling the release of tritium from ceramics within the framework of the diffusion-desorption model have a temperature dependence of $\lg\tau = -1.374+1676/t$. They are in good agreement with the literature data, suggesting that the tritium residence time increases with increasing open porosity.

1. Introduction

It is expected that the development of thermonuclear fusion will be able to easily satisfy the energy needs of humanity. There are enough fuel reserves for the fusion reaction: tritium can be bred via a $^{6}Li(n,\alpha)^{3}H$ reaction from lithium, which is common in nature, and deuterium is quite easily extracted from seawater. One of the problems on the way to implement thermonuclear fusion today is the search for and development of breeder materials that can withstand the high radiation and energy loads that are expected in a long-pulsed fusion devices. In recent years, ceramic materials have seen extensive use in both nuclear and fusion applications. This is largely due to their excellent ability to withstand high radiation levels and temperatures, combined with low neutron absorption and stable chemical behavior. They serve various purposes – ranging from fission reactor fuel to insulation, shielding, and neutron-absorbing elements. Several oxide-based ceramics, such as MgO, Al_2O_3, and $MgAl_2O_4$ have been the subject of research for their potential in diagnostic systems and as optical windows in future fusion devices [1–3].

Various types of lithium-containing ceramic materials are currently being actively studied for use as a breeder material in helium-cooled breeder blanket concepts [4–6]. With the goal of creating a ceramic material with improved characteristics, Knitter et al. [7] first presented a biphasic ceramics based on lithium orthosilicate Li_4SiO_4 (LOS) and

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lithium metatitanate Li_2TiO_3 (LMT) fabricated by a melt-based process, which is meanwhile the European reference material for solid breeder concepts for ITER and DEMO [4]. Further research in this direction was continued by other authors [8–12], which set a general trend for studying the most promising composition of a biphasic compound of ceramics, as well as developing different methods for manufacturing ceramic pebbles for the fusion reactor pebble bed. In addition to the melt-based fabrication process used by Knitter et al. [7,13,14], there were also several other methods developed for producing biphasic ceramic pebbles based on LMT and LOS. Li_2TiO_3-Li_4SiO_4 ceramics samples were manufactured by the wet method from powders synthesized by the hydrothermal method [15], by the microwave sintering method [16], and the graphite bed method, which was used to produce samples of biphasic ceramic pebbles coated with a Li_2TiO_3 layer [17].

To assess the prospects for using the lithium ceramics being developed as a breeder material, it is preferable to conduct irradiation experiments at research nuclear reactors, when tritium is formed in lithium under the influence of neutron irradiation. Today, in-situ reactor tests are practically the only available way to assess the parameters of tritium generation, retention and release from lithium-containing materials. Neutrons interacting with ceramic oxides can cause induced activation, formation of gas bubbles, reduction in thermal conductivity, embrittlement and other material-degrading effects [18,19]. In turn, tritium retention in the material is one of the key concerns for the operation of future fusion reactors. Large values of tritium residence time can lead to significant levels of radioactive contamination of the breeder material and the blanket as a whole, thereby complicating the processes of operation and waste management.

The authors of this paper previously conducted a series of experiments on the irradiation of biphasic lithium ceramics at the WWR-K reactor [20–24]. This paper is a continuation of a series of works devoted to the analysis of the interaction of tritium with biphasic lithium ceramics, carried out by the authors since 2021. The article describes the methodology and the results of calculating the residence time of tritium in a sample of lithium ceramics containing nominal 65 mol % lithium orthosilicate and 35 mol % lithium metatitanate. As is known, such assessments for two-phase ceramics have not yet been carried out.

2. Materials and method

A ceramic pebble bed sample weighing 5 g containing nominal 65 mol % lithium orthosilicate and 35 mol % lithium metatitanate (pebble diameter 0.25–1.25 mm) was fabricated at KIT (Karlsruhe Institute of Technology) using the KALOS process. Detailed information on the pebble fabrication method is given in [14].

The ceramic sample irradiation experiments were conducted using the vacuum extraction method at the Complex of In-Reactor Gas Release Analysis facility (CIRRA) [25–26] located at the WWR-K research reactor in Almaty, Kazakhstan. The total duration of the reactor experiment was 21 days (one reactor campaign). The reactor power output of finally 6 MW was achieved sequentially: initially, the power levels reached were 10 kW, 60 kW, 1 MW, 3 MW, 4.8 MW, and 6 MW. On the 8th and 15th day of irradiation, a series of experiments with heating the sample at a reactor power of 6 MW were conducted. Heating was performed using an electric heater wound around the outer wall of the capsule with the sample. During the irradiation on 6 MW, the ceramic sample temperature was maintained at about 665 °C. An extended description of the experiments is given in [20,21,24]. The composition of gases released from the sample during irradiation experiments was monitored using a quadrupole mass spectrometer. Three thermocouples of chromel-alumel type (with accuracy ± 1 °C) were installed on the walls and bottom of the irradiation ampoule device to measure the temperature (Fig. 1).

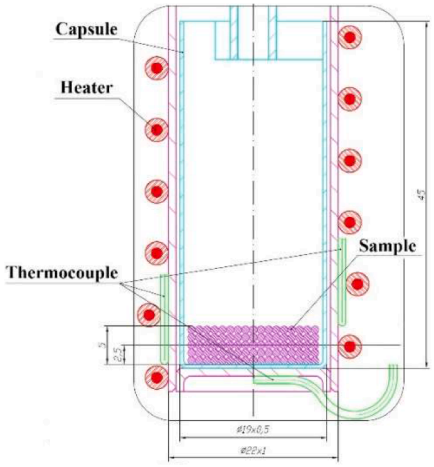


Fig. 1. Irradiation ampoule device with capsule.

3. Results and discussion

3.1. Experimental results

Fig. 2 shows the complete diagram of the irradiation experiment. The graph shows the time dependences of the gas release from the sample with the following masses: M2, which corresponds to the evolution of hydrogen H_2; M4, which corresponds to the evolution of HT and He gases; M6, which corresponds to the evolution of tritium in the form of a T_2 molecule and M18, which corresponds to the evolution of water vapor H_2O.

To estimate the tritium residence time in the sample of biphasic lithium ceramics during irradiation, the sections of the experiment corresponding to short-term heating of the sample on the 8th and 15th day, respectively, were considered. Fig. 3 shows a typical section of the experiment consisting of a series of heatings of lithium ceramics on the 15th day of irradiation. With an increase in the temperature of the sample, an increase in the pressure of hydrogen and gases with the mass number M4 (He + HT) is observed, as well as a drop in the pressure of tritium in the form of the T_2 molecule.

The total amount of released tritium atoms was taken as the sum of molecules with the mass number M4 and the doubled number of molecules with the mass number M6: M4 + 2M6. It can be observed from Figs. 2 and 3 that, despite the decrease in the level of the M4 mass,

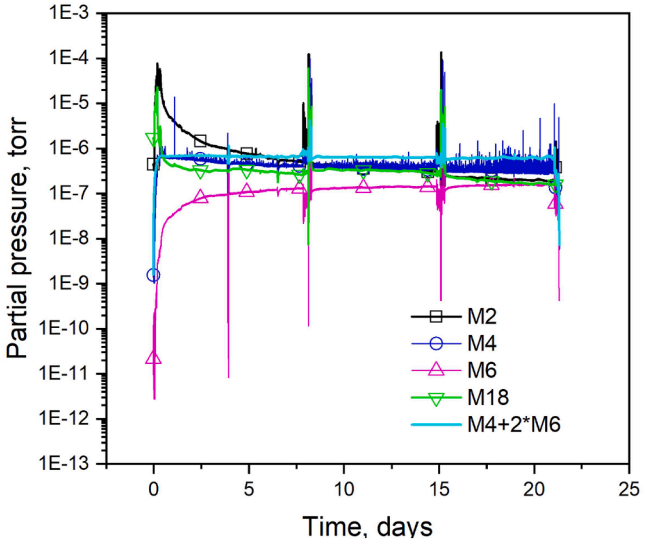


Fig. 2. In situ gas release of selected masses during the reactor experiment.

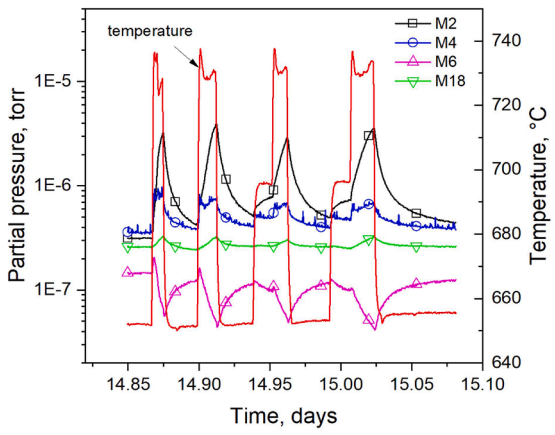


Fig. 3. Heating of the sample on the 15th day of irradiation.

responsible for the release of HT and He, and the increase in the level of tritium in the form of the T_2 molecule, the total tritium level, accounting for the release of tritium atoms in the form of HT and T_2 molecules (turquoise line), remains approximately constant. Fig. 4 shows the section with the first heating on the 15th day of irradiation, taking into account the total release of tritium. As can be seen from Figs. 3 and 4, tritium was released in several peaks, and during heating the release of tritium did not have time to reach equilibrium values.

3.2. Calculation of residence time based on the experimental peak of tritium release from ceramics

First, an attempt was made to estimate the residence time of tritium τ in biphasic lithium ceramics based on the experimental peak of tritium release (Fig. 5) as the ratio of the amount of released tritium Q to the average release G_0 level during the temperature transient (as shown in [27–28]):

$$\tau = \frac{Q}{G_0}, \quad (1)$$

where Q is the area under the peak of tritium release, G_0 is the equilibrium value of tritium release before heating.

On the general diagram of the experiment, the peak of tritium release during temperature transient at a reactor power of 6 MW was selected (Fig. 5 shows a characteristic peak of tritium release in the form of HT and T_2 molecules (M4 + 2M6) during the first heating). Since the experimental peak is not complete, it was extrapolated using a curve $y = y_0 + A_1 e^{-(x-x_0)t}$, where x_0 corresponded to the start point of

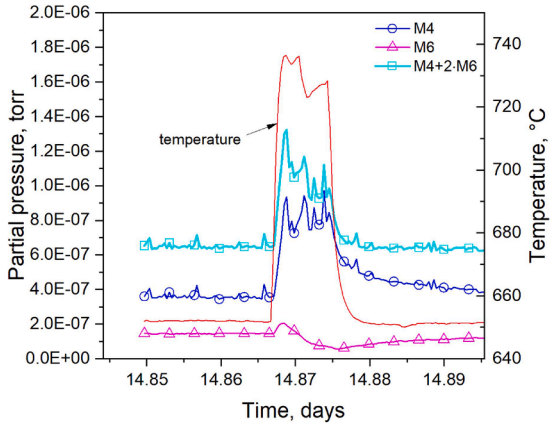


Fig. 4. Peak release of tritium atoms in the form of HT and T_2 molecules (M4 + 2M6) during heating on the 15th day of irradiation (first peak in Fig. 3).

extrapolation, and the value of the extrapolated level y_0 practically coincided with the equilibrium value of tritium release before heating G_0 (with a difference of no >1 %), A_1 is a constant. The integral amount of released tritium Q under the approximated curve was determined taking into account the specified asymptotic conditions of the exponential function, while the equilibrium level Q_0 was not taken into account.

Tritium residence time τ was estimated using the above algorithm for all peaks of tritium release on the 8th and 15th day of irradiation. Unfortunately, the obtained values were assessed to be incorrect due to the tremendous range of τ = 4–30 min and did not show an adequate dependence on temperature. The authors attribute this to the fact that the dependence of tritium release was extrapolated incorrectly (although the approximation seems appropriate) and the actual decrease in the tritium flux from the sample occurs much longer. Thus, it can be concluded that the sample was not heated long enough, or the heating temperatures should be higher.

3.3. Modeling tritium release from ceramics during temperature transients

To more thoroughly assess the residence time of tritium in lithium ceramics, we simulated the release of tritium during the establishment of its equilibrium flux and with a subsequent series of heatings (temperature transients). The model describes a lithium ceramic cluster in the form of non-contacting spheres of five dominant diameters in a vacuum, in which tritium is generated under the influence of neutron irradiation [24]. The tritium generation rate is uniform over the volume of each ball and is proportional to the density of lithium nuclei in the ceramics and the neutron flux (or reactor power), which changes stepwise over several hours as the reactor reaches a steady-state power of 6 MW. The simulation took into account that tritium release reaches a steady state after 50,000 s. Figs. 6 and 7 show the results of modeling the release of tritium in the form of HT and T_2 molecules (M4 + 2M6). The experimental curves are satisfactorily described by a number of sets of desorption and diffusion parameters.

Next, the obtained coefficients of pre-exponential constants and activation energies of diffusion and desorption were used to construct a diagram of tritium release from a lithium ceramic pebble bed sample upon heating from 600 °C with temperature plateaus at 700, 800, 900 and 1000 °C, respectively, at a reactor power of 6 MW. In this way, the model experiment was reproduced, the results of which are shown in Fig. 8.

As a result of the modeling, the dependences of the change in tritium fluxes from each pebble were also obtained. Pebbles with diameters D1-D5, the mass fraction of which prevailed in the pebble bed, were taken as the main ones in the calculations (Fig. 9). It can be concluded from Figs. 8 and 9 that the amount of tritium released from the ceramics gradually decreases. This fact is confirmed by the results of calculating the amount of tritium contained in the ceramic sample for both the entire pebble bed and for each pebble with diameters D1-D5, respectively (Figs. 10 and 11). From the graphs, it can be seen that with an increase in temperature, the amount of tritium dissolved in the sample decreases insignificantly. With each heating by 100 °C, <50 % of the current amount of dissolved tritium is released from the sample.

3.4. Calculation of tritium residence time based on the results of tritium release modeling from ceramics

The tritium residence time in the ceramic pebble bed was calculated as the ratio of the amount of tritium contained in the ceramic sample Q_1 (Fig. 10) to the equilibrium value of tritium release G_0:

$$\tau = \frac{Q_1}{G_0},$$

The obtained values in comparison with the literature data for lithium metatitanate are shown in Fig. 12. Good agreement with the literature data is noted, and the tritium residence time for biphasic

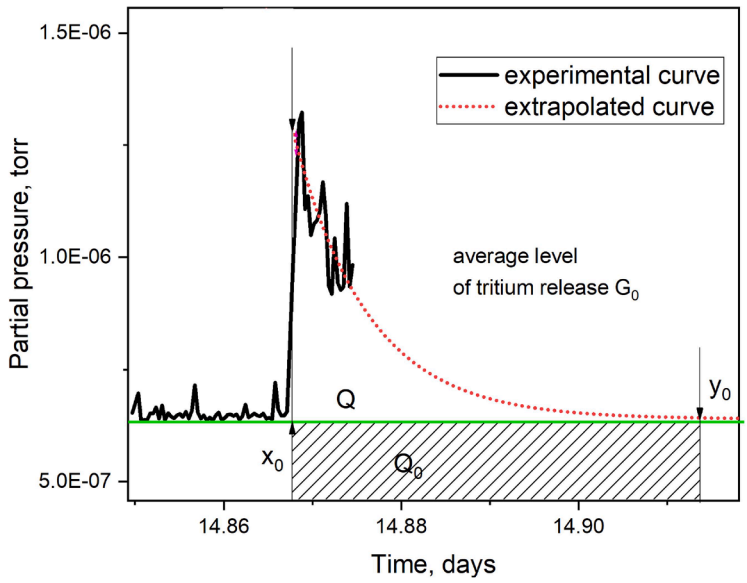


Fig. 5. Fitting of the experimental curve of total tritium release during temperature transient.

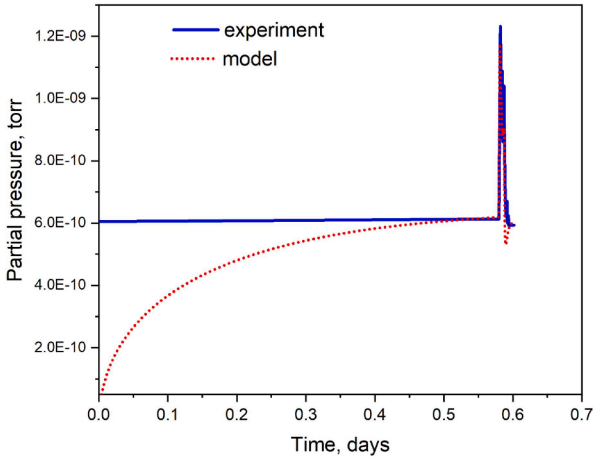


Fig. 6. Modeling of tritium release during temperature transient in the lithium ceramic sample from 665 to 735 °C on the 15th day of irradiation.

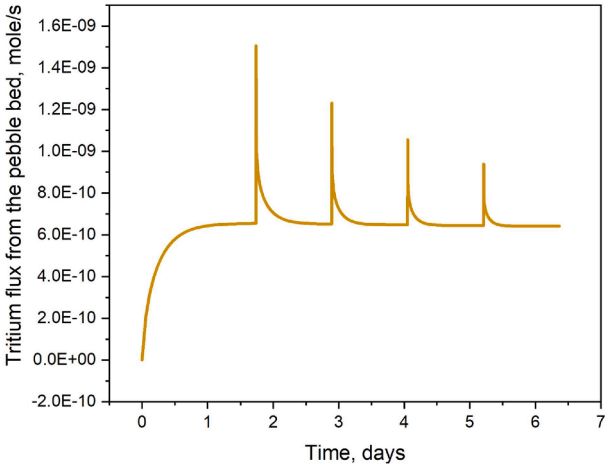


Fig. 8. Simulation of tritium release from the pebble bed.

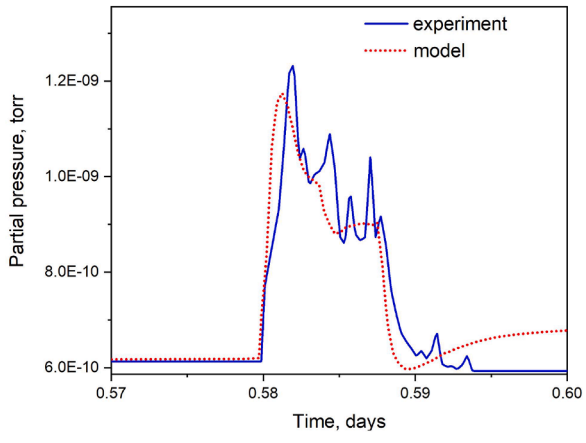


Fig. 7. Enlarged section of the modeled peak of tritium release (from. Fig. 5).

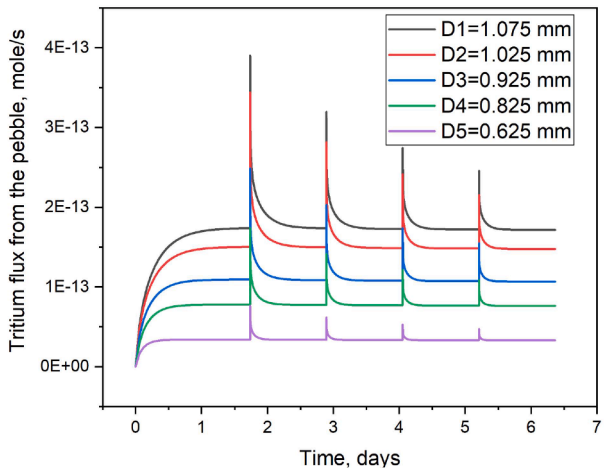


Fig. 9. Simulation of tritium release from single pebbles.

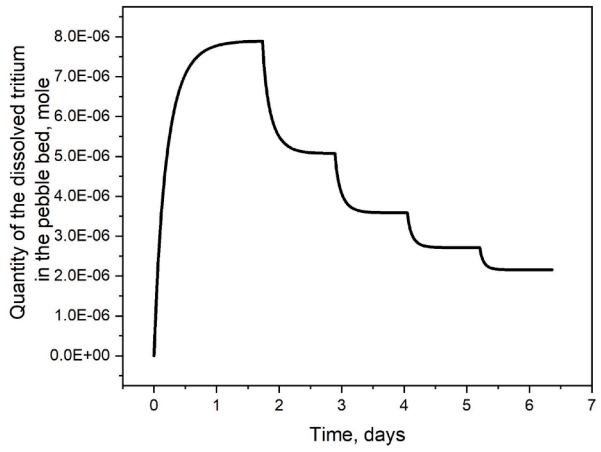


Fig. 10. Quantity of dissolved tritium in the pebble bed.

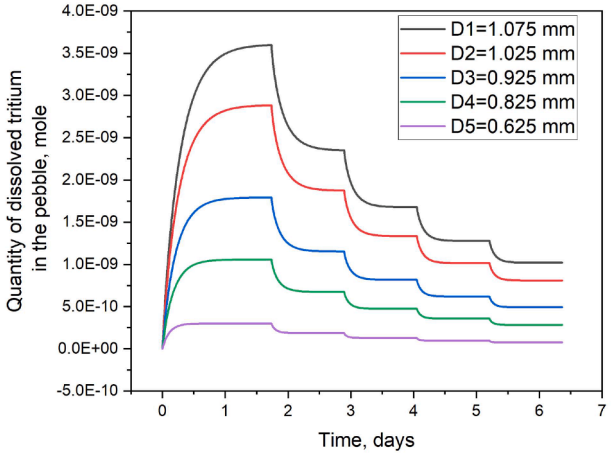


Fig. 11. Quantity of dissolved tritium in the single pebbles.

lithium ceramic pebble bed is lower than for LMT in the EXOTIC-9 experiment [27] and LMT from Zhao et al. [30]. The open porosity index of these pebbles is lower than that of the test sample, which confirms the fact that the tritium residence time increases with a decrease in the open porosity of the ceramic pebbles [29].

The temperature dependence of the tritium residence time in biphasic lithium ceramic sample can be written as $lg\tau = -1.374+1676/t$, which practically coincides in its angular coefficient (as can be seen in Fig. 12) with the τ values for LMT pebbles ($lg\tau = -0.74+1622.4/t$) manufactured by the freeze-drying method [30].

It is worth noting that the experiments were carried out in the vacuum extraction mode, while the presence of a purge gas and the addition of hydrogen can have a significant effect on the tritium release parameters. This is because the presence of hydrogen results in an increase in the surface coverage and promotes isotope exchange such as HT recombination and release. Thus, in the work from Ying et al. [31] it is shown that tritium reaches a steady-state release level faster when hydrogen is added to the helium purge gas. It was found that the tritium residence time increases by 33 % when the hydrogen level is reduced from 300 to 200 Pa at a hydrogen volume fraction of 0.1 % in the helium purge gas. In other words, when the hydrogen pressure with a volume fraction of 0.1 % in the helium purge gas is increased from 160 to 200 Pa, the tritium residence time decreases by 19 %.

Figs. 13 and 14 show the results of comparing the tritium residence time in ceramics depending on the pebble size and the tritium generation rate. As can be seen from the graphs, the tritium residence time in

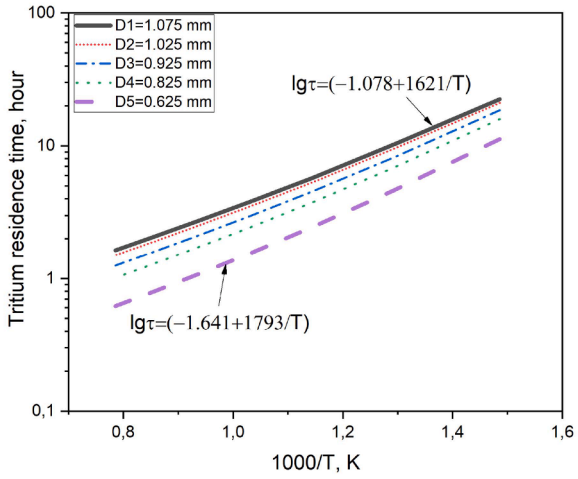


Fig. 13. Tritium residence time for pebbles of different sizes (diameters).

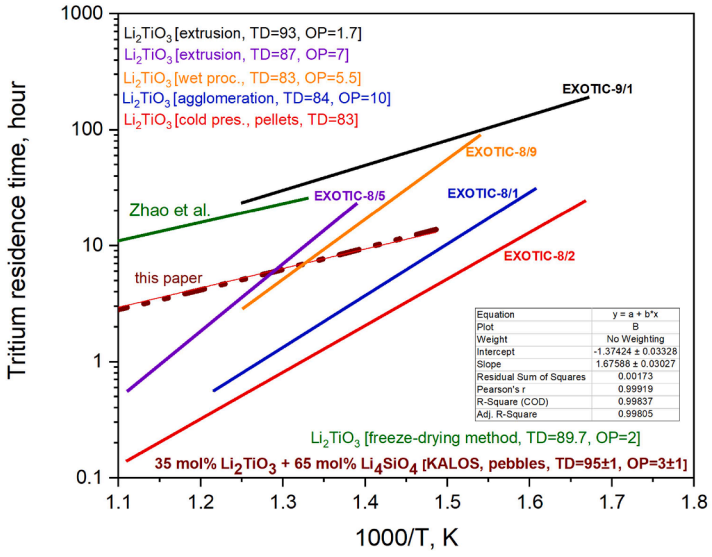


Fig. 12. Tritium residence times of different studies in comparison (reproduced from [29,30]).

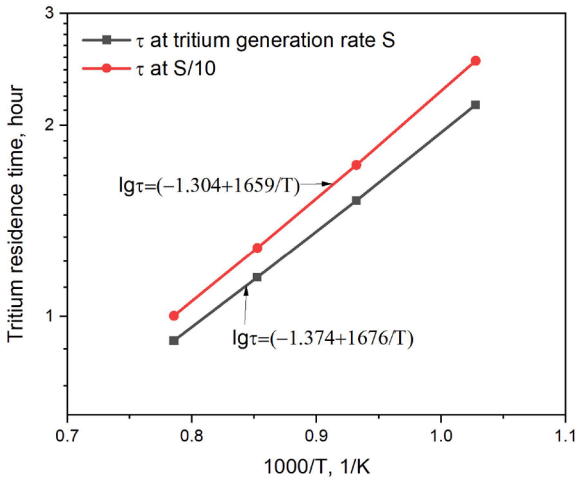


Fig. 14. Dependence of tritium residence time on tritium generation rate (S) in lithium ceramics pebble bed.

the pebble is directly proportional to its size. In turn, an increase in the tritium generation rate reduces its residence time in the pebble.

4. Conclusions

The tritium residence times were determined in a biphasic lithium ceramic pebble bed containing nominal 65 mol % lithium orthosilicate and 35 mol % lithium metatitanate (pebble size 0.25 −1.25 mm) during in-situ irradiation at the WWR-K reactor. When determining the tritium residence time based on the extrapolated experimental peak of tritium release, the values were assessed to be incorrect due to the tremendous range of τ = 4–30 min and did not show an adequate dependence on temperature. The disadvantage of this method for determining the residence time was that the calculation assumed that the main amount of tritium from the ceramics is released from the one considered peak, which may not actually has happened. To eliminate this error, tritium release was simulated based on the diffusion-desorption model. The obtained values of tritium residence time for the biphasic lithium ceramics pebble bed are in good agreement with the literature data, taking into account the fact that the tritium residence time increases with increasing open porosity.

The authors of this article attempted to analyze the results of experiments on the irradiation of biphasic lithium ceramics and to estimate the residence time of tritium in ceramics. The analysis showed that there are significant factors affecting the correctness of the assessment of the residence time of tritium in ceramics, namely:

- for a more accurate experimental determination of the amount of tritium contained in ceramic pebbles, the differences between the temperature levels during heating should be as big as possible;
- to achieve equilibrium values for the tritium emission flow, it is necessary to have longer holding periods of the sample at constant temperatures.

CRediT authorship contribution statement

Timur Kulsartov: Writing – original draft, Methodology, Investigation, Conceptualization. **Zhanna Zaurbekova:** Writing – original draft. **Regina Knitter:** Supervision, Resources, Formal analysis, Data curation, Conceptualization. **Julia Leys:** Supervision, Resources, Formal analysis, Conceptualization. **Asset Shaimerdenov:** Supervision, Project administration, Methodology, Investigation. **Yevgen Chikhray:** Methodology, Investigation, Data curation. **Saulet Askerbekov:** Investigation. **Inesh Kenzhina:** Investigation. **Alexandr Yelishenkov:** 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.

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Data availability

The authors do not have permission to share data.

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