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Studies of two-phase lithium ceramics Li_4SiO_4-Li_2TiO_3 under conditions of neutron irradiation

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ABSTRACT

This work presents the preliminary experimental data on the study of gas release from two-phase lithium ceramics Li_4SiO_4-Li_2TiO_3 under neutron irradiation conditions. Experiments were carried out at the WWR-K research reactor (Almaty, Kazakhstan) for ~4.3 days. The total neutron fluence during the irradiation was $\sim 1.8 \cdot 10^{19} n/cm^2$. In the course of irradiation, two experiments on ceramics heating during irradiation and two experiments with hydrogen isotopes (H_2 and D_2) supply into the experimental chamber with the sample were performed at a temperature of 680 °C and reactor power of 6 MW. During the entire irradiation, the gas composition in the continuously evacuated ampoule device with samples was recorded. The obtained dependences of the release of tritium-containing molecules and helium during the experiment were qualitatively analyzed.

1. Introduction

Lithium ceramics are considered promising functional materials for breeder blankets of future fusion reactors [1–3]. Tritium is produced in lithium under neutron irradiation according to the $^6Li(n,\alpha)T$ reaction. Further, this tritium is extracted from the blanket with a purge gas and is returned to the fusion zone, implementing the concept of a closed thermonuclear cycle.

Lithium orthosilicate Li_4SiO_4 and lithium metatitanate Li_2TiO_3 deserve special attention among the well-known lithium ceramics. According to [4–8], these ceramics show high mechanical, thermal and chemical resistance. However, lithium metatitanate has a lower lithium density than orthosilicate, making it necessary for additional enrichment with the lithium-6 isotope in pebbles production.

Several years ago, to combine the advantages of these two materials, the idea of a two-phase ceramic compound based on Li_4SiO_4 and Li_2TiO_3 was proposed [9] and later also used by others [10–11].

To date, very little information is available on evaluating the release of tritium from lithium ceramics. However, this criterion is one of the keys to the final choice of breeder material. Studies carried out to determine the parameters of tritium yield from lithium ceramics were carried out "in-situ" or with irradiated samples [12–18]. The small part of "in-situ" experiments involved using a purge gas (helium + hydrogen), which transferred the tritium extracted from the breeder zone. Thus, when analyzing the results of such experiments, difficulties arose associated with the need to consider the release of tritium in the form of tritium water (T_2O and HTO).

The experiments presented in this paper were performed "in-situ" with new promising material - two-phase Li_4SiO_4-Li_2TiO_3 ceramics by vacuum extraction method with continuous pumping. The content of

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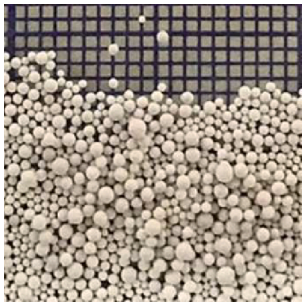

Fig. 1. Pebbles of Li_4SiO_4-Li_2TiO_3 two-phase ceramics.

Table 1
Main parameters of Li_2TiO_3-Li_4SiO_4 pebbles.

Parameter	Value
Li_2TiO_3 content	25.5 mol% [20]
Density	95 ± 1 %
Open porosity	2.2 ± 0.6 % [20]
Closed porosity	2.6 ± 0.2 % [20]
Enrichment by ^{6}Li isotope	natural
Sphericity	1.12

residual gases in the active cell with the sample is kept to a minimum (less than 10^{-6} Torr). This mode provides more opportunities for analyzing the results without unwanted inclusions.

2. Materials and method

The samples of lithium orthosilicate with 25 mol% lithium metatitanate (pebbles with sizes from 250 to 1250 μm and a total weight of 5.027 g) were made using KALOS method [19] and provided by KIT (Karlsruhe Institute of Technology) (Fig. 1). The main parameters of samples are shown in Table 1, more details on the material is given in [20].

The experiments were carried out at the CIRRA (Complex of In-Reactor gas Release Analysis) facility [21], located at the WWR-K research reactor in Almaty, Kazakhstan (Fig. 2). The facility can be structurally divided into four key elements: the reactor ampoule device (AD) with the samples; the vacuum path and the pump-out system (P1 and P2 – active wide-range pressure sensors EdwardsWRG-NW25 with an accuracy of 15 % for pressures below 100 mbar and 30 % for pressures below 1 μbar); the RGA-100 residual gas analyzer (with an accuracy of 10 %), as well as the gas inlet system, which uses a leaking valve to supply controlled gas flows into the chamber with samples during the experiment.

Fig. 2 shows an enlarged fragment of the active part of the reactor irradiation device. The test samples are placed inside the loading capsule, which is placed in the evacuated ampoule device. The AD was equipped with a heater to provide the required temperature conditions. Three thermocouples of chromel–alumel type (with accuracy ±1 °C) were installed on the walls and bottom of the AD to measure the temperature. Then this assembly was placed in the dry experimental channel of the reactor. The design makes it possible to use the AD several times. The samples were located in AD at the core center level.

In Fig. 3, photographs of the main elements of the reactor irradiation device are presented. Before the experiments, the vacuum conditioning of the AD was conducted, consisting of annealing the ampoule and the pipelines of the CIRRA facility.

The annealing procedure was as follows:

1. Technological annealing of the ampoule device and capsule at 700 °C for 5 h;
2. Filling the test sample into the capsule;
3. Loading a capsule with sample into the ampoule device;
4. Pumping out an ampoule device with a sample when the primary pumping path is heated up to 150 °C for 3 h.

Then the reactor experiments were conducted using the vacuum extraction method: two-phase lithium ceramics was irradiated under conditions of continuous pumping out, while the quadruple mass-spectrometer recorded the release of tritium-containing molecules [22,23].

Fig. 4 shows a general diagram for the entire irradiation time: the time dependence of sample temperature change, and the graph of the reactor power change.

The whole experiment was divided into six stages:

I - beginning of the experiment (stepwise reaching of the reactor power level of 1, 3, 4.8 and 6 MW. The ampoule temperature was increased up to 330, 475, 600 and 680 °C, accordingly);

II - section without changes in the parameters of the experiment (reactor power is 6 MW, the sample's temperature is 680 ℃);

III - the experiment with cyclic heating of the sample up to temperature shelves from 720 to 790 °C (heating rate is 50 ℃/min) and it's cooling; the reactor power is 6 MW;

IV – section without changes in the parameters of the experiment (reactor power is 6 MW, the sample's temperature is 680 ℃);

V – the second experiment with heating of the samples and next experiment with supply of H_2 and D_2 gases into the chamber;

VI - section without changes in the parameters of the experiment and stepwise power decrease (from 6 to 3, 2, and 1 MW).

The total irradiation time was about 4.3 days. The total neutron fluence during the irradiation was ~$1.8{\cdot}10^{19}$n/cm^2. The neutron spectrum of the WWR-K reactor is presented in Fig. 5.

3. Results and discussion

In this paragraph, each stage of the experiment is described in more detail, and a preliminary analysis of the obtained results is conducted.

During the initial heating (Fig. 6), when the reactor power was changed, an increased release of gases with mass numbers M2 (H_2), M18 (H_2O), M28 (CO) and M44 (CO_2) was observed.

Then, when the power and, correspondingly, the temperature increased, the pressure of these gases decreased. Emission of gases with the mass number M4 - helium and HT began almost from the beginning of the experiment. The release of gas with mass number M6, which is responsible for T_2, was observed with some delay. That is, the appearance of this mass begins from the reactor power of 3 MW. The release of gases with mass numbers M4 and M6 reached stationary values for each reactor power level.

Due to the relatively high gas pressures in the experimental chamber at this initial stage of the experiment, the mode of operation of the mass spectrometer was periodically changed to the so-called "protection mode". This circumstance made it difficult to analyze some parts of this experiment area correctly. It is necessary to make several remarks concerning gases with mass numbers M20 (presumably tritium-containing gas HTO) and M22 (presumably T_2O heavy water vapours). For this purpose, correct analysis and interpretation of the whole spectrum are needed since other masses can cause these mass spectra peaks. They are probably just additional peaks of some gases and are caused by complex and unlikely processes during the ionization of different gas molecules. As a confirmation, the behaviour of the M22 peak (blue) (Fig. 3) is similar to that of the M44 peak (green). Apparently, it is a CO_2 molecule ionized twice and therefore, as for the peaks for M20 and M22, it cannot be concluded that they are peaks of tritiated water.

As power changes from 3 to 4.8 MW and the ampoule temperature increases from 450 to 600 °C, accordingly, there is a sharp increase in the release of CO molecule and a drop in the CO_2 molecule peak. It is connected with the increase of the ampoule temperature and, accordingly, the rate of CO desorption also increase either from ampoule materials or from samples. It is not yet clear and the assumption is mainly based on the elements of ampoule and capsule materials.

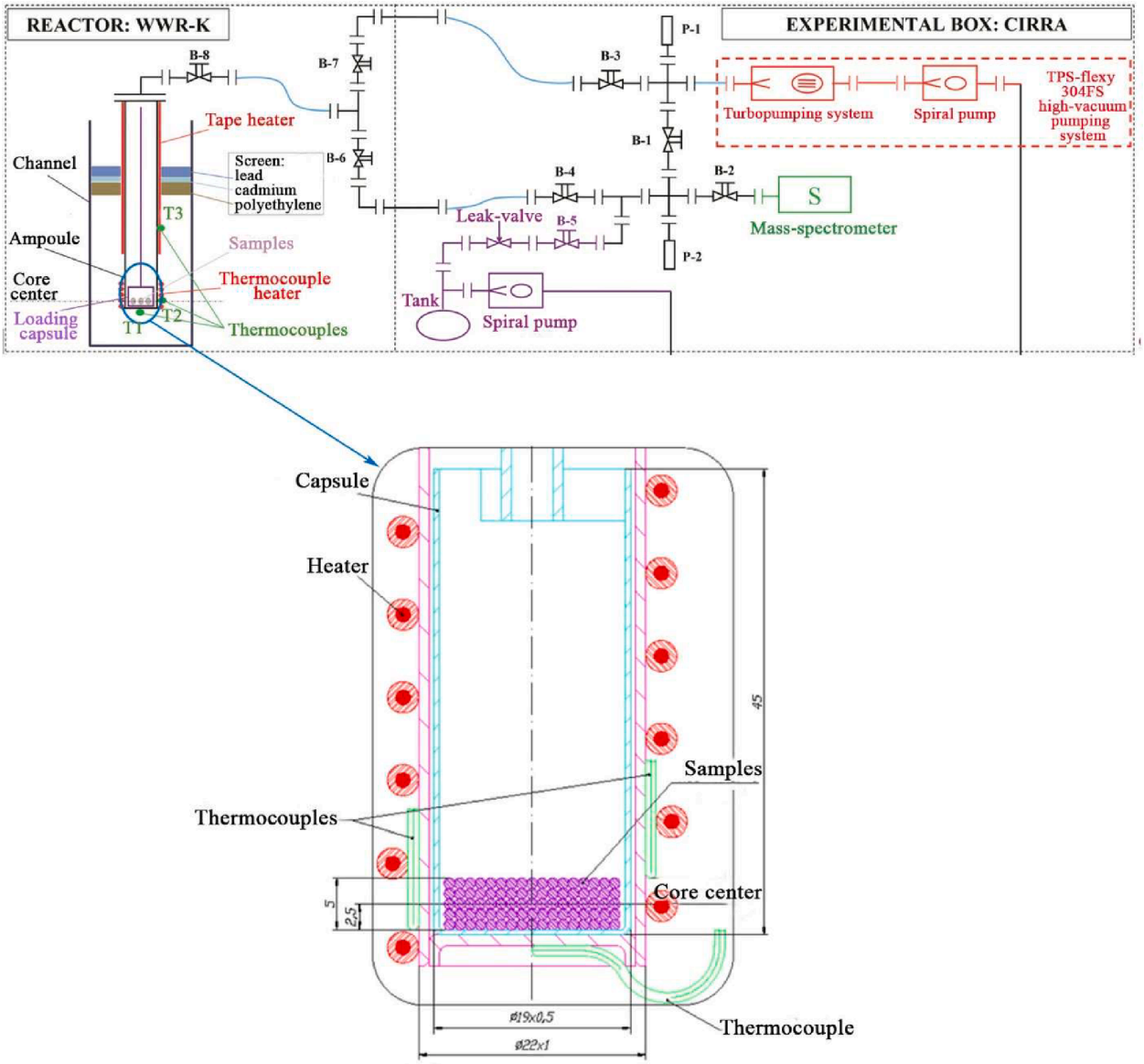

Fig. 2. Diagram of the CIRRA experimental facility and scheme of the ampoule device.

Stages II (Fig. 7) and IV are the regions with no change in experimental parameters: the reactor runs at constant power of 6 MW, the ampoule temperature is 680 °C. A decrease in the release of hydrogen, CO, and H_2O was observed. There is also a noticeable relative increase in T_2 pressure and a slight decrease in molecular pressure of mass number M4. In this case, the gases with mass number M4 may be helium and HT. For gases with mass number M4, the gas evolution occurs in the form of frequent peaks. The authors suppose that these emissions are determined only by He, which leaves the inner cavities of the ceramic when free paths appear.

Further, there is stage III of the experiment with several heating/cooling cycles. Heating the AD was performed using a heater (Fig. 4) up to temperature shelves from 720 to 790 °C (Fig. 8). A substantial increase in hydrogen release and a decrease in T_2 molecule release was observed during heating. An increase of M4 (helium and HT) was also observed, and it is assumed to be due to the increase of samples temperature and because tritium begins to interact with hydrogen on the samples surface, forming HT molecule.

Stage V is the region of the second experiment with heating (Fig. 9). Here, the same trends that were observed in the first experiment with sample heating are practically repeated. A noticeable increase of hydrogen yield during heating and a simultaneous significant drop in the peak of the T_2 molecule is observed. That occurs because some tritium atoms on the surface bind to the hydrogen atom, so there is an increase in the pressure of M4 mass gases – namely, HT.

Then, a series of experiments with gas supply into the chamber was conducted (Figs. 10 and 11). Gas from tank V through leak-valve L (Fig. 3) was supplied into the capsule with the test sample. In the process of hydrogen supply (Fig. 10), the T_2 molecule peak decreases. The authors believe, that this fact is due to release of tritium in the form of HT molecule. It is slightly visible in the figure due to emissions of He, but the average pressure value qualitatively shows the growth of M4 peak. In this case, the hydrogen pressure in the chamber was increased without temperature change.

The experiment with deuterium supply (Fig. 11) into the chamber displays some particularities. It was difficult to determine the pressure changes for the molecule HT and He, as the supply of deuterium to the chamber was followed by an increase in the peak for gases with M4 mass, which was mainly determined by the supplied deuterium. Also, supplying the chamber with deuterium resulted in an additional M6 peak caused by the ionized D_3 molecule complex (this 6-mass peak is about 1% of the M4 peak (caused by deuterium). So, only the increase in

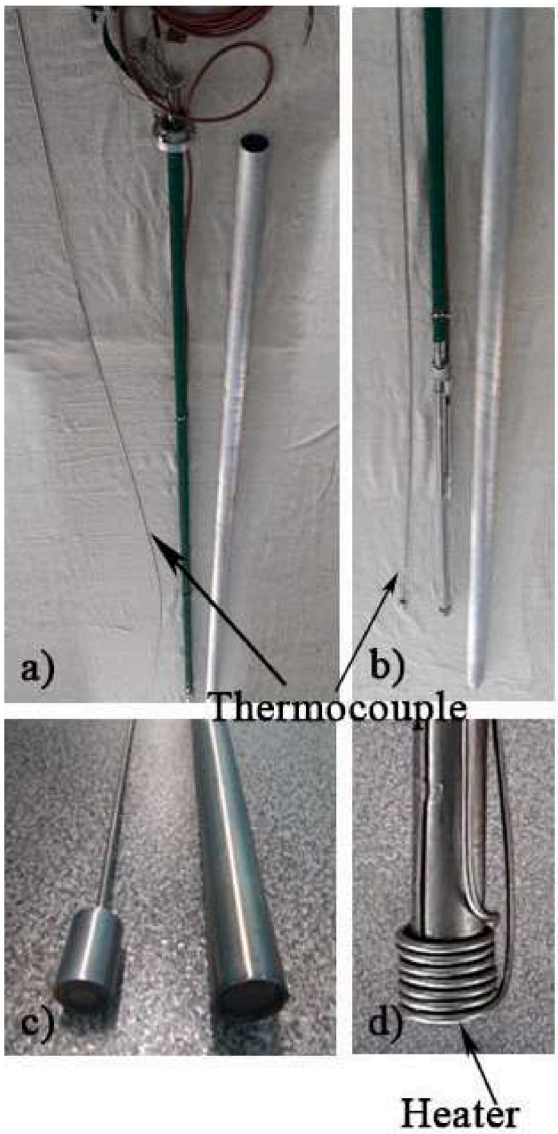

Fig. 3. View of the irradiation device: a – thermocouple, the upper part of the AD (green) and a tube of the reactor channel, b – bottom part of the AD, c – loading capsule and AD, d – AD with mounted heater and thermocouple. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

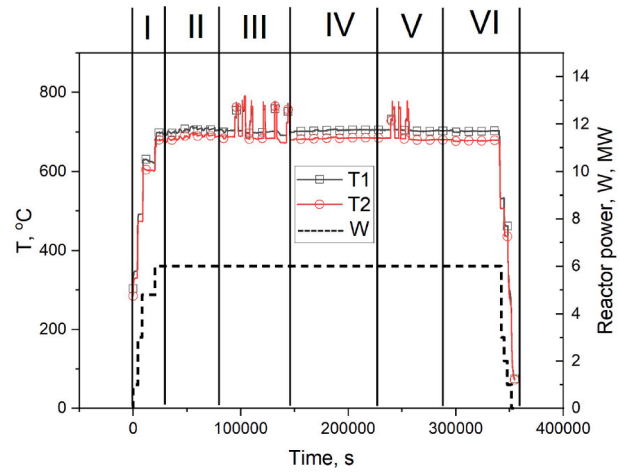

Fig. 4. The diagram of the experiment for the entire irradiation time.

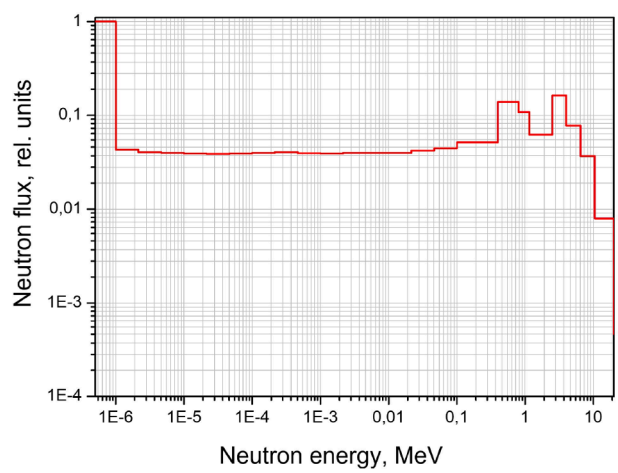

Fig. 5. Neutron spectrum of the WWR-K reactor.

the M5 peak responsible for the DT molecule can be analyzed.

Part VI (Fig. 12) is the second region without changes in experimental parameters of irradiation and the region with a stepwise decrease in reactor power. The values of the steady-state power levels at the step-down were 3, 2, and 1 MW. Accordingly, we also observed a decrease in the temperature of the sample. At that time, the M4 mass decreases much faster (when the power decreases from 6 to 3 MW) than T_2. And the authors associate this with a significant drop in the hydrogen pressure in the chamber. At this hydrogen pressure, the peak values for gases with masses M4 and M6 become comparable.

The total release of tritium in experiments can be estimated as the sum of HT + 2 T_2 + HTO. It should be noted that after 2 days of irradiation, this value practically does not change.

As for the release of tritium in the form of HTO, there is a certain relationship between the magnitude of the M20 peak and the absolute values of the pressure of water and hydrogen in the chamber (their ratio is also important). The general increase in M20 peal is determined by HTO, while its sharp changes in experiments with deuterium supply are not associated with the vapor pressure of HTO (but with the pressure of heavy water D_2O, which was present in the supplied deuterium). It is also worth noting that from the second day of irradiation, the M20 peak itself is noticeably less than M6 (and, from the point of view of tritium release, it is at least two times less significant, since T_2 has 2 tritium atoms). One way or another, the release of tritium in the form of tritiated water at the end of the irradiation is about 5 %.

4. Conclusions

The dependences of the gas composition change in the chamber with two-phase lithium ceramics during its irradiation in the WWR-K reactor are obtained.

For all stages of experiments (including heating/cooling cycles, supply of hydrogen and deuterium into the chamber, changes in the reactor power), the features of the tritium-containing molecules and helium release from the test sample were considered.

As a general conclusion it can be said that tritium is released mainly in the form of HT, T_2 and HTO molecules. The total release of tritium can be estimated as the sum HT + 2 T_2 + HTO, and after ~2 day of irradiation this value practically does not change. At the end of irradiation, the fractions of released tritium in the form of HT, T_2 and HTO molecules are 0.65, 0.3 and 0.05, respectively. The ratio of the number of released tritium atoms in the form of HT, T_2 and HTO depends on the concentrations of tritium, hydrogen atoms and adsorbed water molecules on the surface. The release of helium has the character of release pulses

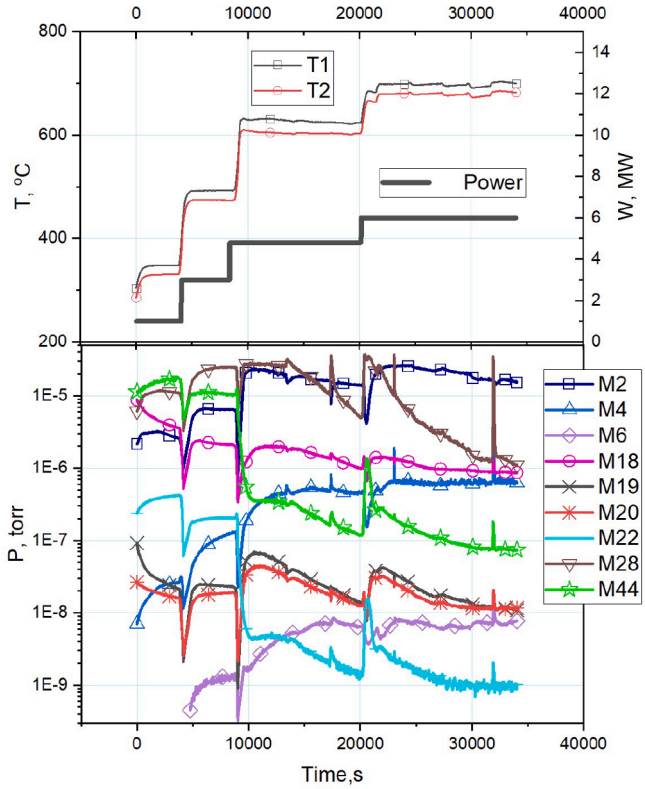

Fig. 6. Initial heating of the sample (Stage I).

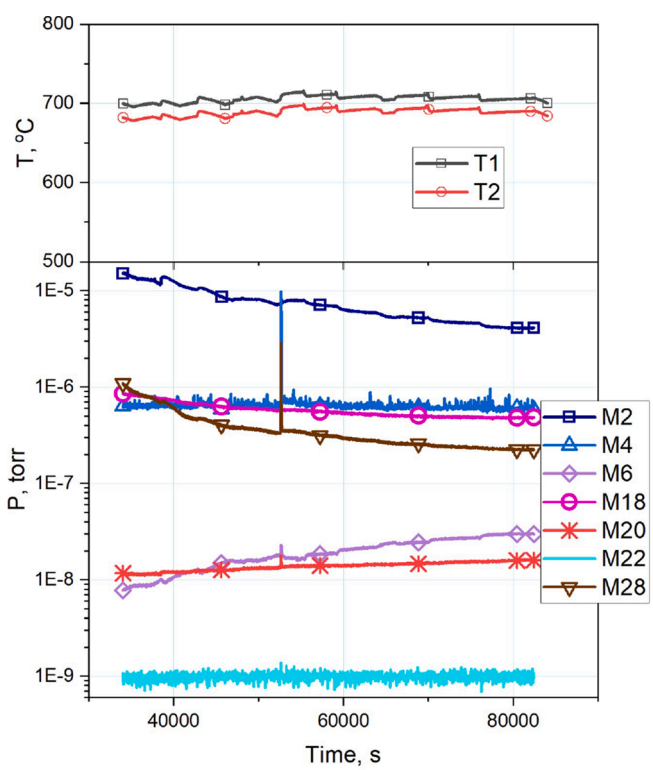

Fig. 7. Stage II of the experiment without change of the parameters.

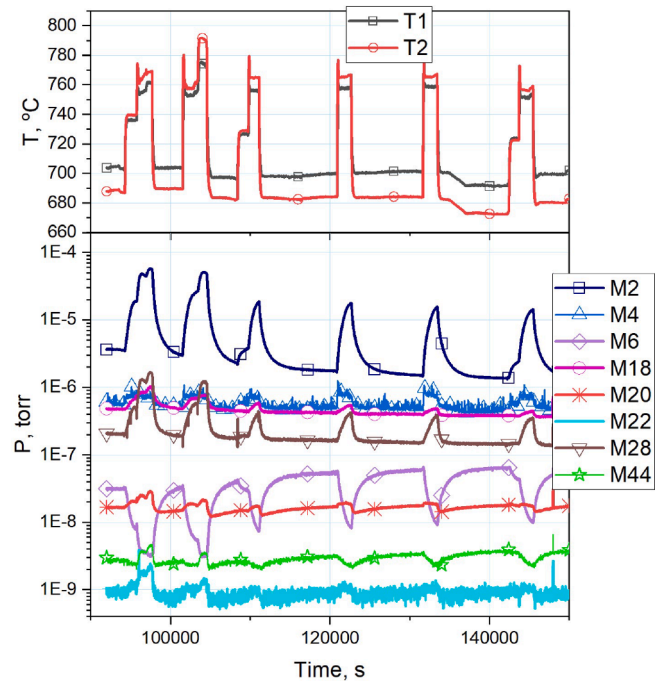

Fig. 8. Stage III of the experiment with cyclic heating/cooling of the sample.

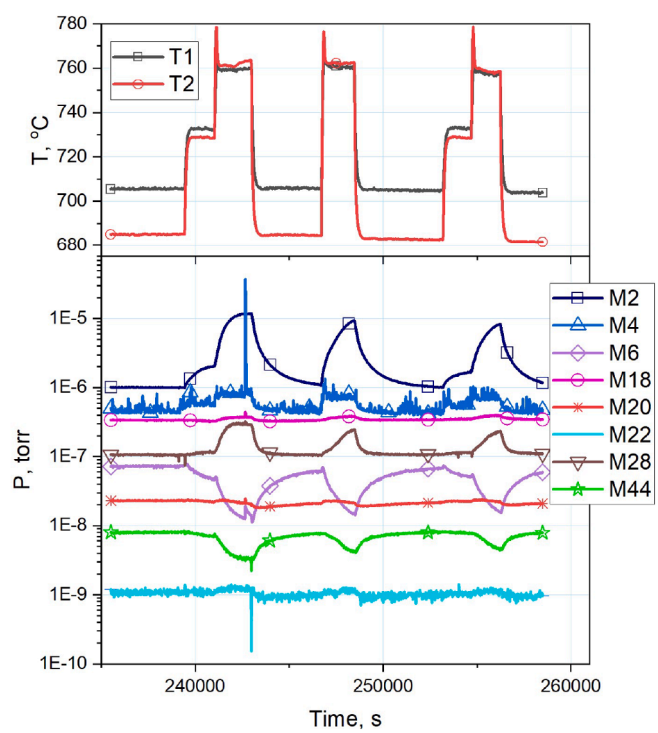

Fig. 9. Stage V of the experiment with heating/cooling cycles.

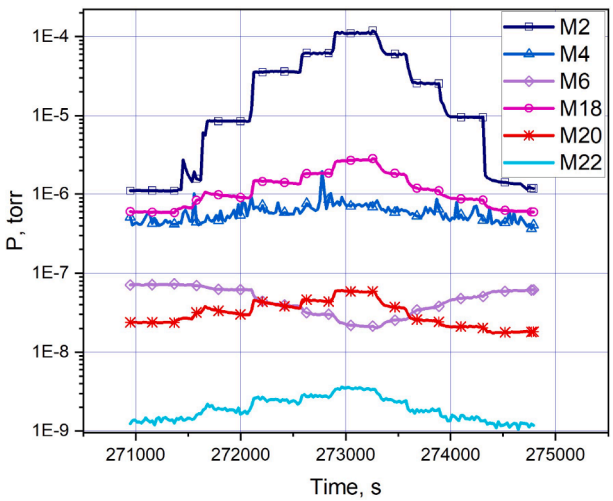

Fig. 10. Stage V of the experiment with hydrogen supply into the chamber.

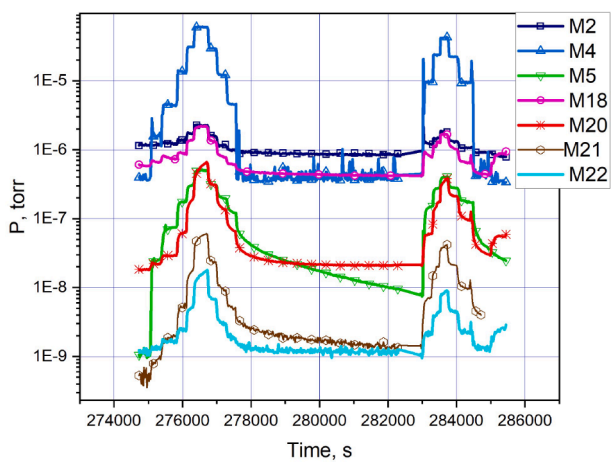

Fig. 11. Stage V of the experiment with deuterium supply into the chamber.

(peaks) and increases in amplitude with irradiation time.

The kinetics of changes in the ratio of tritium-containing molecules and the release of helium from the samples depends on the experimental conditions and the total irradiation time.

According to estimates, the amount of residual tritium should be less than 1% of the released one, but it can be correctly determined only in the post-irradiation experiments, which are planned to be carried out in future.

The results obtained for each irradiation stage are planned to be analyzed in more detail in future publications. This will make it possible to determine the parameters of tritium and helium production and release from ceramics under irradiation conditions.

CRediT authorship contribution statement

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

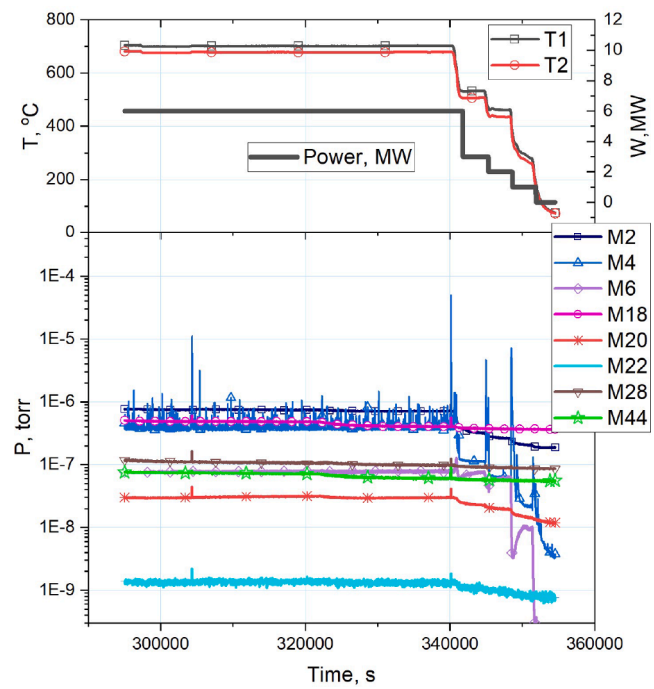

Fig. 12. Stage VI of the experiment with reactor power decrease.

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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