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IRRADIATION EXPERIMENTS WITH Li_2TiO_3/Li_4SiO_4 TWO-PHASE LITHIUM CERAMICS AT THE WWR-K REACTOR

Advanced two-phase lithium ceramics Li_2TiO_3-Li_4SiO_4 are considered as a potential candidate for use in solid-state fusion reactor blankets. This phase composition makes it possible to combine the high lithium content in orthosilicate Li_4SiO_4 with the mechanical strength of lithium metatitanate Li_2TiO_3.

The study of the effect of neutron irradiation on two-phase lithium ceramics, as well as the correlation with its structural and physical properties, remains quite relevant, taking into account the technological diversity in the manufacture of the ceramics themselves and ceramic pebbles. This paper presents the results of an experiment on reactor irradiation of two-phase ceramics 25mol% Li_2TiO_3 + 75mol% Li_4SiO_4 obtained on the KALOS installation (KArlsruhe Lithium OrthoSilicate) using a modified melt-spraying technique. Neutron irradiation was carried out at the WWR-K research reactor (Almaty, Kazakhstan) for 21 days at a sample temperature of 50 °C. The accumulated fluence for thermal neutrons was $3.7\cdot10^{19}$ n/cm^2. The nuclear reaction rate was $2.16\cdot10^{13}$ reactions/cm^3·s; the damage rate was $7.63\cdot10^{-8}$ dpa/s, the damage during the irradiation period was 0.14 dpa.

Keywords: two-phase lithium ceramics, neutron irradiation, tritium, tritium release.

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ВВР-К реакторында екіфазалы литий керамикасын Li_2TiO_3/Li_4SiO_4 сәулелендіру бойынша эксперименттер

Жетілдірілген екіфазалы литий керамикасы Li_2TiO_3 - Li_4SiO_4 термоядролық реакторлардың қатты бланкеттерінде қолдануға перспективалы үміткер ретінде қарастырылады. Мұндай фазалық құрам Li_4SiO_4 ортосиликатының жоғары литий мөлшерін Li_2TiO_3 литий метатитанатының механикалық беріктігімен үйлестіруге мүмкіндік береді.

Екіфазалы литий керамикасына нейтрондық сәулелендірудің әсерін зерттеу, сондай-ақ оның құрылымдық және физикалық қасиеттерімен корреляциясы өзекті мәселе болып қала беруде, өйткені керамиканың және керамикалық сфералық түйіршіктердің өндіріс технологиялары әртүрлі. Бұл ұсынылған жұмыста балқыманы бүрку әдісі бойынша модификацияланған технологиямен KALOS (KArlsruhe Lithium OrthoSilicate) қондырғысында алынған Li_2TiO_3 + 75 моль % Li_4SiO_4 екіфазалы керамикасына реакторлық сәулелендіру экспериментінің нәтижелері келтірілген. Сондай-ақ, Нейтрондық сәулелендіру зерттеу реакторы ВВР-К-де (Алматы қ., Қазақстан Республикасы) 21 күн бойы 50 °С температурада жүргізілді. Жиынтық жылулық нейтрон флюенсі $3.7\cdot10^{19}$ н/см² құрады. Ядролық реакциялардың жылдамдығы $2.16\cdot10^{13}$ реакция/см³·с, ал атомдардың ығысу жылдамдығы $7.63\cdot10^{-8}$

dpa/c (бір атомға шаққандағы ығысу саны) деңгейіне жетті. Сәулелендіру кезеңіндегі жиынтық сәулелік зақым 0.14 dpa құрады.

Түйін сөздер: екіфазалы литий керамикасы, нейтрондық сәулелендіру, тритий, тритий бөлінуі.

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Эксперименты по облучению двухфазной литиевой керамики Li_2TiO_3/Li_4SiO_4 на реакторе ВВР-К

Усовершенствованная двухфазная литиевая керамика Li_2TiO_3-Li_4SiO_4 рассматривается как потенциальный кандидат для использования в твердотельных бланкетах термоядерных реакторов. Такой фазовый состав позволяет сочетать высокое содержание лития в ортосиликате Li_4SiO_4 с механической прочностью метатитаната лития Li_2TiO_3.

Исследование воздействия нейтронного облучения на двухфазную литиевую керамику, а также корреляция с ее структурными и физическими свойствами, остается актуальной задачей, учитывая технологическое разнообразие методов изготовления самой керамики и керамических сферических гранул. В данной работе представлены результаты экспериментального исследования по реакторному облучению двухфазной керамики Li_2TiO_3 + 75моль% Li_4SiO_4, полученной на установке KALOS (KArlsruhe Lithium OrthoSilicate) по модифицированной технологии распыления расплава. Нейтронное облучение проводилось в исследовательском реакторе ВВР-К (г. Алматы, Казахстан) в течение 21 дня при температуре образцов 50 °C. Накопленный флюенс тепловых нейтронов составил $3.7 \cdot 10^{19}$ н/см². Показано, что скорость ядерных реакций достигала $2.16 \cdot 10^{13}$ реакций/см³·с; скорость накопления повреждений $7.63 \cdot 10^{-8}$ dpa/c (смещения на атом в секунду), а суммарное повреждение за период облучения составило 0.14 dpa.

Ключевые слова: двухфазная литиевая керамика, нейтронное облучение, тритий, выделение трития.

Introduction

Irradiation experiments with two-phase lithium ceramics Li_2TiO_3-Li_4SiO_4 are attracting the attention of researchers in the field of nuclear/fusion energy and materials science. This material is considered as a potential candidate for use as solid fuel in advanced nuclear reactors, as well as a blanket material for use in thermonuclear reactors.

In the field of thermonuclear energy, the use of ceramic materials for the reproduction of tritium in the blanket of a thermonuclear installation is a strategically important area of research. Due to their unique thermophysical and thermomechanical characteristics, as well as high efficiency of tritium formation and release, lithium-containing ceramics are becoming a central element for the implementation of fusion reactor blanket concepts. Among the many material options, such as Li_2TiO_3, Li_4SiO_4, Li_2O, Li_2ZrO_3 and Li_2AlO_2, composite materials that combine the positive qualities of several chemical compositions are attracting particular interest from researchers.

In a number of studies carried out in this direction, great attention is paid to studying the properties of ceramic pebbles, which are potential candidates for use as a blanket material for the future DEMO reactor [1,2]. The work of M. Xiang et al [3] stands out for its claim that two-phase composites consisting of Li_2TiO_3-Li_4SiO_4 pebbles, where the second phase is dispersed in a Li_2TiO_3 matrix, exhibit noticeable differences in grain structure compared to single-phase materials. The noted granularity of the composite is a key factor, given that grain size directly influences the tritium release properties of the ceramic material. Thus, increasing the yield of tritium from pebbles can be a decisive factor in optimizing tritium production and increasing the efficiency of a thermonuclear reactor blanket.

This aspect becomes the basis for further research and experiments aimed at determining the optimal characteristics of the blanket material and expanding the understanding of the mechanisms of tritium formation, release and transfer in ceramic materials. It is in this context that the study of the effect of neutron irradiation on two-phase lithium ceramics, as well as the correlation with its structural and physical properties, comes to the fore, providing the basis for the development and improvement of technologies in the field of fusion.

To date, a significant amount of research has been conducted on two-phase lithium ceramics under reactor conditions [4-9], including research conducted by authors of this article [10-13]. Most of these studies were performed *in situ*. These studies gave an understanding of how important it is to know the structural changes occurring in the ceramics themselves for understanding the processes of tritium production and release.

At the same time, accurate detection of tritium and helium release under reactor irradiation conditions requires carefully validated methodological approaches, as outlined in [14]. Equally crucial is the consideration of thermomechanical behavior at elevated temperatures, since significant stresses may arise in tightly packed pebble beds and the walls of steel capsules [15]. These factors must be taken into account when planning further high-temperature experiments with lithium ceramics.

This article provides a description of the first stage of the study, namely a description of the methodology of the already completed irradiation experiment, with the necessary calculated data on the irradiation parameters. This article is useful for further analysis (PIE), and, in the experience of the authors, is necessary for comparative assessments with the results of other reactor irradiation experiments of lithium ceramics. The methodological conditions of an experiment, especially such a multifactorial one as a reactor irradiation experiment, often affect some PIE results and are necessary for adequate interpretation of the results of future studies of irradiated lithium ceramics.

Methodology of irradiation experiments at the WWR-K reactor

The WWR-K reactor, as a source of neutron radiation, provides unique opportunities for conducting irradiation experiments [16]. Lithium ceramic samples were irradiated in the reactor core to study the following parameters:

• Physical and mechanical properties: Changes in mechanical properties, including hardness, strength and resistance to cracking due to radiation, as well as physical properties such as density, and changes in dimensional characteristics, which may vary due to exposure to neutron radiation.

• Properties of tritium generation: Study of the dynamics of generation, accumulation and transfer of tritium in a material, providing a deep understanding of the mechanisms occurring under the influence of various types of radiation and temperature load.

The authors envisage the preparation of a number of additional publications, each of which will reveal the nuances of the above research points.

The main parameters of the conducted reactor experiment:

• Thermal neutron flux density in lithium ceramics is $2 \cdot 10^{13}$ n/(cm^2·s);

• Duration of irradiation 21.5 effective full power days;

• The temperature of the samples during irradiation did not exceed 50 °C;

• Reactor power 6 MW;

• The accumulated fluence for thermal neutrons is $3.7 \cdot 10^{19}$ n/cm^2.

The samples were placed in the center of the reactor core as shown in Figure 1.

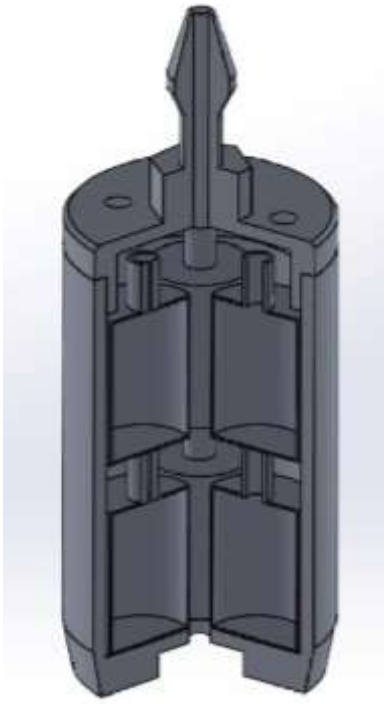

Figure 1 – Three dimensions view of irradiation device

The irradiation device (ID) is a WWR-K's standard irradiation ampoule, inside of which there are capsules with samples of different types of lithium ceramics (Figure 1). The capsules are cylindrical cases with a height of 30 mm, a diameter of 20 mm, and a wall thickness of 1 mm. It is worth noting that

before being placed in the appropriate capsules, the samples were wrapped in primary packaging made of aluminum foil. The capsules inside the ampoule are arranged in such a way that three capsules with samples (No. 1, No. 2, No. 3) are located in the lower part of the cell, and the rest (No. 4, No. 5, No. 6) are in the upper part. Capsules with samples are filled with argon and sealed, the capsules themselves and the ampoule are cooled with water. The standard ampoule is located in the irradiation channel of the WWR-K core. The ampoule and capsules are made of SAV-1 aluminum alloy. The irradiation channel itself is located on the periphery of the WWR-K core and is surrounded by beryllium blocks.

The test samples

In the presented article, the emphasis is on the results with samples of two-phase lithium ceramics, namely 25mol%LMT+75mol%LOS (capsule No. 1), produced by specialists from KIT (Karlsruhe Institute of Technology). Here LMT indicates Li_2TiO_3 – lithium metatitanate, and LOS stands for Li_4SiO_4 – lithium orthosilicate.

The selection of such a phase composition combines the valuable characteristics of both ceramic phases: the susceptibility and lithium density from LOS, taking into account its dominant contribution of 75mol%, and the mechanical strength, chemical and thermal stability from LMT, with its share of 25mol%. This unique combination provides significant advantages in terms of radiation resistance and tritium production efficiency.

The physical characteristics are listed in Table 1, which also shows the atomic concentrations of various elements in the samples.

Table 1 – Main sample's parameters

Formula	25 mol% Li_2TiO_3 + 75 mol% Li_4SiO_4
Atomic concentrations of elements, 10^{-3} at/(barn·cm)	*Li-6*: 1.51, *Li-7*: 18.61, *Ti*: 6.03, *O*: 26.15, *Si*: 2.01
Pebbles diameter, microns	250-1250
Average pebble bed height, mm	0.7061
Density (pebble), g/cm^3	2.836
Density (pebble bed), g/cm^3	1.5

Neutronic calculations

Based on a detailed model of the WWR-K reactor core and the irradiation device with samples, and using the MCNP code for modeling particle transport, an accurate prediction was obtained for the formation and accumulation of nuclear reaction products in the studied lithium ceramic samples.

The main channels of nuclear reactions occurring with lithium are as follows:

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

$$^7\mathrm{Li} + \mathrm{n} \rightarrow \mathrm{T} + {}^4\mathrm{He} + \mathrm{n}' - 2.47\ \mathrm{MeV} \quad (2)$$

As calculations have shown, due to the high cross section of reaction (1) and the predominantly thermal spectrum of neutrons in the reactor [17], tritium and helium are generated with a very noticeable intensity.

The production of tritium and helium in lithium ceramics by the end of irradiation was calculated. In particular, the recorded amount of produced tritium is 0.37 Ci, which is equivalent to 0.039 mg, and the amount of helium reaches 0.051 mg. These data are important for understanding the dynamics of processes occurring under the influence of neutron radiation. The reaction rates in lithium ceramics are also determined, which is determined by (3) formula (see Table 2).

Reaction rate Q [reaction/(cm^3·s)]:

$$Q = n_0 \int \Phi(E)\sigma(E)dE, \quad (3)$$

where n_0 is the nuclear concentration of the original isotope [nucleus/cm^3]; Φ(E) – neutron flux density [n/(cm^2·s)] and σ(E) – microscopic cross section of the reaction [barn].

Table 2 – ^{6}Li (n,α)T reaction rate

Sample volume, cm^3	Pebble density, g/cm^3	Reaction rate, reaction/(cm^3·s)
0.20	2.84	$2.16 \cdot 10^{13}$

The results of calculations of the total heat release and specific heat release from neutrons and gamma radiation (including delayed gamma) for the device elements are shown in Table 3. The height of the registration zone (cylinder) of the capsule and the canister was 5 mm.

For calculations of radiation damage in lithium ceramics, cross sections of ceramic breeders [18,19] were used, provided in a predefined structure of 100 energy groups for discrete levels of ^{6}Li enrichment.

Table 3 – Total heat release and specific heat release of device elements with a sample of lithium ceramics

Material	Total heat, W	Specific heat, W/g
Sample (pebble bed)	3.6	12.09
Aluminum alloy (capsule)	0.130	0.31
Aluminum alloy (ampoule)	1.8	0.31

First, the neutron flux densities in the samples were calculated for the 100-group energy spectrum. Then, using formula (4), the rate of damage and dpa for entire irradiation period at the WWR-K reactor was calculated

$$\frac{R}{N} = DPA/\sec = \int_0^{E_{max}} \Phi(E_i)\sigma_D(E_i)dE_i, \qquad (4)$$

where R – the rate of damage [dpa/(m^3·s)], N – atomic density [atom/m^3], σ_D – cross section of atom displacement, $\Phi(E_i)$ – flux distribution on energy.

The obtained calculation results were:
- Damage rate –$7.63 \cdot 10^{-8}$ dpa/s
- Damage per 21 days – 0.14 dpa

Thermophysical calculations

To verify the absence of a critical thermal load on the samples, it was necessary to determine the temperature distributions throughout the one-layer pebble bed. This was performed using numerical calculations of the temperature distribution throughout the volume of the working chamber, carried out in accordance with the previously described geometry. The calculation was carried out for a 3-dimensional model shown in Figure 2. The internal part of the ID (canister, capsules with samples and argon, water washing the canister and capsules) in Figure 2 is shown on the right for clarity.

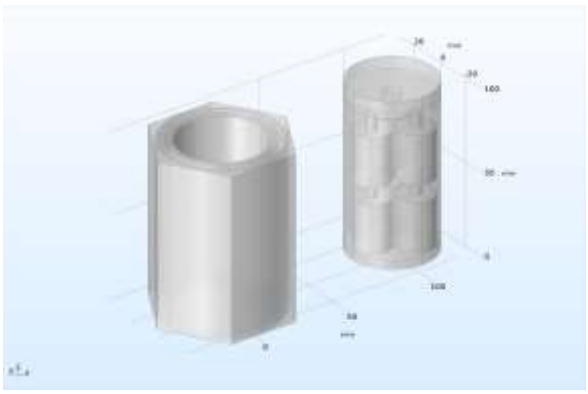

Figure 2 – 3D model of irradiation device

Initial and boundary conditions

The initial temperature of all domains of the model is 40 °C. There is no heat exchange from the ends of the canister (top and bottom). The temperature of the outer surface of the hexagon and the incoming water is constrained to 40 °C.

Ceramics samples are located at the bottom of the capsules in one layer 0.7 mm thick. Capsules with samples are filled with argon at 1 atm and sealed. Water at a temperature of 40 °C is supplied to the canister water cooling cavities (shown in blue in Figure 3) from top to bottom at a speed of 0.1 m/sec. It is also assumed that the temperature of the outer surface of the irradiation channel (aluminum alloy hexagon) is constant and equal to 40 °C.

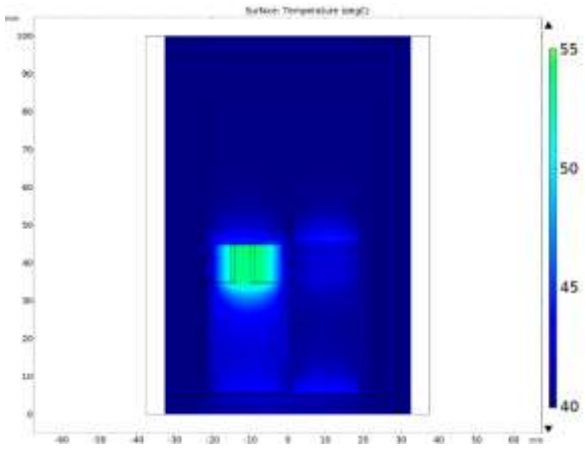

Figure 3 – Temperature distribution in capsules (capsule №1 is in lower left part)

The calculation was carried out in a stationary mode.

The governing equations in the constructed model for calculating heat distribution were:

Equation of heat transfer between gas and surfaces of the sample and capsule wall:

$$\rho C_P \vec{u} \cdot \nabla T = \nabla \cdot (k \nabla T) + Q, \qquad (5)$$

where C_p – heat capacity at constant pressure, J/(kg·K); Q – total specific heat of the material, W/m^3.

Heat transfer by radiation was not taken into account due to the low temperature of the samples and the irradiation device.

Standard temperature-dependent parameters for gases and materials were used for the calculation.

Calculation results

The results of calculated temperature distribution over the sample and in the cavity of the capsule №1 are presented in the colors in Figure 3.

Table 4 – Radial temperature distribution in pebble bed (capsule №1)

Radius, mm	Temperature, °C
0	41.5
3	43.1
6	43.6
9	43.4
12	43.1
15	42.2
18	41.1

The calculation results showed that for a given irradiation configuration and number of samples, the maximum heating temperature in the area of the capsule with samples will not exceed 55 °C. In this case, the maximum spread (gradient) of temperature along the radius of pebble bed will be no more than 2.5 °C.

Initial samples Irradiated samples

Figure 4 – Comparison of samples before and after irradiation

Post-irradiation characterization

After the tests, samples were removed from the capsules and primary packaging, subsequently sorted into pebble-shaped and powdery fractions, as well as separate placement of the primary foil packaging. Since it is planned to conduct experiments with aluminum foil using liquid scintillation to determine residual tritium in it, the primary packaging was retained for additional studies.

Conclusions

The results of neutron-physical calculations are presented: the amount of produced tritium and helium from lithium ceramics with a volume of 0.20 cm^3 were estimated as 0.039 mg and 0.051 respectively; the rate of ^6Li (n,α)T reaction was $2.16 \cdot 10^{13}$ reactions/cm^3·s; the damage rate was $7.63 \cdot 10^{-8}$ dpa/s, total damage per irradiation period was 0.14 dpa.

The results of thermophysical calculations revealed that with the considered irradiation configuration and a given number of samples, the peak temperature along the capsule with samples will not exceed 55 °C, while the maximum temperature gradient along the sample filling will be limited to 2.5 °C.

Experiments with two-phase lithium ceramics Li_2TiO_3-Li_4SiO_4, carried out at the WWR-K reactor, provide unique data on the interaction and behavior of samples under the influence of radiation load.

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