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Effect of irradiation temperature on the mobility of structural and vacancy defects in the damaged layer of Li_2ZrO_3 ceramics

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ABSTRACT

The paper presents the assessment results of the irradiation temperature effect on the change in the type of structural defects caused by irradiation with helium ions in the near-surface layer of Li_2ZrO_3 ceramics, as well as determining the nature of structural damage using the electron paramagnetic resonance method in the case of irradiation fluence variation. During characterization of the structural changes caused by helium ion irradiation, the main attention was paid to detailing the type of structural defects and radiolysis products arising during irradiation, alongside alterations in their concentration using the electron paramagnetic resonance method. During the experiments, it was determined that the observed effects of thermally stimulated mobility of vacancy defects in the damaged layer caused by irradiation with helium ions indicate a positive effect of thermal heating of samples during irradiation at low fluences. This effect consists in a decline in the number of oxygen vacancies in the damaged layer at low irradiation fluences (10^{15}–10^{16} cm^{-2}), the concentration of which was determined using the EPR method, and the observed structural changes at low irradiation fluences in the case of an elevation in irradiation temperatures are associated with deformation distortion of the crystalline structure of ceramics caused by transformation energy processes, and thermal expansion effects.

1. Introduction

Interest in alternative methods of obtaining energy, in particular, using nuclear and thermonuclear energy methods, is due to a number of reasons, the key ones of which include the annual reduction of fossil resources used to obtain energy in order to close the deficit in energy consumption, the deterioration of the environmental situation due to harmful emissions leading to climate change, felt on a global scale, the development of technological processes associated with the global development of all industries [1–3]. At the same time, the energy shortage observed at the moment will only increase in the near future due to the expansion of consumption, in connection with which much attention in the energy sector is paid to the search for new ways of obtaining energy, which are based on technologies for obtaining energy using nuclear and, in the foreseeable future, thermonuclear reactors, work on which has been actively underway for several years (among the most significant projects in the field of thermonuclear energy, one can single out the ITER project, the construction of which began in 2010) [4–6]. It should be noted that much attention in the development of the use of alternative methods of energy production is given to technological solutions related to new types of fuel, the use of which will increase the efficiency of energy production, reduce the amount of long-lived nuclear waste, and also increase energy output [7–9]. In the case of thermonuclear reactors, much attention is currently being paid to finding materials capable of producing tritium, which is the basis for maintaining thermonuclear fusion reactions [10,11]. The problem of tritium production in this case is one of the limiting factors, since a fairly large amount of tritium is required to maintain the stability of a thermonuclear reactor, while the technological solutions available today do not allow for all the needs for tritium, as a result of which much attention has been paid in recent years to technologies for creating materials in which, as a result of nuclear reactions, tritium can be produced directly

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in the reactor core, thereby ensuring a continuous flow of tritium to maintain thermonuclear reactions [12–15]. The key materials in this direction, with which the most promising solutions to the problem of tritium production are associated, are lithium ceramics based on zirconates, orthosilicates or titanates, in which there is a sufficiently large amount of lithium used to produce tritium, the production of which occurs as a result of nuclear reactions of lithium with neutrons [16,17]. At the same time, the processes of tritium production in lithium-containing ceramics as a result of nuclear reactions are accompanied by the accumulation of nuclear reaction products in the form of helium and hydrogen, the accumulation of which in the near-surface layer can have a negative impact on the structure of the ceramics, the result of which can be the destruction of the near-surface layers as a result of swelling and the formation of blisters (gas-filled inclusions) [18–20].

The main objective of this study is to determine the defect formation mechanisms in the near-surface layer caused by high-dose irradiation of lithium-containing ceramics with helium ions, and to establish the effect of irradiation temperature variations on the stability of defect formation processes. Li_2ZrO_3 ceramics with high hardness and crack resistance [21,22], high corrosion resistance during interaction with aggressive environments [23,24], and low thermal expansion during exposure to high temperatures for a long time were selected as objects of study. This type of ceramics is considered as one of the candidate materials to replace traditional Li_4SiO_4 ceramics used for tritium breeding [25], alongside to create two-phase ceramics [26] with high resistance to external influences, especially to radiation damage and the accumulation of products of nuclear reactions and radiolysis processes initiated as a result of long-term operation modes under radiation exposure. It should also be noted that Li_2ZrO_3, unlike other types of ceramics, has fairly good compatibility with other types of structural materials, which allows them to be combined with various materials, including those used in the active zone. Low thermal expansion of Li_2ZrO_3 ceramics allows them to be used in high-temperature conditions, as well as long-term thermal exposure. It should be noted that much attention is currently being paid to the study of the defect formation mechanisms in ceramics, especially in cases of high-dose irradiation, in which not only single defects arise in the structure of the damaged layer, but also processes of formation of complexes in the form of gas-vacancy defects ($He – V_O$) are initiated, the accumulation of which results in appearance of blisters in the near-surface layer due to the agglomeration of defects in the pores. In turn, during simulation of the radiation damage processes in ceramics, in order to obtain data on the degradation kinetics of materials under conditions as close as possible to real operating conditions, attention should be paid to high-temperature operating conditions, which, as a rule, are accompanied by processes of diffusion of defects and vacancies, which results in acceleration of defect formation processes, alongside associated structural distortions and deformations. In this case, the structural damage accumulation mechanisms caused by irradiation at elevated temperatures are usually accompanied by diffusion processes caused by thermal action, which in turn leads to their more intensive agglomeration in voids and subsequent growth in the volume of blisters. In the case of small doses, the mobility of point and vacancy defects can result in acceleration of defect annihilation processes or the reverse effect of accumulation of deformation distortions associated with distortion of chemical bonds due to the transformation of the ionization loss energy of interacting ions from kinetic to thermal, which in the case of high-temperature exposure can result in amplified distortions due to alterations in the amplitude of thermal oscillations.

2. Materials and methods

Li_2ZrO_3 ceramics were synthesized using the method of mechanochemical grinding of $LiClO_4 \times 3H_2O$ and ZrO_2 components in an equal molar ratio, followed by thermal sintering of the ground mixtures in a muffle furnace at a temperature of 1200 °C for 5 h, followed by cooling together with the furnace. As a result of using these synthesis conditions, it was possible to obtain ceramics with a highly ordered phase of Li_2ZrO_3 with a monoclinic phase. The phase composition of the studied ceramics was determined using the X-ray phase analysis method, on the basis of which the values of the parameters of the crystal lattice, as well as its volume, were obtained. The choice of synthesis conditions is determined by the possibilities of obtaining single-phase, well-structured ceramics. Before thermal annealing, the samples were pressed into tablets with a diameter of 10 mm and a thickness of about 1 mm, which were subsequently used for experiments on irradiation and simulation of defect formation processes associated with the formation of oxygen vacancies, as well as gas-vacancy complexes.

Simulation of the kinetics of accumulation of structural defects and radiolysis products in the near-surface layer of ceramics was carried out by irradiation of ceramics with helium ions (He^{2+}) with an energy of about 40 keV in a wide range of irradiation fluences from 10^{15} to 5×10^{17} cm^{-2}, the choice of which is due to the possibility of forming large concentrations of structural defects in the structure of the damaged layer, which under certain conditions are capable of initiating blister formation processes due to the agglomeration of complex defects and radiolysis products in the near-surface layer. Irradiation was carried out at four different temperatures (300 K, 500 K, 700 K and 1000 K), temperature control was carried out using a special system of thermocouples placed on the heater and the sample on both sides, which allows for high-precision control of the sample heating degree during irradiation. The samples were heated in a vacuum on a special target holder; after experiments were conducted to set radiation doses, the resulting samples were cooled together with the target holder for 10–15 h in a vacuum until room temperature was reached. The choice of irradiation temperatures is determined by the possibility of modeling radiation damage processes under conditions as close as possible to the actual operating conditions of this type of ceramics when they are used as materials for tritium breeding. The samples were irradiated using the DC-60 accelerator. To irradiate the samples in order to initiate the destruction processes and to avoid oxidation processes, the samples were irradiated in a vacuum. The use of helium ions for irradiation was chosen to simulate the processes of structural changes characteristic of the processes of accumulation of nuclear reaction products in the near-surface layers, and due to the weak solubility of helium ions in the near-surface layer, the formation of gas-filled inclusions in the form of blisters in the near-surface layer. In turn, an alteration in the irradiation temperature can result in elevation in the intensity of diffusion of the resulting structural defects in the damaged layer, due to a change in the amplitude of thermal vibrations and thermal expansion of the crystalline structure of ceramics.

The establishment of dependencies of the change in the degree of structural disorder caused by deformation swelling of the crystalline structure during irradiation was carried out using the method of X-ray diffraction of the studied samples associated with the determination of the kinetics of changes in the parameters of the crystal lattice depending on the atomic displacement value (dpa). The magnitude of atomic displacements was estimated by modeling the processes of interaction of incident ions, as well as the formation of vacancy defects, leading to structural changes associated with atomic displacements. Modeling was carried out in the SRIM Pro 2013 software code.

Determination of the concentration of defects caused by irradiation depending on the irradiation conditions (in the case of variation in fluence and irradiation temperature), alongside their type with a change in irradiation fluence, was carried out using the electron paramagnetic resonance (EPR) method, based on the determination of paramagnetic effects arising as a result of the formation of point and vacancy defects, alongside their evolution associated with their accumulation and subsequent agglomeration [27].

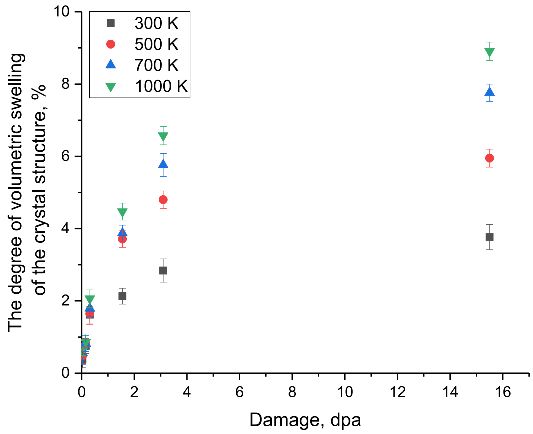

Fig. 1. Comparative analysis results of the assessment of the degree of structural disorder associated with deformation swelling of the crystal lattice as a result of irradiation with He^{2+} ions depending on the irradiation conditions.

3. Results and discussion

Fig. 1 reveals the assessment results of the change in the structural disorder degree associated with deformation swelling of the ceramics crystal lattice, associated with the effects of radiation exposure on the crystal structure of the damaged layer. The magnitude of volumetric swelling was estimated by comparative analysis of changes in the structural parameters of irradiated samples with the data of the original samples; based on the comparison results, the magnitudes of changes in the volume of the crystal lattice associated with deformation swelling were determined. These dependencies were constructed based on alterations in the crystal lattice parameters at the irradiation fluence growth in comparison with the data of the initial sample, not subjected to irradiation. The general appearance of the presented dependencies of certain changes in the structural disorder degree has an exponential trend with clearly distinguishable differences in the region of high irradiation fluences (when the value of atomic displacements exceeds 1 dpa). In the case of small fluences (in this context meaning fluences of 10^{15}–10^{16} cm^{-2}), the main alterations in the structural parameters depending on the irradiation temperature have minor differences, which in this case can be caused by a change in the amplitude of the crystal lattice oscillations as a result of thermal expansion, the value of which, according to estimates, is about 1–3 × 10^{-6} K^{-1} depending on the exposure temperature in the case of non-irradiated samples. Thus, the deformation distortion of the crystal lattice at low fluences (10^{15}–10^{16} cm^{-2}) has a weakly expressed dependence on temperature, and the swelling value itself is no more than 0.5–1.8 %, which is within the limits of permissible changes in structural parameters. The irradiation fluence growth above 10^{16} cm^{-2} leads to the formation of clearly expressed differences in the trends of the change in the structural disorder degree, which has a direct dependence on the irradiation temperature. In the case of irradiation at a temperature of 300 K, the maximum change in the crystal structure disorder value is about 2.8–3.7 % at maximum irradiation fluences, while with an elevation in the irradiation temperature, similar changes in structural parameters are more than 1.5–2.5 times higher. In this case, an increase in the irradiation temperature leads to an acceleration of diffusion processes, which, in the case of high concentrations of defective inclusions in the damaged layer, lead to an acceleration of the processes of destabilization of the damaged layer, which in turn leads to swelling and accelerated deformation.

In this case, the disordering mechanisms may be effects caused by the acceleration of diffusion of structural defects arising as a result of irradiation and their subsequent agglomeration, which leads to the formation of not only He – V_O type complexes, but also more complex defects, in particular, radiolysis products, the accumulation of which can result in accelerated destruction of the damaged layer. However, using standard analysis methods (microstructural or X-ray methods), it is impossible to directly determine what type of defects are formed in the damaged layer and have the greatest impact on destruction and disordering. For these purposes, the most optimal method of research is EPR spectroscopy, the use of which with high accuracy allows to determine not only the kinetics of accumulation of structural defects, but also changes in their concentration in the damaged layer, which allows to determine more accurately the mechanisms of structural disorder and their role in changing the properties of ceramics subject to high temperature irradiation.

Fig. 2 demonstrates the results of EPR spectroscopy of the studied ceramic samples depending on the conditions of irradiation with He^{2+} ions (in this case, the results are presented depending on the irradiation fluence at different irradiation temperatures). Based on the presented alterations in the EPR spectra depending on the irradiation conditions (in this case, both factors are considered: irradiation temperature and fluence), it is possible to distinguish three clearly expressed types of changes associated with the formation of E-centers, characterizing the formation of oxygen vacancies (V_O) in the structure, gas-vacancy complexes of the He – V_O type, arising at high irradiation fluences, and Zr^{3+} defects, characteristic of the formation of radiolysis products in the damaged layer. At the same time, the dynamics of alterations in the intensities of the singlet line characteristic of E-centers, as well as spectral bands for two other types of defects He – V_O and Zr^{3+} defects, depending on the irradiation temperature during comparison of the results at the same irradiation fluences, indicates a significant influence of the temperature factor on the concentration dependences and defect formation mechanisms in the damaged layer. According to the data obtained, in the case of high-temperature irradiation at fluences above 10^{16} cm^{-2}, an elevation in the intensities of spectral lines characteristic of He – V_O and Zr^{3+} defects is observed in the EPR spectra, which indicates a more pronounced structural deformation caused by thermally induced diffusion of oxygen vacancies in the damaged layer.

A comparative analysis of the kinetics of changes in defect concentration was carried out by determination of the concentrations of defects of various types in the damaged layer structure, based on the amplitudes of the spectral lines characteristic of each type of detected structural defects. The assessment results are presented in Fig. 3 as dependencies of the change in the concentration of the E center (associated with oxygen vacancies), gas-vacancy complexes and Zr^{3+} - defects characteristic of radiolysis products, on the value of atomic displacements associated with the irradiation fluence.

In the presented dependences of alterations in defect concentration depending on the value of atomic displacements, two clearly distinguishable areas, characterized by different types of defects, and a change in their concentration in the damaged layer, can be distinguished. With damage less than 1 dpa, an accumulation of oxygen vacancies (E-centers) occurs in the structure of the damaged layer, the change in the concentration of which has a clearly expressed temperature dependence. With a rise in irradiation temperature, according to the data presented, a reduction in the concentration of E-centers in the damaged layer in the case of comparison at the same irradiation fluence values is observed. This difference may be due to the fact that at low irradiation fluences, the effect of irradiation temperature affects the migration of oxygen vacancies, a change in the rate of which at low structural damage can lead to partial annihilation of oxygen vacancies, or the formation of gas-vacancy complexes of the He – V_O type, which at low fluences are isolated from each other and cannot be registered. The dominance of the mechanisms of formation of these complexes at lower irradiation fluences is supported by the results of the concentration dependences of defects obtained at an irradiation temperature of 1000 K, for which the registration of peaks characteristic of gas-vacancy complexes is observed

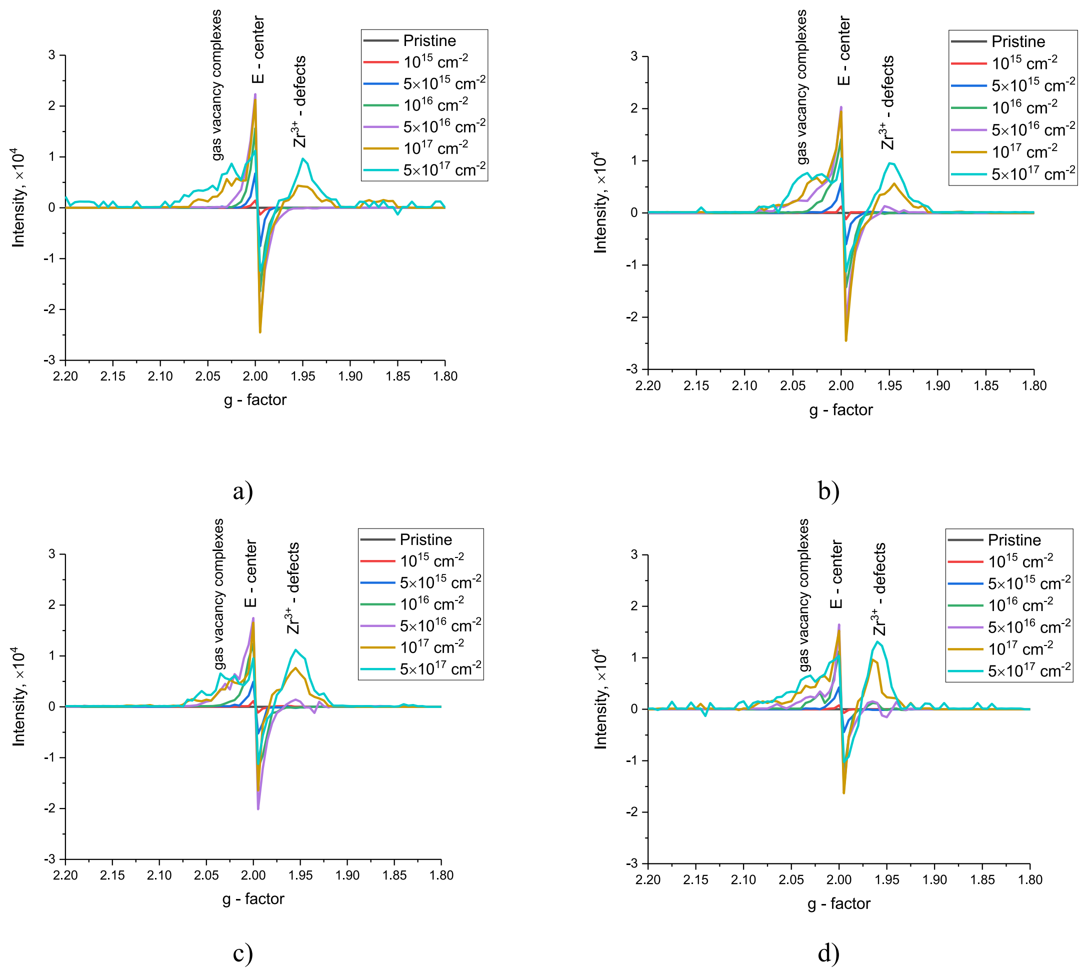

Fig. 2. Results of EPR spectra of the studied ceramics contingent upon irradiation conditions: a) irradiation at T = 300 K; b) irradiation at T = 500 K; c) irradiation at T = 700 K; d) irradiation at T = 1000 K.

at a fluence of about 10^{16} cm^{-2}, which indicates the acceleration of defect formation processes associated with the formation of complex defects, and radiolysis products in the case of high-temperature irradiation. Thermally stimulated processes of diffusion of oxygen vacancies and implanted helium in the case of high irradiation temperatures lead to a more pronounced process of formation of complex defects of the He-V_O type, with the possibility of their subsequent agglomeration, which was confirmed in several works [28–31] related to the mechanisms of blister formation and gas swelling in ceramics. Moreover, the observed decline in the concentration of oxygen vacancies for samples irradiated at temperatures of 700–1000 K in the case of fluences above 5 × 10^{17} cm^{-2} may be associated with their combination into He-V_O complexes, the density of which grows, according to the EPR spectra (see data in Figs. 2 and 3). In this case, the observed alteration in the structural disorder degree (see data in Fig. 1), given for samples irradiated at high temperatures, can be explained by the dominance in the structure of the damaged layer of structural defects associated with the formation of He–V_O, as well as radiolysis products in the form of Zr^{3+} defects, the concentration of which is also significantly higher at high fluences in the case of samples irradiated at temperatures of 500–1000 K in comparison with the evaluation results obtained for samples irradiated at a temperature of 300 K.

Based on the obtained dependencies, it can be concluded that in the case of high-temperature irradiation at low fluences (10^{15} - 10^{16} cm^{-2}), for which the characteristic accumulation of structural defects is less than 1 dpa, the dominant role is played by thermal effects, leading to an elevation in the mobility of oxygen vacancies and point defects, some of which are capable of annihilating or relaxing due to thermal exposure. In the case of high-dose irradiation, the processes of thermally stimulated diffusion of oxygen vacancies and implanted defects result in acceleration of deformation disordering processes due to the formation of gas-filled inclusions in the structure, alongside the accumulation of radiolysis products, which is accompanied by the rupture of crystalline and chemical bonds in the structure of ceramics. In the case of irradiation at a temperature of 300 K, the main processes of defect formation during high-dose irradiation are associated with the formation of complexes of the He – V_O type, the concentration of which is significantly lower than during high-dose irradiation, and the main contribution to deformation distortion is made by oxygen vacancies.

4. Conclusion

Based on the experiments conducted, the following conclusions were formulated:

Analysis of alterations in the structural parameters of Li_2ZrO_3 ceramics depending on the irradiation conditions revealed that the main changes associated with the occurrence of deformation distortions in the structure as a result of the interaction of incident ions with the crystal structure and the associated energy processes of transformation of kinetic energy into thermal energy, in the case of small fluences (10^{15}–10^{16} cm^{-2}) with a change in the irradiation temperature have small differences associated mainly with the thermal expansion of the crystal structure. Moreover, in the case of irradiation fluences of 5 × 10^{16}–5 × 10^{17} cm^{-2}, the observed differences in the structural

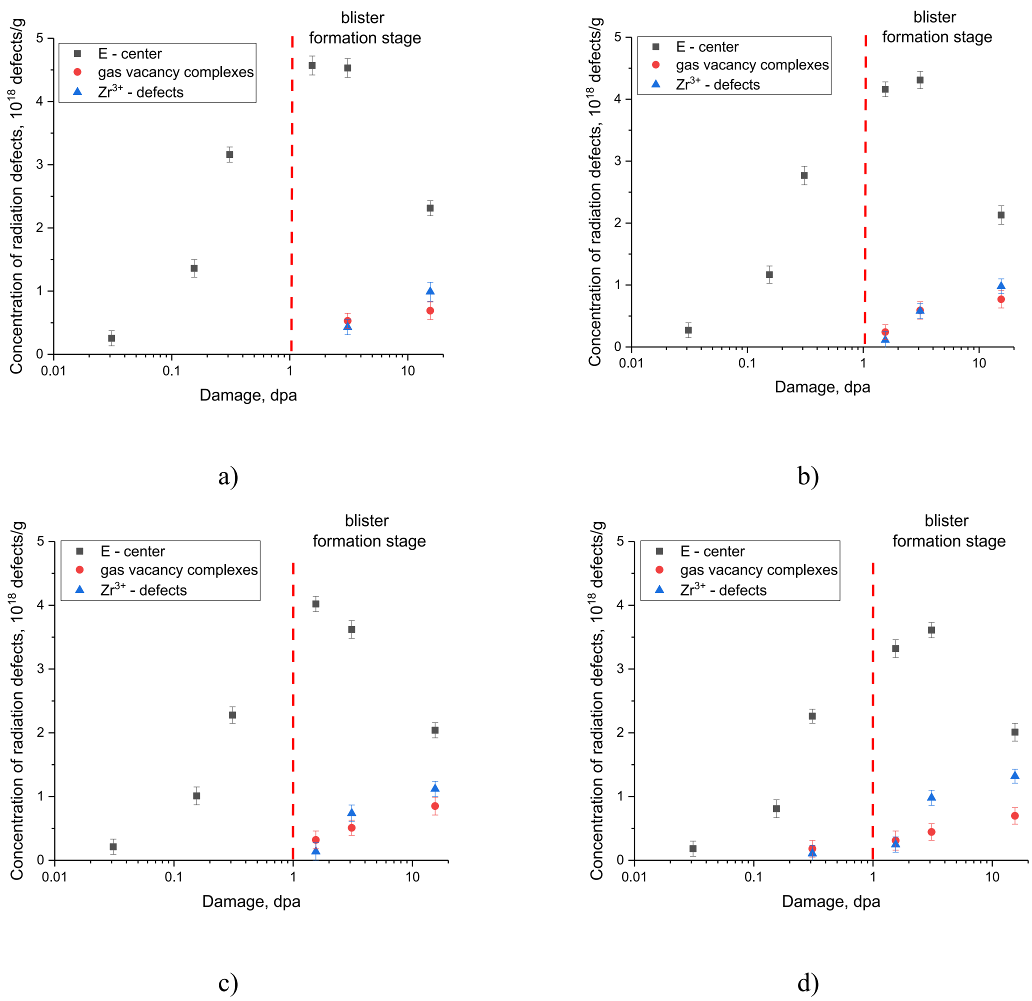

Fig. 3. Assessment results of the change in the concentration of defects in the structure of the damaged ceramic layer depending on the variation of irradiation conditions (The dotted line in the figure highlights the area characteristic of high-dose irradiation, which causes clusters of defects to form, leading to swelling and degradation): a) irradiation at T = 300 K; b) irradiation at T = 500 K; c) irradiation at T = 700 K; d) irradiation at T = 1000 K.

degradation degree of the crystal structure have a clearly expressed dependence on the irradiation temperature, a growth in which results in large deformations and swelling of the crystal lattice, caused by the diffusion of He-V_O complexes in the damaged layer and their agglomeration.

According to EPR spectroscopy data, it was established that in the case of irradiation fluences of 10^{15}–5×10^{16} cm^{-2}, the main contribution to the change in structural parameters is made by oxygen vacancies, the concentration of which has a clearly expressed dependence on the irradiation temperature, an elevation in which leads to a decrease in the concentration of oxygen vacancies, a reduction in which in this case can be due to an alteration in the rate of their diffusion in the damaged layer, which can result in relaxation.

It was found that in the case of high temperatures, the concentration of gas-vacancy complexes, as well as Zr^{3+} defects in the structure of the damaged layer is higher than at lower irradiation temperatures, which may be due to a rise in the mobility of implanted helium at high temperatures, which leads to the formation of the He – V_O type complexes, with their subsequent agglomeration and filling of porous voids in the damaged layer.

According to the obtained results, it can be concluded that during consideration of lithium-containing ceramics as materials for tritium propagation, during assessment of the prospects for their use, it is necessary to take into account the operating conditions, and thermally stimulated processes of helium diffusion and oxygen vacancies, which at high temperatures are capable of forming He-V_O complexes, the accumulation of which results in structural disorder degree growth.

CRediT authorship contribution statement

Bauyrzhan K. Abyshev: Writing – review & editing, Writing – original draft, Visualization, Resources, Methodology, Funding acquisition, Data curation, Conceptualization. **Sholpan G. Giniyatova:** Software, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Artem L. Kozlovskiy:** Writing – original draft, Visualization, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization.

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

No data was used for the research described in the article.

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