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Study of thermally induced diffusion mechanisms of implanted helium in the near-surface layers of ceramics based on lithium metazirconate

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Abstract. The aim of this study is to determine the thermal exposure effect on the destruction processes of of near-surface layers of ceramics based on lithium metazirconate associated with the diffusion of implanted helium, alongside to determine the effect of the magnesium oxide dopant in low concentrations on restraining diffusion mechanisms during high-temperature irradiation. Interest in this type of ceramics is primarily due to the possibility of expanding the types of lithium-containing ceramics for the production and accumulation of tritium, as well as an increase in stability to degradation processes characteristic of high-temperature operating modes, alongside the accumulation of fission products of nuclear reactions of neutrons with lithium in the form of helium. According to studies carried out using the depth nanoindentation method (measurements along the trajectory of ion movement on a side cleavage), it was found that an elevation in the irradiation temperature leads to a rise in the thickness of the damaged layer, the alteration in which is due to the effects of helium diffusion into depth due to thermal effects. The softening of the near-surface layer during high-temperature irradiation is due to structural changes associated with a growth in deformation distortion of the crystal structure and its swelling, which is most pronounced during high-dose irradiation. At the same time, alterations in the structural features of lithium metazirconate due to the dopant concentration growth during the synthesis of lithium-containing ceramics leads not only to an increase in resistance to softening of the damaged layer, but also to a reduction in the thickness of the diffusion layer in ceramics under high-temperature irradiation.

Keywords: helium diffusion; lithium-containing ceramics; blankets; radiation damage; helium swelling

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1. Introduction

Current trends in the development of alternative energy sources in order to reduce the dependence of the energy sector on fossil fuels cannot be considered without nuclear, hydrogen and, in the near future, thermonuclear energy [1-3]. Moreover, while development trends in nuclear energy are aimed at finding opportunities to enhance the efficiency of nuclear fuel burnup, and to increase the temperature of the core, the main efforts in hydrogen energy are aimed at finding technological solutions to reduce the cost of energy production by reducing the operating temperatures of fuel cells or simplifying hydrogen production processes, in thermonuclear energy, in addition to finding solutions to problems associated with plasma confinement, research aimed at finding ways to produce tritium plays an important role [4,5]. In this case, tritium is one of the key types of fuel for thermonuclear power plants, and the methods available today for its production do not allow us to fully meet the needs for its volumes.

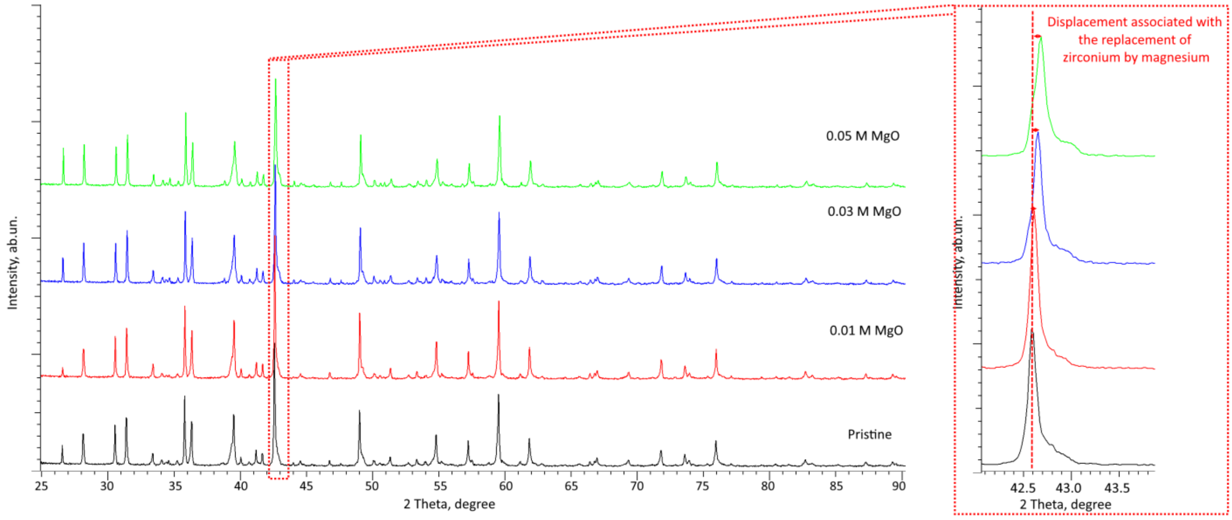
One of the solutions to the problem of tritium production in thermonuclear energy is the use of lithium-containing ceramics as blankets for the propagation of tritium, which is an essential fuel for maintaining thermonuclear reactions [6,7]. The technology of using lithium-containing ceramics for tritium propagation is based on the nuclear reaction of lithium with a neutron (Li(n,t)He), which results in the formation of tritium and helium (He) as a fission product, the accumulation of which also occurs in the blanket material [8,9]. At the same time, the existing technical groundwork in the field of producing lithium-containing ceramics opens up wide opportunities for varying the compositions of ceramics by using various compounds, the variation of which causes changes in the structural, strength and thermophysical parameters [10-13]. Among the fairly large number of variations in the compositions of lithium-containing ceramics, lithium metazirconate has recently been distinguished, which has fairly high resistance to external influences, such as mechanical pressure, thermal expansion, etc., and also has good compatibility with other types of structural materials [14-16].

Deformation processes in the near-surface layer are usually caused by the accumulation of nuclear reaction products in the form of helium and hydrogen, which accumulate in pores and voids, thereby creating additional deformation distortions in the structure. It should also be noted that migrating helium, due to its mobility and low solubility, is capable of forming clusters together with oxygen vacancies, which also contributes to a growth in the deformation of the crystalline structure of the near-surface layer. At the same time, according to a number of fundamental works [17-19], aimed at studying the mechanisms of gas swelling of near-surface layers in steels and alloys (traditional structural materials in nuclear energy), temperature exposure contributes to the acceleration of helium diffusion processes in the near-surface layer [11], which results in more pronounced agglomeration into bubbles, and as a result, an elevation in the destruction effect. In the case of ceramic materials, these effects have not been fully studied, which opens up opportunities for their detailing, in particular, the need to study the effects of destruction that occur during the simultaneous impact of high temperatures and radiation damage on the near-surface layer of ceramic materials exposed to irradiation. The key differences between such effects in steels and alloys and ceramics consist in the structural features of the ceramics, and in most cases, the dielectric nature of the ceramics, which leads to the need to consider ionization effects associated with the redistribution of electron density, as well as the subsequent athermal effects and the emergence of metastable states following these changes [20,21].

The aim of this study is to determine the effect of magnesium oxide addition in small concentrations (0.01 – 0.05 M) to the composition of ceramics based on lithium metazirconate (Li_2ZrO_3) on the resistance to the accumulation of deformation distortions and softening (decrease in hardness) of the near-surface layer of ceramics when irradiated with He^{2+} ions. At the same time, the emphasis in the study of radiation damage is on determination of the influence of irradiation temperature on diffusion processes caused by the high mobility of He^{2+} ions in the near-surface layer [11]. Conducting such studies is aimed at expanding the understanding of the mechanisms of degradation of near-surface layers of ceramics, which during operation are subject to external influences (mechanical, thermal effects during heating), alongside due to the high mobility of nuclear reaction products n(Li,t)He, accumulation of helium, tritium and hydrogen in the near-surface layer, which results in destabilization of the structure and, as a consequence, softening. The choice of Li_2ZrO_3 ceramics with the addition of a low concentration of MgO as objects of study is due to the possibility of modification of the properties of lithium metazirconate, without the formation of impurity phases in the composition in the form of a $MgLi_{2Z}rO_4$ substitution phase, which contributes to the preservation of the structural motif of the Li_2ZrO_3 phase, with changed parameters, as well as heightened resistance to destruction.

2. Materials and methods

Ceramics based on lithium metazirconate with the addition of magnesium oxide in low concentrations were chosen as objects of study. The ceramics were obtained using solid-phase grinding technology and subsequent thermal sintering. Grinding was carried out in a planetary mill PULVERISETTE 6 classic line, at a grinding speed of 400 rpm for 30 minutes. After grinding, the main purpose of which was to obtain a mixture of homogeneous composition, the resulting powders were pressed into tablets with a diameter of 10 mm and a thickness of 1 mm, which were subsequently subjected to thermal annealing for 8 hours at a temperature of 1300 °C. A RUS-universal muffle furnace was used to sinter the samples. To obtain ceramics based on lithium metazirconate, $LiClO_4 \times 3H_2O$, ZrO powders were used in a molar ratio of 1 to 1, which, under the selected synthesis conditions, made it possible to obtain ceramics with a monoclinic Li_2ZrO_3 phase (see data presented in Figure 1). MgO was added to the ceramic composition at the grinding stage in molar fractions of 0.01, 0.03 and 0.05 M. The choice of these concentrations is based on the results of [22], in which it was revealed that the MgO dopant concentration growth above 0.1 M results in the formation of two-phase ceramics, in which, under the selected thermal annealing conditions, the formation of a tetragonal $MgLi_2ZrO_4$ phase occurs, the formation of which leads to an alteration in strength characteristics. In this case, the use of low MgO concentrations results in the formation of single-phase ceramics, and the main structural changes are associated with the effect of partial substitution of zirconium by magnesium, which results in the crystal lattice parameters and volume reduction, the data of which are presented in the table on an inset 1b. Such changes in parameters are due to the difference in the ionic radii of Mg^{2+} (74 pm) and Zr^{4+} (79 pm) (see the inset in Figure 1, reflecting the change in the position of the reflection associated with the substitution effect, as well as the parameter data in Inset 1b).



a)

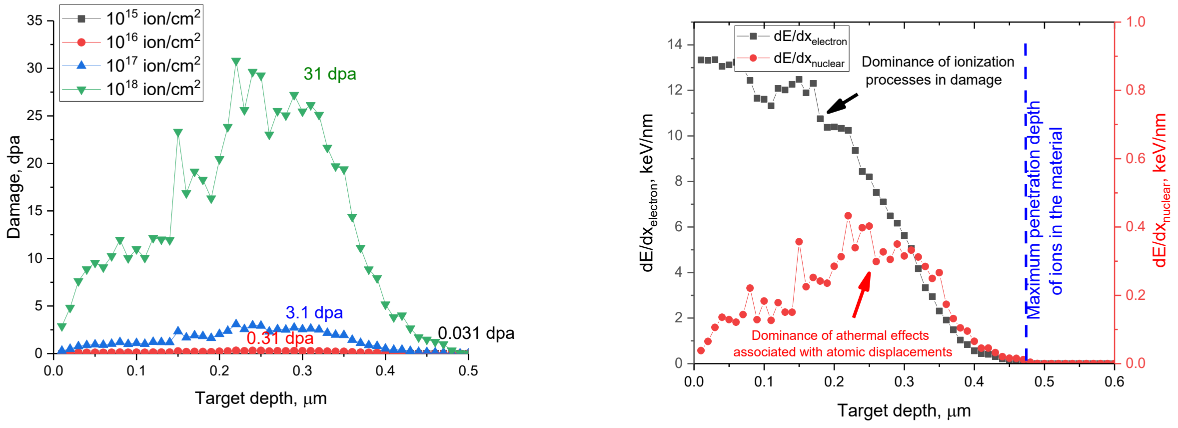
Lattice parameter	MgO concentration, M			
	0	0.01 M	0.03 M	0.05 M
Li_2ZrO_3 (PDF-00-033-0843), Monoclinic, C2/c(15)	a=5.40839 Å, b=8.90597 Å, c=5.39074 Å, β=111.704° V=241.25 Å^3	a=5.40678 Å, b=8.90153 Å, c=5.39564 Å, β=111.732° V=241.23 Å^3	a=5.40251 Å, b=8.89464 Å, c=5.39743 Å, β=111.737° V=240.92 Å^3	a=5.39758 Å, b=8.89757 Å, c=5.39826 Å, β=111.707° V=240.87 Å^3

b)

Figure 1. a) Results of X-ray diffraction of the studied Li_2ZrO_3 ceramics depending on the MgO concentration (the inset shows a comparison of the alteration in the position of the reflection at 2θ = 42.7°, reflecting the substitution effect of zirconium by magnesium); b) Data on crystal lattice parameters depending on variations in MgO concentration during the manufacture of ceramics

Simulation of helium accumulation processes in the near-surface layer of ceramics with a thickness of about 300-500 nm was carried out by irradiating the samples under study with He^{2+} ions at the DC – 60 accelerator (Institute of Nuclear Physics of the Ministry of Energy of the Republic of Kazakhstan), the use of which, as was shown in the works [23,24], allows one to simulate the near-surface layer degradation processes in a wide range of irradiation fluences and, as a consequence, atomic displacement values. Irradiation was carried out at fluences from 10^{15} to 10^{18} cm^{-2}, which, according to estimates, corresponds to about 30 dpa at the maximum irradiation fluence (see evaluation data presented in Figure 2). The samples were irradiated at different temperatures, the particle flux density was about 40 μA. Temperature control was performed using a special target holder that allowed maintaining a constant target temperature during irradiation.

As can be seen from the presented calculated data, the value of atomic displacements of the order of 30 dpa can be achieved at an irradiation fluence of 10^{18} cm^{-2}, while the maximum of this value is observed at a depth of about 250-300 nm, with a maximum ion travel depth of about 500 nm. It should be noted that due to the high resistance to external influences of the selected ceramics, as well as the low ion flux density, the effect of possible sputtering under ion exposure was excluded in the process of simulation of the helium ion accumulation in the near-surface layer. Figure 2b demonstrates the estimation results of the value of ionization energy losses of incident ions during interaction with the crystal structure of the target, according to which, over most of the path length, the main contribution to changes is made by ionization losses during interaction with electron shells (ionization effects), and at a depth above 250 nm, ionization losses associated with collisions with nuclei play a role, which leads to the formation of primary knocked out atoms in the structure if the transferred energy exceeds the binding energy.



a) b)

Figure 2. Simulation results of the interaction of incident He2+ ions with a near-surface layer of ceramics: a) evaluation results of the distribution of atomic displacements (dpa) in the near-surface layer in depth depending on the irradiation fluence; b) results of the distribution of changes in ionization losses during interaction with electrons (dE/dxelectron) and nuclei (dE/dxnuclear)

The irradiation temperatures were chosen to be 300, 500, 700 and 1000 K, which made it possible to evaluate the influence of thermal effects caused by alterations in the amplitude of thermal vibrations of atoms in the crystal lattice, as well as acceleration of the diffusion processes of implanted He^{2+} ions in the near-surface layer, which can lead to both more pronounced agglomeration of helium in voids with a subsequent rise in deformation distortions, and an elevation in the penetration depth of He^{2+} ions due to diffusion deep into the sample [25,26].

The effect of irradiation and changes in irradiation conditions (temperature variations) on the structural characteristics of ceramics was determined by assessing changes in the volumetric swelling value ($\Delta V = V_{irradiated} - V_{pristine}$), which was determined by calculating the crystal lattice volumes in the initial state (for non-irradiated samples) and after accumulation of a certain radiation dose. In this case, the value of ΔV(dpa) reflects the destructive swelling of the crystal lattice, caused by both deformation mechanisms associated with the accumulation of structural distortions caused by irradiation, and the introduction of He^{2+} ions into voids, which is accompanied by the formation of gas-filled inclusions in the near-surface layer.

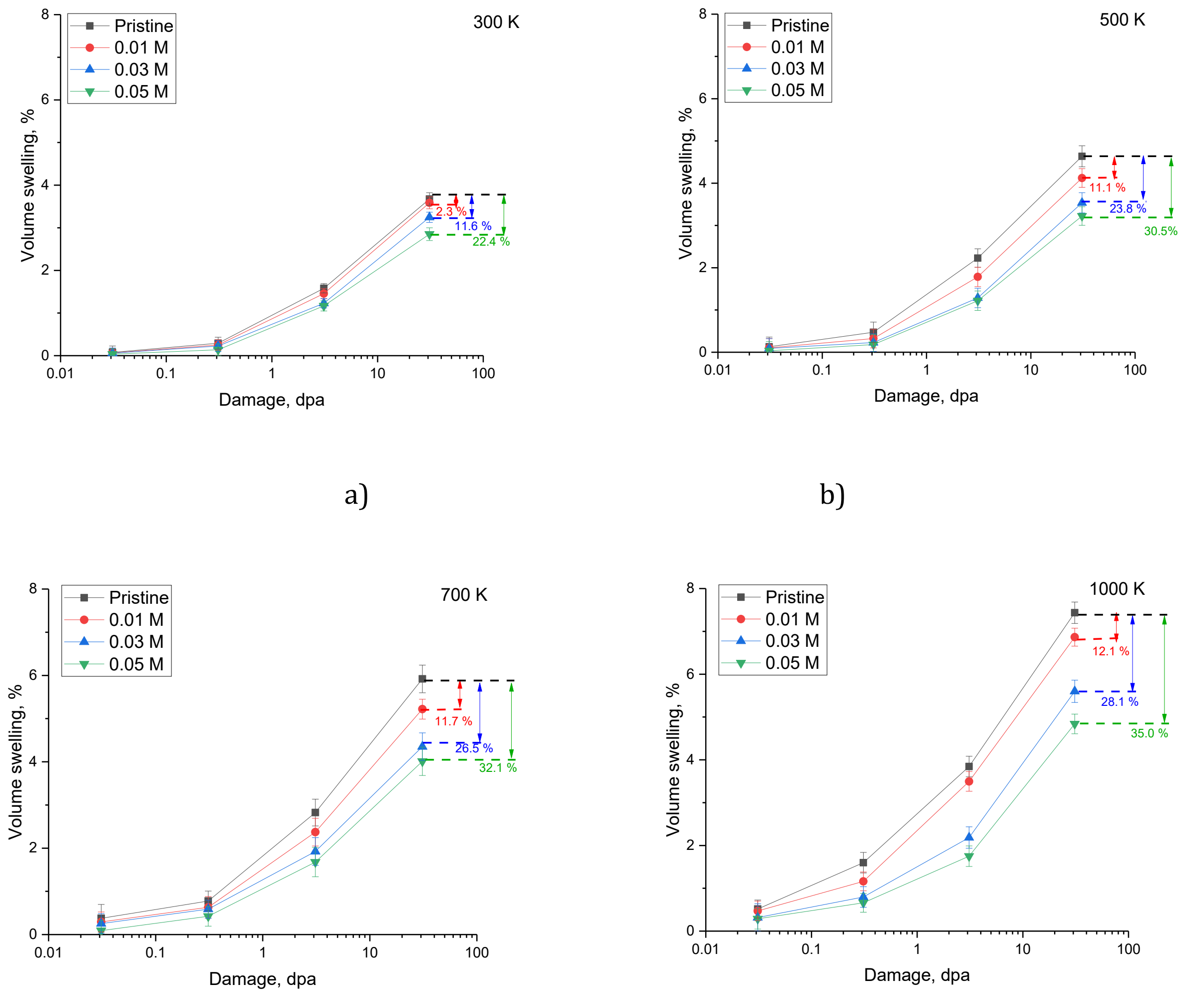
The irradiation effect on the mechanisms of softening (decrease in the hardness of the near-surface layer) contingent upon the value of atomic displacements and irradiation temperature was determined by the nanoindentation method. When determining the hardness values of the samples, the number of measurements at a given depth was at least 10 points. In this case, the measurements were carried out taking into account the need to avoid overlapping traces from the indenter during measurement, which can lead to large deviations. The measurement error was determined by calculating the standard deviation of the hardness value in the average value obtained during the measurements. To assess the mechanisms of diffusion of implanted He^{2+} ions in the damaged layer, as well as the restraining factors caused by the structural features of doped ceramics, measurements on side cleavages with a step of 50 nm were carried out, which made it possible to evaluate the hardness degradation in depth, the characteristic ion travel depth in ceramics, alongside the diffusion of ions to a depth exceeding 500 nm (the maximum depth of travel of He^{2+} ions in ceramics, according to SRIM Pro 2013 calculations presented in Figure 2a).

The study of the kinetics of changes in the thermophysical parameters of the studied ceramic samples contingent upon the dopant concentration, as well as in the case of variations in irradiation conditions and irradiation fluence, was carried out by determining changes in the thermal conductivity coefficient of ceramics using the longitudinal heat flow method implemented on the KIT-800 universal thermal conductivity meter.

3. Results and discussion

Figure 3 illustrates the determination results of the volumetric swelling of the damaged ceramic layer crystal structure depending on the value of atomic displacements (dpa), calculated based on simulation using the SRIM Pro 2013 program code, as well as irradiation temperature. The overall appearance of the presented data on the change in the ΔV(dpa) value indicates the negative effect of the structural damage accumulation on the crystalline structure of the irradiated layer, expressed in deformation distortion and a rise in volumetric tensile stresses in the damaged layer. At the same time, consideration of these dependencies should be carried out in two contexts: depending on the phase composition of the ceramics, more precisely in determining the influence of the MgO dopant concentration on the change in the ΔV(dpa) value; depending on the irradiation temperature, a change in which causes thermal effects associated with volumetric broadening and the influence of thermal vibrations on the structural disorder degree.

During analysis of the obtained dependences ΔV(dpa) on the structural features of ceramics, the change of which is due to variations in the dopant concentration, it can be concluded that the MgO dopant concentration growth, leading to structural ordering of the crystal lattice, results in elevation of resistance to volumetric tensile deformation, the alteration of which is observed in the case of variations in irradiation fluence. In this case, for modified ceramics, the dopant concentration growth, as is clearly seen from the data presented in Figure 3, leads to less pronounced changes in the crystal lattice volume associated with the accumulation of structural distortions and the concentration of implanted ions. This behavior of the ΔV(dpa) value in the case of a change in the dopant concentration can be explained by the structural ordering effects associated with the partial substitution of zirconium by magnesium, which, according to X-ray diffraction analysis data, results in structural ordering growth due to the crystal lattice parameters and volume reduction, as well as a reduction in the concentration of structural and vacancy defects in ceramics. During irradiation, especially at high temperatures, the concentration of initial defects and vacancies in the ceramic composition plays a key role in determination of the degradation kinetics of the damaged layer structure and its swelling rate. It should be noted that in this case, the absence of impurity inclusions in the composition of the synthesized ceramics eliminates the effect of interfacial hardening characteristic of two-phase lithium-containing ceramics [27-29], and the elevation in resistance to destructive swelling in this case is due to structural ordering caused by substitution effects. It should also be noted that when analyzing the obtained dependences ΔV(dpa) in the case of high irradiation temperatures (700 – 1000 K) a change in the dopant concentration in the ceramic composition has a more significant effect on the resistance to volumetric swelling in comparison with Li_2ZrO_3 ceramics obtained without the addition of a dopant. In the case of irradiation temperatures of 700 – 1000 K, the addition of 0.03 – 0.05 M MgO dopant to the composition of Li_2ZrO_3 ceramics leads to a decline in the ΔV(dpa) value of the order of 26 – 35 % in comparison with a similar value obtained for unmodified Li_2ZrO_3 ceramics. It is important to highlight that the elevation in resistance to volumetric swelling is nonlinear and is most pronounced at high irradiation fluences (1017 – 1018 cm^{-2}), which indicates the cumulative effect of structural distortions, most pronounced at high irradiation doses, for which deformation distortions are caused not only by the accumulation of point and vacancy defects, alongside the clusters formed by them, but also by the formation of gas-filled inclusions, the appearance of which in the damaged layer structure is associated with the implanted helium concentration growth. In this case, more pronounced changes in the ΔV(dpa) value, especially for samples irradiated at high temperatures, are due to a combination of two effects: the accumulation of structural distortions and the high mobility of implanted helium, the accelerated diffusion of which at high temperatures contributes to the filling of voids with their subsequent deformation stretching [11,30]. The containment of these effects is clearly demonstrated by the presented ΔV(dpa) dependences for modified ceramic samples.

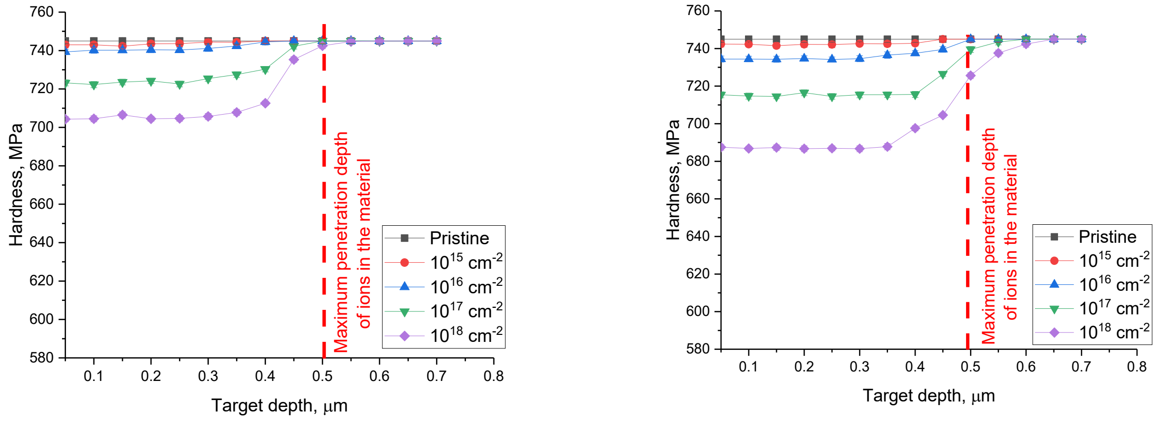


a) b)

c) d)

Figure 3. Results of alterations in the ΔV value, reflecting the volumetric change in the structural parameters of the crystalline structure of the damaged layer as a result of variations in irradiation conditions (fluence and irradiation temperature): a) 300 K; b) 500 K; c) 700 K; d) 1000 K

Figures 4 – 7 demonstrate the assessment results of changes in the hardness values of the studied samples by depth, reflecting the effect of irradiation fluence, irradiation temperature, and dopant concentration at its variation on the strength properties and diffusion processes caused by the implanted helium migration. The overall appearance of the presented dependences indicates the cumulative effect of softening, expressed both in the hardness value reduction with an irradiation fluence growth, and in a change in the trend of hardness degradation by depth, reflecting the diffusion of implanted ions to the surface (in the case of low irradiation fluences) and deep into the damaged layer, to a depth exceeding the ion travel depth in the material (this boundary is reflected by the red dotted line in Figures 4 – 7). At the same time, the diffusion of implanted helium ions is most pronounced for high-dose irradiation of samples at an irradiation temperature growth, which clearly reflects the fact of the presence of the effect of migration of implanted helium ions deep into the samples, indicating the destructive nature that accompanies these processes. Analysis of the profiles of changes in hardness by depth in the case of high-temperature irradiation for unmodified Li_2ZrO_3 ceramics revealed an increase in the depth of the layer in which lower hardness values are observed, and it was determined that the most pronounced changes were observed for samples irradiated at a temperature of 1000 K. Comparing the depth profiles of hardness, it can be concluded that the implanted helium diffusion is most pronounced when atomic displacements reach values of the order of 1 – 3 dpa (at a fluence above 10^{17} cm^{-2}), which can be explained by the effects of structural deformation distortion, as well as the formation of gas-filled inclusions with implanted helium, which at high temperatures agglomerate into larger formations, amplifying deformation distortion by stretching the crystalline and chemical bonds of the crystal lattice [31,32]. At the same time, modification of the structure of ceramics by increasing the MgO dopant concentration leads to a reduction in the overall trend of changes in hardness by depth both at low irradiation temperatures and at high temperatures, for which the diffusion of implanted helium ions is more pronounced. It should also be noted that minor deviations in hardness values can be explained by local structural defects, the presence of which can have a slight increase in softening resistance. According to the obtained data on the change in the values of volumetric swelling of the surface layer when comparing them in the case of variation in the irradiation temperature, it can be concluded that an increase in the irradiation temperature leads to a more intensive increase in the difference ΔV. At the same time, the growth of this value in the case of the original unmodified ceramics indicates destabilization of the crystal structure due to its disordering caused by the effect of thermal expansion, as well as accelerated diffusion of implanted ions in the damaged layer. The addition of the stabilizing additive MgO, as can be seen from the presented data, leads to an increase in resistance to volumetric swelling caused by the accumulation of helium in the pores with a subsequent increase in gas-filled inclusions due to the accumulation of helium.



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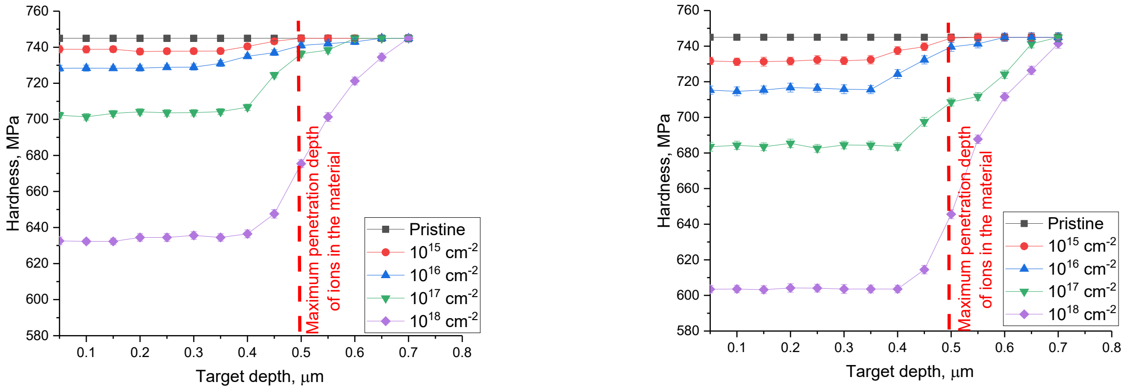
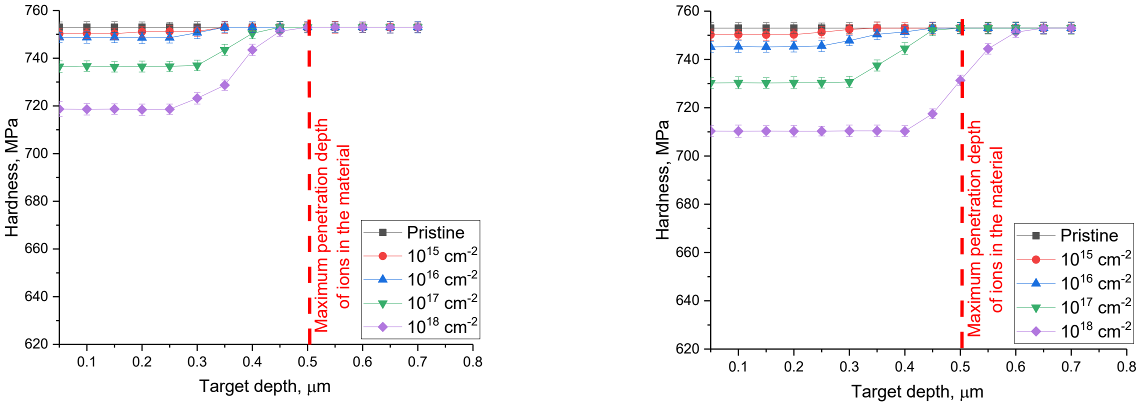


c) d)

Figure 4. Change in the hardness of Li_2ZrO_3 ceramics by depth in the case of irradiation at different temperatures: a) at irradiation temperature of 300 K; b) at irradiation temperature of 500 K; c) at irradiation temperature of 700 K; d) at irradiation temperature of 1000 K

According to the data presented in Figure 5, when comparing trends in changes in hardness of the studied unmodified ceramics and with the addition of 0.01 M MgO, it is clear that the decline in hardness is less pronounced both at an irradiation temperature of 300 K and at higher temperatures. This behavior of trends in hardness degradation by depth indicates a positive effect of the dopant on the resistance of ceramics to deformation distortions caused by exposure to irradiation.



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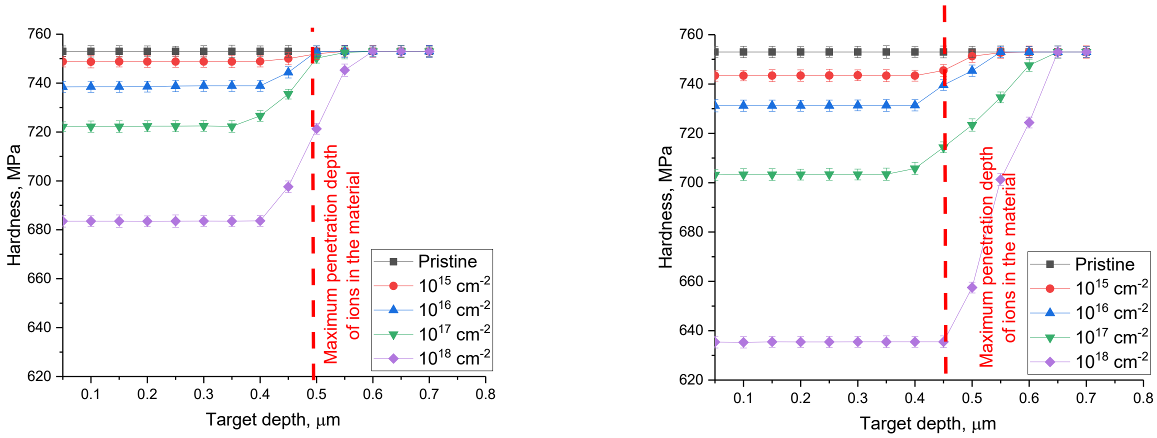
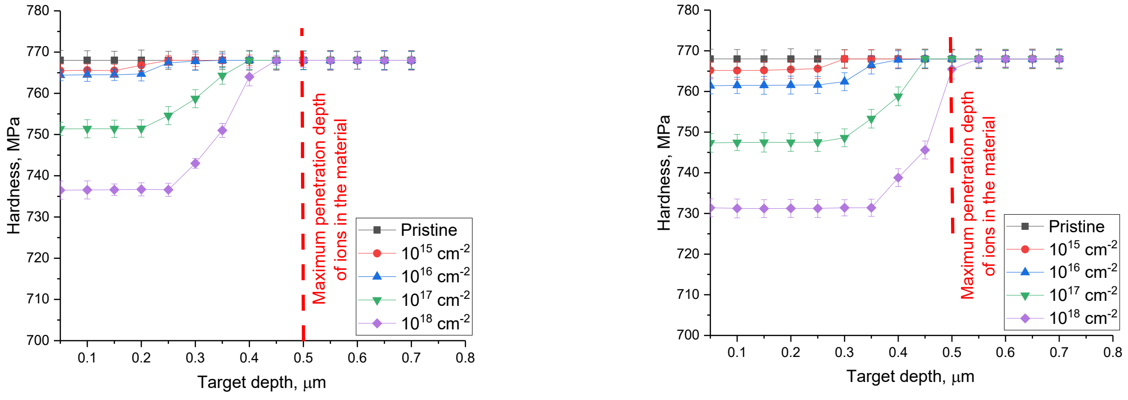


c) d)

Figure 5. Change in the hardness of Li2ZrO3 ceramics with the addition of the 0.01 M MgO dopant by depth in the case of irradiation at different temperatures: a) at irradiation temperature of 300 K; b) at irradiation temperature of 500 K; c) at irradiation temperature of 700 K; d) at irradiation temperature of 1000 K

Similar changes in trends in the degradation of hardness values with depth, reflecting an increase in the resistance of ceramics to strain softening in comparison with unmodified ceramics, are also observed for ceramics with a dopant concentration of 0.03 M. It should be noted that an elevation in the dopant concentration results in resistance growth, and, as can be seen from the data presented, a decrease in the effect of helium ion diffusion deep into the sample at high irradiation temperatures, which indicates a limiting factor due to the structural features of the modified ceramics.



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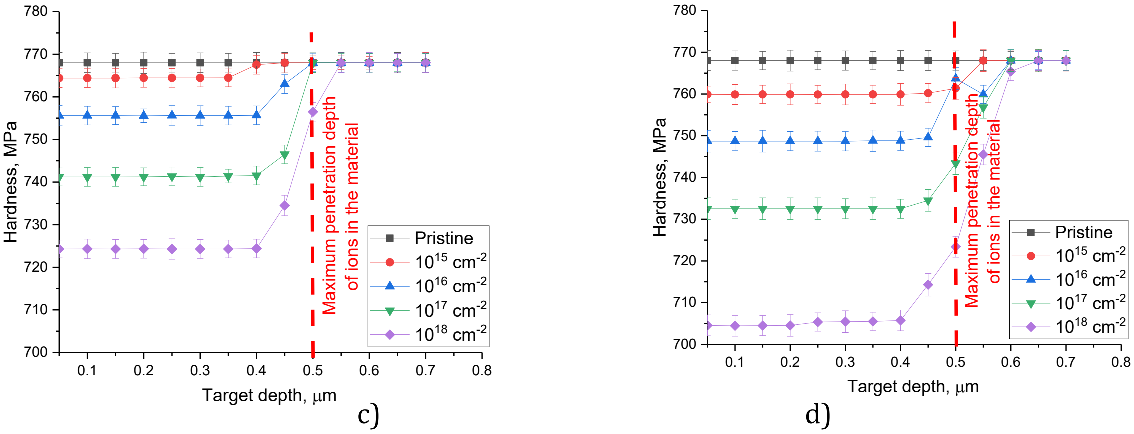
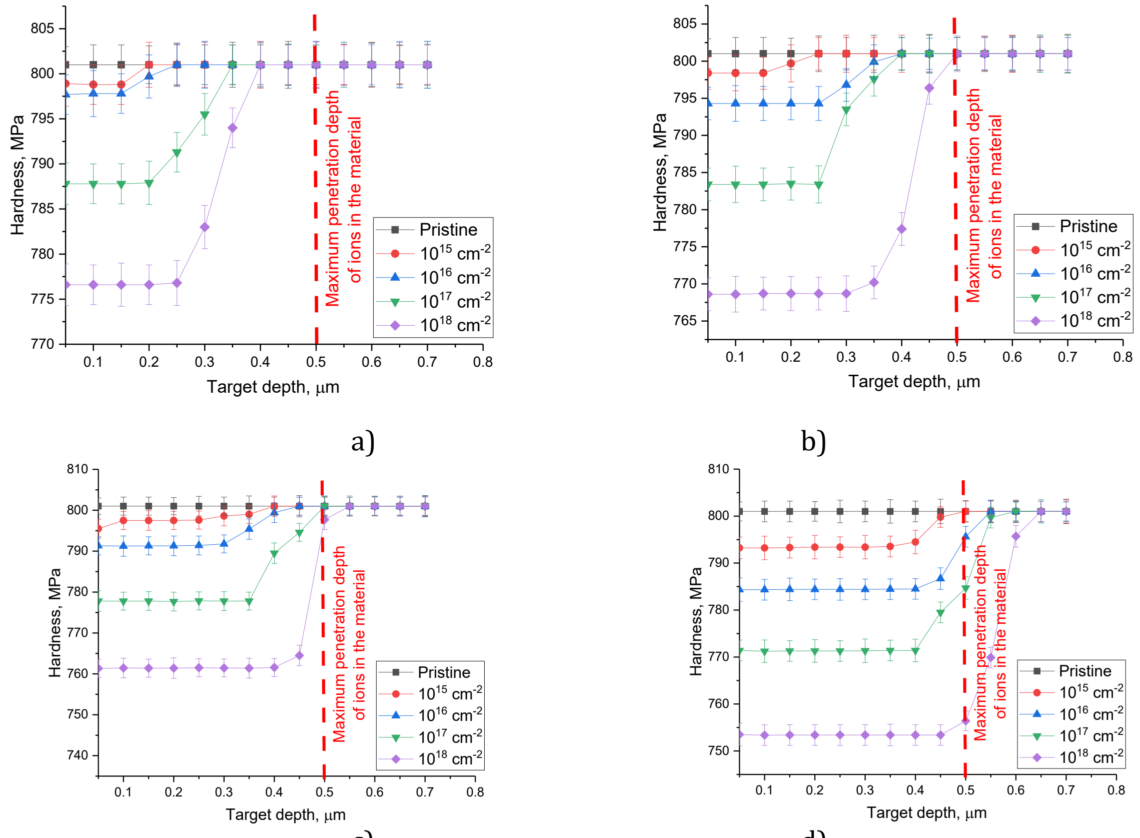


c) d)

Figure 6. Change in the hardness of Li_2ZrO_3 ceramics with the addition of the 0.03 M MgO dopant by depth in the case of irradiation at different temperatures: a) at irradiation temperature of 300 K; b) at irradiation temperature of 500 K; c) at irradiation temperature of 700 K; d) at irradiation temperature of 1000 K



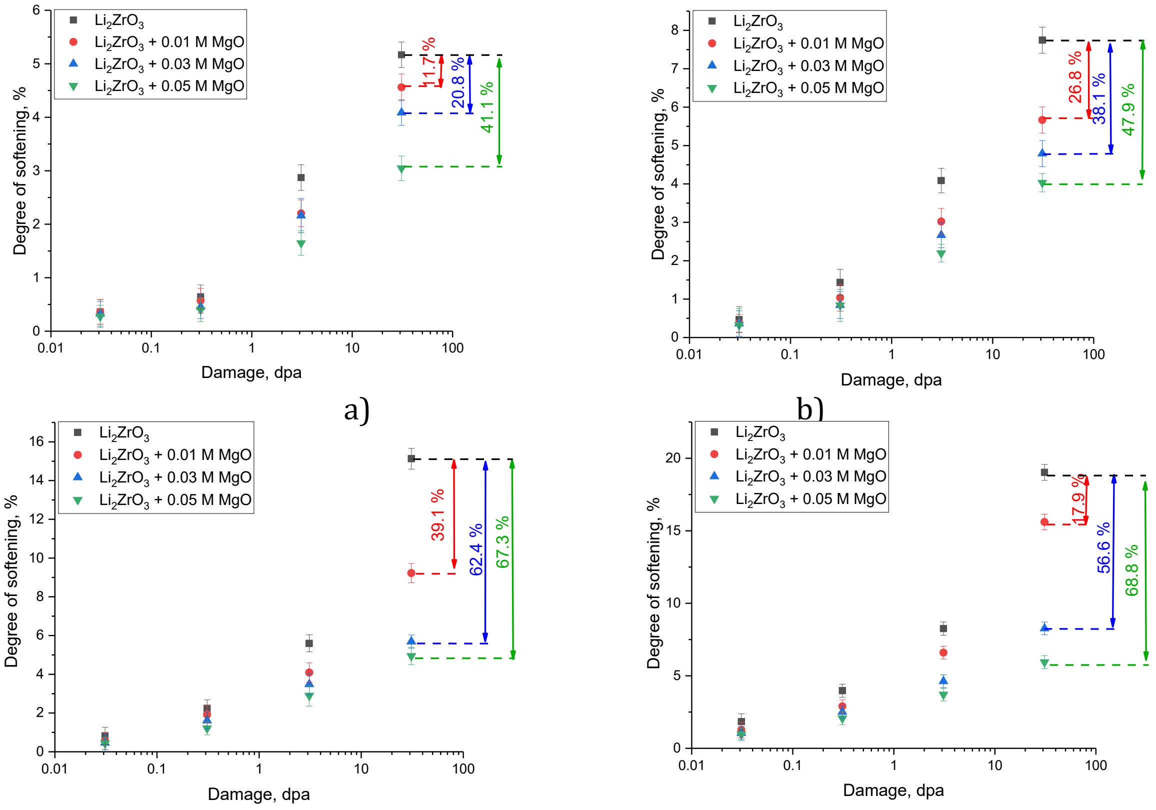
a) b)

c) d)

Figure 7. Change in the hardness of Li_2ZrO_3 ceramics with the addition of the 0.05 M MgO dopant by depth in the case of irradiation at different temperatures: a) at irradiation temperature of 300 K; b) at irradiation temperature of 500 K; c) at irradiation temperature of 700 K; d) at irradiation temperature of 1000 K

Analysis of the dependences of the values of change in hardness by depth for samples of Li_2ZrO_3 ceramics with the addition of 0.05 M MgO dopant indicates that an elevation in the dopant concentration in the ceramic composition leads to a rise in the softening resistance of the near-surface layer, and a reduction in the thickness of the damaged softened layer both in the case of temperatures of 300 – 500 K, and a slight excess of the maximum path length of He^{2+} ions in the case of irradiation at a temperature of 700 K. The results obtained indicate fairly high stability of Li_2ZrO_3 ceramics with the addition of 0.05 M MgO dopant to the deformation-induced disordering of the near-surface layer, alongside the inhibition of the diffusion of implanted helium deep into the sample, which was observed under similar irradiation conditions for unmodified ceramic samples. It should also be noted that the observed alterations in the hardness values of ceramic samples irradiated at a temperature of 1000 K, which exceed the maximum ion travel depth of the order of 100 – 150 nm (in the case of maximum irradiation fluence), are significantly less than the observed changes under similar irradiation conditions for unmodified ceramics and modified ones but with a lower dopant concentration (0.01 – 0.03 M).

Figure 8 illustrates the assessment results of the softening degree ΔHV= HVpristine – HVirradiated, reflecting the near-surface layer hardness degradation depending on the value of atomic displacements. The data are calculated at a depth of the order of 250 – 300 nm, corresponding to the maximum value of atomic displacements, according to the calculated data of SRIM Pro.



a) b)

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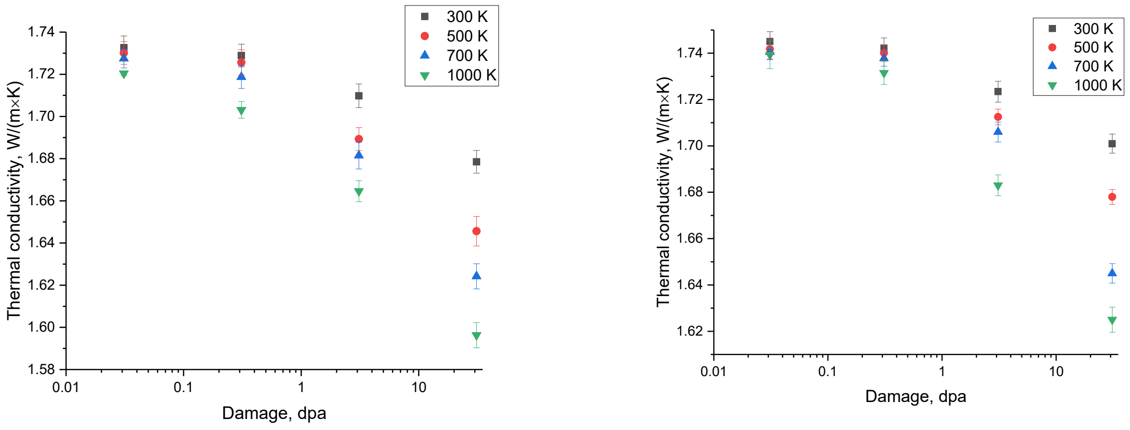
Figure 8. Results of a comparative analysis of the softening degree (ΔHV= HVpristine – HVirradiated) of the ceramics under study contingent upon the value of atomic displacements with variations in irradiation temperature: a) at irradiation temperature of 300 K; b) at irradiation temperature of 500 K; c) at irradiation temperature of 700 K; d) at irradiation temperature of 1000 K

According to the presented dependences of changes in the ΔHV value, it is clear that the addition of the MgO dopant to the composition of Li_2ZrO_3ceramics leads to an increase in resistance to destructive softening, which is most manifested under high-dose irradiation (about 30 dpa). It should be noted that for doped samples, an irradiation temperature growth, as can be seen from the data presented, leads to an elevation in the difference in the softening values, which indicates higher rates of resistance of modified ceramics to thermally induced processes of degradation and softening associated with the processes of diffusion of implanted helium ions in the near-surface layer. This effect is most clearly manifested for samples with a dopant concentration of 0.05 M, for which the difference in ΔHV values under high-temperature irradiation (700 – 1000 K) in comparison with unmodified ceramics is about 67 – 68 %, which indicates a higher resistance of ceramics to softening. Moreover, in the case of unmodified Li_2ZrO_3 ceramics, the most significant changes, close to the critical parameters of degradation of strength parameters, are observed under high-temperature irradiation (700 – 1000 K), for which the maximum value of ΔHV is more than 15 – 18 %, which exceeds the critical permissible value of a reduction in hardness of 10 %. In turn, doping with MgO results in ΔHV value reduction under high-temperature irradiation, and the ΔHV value itself for samples with a dopant concentration of 0.03 – 0.05 M at an atomic displacement of about 30 dpa is no more than 5 – 8 %, which is below the critical permissible value of hardness reduction. The observed effect of hardening and heightened resistance to radiation-strain softening of modified ceramics in the context of variations in dopant concentration reflects a direct relationship between the degradation resistance and the structural ordering degree, changes in which are due to substitution effects at dopant concentration growth. In this case, modification of MgO lithium ceramics leads to a rise not only in resistance to volumetric deformation swelling (see data in Figure 3), but also in an elevation in the resistance of ceramics to softening under high-dose high-temperature irradiation, as well as a decrease in the effect of diffusion of implanted helium ions deep into the sample (see data on changes in hardness values with depth, presented in Figures 4-7).

Figure 9 reveals the assessment results of the change in the thermal conductivity coefficient of the ceramics under study, reflecting the change in thermophysical parameters and their relationship with the accumulation of structural distortions caused by irradiation. The data are presented in the form of dependencies for each type of ceramics under study with variations in the temperature at which irradiation was carried out, in order to reflect the influence of temperature on the destructive change in thermophysical properties. It is important to highlight that in the initial (non-irradiated state) the addition of a dopant to the composition of Li_2ZrO_3 ceramics results in the thermal conductivity coefficient growth, which is due to the structural ordering effects, as well as the properties of the MgO dopant, which determine an increase in thermal conductivity and heat transfer due to the higher thermal conductivity of the dopant. As is evident from the data presented, in the case of Li_2ZrO_3 ceramics, the thermal conductivity coefficient is about 1.734 W/(m×K), while the addition of the MgO dopant results in the thermal conductivity coefficient growth to 1.746, 1.801 and 1.943 W/(m×K) for dopant concentrations of 0.01, 0.03 and 0.05 M, respectively. In the case of irradiation of the studied Li_2ZrO_3 ceramic samples without addition of MgO to the composition at temperatures of 300 – 500 K, the most significant alterations in the thermal conductivity coefficient are observed when the radiation dose accumulates above 1 dpa, for which, according to the assessment of changes in the ΔTCC value, the thermal conductivity reduction is more than 1.5 %, and a further radiation dose elevation results in reduction of ΔTCC to 2-4 %. For high-temperature irradiation, the decrease in thermal conductivity values at the maximum irradiation dose is about 7 – 8 %, which is more than 2 times higher than similar changes during irradiation at lower temperatures, and also indicates the negative impact of temperature exposure on the decrease in the thermophysical parameters of the ceramics under study. For Li2ZrO3 ceramic samples with the addition of the MgO dopant, it was found that an increase in the dopant concentration leads not only to a rise in the initial thermal conductivity values, but also to less pronounced changes in the value of ΔTCC both in the case of low irradiation temperatures (300 and 500 K) and during high-temperature irradiation, for which the maximum change in the ΔTCC value is of the order of 4 – 6 %. This difference in alterations in the thermal conductivity coefficient of the ceramics under study, associated with the addition, as well as variations in the MgO dopant concentration, is due to several factors. Firstly, as was established using the X-ray diffraction analysis, a dopant concentration growth in the composition of the ceramics results in structural ordering and, as a consequence, compaction of the ceramics, and the possible partial substitution of zirconium by magnesium in the crystal lattice of the Li_2ZrO_3 phase contributes to a rise in resistance to thermal expansion associated with the temperature effect on the samples during irradiation. Secondly, the elevation in resistance to volumetric swelling (see data in Figure 3) in the case of modified ceramics indicates that the concentration of defective inclusions in the damaged layer structure, and areas of disorder, causing volumetric-strain distortion of the crystal structure is significantly less, which, as a consequence, results in greater stability of the thermophysical parameters of the ceramics under study under high-dose irradiation. It should also be noted that modification of Li_2ZrO_3 ceramics by changing the dopant concentration, leading to an elevation in the initial thermal conductivity values, provides higher heat transfer due to phonon heat transfer mechanisms, due to a reduction in structural defects, which are scattering centers for thermal phonons.



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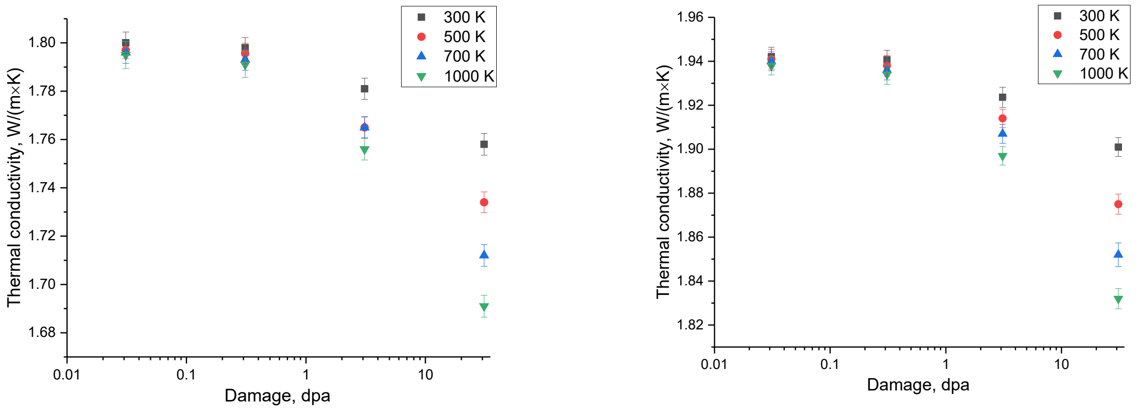
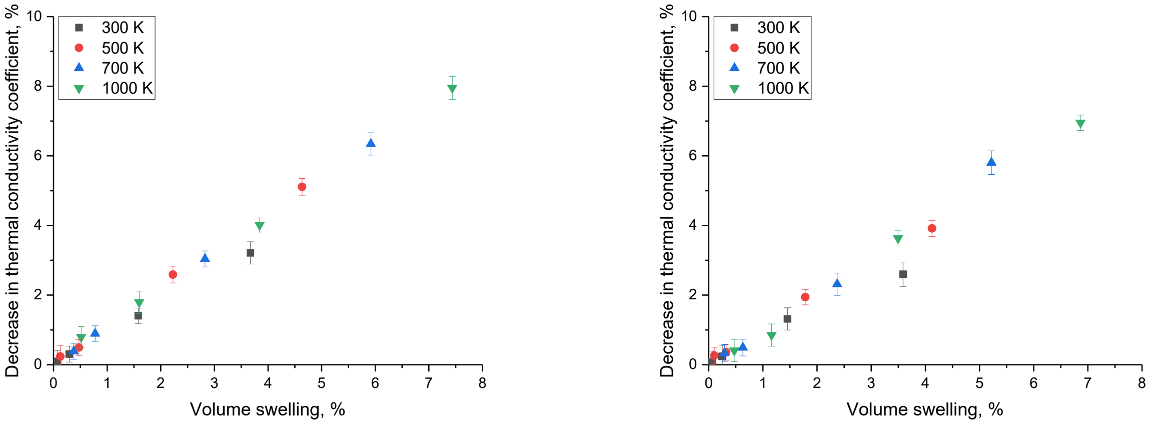


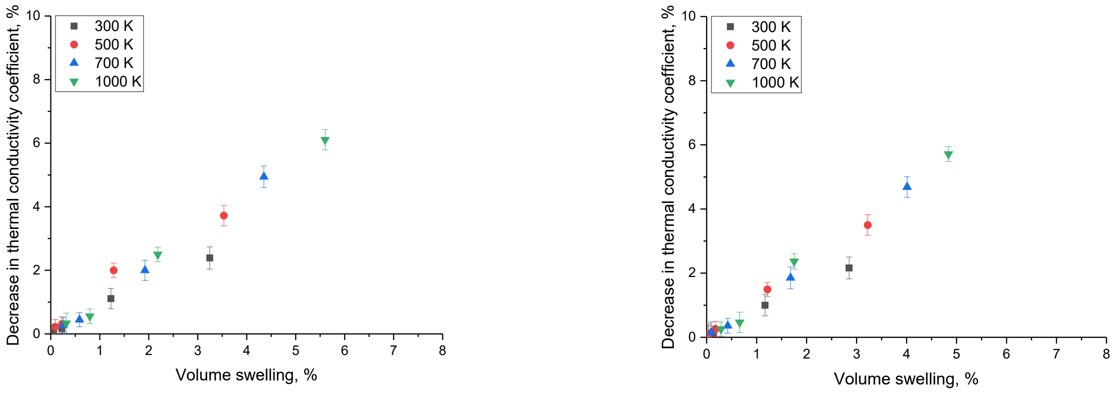
c) d)

Figure 9. Results of changes in the thermal conductivity coefficient of the studied Li_2ZrO_3 ceramics contingent upon alterations in irradiation conditions (the value of atomic displacements and temperature) with variations in the MgO dopant concentration: a) without dopant; b) at a concentration of 0.01 M; c) at a concentration of 0.03 M; d) at a concentration of 0.05 M

Figure 10 demonstrates the dependence of the change in thermal conductivity coefficient on volumetric swelling (ΔTCC(VS)), reflecting the connection between destructive volumetric swelling of ceramics and degradation of thermal conductivity, the change of which is associated with structural changes. The data are presented in the form of dependences ΔTCC(VS) obtained for each type of ceramic under study when the irradiation temperature changes, which reflect the influence of variations in irradiation conditions on both destructive swelling and thermal conductivity reduction, expressed as a comparison of the thermal conductivity coefficient values of the ceramics under study in the initial and irradiated states.



a) b)



c) d)

Figure 10. Results of a comparative analysis of ΔTCC(VS) for the ceramics under study: a) without dopant; b) at a concentration of 0.01 M; c) at a concentration of 0.03 M; d) at a concentration of 0.05 M

The presented dependences ΔTCC(VS) have a direct correlation with each other and reflect the relationship between the volumetric deformation of the crystal structure, caused by the accumulation of structural distortions and areas of disorder in the damaged layer and the degradation of the thermophysical parameters of the ceramics under study, the mechanisms of change during phonon heat transfer have a direct correlation with the density of structural defects. According to the data presented, the most pronounced changes in thermal conductivity are observed for samples irradiated at high temperatures (700 – 1000 K), for which the accumulation of structural distortions is associated not only with effects caused by the mechanisms of interaction of incident ions with the crystal structure and implantation processes, but also with thermal expansion caused by an alteration in the amplitude of thermal vibrations of atoms in the crystal lattice, an elevation in which promotes their mobility, alongside the migration of point and vacancy defects in the structure. Moreover, the substitution of zirconium by magnesium with a rise in the dopant concentration in the composition of ceramics, which causes a growth in the structural ordering degree (changes in the crystal lattice parameters and volume), helps to restrain the effects of structural disorder, which are most pronounced during high-temperature irradiation.

4. Conclusion

During the experiments carried out related to the assessment of the diffusion mechanisms of implanted helium in the near-surface layers of lithium-containing ceramics, the following results were obtained:

1. During determination of the effect of variations in the phase composition of ceramics on resistance to deformation volumetric swelling, carried out by analyzing the ΔV(dpa) dependences, it was found that the stability of ceramics is affected not only by structural changes caused by variations in the dopant concentration in the composition, but also by the temperature effect, causing an elevation in deformation distortions associated with thermal effects and accelerated diffusion of implanted He^{2+} ions.

2. It has been established that with irradiation temperature growth in the case of Li_2ZrO_3 with high-dose irradiation, a more intense reduction in the hardness of the near-surface layer is observed at the depth of maximum atomic displacements (depth of the order of 200 – 250 nm), which indicates an adverse effect of irradiation temperature on the stability of strength properties. At the same time, the irradiation temperature growth leads not only to more pronounced changes in hardness values, but also to a rise in the damaged layer thickness, which indicates the occurrence of diffusion processes associated with the migration of implanted He^{2+} ions into the depth, which results in deformation distortion of the near-surface layer and, as a consequence, its softening.

3. During assessment of changes in the thermophysical parameters of the studied ceramics exposed to irradiation with He^{2+} ions, it was established that the irradiation temperature growth during high-dose irradiation (about 3 – 30 dpa) has a negative effect on a decline in the thermal conductivity coefficient, indicating an increase in heat losses in irradiated samples. Moreover, it should be noted that for doped samples, less pronounced trends in thermal conductivity degradation are observed, which indicate a direct relationship between destructive volumetric swelling and a reduction in the thermal conductivity of ceramics.

Based on the research carried out, and conclusions drawn from the results of experiments and analysis of the obtained dependencies, we can draw a general conclusion about the prospects of using the MgO dopant in low concentrations not only to stabilize the strength and thermophysical parameters of Li_2ZrO_3 ceramics, but also to enhance degradation resistance of properties under high-dose irradiation, alongside the accumulation of helium in the near-surface layer, large concentrations of which contribute to destructive embrittlement and softening of ceramics.

Authors' contributions

A.A. Khametova – conducting experiments, working out the modes of obtaining ceramics, characterizing structural changes.

D.I. Shlimas – conducting experiments on irradiation, interpreting structural changes caused by irradiation, assessing the depth of ion penetration in the damaged layer.

Sh.G. Giniyatova – conducting experiments, interpreting the obtained data, analyzing the state of the problem, processing the obtained results.

A.L. Kozlovskiy – conducting experiments, general analysis of the relationship between structural changes and strength parameters, determining the kinetics of material degradation.

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Литий мета-цирконаты негізіндегі керамикалардың беткі қабаттарындағы ендірілген гелийдің термиялық-индуцияланған диффузиялық механизмдерін зерттеу

Аңдатпа. Зерттеудің мақсаты – жоғары температуралық сәулелену кезінде ендірілген гелийдің диффузиясымен байланысты литий мета-цирконаты негізіндегі керамикалардың беткі қабаттарының деструкция процестеріне жылулық әсердің ықпалын анықтау, сондай-ақ аз концентрациядағы магний оксиді қосымшасының диффузиялық механизмдерді тежей алатын әсерін бағалау. Мұндай типтегі керамикаға қызығушылық, ең алдымен, тритийді өндіру және жинақтау үшін литий құрамындағы керамикалардың түрлерін кеңейту мүмкіндігімен, сондай-ақ жоғары температуралық пайдалану режимдеріне тән деградациялық процестерге тұрақтылығын арттырумен және нейтрондардың литиймен ядролық реакциясы нәтижесінде пайда болатын гелий тәрізді бөліну өнімдерін жинақтаумен байланысты. Наноиндентация әдісін пайдаланып (иондардың қозғалыс траекториясы бойымен және бүйірлік жарылыс бойынша тереңдігін өлшеу арқылы) жүргізілген зерттеулерге сәйкес, сәулелену температурасының жоғарылауы зақымданған қабаттың қалыңдығының артуына әкелетіні анықталды, бұл өзгеріс гелийдің термиялық әсерлер есебінен ішке қарай диффузиялануымен байланысты. Жоғары температуралық сәулелену кезінде беткі қабаттың беріктігінің төмендеуі кристалдық құрылымның деформациялық бұрмалануының артуы және оның ісінуімен байланысты құрылымдық өзгерістерге негізделген, бұл әсіресе жоғары дозалы сәулелену жағдайында айқын байқалады. Синтез кезінде литий құрамындағы керамикаларға қосымша заттың концентрациясын арттыру арқылы литий мета-цирконатының құрылымдық ерекшеліктерінің өзгеруі зақымданған қабаттың беріктік жоғалтуға төзімділігін арттырып қана қоймай, жоғары температуралық сәулелену кезінде керамикалардағы диффузиялық қабаттың қалыңдығын азайтуға да әкеледі.

Түйінді сөздер: гелий диффузиясы; литий құрамындағы керамикалар; бланкеттер; радиациялық зақымданулар; гелийден ісіну

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Изучение термически – индуцированных диффузионных механизмов имплантированного гелия в приповерхностных слоях керамик на основе метацирконата лития

Аннотация. Цель данного исследования заключается в определении влияния термического воздействия на процессы деструкции приповерхностных слоев керамик на основе метацирконата лития, связанных с диффузией имплантированного гелия, а также определение влияния допанта оксида магния в малых концентрациях на сдерживание диффузионных механизмов при высокотемпературном облучении. Интерес к данного типа керамикам обусловлен в первую очередь возможностью расширения типов литийсодержащих керамик для производства и накопления трития, а также увеличением стабильности к деградационным процессам, характерным для высокотемпературных режимов эксплуатации, а также накопления продуктов деления ядерных реакций нейтронов с литием в виде гелия. Согласно проведенным исследованиям с применением метода наноиндентирования по глубине (измерений вдоль траектории движения ионов на боковом сколе) было установлено, что увеличение температуры облучения приводит к увеличению толщины поврежденного слоя, изменение которой обусловлено эффектами диффузии гелия вглубь за счет термических эффектов. Разупрочнение приповерхностного слоя при высокотемпературном облучении обусловлено структурными изменениями, связанными с увеличением деформационного искажения кристаллической структуры и ее распухания, которое наиболее выражено при высокодозном облучении. При этом изменения структурных особенностей метацирконата лития за счет увеличения концентрации допанта при синтезе литийсодержащих керамик, приводит не только к увеличению устойчивости к разупрочнению поврежденного слоя, но и уменьшению толщины диффузионного слоя в керамиках при высокотемпературном облучении.

Ключевые слова: диффузия гелия; литийсодержащие керамики; бланкеты; радиационные повреждения; гелиевое распухание

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