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Temperature effects of defect formation in BeO ceramics during helium blistering

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ABSTRACT

The work is devoted to the study of the processes of defect accumulation in the near-surface layer of BeO ceramics associated with irradiation with low-energy He^{2+} ions, as well as to the establishment of the role of temperature effects on changes in thermal conductivity. Identification of the role of temperature effects in acceleration of the processes of distortion and disordering of the near-surface layers is one of the key tasks, the solution of which will allow more accurate prediction of the behavior of materials under extreme conditions combining the effects of high temperatures and radiation damage. During assessment of changes in structural parameters depending on variations in irradiation conditions, anisotropic distortion of the crystal lattice, the degree of deformation of which has a direct relationship with the temperature effect, was established. It has been determined that an increase in the irradiation temperature leads to an acceleration of the processes of accumulation of deformation distortions, the growth of which in the damaged layer leads to a more pronounced destabilization and broadening of the damaged layer depth. Such changes are associated with the acceleration of the diffusion processes of point and vacancy defects, the migration of which leads not only to an increase in deformation distortions and amorphous inclusions, but also to an increase in the damaged layer thickness. During determination of changes in the concentration of vacancy and structural defects in the damaged layer, relationships between the concentration of oxygen vacancies and the degradation of thermal conductivity, the reduction of which is due to an increase in the effects of phonon scattering and a decrease in the rate of heat transfer, were established.

1. Introduction

Expansion of the potential for the use of ceramic materials as nuclear materials used as first wall materials, inert matrix materials, absorbers or reflectors, and spent nuclear fuel storage materials requires a large fundamental and experimental base determining the potential for use in extreme conditions [1–3]. At the same time, the greatest interest in the last few years has been in studies that simulate operating conditions as close as possible to real ones, allowing evaluation of the behavior of materials when exposed to a combination of factors, such as the accumulation of radiation damage, exposure to aggressive environments or high temperatures, high mechanical loads, etc. [4,5] Such studies are primarily important from the point of view of expansion of the understanding of the processes of degradation of the near-surface layers of structural materials under extreme influences, as well as the creation of a database on the properties of materials, the main purpose of which is primarily associated with the need to select the optimal compositions of structural materials, as well as the search for opportunities to enhance their resistance to external influences [6,7]. As a rule, during operation under extreme conditions, especially in the case of simultaneous exposure to radiation and temperature, structural materials experience the combined effect of thermal expansion and deformation distortion of the crystalline structure, the degree of which is directly related to the thermal effects and the stability of the material to thermal expansion.

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Under such influences, the degradation rate of the material can grow significantly due to the acceleration of diffusion and migration of radiation-stimulated point and vacancy defects, which, migrating in the damaged layer, increase the deformation distortion of the crystal structure [8–11]. The growth of deformation distortions and stresses caused by the effects of radiation and temperature influences contribute to the growth of microcracks, as well as the formation of highly disordered or amorphous inclusions in the damaged layer, which negatively affects the resistance of materials to external influences, as well as the loss of heat-conducting properties, the deterioration of which can lead to non-uniform heat exchange, the emergence of areas of local overheating, which in turn increases the destabilization of structural materials used in extreme conditions [12–15].

The choice of beryllium oxide ceramics as structural materials allows for the expansion of the operating capabilities of new generation nuclear installations by increasing the operating temperatures of the core, as beryllium oxide has a fairly high melting point (around 2500 °C) and high thermal conductivity (around 200 W/($m \times K$) in contrast to other types of oxide ceramics, such as zirconium dioxide or tungsten oxide [16,17]. It is worth to highlight that the high thermal conductivity of beryllium oxide, the value of which is comparable to a number of metal alloys, makes it possible to use these ceramics to remove heat from the active zone, which reduces the risk of local thermal stresses in dispersed fuel, the presence of which in the case of high-dose irradiation can result in destabilization of fuel elements and their embrittlement [18,19]. At the same time, high values of thermal conductivity and melting point make ceramics based on beryllium oxide competitive with nitride ceramics, such as aluminum or silicon nitride, which are considered the most promising materials for high-temperature applications in nuclear power engineering. Also, in turn, the high stability of beryllium oxide ceramics to the effects of high temperatures due to low values of thermal expansion coefficients in combination with a low value of the neutron capture cross-section allows this type of ceramics to be considered as structural materials for new-generation high-temperature reactors [20, 21]. However, as is known, during operation, ceramics can be exposed to high levels of radiation backgrounds and simultaneous exposure to high temperatures, which can lead to acceleration of the destabilization processes of the near-surface layers, which are most susceptible to external influences. The combination of such effects at high doses of radiation can lead to the acceleration of destabilization processes of the near-surface layers, loss of thermal conductivity, which in turn will contribute to the formation of non-uniform areas of local overheating, capable of accelerating destabilization processes and leading to catastrophic consequences [22,23].

Interest in study of the stability of the structural and thermal conductivity properties of BeO ceramics to high-dose irradiation with He^{+} ions, and consequently, the accumulation of radiation defects and associated changes, is not only due to the possibilities of using ceramics as materials in contact with various heat carriers, in particular with helium or other gases. Moreover, helium accumulation in the near-surface layers can occur not only upon contact with the coolant, but also as a result of the interaction of neutrons with beryllium, the reaction (n, α) can occur. In this case, helium formation occurs in the entire volume of the ceramics [24], which leads to the initialization of helium diffusion processes from the volume to the surface. Previously, in [25, 26], we studied the mechanisms of defect formation in BeO ceramics in the case of irradiation with low-energy He^{+} ions and their influence on the change in strength, optical and thermal properties. In this case, the main emphasis in the study in [25] was placed on determination of the role of defect formation processes on the change in the strength properties of the near-surface layer, resulting in destabilization of the crystal structure and softening (loss of hardness and wear resistance). In work [26], the main emphasis was placed on determination of the relationship between changes in optical and thermal properties with the results of assessment of the values of residual mechanical stresses arising as a result of irradiation in the near-surface layer. Moreover, during the study using the Raman spectroscopy method, an anisotropic nature of the deformation of the crystal structure was established during irradiation with He^{+} ions, associated with a change in the contributions of tensile and compressive stresses in the damaged layer with an increase in the irradiation fluence. It should be noted that in [26] a diffusion mechanism of structural disordering of the near-surface layer was established with an increase in irradiation fluence, which is caused by the migration of point and vacancy defects into the sample, to a depth exceeding the range of He^{+} ions. This effect may play a negative role in determination of the potential applicability of these ceramics as structural or functional materials and may also be enhanced by additional thermal action during irradiation. The influence of temperature effects on the change in the properties of the near-surface layer of BeO ceramics was considered in the presented work, the distinctive feature of which from previously conducted studies is the determination of the role of temperature effects during irradiation on the change in the thermophysical properties of ceramics, as well as the establishment of the role of vacancy defects depending on the irradiation conditions on the change in the mechanisms of heat transfer and amorphization of the near-surface layer. In this case, the problem of gas swelling associated with the accumulation of helium in pores and microcracks can lead, in the case of prolonged temperature exposure, to the acceleration of the processes of destabilization of the structural properties of the damaged layer, and due to the high mobility and inertness of helium, prolonged temperature exposure, as well as to the acceleration of the processes of destruction of the near-surface layer due to the growth of gas-filled inclusions and swelling, resulting in partial amorphization due to the high concentration of deformation distortions and stresses. At the same time, the stability of the near-surface layer plays a very important role in determination of the stability and compatibility of ceramic structural materials, since the near-surface layers interact most actively with other materials.

2. Materials and methods

The objects of the study were polycrystalline ceramics based on beryllium oxide (BeO) with a hexagonal type of crystal lattice and a wurtzite-type structure. The interest in this type of ceramics is due to the possibility of using it as microwave windows, neutron moderators, as well as high thermal conductivity in comparison with other types of oxide ceramics considered as structural materials, its use in heat-removing material, which, together with high radiation resistance, makes it one of the promising materials in nuclear power engineering. Fig. 1 shows the results of characterization of the initial BeO ceramic sample in the form of an X-ray diffraction pattern reflecting the type of crystal structure and the degree of crystallinity, a Raman spectrum with modes characteristic of this type of structure, characterizing chemical bonds, as well as an optical transmission spectrum. According to the presented X-ray diffraction and Raman spectroscopy data, the studied ceramics are BeO with a hexagonal type of crystal lattice, the parameters of which are $a = 2.6857$ Å $c = 4.3592$ Å, wurtzite type structure. According to X-ray diffraction data, the packing density of the hexagonal crystal lattice, estimated by the ratio of the parameters c/a, is 1.6231, which has a slight deviation from the reference value of c/a – 1.62305, the deviation of the c/a value from the reference value is associated with the formation of anisotropic distortion of chemical bonds, as well as deformation distortions of the coordination spheres. Moreover, the A_1(TO) and E_1(TO) modes, which have the highest intensities, indicate a high degree of crystallinity and structural ordering. According to optical spectroscopy data, the initial sample has a fairly good transmittance in the visible and near IR range, and the absence of any additional absorption bands indicates a high degree of structural order and a low concentration of defects in the ceramics, which is in good agreement with the results of Raman spectroscopy and X-ray diffraction.

Irradiation of samples with helium ions (He^{2+}) was carried out on the DC-60 accelerator (Institute of Nuclear Physics). Irradiation with helium

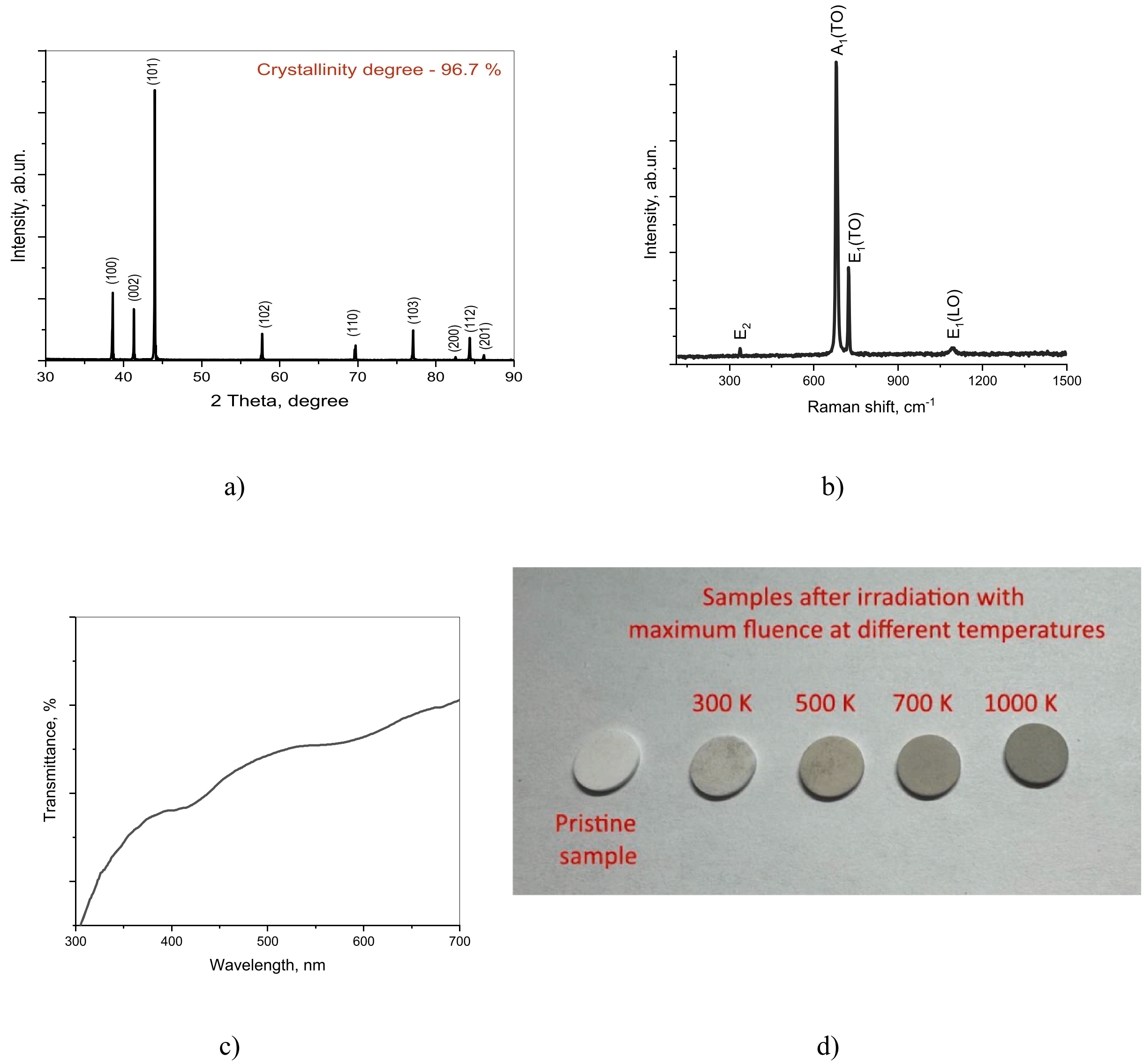

Fig. 1. Characterization of the initial BeO ceramic sample: a) X-ray diffraction pattern; b) Raman spectrum; c) optical transmission spectrum; d) Image of ceramic surfaces before and after irradiation, showing changes caused by variations in irradiation conditions.

ions was chosen to simulate the processes of helium accumulation in the near-surface layer of ceramics. Variation of the irradiation fluence made it possible to establish the kinetics of changes in the near-surface layer associated with the formation of gas-filled inclusions. Irradiation was carried out using low-energy He^{2+} ions with an energy of 40 keV, the particle flux density was about 50 nA/cm^2 $\times$ *s*, the irradiation fluences were chosen considering the possibility of modeling damage in the range from 1 to 50 dpa and ranged from 10^{16} to 3×10^{17} cm^{-2}. The influence of temperature factors on the degree of disordering, as well as the associated diffusion mechanisms leading to the migration of defects into the depths of the samples, leading to the destabilization of structural parameters, was determined by irradiation with He^{2+} ions at various temperatures in the range from 300 K to 1000 K. Heating of samples during irradiation was carried out using a ceramic heater. Samples were placed on the heated surface of the holder and secured with three clamps along the edges of the sample, which allowed the sample to be held tightly pressed to the heating surface during irradiation. Thermocouples were placed on both sides of the sample at four points: on the side pressed against the heater surface and on the front side exposed to irradiation. Such a scheme of thermocouple placement allows monitoring the uniformity of heating of the samples, as well as any changes in temperature during irradiation.

Fig. 1d shows the results of the images of the surface of the studied ceramics before and after irradiation with a fluence of 3×10^{17} cm^{-2} at different temperatures. The obtained data reflect the role of the irradiation temperature on the change in color, and consequently, the optical properties of the near-surface layers. The result of irradiation at different temperatures is a change in the color of the near-surface layer from white to dark gray, which in turn can be explained by the formation of defects and vacancies in the near-surface layer of ceramics. Moreover, the change in color depending on the irradiation temperature indicates the intensification of the processes of accumulation of defects in the damaged layer due to the thermal effect on the structure of the damaged layer.

The structural degradation was measured using the X-ray diffraction method, realized by recording X-ray diffraction patterns on a D8 Advance ECO diffractometer (Bruker, Germany). The deformation of chemical bonds and amorphization of the damaged layer by depth depending on the irradiation temperature were determined by analyzing the results of Raman spectra of the studied samples obtained on an Enspectr M532 Raman spectrometer (Spectr-M LLC, Chernogolovka, Russia).

The amorphous fraction concentration in the damaged layer was determined taking into account formula (1):

$$f_A = 1 - \frac{\sum_{n}^{i=1} \frac{A_i^{irradiated}}{A_i^{unirradiated}}}{n}, \tag{1}$$

where $A^{unirradiated}$, $A^{irradiated}$ are the weighted contributions of the intensities of the spectral lines in the Raman spectra of the unirradiated and irradiated samples, n is the number of spectral lines. This formula was proposed in the work [27], describing the effect of irradiation on the structure of oxide ceramics.

Determination of the concentration dependences of vacancy defects was assessed on the basis of changes in induced optical absorption spectra obtained by comparative analysis of the spectra of irradiated samples with the initial samples, on the basis of which the temperature dependences of changes in structural damage in ceramics were determined. Earlier, in [26] it was shown that for irradiated BeO ceramic samples, the main changes in the optical spectra are associated with a decrease in transmission capacity, a change in which indicates the accumulation of radiation damage that has a negative effect associated with the creation of additional scattering centers, as well as the formation of vacancy complexes at 3.4 – 2.84 eV, the presence of which is due to structural changes in the crystal structure of the irradiated samples.

The thermal conductivity of ceramics was determined using the longitudinal heat flow method, which makes it possible to estimate the change in thermal conductivity depending on the irradiation fluence, as well as to establish the role of the irradiation temperature on the change in thermophysical parameters. The measurements were carried out using the universal KIT-800 device.

3. Results and discussion

The influence of variations in irradiation conditions on changes in the properties of ceramics was determined by measuring structural parameters – the parameters of the crystal lattice (a and c), their ratio c/a, which characterizes the degree of anisotropy of the crystal lattice caused by external influences. In this case, the change in the parameters a and c were expressed through the value of the deformation distortion of each parameter, which was calculated by comparing the values of the irradiated samples with the results obtained for the initial samples. The degree of anisotropy was estimated by comparing the values of c/a of the irradiated samples with the results obtained for the initial samples. The combination of these parameters made it possible to determine the influence of both the irradiation fluence and the temperature at which the

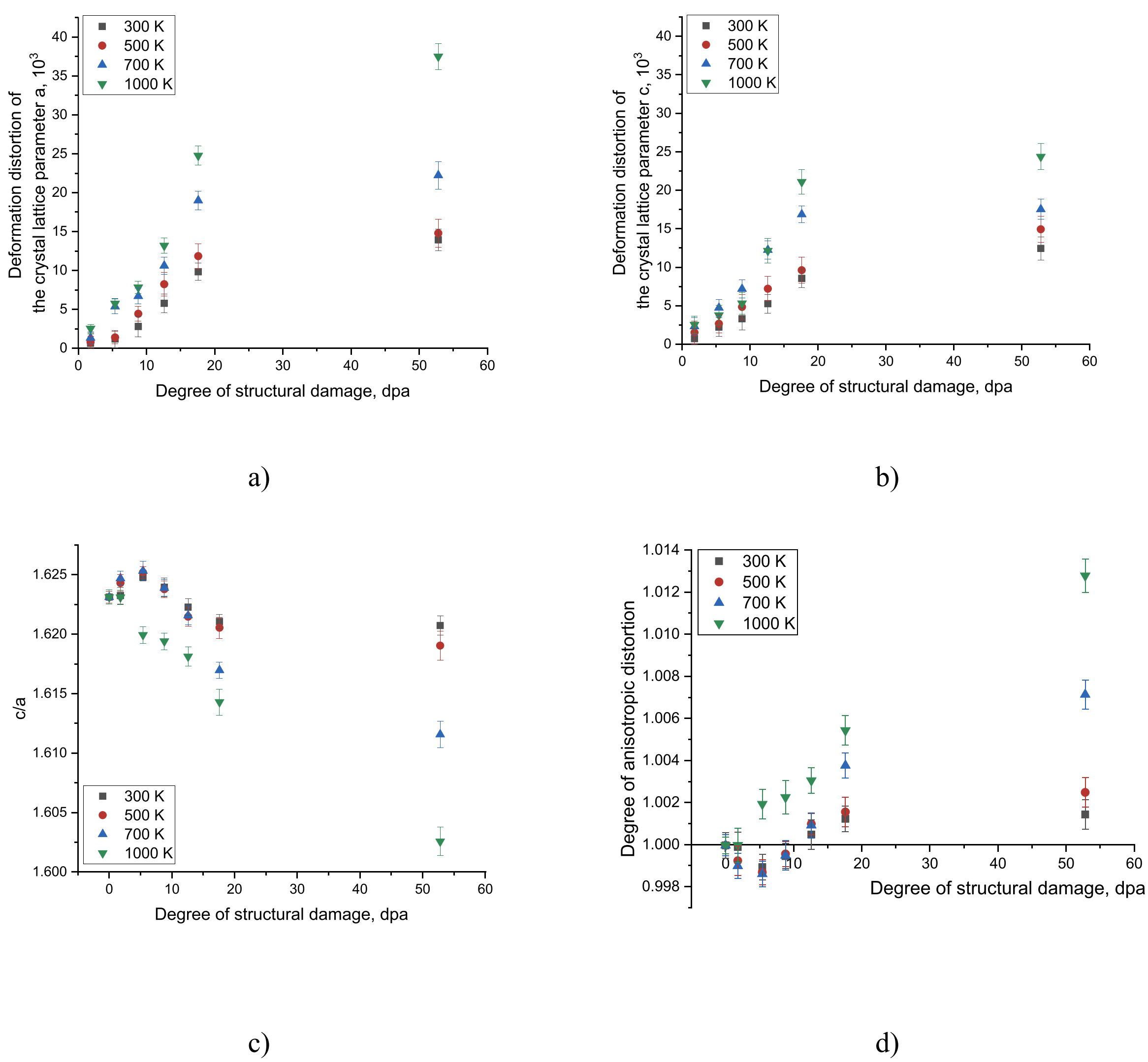

Fig. 2. Results of the assessment of deformation distortions of the crystal structure of BeO ceramics depending on the value of atomic displacements with variations in the irradiation temperature: a) deformation distortion of the crystal lattice along the parameter a; b) deformation distortion of the crystal lattice along the parameter c; c) results of the assessment of the change in the value of c/a, reflecting the packing density of the crystal lattice; d) results of the assessment of the degree of anisotropic distortion of the crystal lattice, calculated on the basis of changes in the value of c/a.

irradiation occurs on the degree of deformation distortion of the crystal structure. The values of deformation distortions of the crystal lattice parameters determined depending on the variation of the fluence conditions and irradiation temperature reflect the distortion of the crystal structure associated with the formation of point and vacancy defects in the damaged layer, as well as their evolution associated with agglomeration and diffusion in the damaged layer.

Fig. 2a-b reveals the evaluation results of the deformation distortions of the crystal lattice parameters of BeO ceramics depending on the data of changes in the irradiation conditions (variation of the irradiation fluence and temperature). The data are presented as a dependence of the deformation of each parameter of the hexagonal lattice axis depending on the value of the atomic displacements. The parameter of the value of atomic displacements was calculated using the method proposed in the work of G.W. Egeland et al. [28], as well as data from simulation of the interaction of He^{2+} ions with the near-surface layer of BeO ceramics, performed in the SRIM Pro 2013 software code. This value characterizes the degree of structural damage caused by atomic displacements during ion irradiation. This parameter also allows one to move from irradiation fluence to dimensionless values, which allows one to compare the degree of damage with other types of irradiation, including neutron irradiation, taking into account the depth of damage.

At the same time, maintenance of a single scale made it possible to compare changes in two parameters depending on the irradiation fluence and to determine the role of temperature effects during irradiation on the degree of deformation distortion of the crystal lattice. As is evident from the presented data on changes in deformation distortions of the crystal lattice axes, the observed trends under the same irradiation conditions (in this case, at the same temperatures) have significant differences, which indicates anisotropic distortion of the crystal lattice caused by irradiation. In this case, such changes are due to the formation of oxygen vacancies (V_O) and interstitial Be_i atoms, as well as complex defects, the formation of which occurs under high-dose irradiation. Moreover, comparing the observed changes depending on the irradiation temperature, we can conclude that the most pronounced effects of anisotropic distortion of the crystal lattice are observed at high irradiation temperatures (700 – 1000 K) and are more pronounced for deformation distortions of the a-axis than of the c-axis. Such effects can be explained by effects associated with the occurrence of local deformation distortions due to the mixing of beryllium and oxygen atoms, which leads to the weakening of chemical and crystalline bonds, as well as the accumulation of oxygen vacancies. The violation of symmetry due to the formation of oxygen vacancies and interstitial atoms leads to an increase in tensile stresses in the crystal lattice, and athermal deformations in the crystal structure, additionally enhanced by the effect of thermal heating, caused by a change in the distribution of electron density, lead to an increase in the deformation of the crystal lattice, which is clearly seen during comparison of the results of deformation distortions at the same irradiation fluence depending on the temperature. The observed increase in deformation distortions of the parameter a at high irradiation fluences in the case of an irradiation temperature of 1000 K indicates a significant contribution of the temperature effect to the processes of distortion of the crystalline structure of the damaged layer. In this case, the irradiation temperature grows from 300 K to 1000 K at an irradiation fluence of 5×10^{17} ion/cm^2, the increase in the degree of deformation of the parameter a is from 12 to 37 %, while the change in the parameter c is from 12 to 24 %. Thus, it can be concluded that an increase in the irradiation temperature leads to a more pronounced destabilization of the crystal lattice along the a axis, which leads to an increase in the anisotropic distortion of the crystal lattice, which is most pronounced with high-dose irradiation.

One explanation for such changes may be the effect of differences in the energies of defect formation in BeO ceramics. From the equation of balance of the BeO crystal lattice, written as $Be^{2+} + O^{2-} \rightarrow V_{Be} + V_O + Be\uparrow + 1/2O_2\uparrow$, it can be concluded that the main mechanisms of defect formation under external influences are associated with the formation of vacancy defects. In this case, differences in the energies of defect formation in the anion and cation sublattices lead to the dominance of the growth of defects of one type (usually oxygen vacancies), which leads to the formation of an uncompensated charge and the emergence of non-stoichiometry. Such differences can lead to an increase in defects in the case of the irradiation fluence growth, in which the effect of the impact is enhanced by an increase in the areas of overlap of deformation-distorted ionized regions that arise along the trajectory of ion movement in the damaged layer, and also be enhanced by the thermal effect on the crystal structure, which contributes to an increase in the mobility of both the atoms themselves in the lattice due to effects associated with a change in the amplitude of oscillations, and an increase in the mobility of the defects formed. In the case of low-energy irradiation with heavy ions, the differences in the values of ionization losses during interaction with electrons and nuclei are significantly smaller than in the case of high-energy ions, for which interactions with electrons play a dominant role at a greater depth of travel. In view of this, the processes of formation of vacancy defects caused by ionization of atoms and their knocking out of nodes are equally probable with the processes of changes in the distribution of electron density along the trajectory of ion movement in the damaged layer. In this case, the formation of oxygen vacancies, which occurs as a result of the interaction of incident ions with the crystal structure, can compete with the processes of anisotropic distortion caused by deformation stresses arising as a result of ionization processes, as well as athermal effects. Also, due to the cumulative effect of changes in electron density with increasing irradiation fluence, additional destabilization of the crystal structure can occur, which leads to the formation of additional oxygen vacancies in the damaged layer, which in turn enhances the effect of anisotropic distortion of the crystal lattice parameters, as well as the packing density of the crystal lattice (see the data shown in Fig. 2c). As can be seen from the dependences of the change in the ratio of the parameters c/a in Fig. 2c, at low irradiation fluences, the changes in the parameters are compensated by differences in deformation distortions, which are most pronounced at low irradiation temperatures. An increase in the irradiation fluence leads to the accumulation of deformation distortions, which, as a consequence, lead to a growth in the anisotropic distortion of the crystal lattice (see the data shown in Fig. 2d), as well as its disordering, expressed in the packing density reduction, a decrease in which indicates an increase in deformation distortions of the crystal lattice due to the formation of vacancy defects and interstitial atoms. It should be noted that at low irradiation fluences, the deformation anisotropic distortion is compensated by the stability of the crystal structure to deformation, while an increase in the irradiation temperature leads to an increase in distortion due to additional thermal broadening, which contributes to the growth of destabilization of the crystal structure.

According to the data presented, the growth of anisotropic distortion of the crystal structure with the irradiation temperature elevation from 300 to 700–1000 K may be due to an increase in the concentration of oxygen vacancies, the formation of which occurs due to thermal expansion and changes in the amplitude of thermal vibrations of the crystal lattice. In this case, thermal expansion in combination with defect formation processes caused by irradiation leads to an increase in structural distortions, as well as an increase in the probability of the formation of not only oxygen vacancies, but also complex defects of the V_O – He type. The growth of the defect fraction in the damaged layer due to thermal effects, as well as an increase in the irradiation fluence, leads to a more pronounced destabilization of the damaged layer, which, according to the data presented in Fig. 1d, leads to a change in the color of the surface, clearly demonstrating a change in properties.

Using the Raman spectroscopy method by conducting a comparative analysis of changes in the shape and intensity of spectral lines between irradiated samples and the initial (non-irradiated) ones, an assessment of structural changes associated with the accumulation of structural distortions and disordering of the crystal structure associated with the formation of highly disordered inclusions (amorphous or amorphous-like inclusions), was made. The determination of the concentration of amorphous inclusions was estimated based on Raman spectra, in particular, the determination of changes in the intensities of spectral lines, and subsequent calculation using formula (1). The determination of deformation distortions was carried out and the values of residual stresses were estimated by determination of the difference in the values of the shifts of the maxima of the spectral lines A_1(TO) and E_1(TO), the shift of which reflects the formation of residual stresses of tensile and compressive types in the structure. It should be noted that, unlike X-ray diffraction, the use of Raman spectroscopy allows for a more accurate comprehensive analysis of structural changes caused by irradiation, as well as determination of the nature of deformation distortions of chemical and crystalline bonds.

The concentration of amorphous inclusions in the damaged layer was estimated based on the obtained Raman spectra of ceramic samples exposed to irradiation with different fluences and at different temperatures. The results of the estimation are shown in Fig. 3a. As is evident from the data presented, the analysis of the concentration of amorphous inclusions shows that the growth trends have different natures, which are influenced by a combination of factors caused by both an increase in the irradiation fluence and temperature effects leading to the growth of disordered inclusions. It should be noted that the temperature factor manifests itself more intensively at low fluences than at high irradiation fluences (above 10^{17} ion/cm^2). In this case, the decrease in the trend of change in the concentration of amorphous inclusions in the damaged layer under high-dose irradiation can be explained by both the effects of accumulation and the diffusion of point and vacancy defects into the material, similar to the extrusion of the damaged volume into the depths and onto the surface in the form of hillocks [29]. According to previously conducted studies aimed at assessing the effect of temperature on the rate of degradation of the crystalline structure expressed in the growth of the value of volumetric swelling [29], it was established that the transition from irradiation at a temperature of 300 K to a temperature of 1000 K leads to a more than twofold increase in the volumetric swelling rate. At the same time, the irradiation temperature alteration from 300 to 500 K leads to a slight increase in the rate of volumetric swelling (less than 10 %), which indicates an acceleration of structural disordering processes at temperatures above 500 K. Moreover, the analysis showed that the changes caused by irradiation are associated not only with volumetric swelling, but also with the formation of hillocks on the surface of ceramics in the form of spherical inclusions, the density and size of which have a clearly expressed dependence on the irradiation temperature.

One of the methods for assessment of structural changes caused by irradiation, as well as determination of the role of temperature effects on the nature and type of structural distortions caused by irradiation, is the method of Raman spectroscopy, combined with the analysis of spectra using the theory of Raman piezospectroscopy, according to which the change in the position of spectral lines is associated with the accumulation of residual mechanical stresses in samples caused by external influences, including irradiation. Fig. 3b demonstrates the assessment

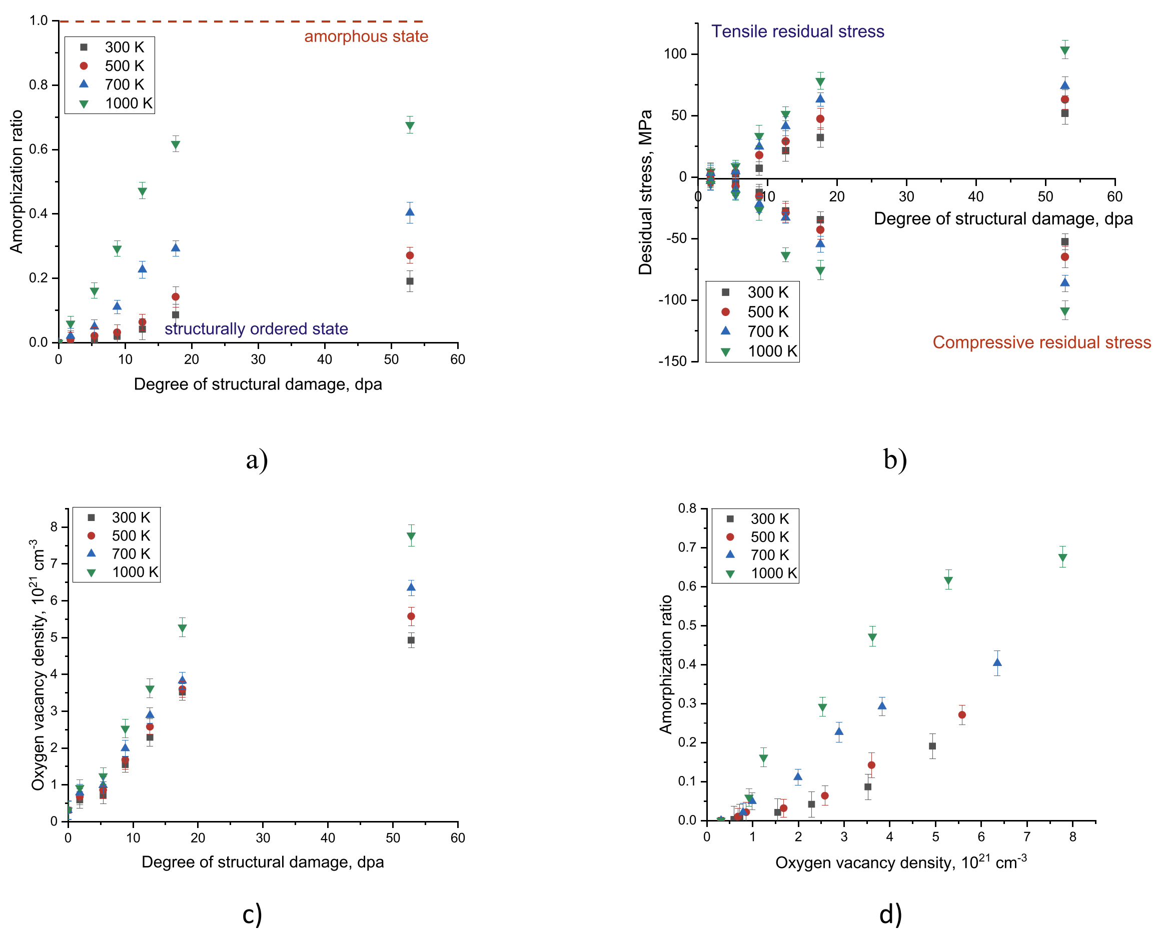

Fig. 3. a) Dependence of the change in the concentration of amorphous inclusions in the damaged layer with a change in irradiation conditions associated with an increase in the irradiation fluence and the temperature at which irradiation occurs; b) Results of the assessment of the values of residual stresses in the damaged layer depending on the variation of irradiation conditions; c) Results of the assessment of the concentration of oxygen vacancies in the structure of the damaged layer of BeO ceramics depending on the variation of irradiation conditions; d) Results of comparative analysis of the relationship between the change in the concentration of oxygen vacancies and amorphous inclusions in the composition of the damaged layer of BeO ceramics.

results of the change in the value of residual stresses obtained by comparative analysis of the shifts of the spectral lines of the Raman spectra, characterized by two spectral lines A_1(TO) and E_1(TO), the shift of which is associated with the accumulation of tensile and compressive stresses in the crystal structure. Calculations of residual stresses were performed taking into account the methodology proposed in [30–32], which is based on the assessment of the displacements of Raman spectral lines relative to the initial position. This effect of multidirectional distortion of the crystal structure was established in the work [26], according to which it was established that with the accumulation of radiation damage, the shift of the spectral band A_1(TIO) is associated with the accumulation of tensile residual stresses, and the shift of the band E_1(TO) with compressive residual stresses. In this case, the nature of changes in the values of residual stresses in the damaged layer depending on the irradiation fluence has a clearly expressed dependence on the irradiation temperature, the growth of which leads to an increase in the destabilization of the damaged layer, as well as an elevation in the amorphous fraction in the composition of ceramics. As can be seen from the data presented, a growth in the irradiation fluence leads to an increase in the differences in the values of shifts of spectral lines associated with deformation tensile and compressive stresses in the structure. At the same time, the analysis of trends in changes in tensile and compressive stresses depending on the irradiation fluence for different temperatures showed an increase in compressive stresses with increasing fluence, which indicates the anisotropic nature of distortions caused by the accumulation of structural distortions. It should also be noted that in the case of irradiation at temperatures of 700 – 1000 K, the trends in changes in residual stresses in the structure are more pronounced than in the case of ceramic samples irradiated at lower temperatures (300 – 500 K). Such differences in trends indicate the influence of temperature effects caused by irradiation during heating of samples on the degree and rate of degradation of the damaged layer. In turn, the accumulation of residual stresses of different directions (compressive and tensile) in the structure causes acceleration of the processes of destabilization of the crystal lattice and its disordering, which is confirmed by the data shown in Fig. 3a. The obtained results make it possible to draw a conclusion about the heterogeneity of the defect distribution in the near-surface layer, associated with the competition of ionization, athermal and ballistic effects that contribute to the degradation of the damaged layer. At the same time, the decrease in the trends of residual stress changes at high irradiation doses (above 10 dpa) can be explained by the ballistic extrusion of some defects deep into the samples, thereby increasing the damage area, which can significantly exceed the ion path depth in the material (about 500 nm, according to SRIM Pro 2013 calculations). In turn, the growth of tensile stresses in the damaged layer at irradiation fluence growth is associated with an increase in the concentration of oxygen vacancies and point defects, the accumulation of which contributes to local distortion and expansion of the crystal lattice. The growth of compressive stresses in this case is due to the formation of local inclusions in the form of gas-filled inclusions during the accumulation of helium in the damaged layer, which in turn results in deformation compression of the crystal lattice.

Fig. 3c illustrates the assessment results of the concentration of oxygen vacancies in the damaged layer of ceramics depending on the variation of irradiation conditions. The data were obtained by assessment of the induced absorption spectra of BeO ceramics and subsequent comparison of the results with the initial data obtained for ceramic samples not subjected to irradiation. According to the conducted assessments of oxygen vacancies in the damaged layer, the observed changes indicate both a cumulative effect caused by an increase in the irradiation fluence and the influence of thermal exposure, which contributes to the intensification of defect formation processes associated with the accumulation of oxygen vacancies. The growth of the concentration of oxygen vacancies and vacancy complexes $He – V_O$ in irradiated ceramics with an increase in the irradiation temperature is due to the fact that the covalent-ionic nature of the Be – O bonds promotes the mobility of oxygen when external influences change, in this case the effect of temperature, leading to an increase in the amplitude of thermal vibrations. In this case, the formation of oxygen vacancies is associated with the difference in the displacement energies between Be and O, the values of which are $E_d(Be) = 30 – 50$ eV and $E_d(O) = 15 – 25$ eV, respectively. Such differences in displacement energies, together with the anisotropic distortion of the crystal lattice, established during the analysis of structural parameters, indicate that with an increase in irradiation fluence, the thermal effect associated with external heating of the samples during irradiation leads to an increase in the number of oxygen vacancies and vacancy complexes in the damaged layer. In this case, the change in the irradiation temperature leads to an increase in the mobility of the crystal structure, which in turn promotes the migration of defects, thereby increasing the degree of damage due to the formation of oxygen vacancies when chemical bonds are broken. In turn, an increase in the concentration of oxygen vacancies will contribute to the disordering of the damaged layer, which leads to the growth of amorphous inclusions in the damaged layer. Direct confirmation of this conclusion is the direct correlation between the concentration of oxygen vacancies and the change in the concentration of amorphous inclusions in the composition of the damaged layer, shown in Fig. 3d

Fig. 4a reveals the assessment results of changes in the thermal conductivity of the studied BeO ceramics depending on the irradiation fluence and temperature. These changes in the thermal conductivity coefficient are given in terms of the values of atomic displacements calculated considering the irradiation fluence, which made it possible to conduct a comparative analysis of the destructive change in the thermophysical parameters of the ceramics. According to the data presented, the observed trends in the thermal conductivity coefficient indicate a destructive effect of the accumulation of structural damage on heat transfer processes. The general view of the trends in the thermal conductivity coefficient has a clearly expressed dependence on both the irradiation fluence (the value of atomic displacements) and the temperature at which the samples are irradiated. In the case of small damage doses (less than 10 dpa) at irradiation temperatures of 300 and 500 K, the change in the thermal conductivity coefficient is insignificant, which indicates a fairly high resistance of ceramics to heat losses. However, with an increase in the irradiation temperature, the observed increase in heat losses (see data in Fig. 4b-c) can be due to an increase in the contribution of the concentration of oxygen vacancies in the damaged layer, an increase in which results in rescattering of thermal phonons, which significantly slows down the heat transfer process.

Figs. 4b-c show the results of a comparative analysis of the relationships between changes in thermal conductivity losses and amorphous inclusions and the concentration of oxygen vacancies depending on the irradiation conditions. The data are presented to clearly demonstrate the role of temperature effects during irradiation, leading to the acceleration of the processes of destabilization of the damaged layer on the change in thermal conductivity, established during the experiments. According to the analysis of the obtained dependencies, it can be concluded that there is a direct correlation between changes in thermal conductivity losses and structural damage, the accumulation of which leads to an increase in scattering centers that have a negative impact on phonon heat transfer and reduce the efficiency of heat transfer. At the same time, the observed changes in the degradation of heat-conducting properties under high-temperature irradiation indicate that the processes of anisotropic distortion of the crystalline structure lead to an abnormal growth in heat losses due to the presence of a large number of vacancy and point defects, and in the case of high-dose irradiation, the formation of gas-filled inclusions, the presence of which leads to an increase in deformation distortions of the crystalline structure.

Fig. 4d demonstrates the comparative analysis results of changes in the thermal conductivity of ceramics measured at a given depth and the concentration of amorphous inclusions obtained by assessment of the intensities of Raman spectra. The imaging was carried out with a step of

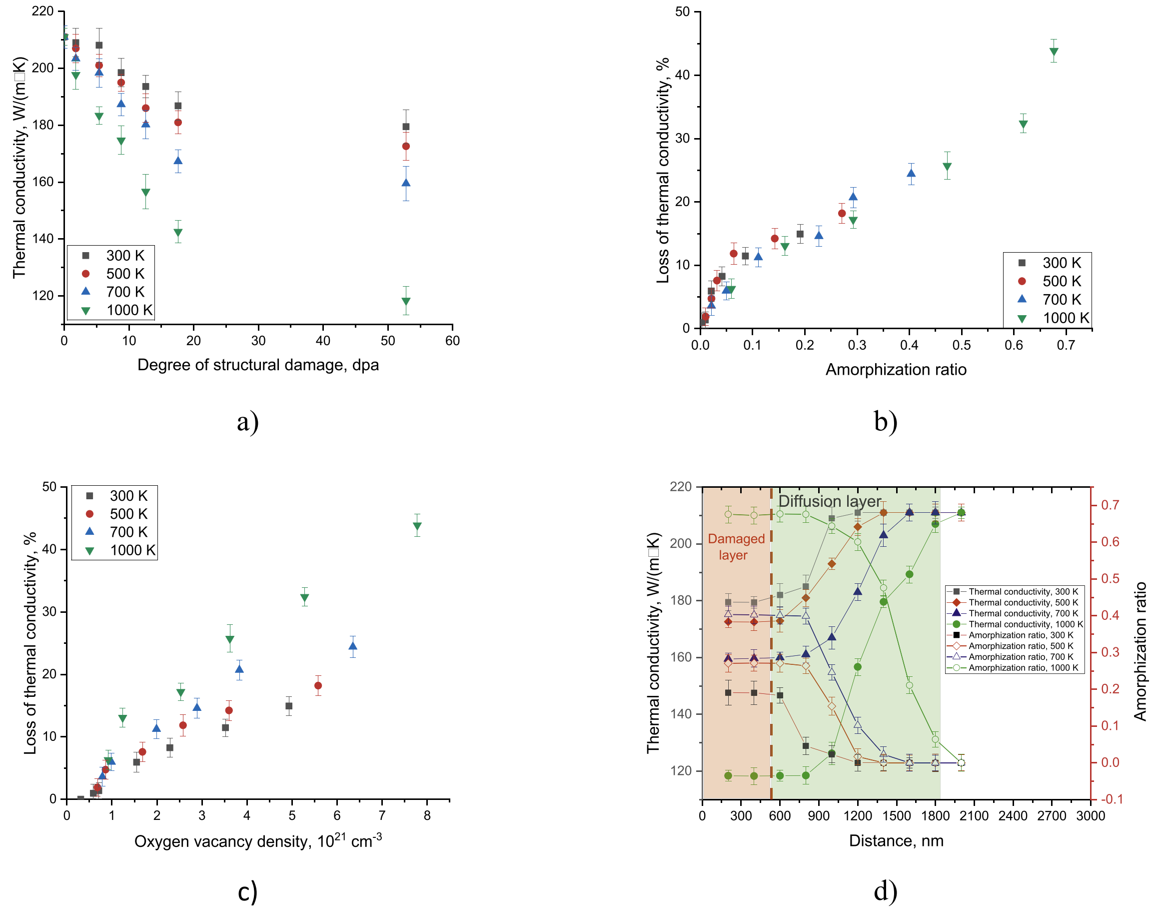

Fig. 4. a) Results of the assessment of the change in the thermal conductivity coefficient of BeO ceramics with changes in irradiation conditions; b) Results of a comparative analysis of changes in thermal conductivity losses from the concentration of amorphous inclusions, determined from the analysis of Raman spectra; c) Results of a comparative analysis of the relationship between the values of thermal conductivity losses and the concentration of oxygen vacancies in the structure of the damaged ceramic layer; d) Results of comparative analysis of measurements of thermal conductivity and concentration of amorphous inclusions at maximum irradiation fluence performed along the depth of the sample, reflecting the diffusion of defects into the damaged layer.

200 nm, which made it possible to assess the changes in these values in the damaged layer, the depth of which is about 500 nm, according to the calculated data of SRIM Pro 2013, and also to determine the effect of irradiation temperature on the diffusion of defects into the sample, leading to amorphization and, as a consequence, to a loss of thermal conductivity. Previously, in several works [33,34] it was shown that an increase in the fluence of low-energy ion irradiation leads to a significant increase in the depth of the damaged layer due to the diffusion of point and vacancy defects into the sample, thereby increasing the thickness of the damaged layer due to disordering and destabilization of the crystal lattice at a depth exceeding the penetration depth of ions in the irradiated material. According to previously conducted studies [26] it was established that the accumulation of structural damage leads to a slowdown in heat transfer processes associated with the formation of additional scattering centers formed as a result of disordering and subsequent amorphization of the damaged layer. Moreover, the authors of the work [26] showed that the change in thermal conductivity occurs not only in the near-surface damaged layer, comparable to the ion range, but also at a greater depth, which indicates degradation processes exceeding the thickness of the damaged layer. At the same time, the nature of changes in the thermal conductivity coefficient with depth showed a clearly nonlinear dependence of changes in thermal conductivity properties on the measurement depth, which indicates a diffusion mechanism for the migration of defects into the damaged layer. With an increase in the irradiation fluence, an increase in the concentration of the resulting defects, as well as vacancy complexes, leads to their displacement into the depths of the sample, as well as to the formation of gas-filled inclusions near the surface, creating additional distortions of the crystal structure. According to the data obtained, an elevation in the irradiation temperature from 300 K to 1000 K leads to an increase in the depth of the damaged layer by more than 1.5 times compared to the calculated data on the ion path depth in BeO ceramics. At the same time, the observed effects of correlation of the concentration of amorphous inclusions and changes in thermal conductivity indicate a ballistic effect of squeezing structural damage into the depth of the sample during intense damage caused by a combination of factors of thermal impact and diffusion of defects in the damaged layer.

The broadening of the damaged layer caused by irradiation, i.e. the layer in which a change in thermal conductivity is observed, depending on the irradiation conditions (changes in irradiation temperature) can be explained as follows. In the case of high-dose irradiation, in addition to oxygen vacancies, vacancy complexes of the V_O – He type, the accumulation of which enhances the destabilization of the damaged layer, can form in the structure. At the same time, the thermal expansion of the crystal structure caused by external temperature influence during irradiation contributes to an increase in the mobility of defects, which in turn leads to an increase in the probability of their agglomeration in voids formed during deformation distortion of the crystal lattice. Also, the established effect of anisotropic distortion of the crystal lattice, associated with the accumulation of tensile and compressive stresses, leads to increased destabilization, thereby displacing part of the defects both deep into the sample, which leads to an increase in the thickness of the damaged layer, and to the surface, resulting in the formation of gas-filled blisters on the surface. Such structural changes caused by irradiation can have a negative impact on heat transfer mechanisms by creating additional obstacles to phonon heat transfer, as well as enhancing the effect of rescattering, thereby slowing down heat transfer. Moreover, the nonlinear nature of the change in the thermal conductivity coefficient at a depth exceeding the ion range in the material indicates the ballistic nature of defect migration, leading to a non-uniform distribution of defects in the damaged layer. According to the data obtained, an increase in the irradiation temperature leads to a more than three- to four-fold increase in the depth of the damaged layer, which must be taken into account during design and assessment of the possibilities of using inert matrices or functional materials operating in extreme conditions.

4. Conclusion

Based on the conducted studies aimed at study of the kinetics of degradation of BeO ceramics irradiated with low-energy He^{2+} ions, the following conclusions have been formulated, making it possible to expand our understanding of the processes of disordering of the near-surface layers of ceramics, as well as to determine the role of temperature effects in the processes of destabilization of the damaged layer.

Analysis of changes in structural distortions and residual stresses in the damaged layer made it possible to determine that at low damage doses (less than 10 dpa), the dominant role is played by tensile stresses associated with the accumulation of vacancy and point defects in the damaged layer, while at damage doses above 10 dpa, the dominant role is played by compressive residual stresses associated with the formation of gas-filled inclusions in the damaged layer during the accumulation of helium and vacancy complexes. It was found that an increase in the irradiation temperature leads to an increase in deformation distortions caused by irradiation due to thermal expansion and acceleration of the diffusion of defects in the damaged layer.

According to the obtained data from a comparative analysis of changes in the thermal conductivity coefficient and the concentration of amorphous inclusions by depth, a direct correlation was established between these parameters. Moreover, the analysis of the obtained dependencies showed that in addition to the general degradation of the thermophysical parameters of ceramics associated with increased disordering due to thermal exposure during irradiation, the observed correlation of the thermal conductivity parameters and the concentration of amorphous inclusions by depth indicates a diffusion expansion of the damaged layer. The observed trends in changes in the concentration of amorphous inclusions at a depth exceeding the ion range in the near-surface layer indicate a diffusion mechanism for the migration of point and vacancy defects, the change of which is ballistic in nature with a clearly expressed decrease in concentration depending on the measurement depth. It is important to highlight that the damaged layer thickness has a clearly expressed dependence on the irradiation temperature, which indicates that thermal heating clearly promotes increased migration of defects in the samples, thereby increasing damage not only in the damaged layer, but also beyond the maximum ion range in the material due to the diffusion of defects into the depth.

According to the conducted studies, it can be concluded that when considering the potential for the use of BeO ceramics as structural materials used in extreme conditions, it is necessary to take into account the effect of anisotropic distortion of the crystal structure caused by a combination of factors of high-temperature exposure and accumulation of radiation damage.

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CRediT authorship contribution statement

Azamat E. Ryskulov: Visualization, Software, Resources, Methodology, Investigation, Data curation, Conceptualization. **Artem L. Kozlovskiy:** Writing – review & editing, Writing – original draft, Validation, Resources, Investigation, Formal analysis, Data curation, Conceptualization. **Igor A. Ivanov:** Project administration, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Inesh E. Kenzhina:** Writing – original draft, Visualization, Validation, Supervision, Investigation, Formal analysis, Conceptualization. **Marina Konuhova:** Software, Resources, 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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