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ORIGINAL STUDY

Determination of the Influence of Temperature Factors on the Softening of the Near-surface Layer of BeO Ceramics Under High-dose Irradiation With Helium Ions

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Abstract

Interest in BeO ceramics as structural materials for new generation reactors, despite the fairly active research of this type of ceramics previously, is primarily due to the combination of their thermophysical, strength and mechanical properties, as well as a low neutron capture cross-section, which allows them to be considered as one of the most promising materials for the creation of new generation fuel elements. The paper defines the main factors and critical irradiation doses at which destruction of the near-surface layer of BeO ceramics exposed to irradiation with low-energy helium ions is observed in the case of irradiation temperature variations in the range from 300 to 1000 K. During experimental work, the influence of temperature on the crystal structure during irradiation and its role in changing the rate of destruction and softening of the near-surface layer of ceramics under high-dose irradiation were revealed. A direct relationship between the change in the strength characteristics of ceramics exposed to irradiation and temperature-induced processes of volumetric deformation swelling of the damaged layer has been determined. According to the data obtained, in the case of high-temperature irradiation it is necessary to consider the role of thermal expansion leading to increased deformation distortions and acceleration of swelling processes of the crystal structure due to accelerated diffusion of point and vacancy defects in the damaged layer.

Keywords: Softening, Swelling, Embrittlement, Destruction, Accumulation of helium, BeO ceramics

1. Introduction

The accumulation of radiation damage during the operation of ceramic structural materials, as is known, leads not only to the destabilization of the crystalline structure, which consists of partial or complete destruction of crystalline and chemical bonds, leading to amorphization and loss of short-range order, but also to effects associated with the softening of the near-surface layer, caused by the accumulation of deformation distortions and stresses [1,2]. In turn, destabilization of the crystalline structure in the near-surface layers due to the accumulation of deformation structural distortions and stresses can lead to a decrease in the resistance of ceramics to external mechanical influences and a loss of strength, including impacts, friction or fracture under external loads [3]. Destructive changes in the properties of the near-surface layer of ceramics during the accumulation of radiation damage, especially if irradiation is accompanied by direct additional external influences, including heating, aggressive environments, can lead to partial or complete destruction of the near-surface layer, consisting of peeling or cracking, which in turn can lead to

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contamination of the coolant or destabilization of the active zone [4,5]. In this regard, any structural material under consideration for new-generation reactors (especially high-temperature nuclear reactors) must have increased resistance to both the accumulation of radiation damage and its evolution, characteristic of high-dose irradiation, and other external effects, including high temperatures (of the order of 500–1000 K) and mechanical loads that arise during operation, as well as thermal expansion of materials. The use of ceramic materials as structural materials, especially when used as inert matrix materials, implies direct contact of these materials with the coolant, as well as their exposure to fission fragments or neutrons, which leads to the accumulation of radiation damage in the near-surface layers [6,7].

The main aim of this experimental work is to identify the effect of heating temperature of samples under high-dose irradiation with low-energy helium ions on the change in the stability of the strength properties of the near-surface layer, as well as to establish the relationship between the mechanisms of helium agglomeration into gas-filled bubbles and the irradiation temperature and their role in the destruction of the near-surface layer. At the same time, identification of the influence of temperature on the softening degree of the near-surface layer, which is most susceptible to external influences in the case of operation associated with both radiation and mechanical impacts, is a determining factor that allows adjustments to be made to the operating conditions of this type of ceramics [8–10]. In turn, the acceleration of destruction processes due to thermal expansion or thermally induced diffusion of defects in the damaged layer is one of the key factors that can have a negative impact on strength properties, as well as significantly reduce the critical doses of radiation at which irreversible destruction of the damaged ceramic layer occurs [11,12]. The choice of ceramics based on beryllium oxide as objects of study is due to the combination of its physical, chemical, strength and thermal characteristics, as well as low values of the neutron absorption cross-section, which allows us to consider this type of ceramics both as matrices for dispersed nuclear fuel and as additional moderators in new-generation reactors, including high-temperature gas reactors in which helium and CO_2 are used as coolants, which in turn requires the use of materials with high thermal expansion, strength and thermal conductivity [12–15]. In this case, the process of ceramics operation is usually accompanied by constant contact with the coolant, which results in the initiation of the processes of penetration of helium from their coolant into the near-surface layers of ceramics, which leads to its destabilization. Due to the inertness of helium, especially when operating at high temperatures, leading to an increase in the mobility of helium, migration and diffusion of helium atoms into the depth of the near-surface layer is possible, which, in the case of mechanical loads, leads to accelerated destabilization of the crystalline structure, as well as its swelling [16,17]. This type of ceramics is also considered as one of the classes of functional structural materials in the composition of heat-conducting protective materials capable of withstanding operation in critical extreme conditions. At the same time, higher thermal conductivity indicators in combination with strength parameters in the case of extreme operating conditions allow for an increased "safety margin" in emergency situations associated with a sharp change in operating temperature conditions, which is a very important parameter for new-generation reactors. However, when considering the prospects for using this type of ceramics as materials used in extreme conditions, it is necessary to take into account temperature factors that can lead to the acceleration of helium diffusion processes, as well as the acceleration of the destabilization of the crystal structure due to thermal expansion and increased mobility of vacancies, which can lead to the growth of microcracks and pores, thereby contributing to the processes of helium swelling and softening. Determination of the role of temperature factors is one of the important criteria for determining the potential of ceramic materials as materials for high-temperature nuclear reactors, given that any materials considered and used in new-generation reactors will be exposed to both high doses of radiation and the effects of high temperatures and mechanical stresses and pressures.

2. Materials and methods

Polycrystalline ceramics based on beryllium oxide (BeO), which have a hexagonal type of crystal lattice with parameters a = 2.6857 Å c = 4.3592 Å and a volume of 27.23 $Å^3$, were chosen as objects for research. Fig. 1 shows the X-ray diffraction pattern of the original sample. The choice of objects of study is based on the possibility of assessment of the resistance of this class of ceramics to radiation damage and the consequences associated with their accumulation in the near-surface layers, as well as their influence on the change in strength characteristics, which will make it possible to assess the prospects for using this class of ceramics as structural materials for nuclear reactors, including as materials of inert matrices that have a large safety margin due to high resistance to external influences.

The experiments on irradiation with low-energy He+ ions were carried out on the basis of the DC-60 accelerator (Astana branch of the Institute of Nuclear Physics, Astana, Kazakhstan). The irradiation of

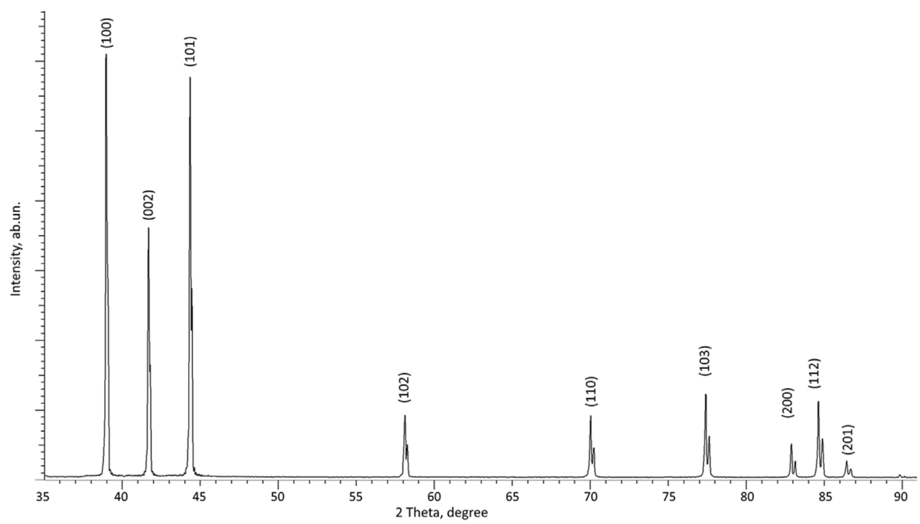


Fig. 1. Results of X-ray diffraction of the studied BeO ceramic sample in the initial state.

ceramic samples was carried out in a special chamber using a target holder capable of heating the samples to a given temperature and then maintaining this temperature throughout the irradiation time. The irradiation fluences were selected from 10^{16} cm^{-2} to 3×10^{17} cm^{-2}, which corresponds to a range of atomic displacements from 1.8 to 53 dpa [18]. Simulation of temperature factors affecting structural changes caused by irradiation was carried out by irradiating samples at temperatures from 300 to 1000 K.

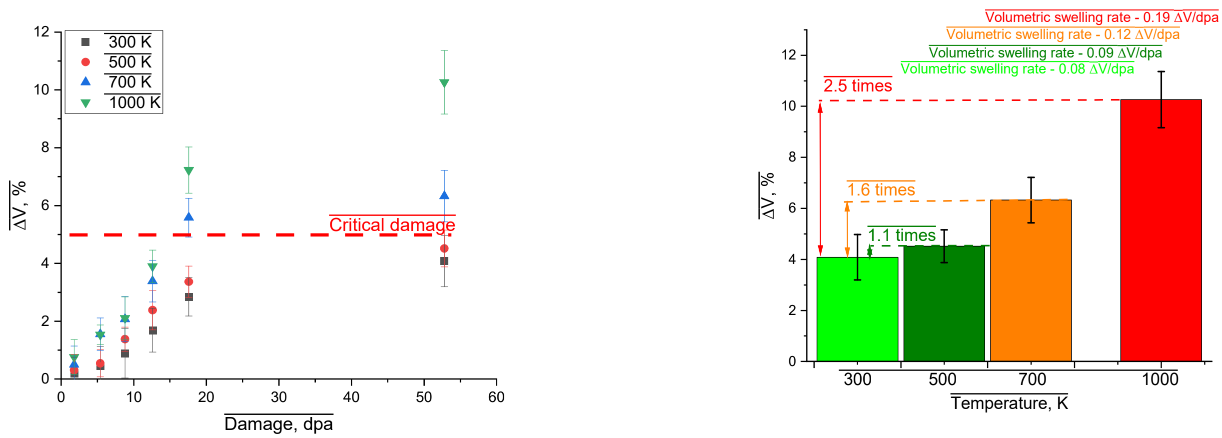
The softening degree depending on the irradiation fluence was determined by estimating the volumetric swelling value of the damaged layer in ceramics by calculating changes in the crystal lattice parameters of the samples before and after irradiation. The volumetric swelling value (ΔV) made it possible to determine the effect of temperature factors on the destructive change in the crystal structure depending on the irradiation fluence, and also to establish the role of temperature factors in the disordering of the damaged layer. At the same time, this calculation of the ΔV value makes it possible to determine the volumetric changes in the structural parameters without consideration of the anisotropic effects of deformation distortion of the crystal lattice parameters, characteristic of hexagonal types, which was reported in the fundamental work of Walker, D. G. et al. [19]. As a criterion for determination of the critical damage dose based on the change in the ΔV value, a value equal to a deviation of the ΔV value by 5 % from the initial value was chosen.

The study of the effect of irradiation temperature on the formation of gas-filled inclusions on the surface at maximum irradiation fluence was carried out by obtaining and subsequently analyzing surface images using the scanning electron microscopy method implemented on a JEOL 7500F microscope.

The strength parameters of the samples under study, as well as their changes as a result of irradiation with He^{+} ions, were studied using the indentation method (hardness determination method) and the bending strength determination method. These methods were implemented using a Duroline M1 microhardness tester (Metkon, Bursa, Turkey) and an LFM-L 10 kH testing machine (Walter + Bai AG, Löningen, Switzerland).

3. Results and discussion

Fig. 2 shows the assessment results of changes in the ΔV value, reflecting the volumetric swelling of the crystal lattice of the damaged layer caused by irradiation at different temperatures. This dependence reflects the effect of the accumulation of deformation distortions of the crystal structure with an increase in the irradiation fluence (the data are presented as a dependence on atomic displacements), as well as the role of temperature effects on the change in the degree of deformation distortion of the crystal structure with the accumulation of damage. The general trend of the presented dependencies reflects the formation of tensile deformation stresses in the damaged layer, the appearance of which leads to destabilization of the crystal structure, expressed in an increase in the volume of the crystal lattice associated with distortion and stretching. The process of destabilization of the crystal structure itself is caused by the fact that during the irradiation process, vacancy defects and interstitial atoms are formed in the damaged layer during the interaction of incident ions with the crystal structure, an increase in the density of which due to an increase in the irradiation fluence leads to the formation of more complex defects in the form of cluster defects, dislocations, vacancy clusters, etc. At the same time, the accumulation of defects and, as a consequence, the increase in



a) b)

Fig. 2. a) Results of trends in ΔV values depending on the variation of atomic displacements caused by irradiation; b) Comparative diagram of the maximum value of ΔV at an irradiation fluence of 3 × 10^{17} cm^{-2}, corresponding to 53 dpa.

their density results in elevation of distorting stresses in the damaged layer, which has a negative effect on the crystalline structure, which together leads to an increase in deformation and volumetric distortion of the crystal lattice. In this case, an increase in the density of local defects in the form of vacancies and interstitial atoms leads to an increase in tensile stresses, and in the case of high irradiation fluences, this effect can lead to destabilization of the crystalline structure due to the formation of microcracks and pores that can be filled with implanted helium migrating in the damaged layer. At the same time, as can be seen from the presented trends of changes in the ΔV value depending on the irradiation temperature, an increase is observed in the case of high temperatures (700–1000 K), which is most pronounced at fluences above 10^{17} cm^{-2} (at values of the order of 20–50 dpa), for which the ΔV value exceeds the threshold of 5 % selected as the critical boundary. It is important to highlight that in the case of irradiation of samples at temperatures below 700 K, the deformation distortion of the crystal structure does not exceed the threshold value of 5 % even in the case of maximum irradiation fluence (3 × 10^{17} cm^{-2}). Thus, it can be concluded that the increase in deformation distortions is associated with a change in the migration rate and mobility of defects in the damaged layer, which leads to a growth in deformation distortions associated with the formation of larger defect clusters in the damaged layer. Also, an elevation in the irradiation temperature leads to an increase in the contribution of thermal expansion of the crystal structure, which contributes to more intense diffusion of defects, and consequently, the creation of additional deformation distortions resulting in destabilization of the crystal structure. The results of a comparative analysis of the ΔV value at a maximum irradiation fluence of 3 × 10^{17} cm^{-2} presented in Fig. 1b indicate that the irradiation temperature growth to 700–1000 K results in more than 1.6–2.5-fold increase in deformation, from which it follows that the rate of accumulation of damage caused by irradiation is more than 1.5–2 times higher due to thermal expansion caused by prolonged thermal exposure during irradiation.

Fig. 3 reveals the results of alterations in the surface morphology of the studied BeO ceramics subjected to high-dose irradiation with He^{2+} ions at a fluence of 3 × 10^{17} cm^{-2}, characteristic of the accumulation of damage of more than 50 atomic displacements. The presented surface images reflect changes associated with the formation of gas-filled inclusions in the near-surface layer, the change in shape, density and size of which depending on the irradiation temperature indicates the influence of temperature factors on the processes of helium agglomeration in the near-surface layer. The presence of such gas inclusions was detected during neutron irradiation in [20], as well as in several other works [21–23], which showed that the formation of such gas inclusions is due to various factors, including grain size and the temperature at which irradiation occurs. According to the presented images, it is evident that at an irradiation temperature of 300 K, the formation of gas-filled inclusions on the surface is quite active, but the sizes of these inclusions are small, which indicates that the helium bubbles that are formed quite actively come to the surface near the grain boundaries. In case of the irradiation temperature growth from 300 to 500 K, enlargement of bubbles is observed, and their density becomes significantly lower than in case of irradiation at temperature of 300 K. Such an effect can be explained by acceleration of

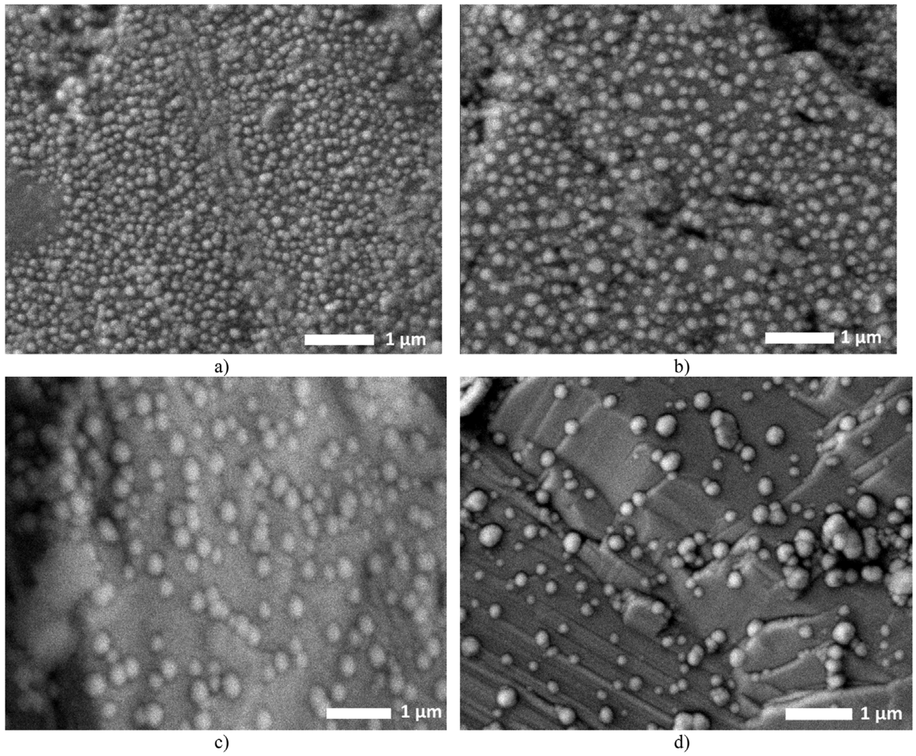
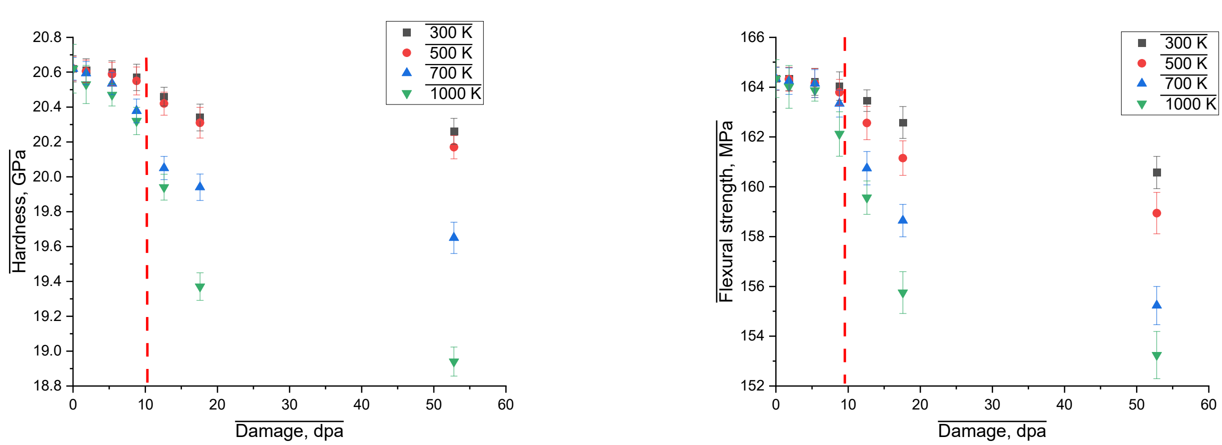


Fig. 3. Results of surface morphology visualization, reflecting the formation of gas-filled bubbles on the surface at maximum irradiation fluence, as well as the role of irradiation temperature on the density and size of gas-filled inclusions: a) 300 K; b) 500 K; c) 700 K; d) 1000 K.

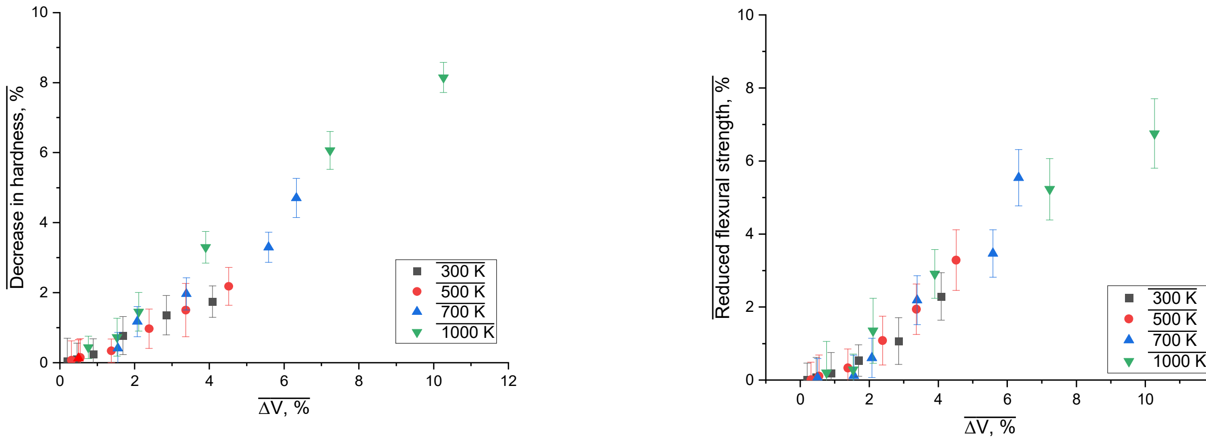
diffusion of implanted helium in near-surface layer, which leads to merging of smaller bubbles into larger ones. Further increase of irradiation temperature from 500 to 700 K and 1000 K leads to enhancement of agglomeration effect, which in case of irradiation at 1000 K leads not only to formation of large bubbles (more than 1.5—2.5 times exceeding in size similar inclusions observed during irradiation at temperature of 300 K), but also their agglomeration into large clusters near micropores, formed as a result of deformation damages caused by irradiation. The observed effects of the size enlargement of gas bubbles at reduction of their density (number per unit area) are explained by the effects of accelerated helium diffusion in the damaged layer, caused by the temperature effect on the crystal lattice associated with thermal expansion, which contributes not only to the acceleration of the migration of implanted helium, but also to an increase in the deformation of the crystal structure. In the case of high temperatures at which irradiation occurs, and as a consequence, helium implantation in the damaged layer, thermal expansion of the crystal structure contributes to easier movement (migration) of defects in the damaged layer, which leads to deformation stretching of the crystal structure, which is clearly evidenced by the results of the trends in changes in the volumetric swelling value (see data in Fig. 1). An increase in volumetric deformation distortions of the damaged layer can promote the growth of micropores formed both during the sintering process of ceramics (technological manufacturing process) and those opening as a result of radiation-stimulated deformation distortions, the density of which increases with the growth of the irradiation fluence (in this case, the growth of the magnitude of atomic displacements). The formation of a structurally disordered layer, the deformation distortions in which have a clearly expressed dependence on temperature factors, leads to the facilitation of the migration of implanted helium, the diffusion of which in the damaged layer leads to its filling of the voids and micropores in the damaged layer. In this case, the higher mobility of helium due to the temperature effect leads to the fact that smaller bubbles merge into larger ones, thereby reducing the internal pressure due to the reduction of the curvature of the bubble surface. An important role in this effect is also played by the mobility of vacancies and point defects, the movement of which in turn as a result of thermal action combined with deformation distortions leads to a more intensive growth of micropores, which leads to additional freedom of helium during migration, creating so-called “corridors” of microcracks along which helium diffusion occurs. The unification of small helium bubbles into larger ones due to their merging and accelerated agglomeration leads to an increase in local tensile stresses near the bubbles, thereby increasing the deformation effect, leading to a decrease in resistance to external influences and a more pronounced destabilization of strength characteristics.

Fig. 4 demonstrates the assessment results of the change in the values of hardness and bending strength of the ceramics under study depending on the value of atomic displacements caused by irradiation with He^{+} ions at different irradiation temperatures. The obtained dependencies are presented in order to assess changes in the strength parameters of the ceramics under study, reflecting the change in the resistance of the ceramics to softening processes associated with the accumulation of structural damage, and, as was established, to the processes of accumulation of gas inclusions in the near-surface layer. The general form of the presented dependencies reflects the presence of two trends in the changes in the values of strength parameters, characterizing different degrees of softening during the accumulation of structural damage caused by irradiation. At atomic displacement values below 10 dpa, the change in the values of hardness and bending strength is less than 1 %, which indicates fairly good indicators of ceramics resistance to external influences during the accumulation of structural damage caused by irradiation. In this case, the resistance of ceramics to softening processes is due to the fact that at given irradiation fluences (or values of accumulated damage in the form of atomic displacements), deformation distortions do not lead to a clearly expressed destabilization of strength characteristics. At the same time, a change in the irradiation temperature leads to more pronounced changes in strength characteristics even in the case of small values of atomic displacements (less than 10 dpa), from which one can conclude that temperature effects have a negative effect on softening. In this case, it should be taken into account that in the case of high irradiation temperatures, the processes of structure degradation are more pronounced due to the acceleration of diffusion of vacancy and interstitial defects, as well as the agglomeration of helium in the resulting microcracks and voids, which in turn leads to the acceleration of softening processes, expressed in a decrease in strength characteristics. Moreover, the most pronounced temperature effect occurs at maximum irradiation doses (3×10^{17} cm^{-2}), at which, according to the data on hardness changes, the difference in hardness values is about 1.1—1.5 GPa, which is more than 4.5—8 % of the initial value. It should also be noted that in the case of assessment of the changes in the bending strength, the changes are less pronounced than the results of changes in hardness, which can be explained by the difference in the mechanisms of softening caused by the types of external effects and associated with the accumulation of structural damage. In the case of changes in hardness, more pronounced degradation can be due to the fact that when helium bubbles accumulate, the deformation stresses they create, especially in the case of high-temperature irradiation, can lead to the formation of metastable states, which, under mechanical loads, lead to accelerated degradation. When bending, these deformation inclusions can prevent the propagation of microcracks, which leads to a slight slowdown in softening, but in the case of high concentrations of deformation distortions, especially in the case of high-temperature irradiation, these effects cannot be restrained by external influences, which leads to destabilization of strength.



a) b)

Fig. 4. Data on changes in the strength parameters of ceramics exposed to irradiation: a) Assessment results of alterations in the hardness value during the accumulation of damage caused by irradiation; b) Assessment results of alterations in the bending strength of ceramics with the accumulation of damage caused by irradiation.



a) b)

Fig. 5. Comparative analysis of the values of change in hardness (a) and bending strength (b) depending on the value of volumetric swelling caused by irradiation.

Fig. 5 illustrates the dependences of the change in the values of the reduction in hardness and bending strength depending on the value of ΔV, reflecting the degradation of the crystalline structure of ceramics exposed to irradiation with He^{+} ions. The results of the assessment of the reduction in hardness and strength are presented in percentage terms, obtained by comparing the measured values in the initial state and after irradiation, which made it possible to assess the degree of degradation of the strength parameters associated with the degradation of the crystalline structure as a result of irradiation.

The obtained dependencies, presented in Fig. 5, indicate a direct relationship between the degree of structural disorder associated with volumetric deformation expansion and the degradation of strength parameters. At the same time, as is evident from the analysis of the obtained dependencies, a change in the rate of accumulation of volumetric deformation distortions associated with a change in the irradiation temperature leads to an acceleration of the degradation processes of strength properties, which is most pronounced at high irradiation fluences (see data in Fig. 3). In this case, the increase in the size of helium bubbles during their agglomeration due to the temperature effect, which has an additional thermally and deformation-induced distortion of the crystalline structure, leads to a more intensive decrease in strength parameters, the decrease of which directly indicates a change in the resistance of ceramics to softening processes and a decrease in resistance to destruction caused by irradiation. The formation of large helium bubbles, as is known, according to the works of Evans J. [24,25], leads to an increase in tensile deformation stresses near the formed bubbles, and thermal expansion promotes the growth of not only bubbles due to the agglomeration and merging of small bubbles, but also the growth of microcracks due to the migration of vacancies, dislocations and interstitial atoms. Thus, the destabilization of the damaged layer occurs more intensively due to the thermal effect, accelerating the processes of structural disordering and, consequently, the softening of the damaged layer. The result of such an impact is not only more pronounced structural changes, leading to a decrease in the resistance of ceramics to external influences, but also a decrease in the irradiation fluence at which critically permissible parameters of irradiation-induced softening are achieved. It should also be noted that during assessment of the potential for using ceramics as structural materials, temperature factors should be considered, especially in the case of high-dose irradiation, in which the temperature effect also has a long-term effect on the properties and, consequently, the degradation rate.

4. Conclusion

During the analysis of the obtained images of the ceramic surface under high-dose irradiation, a direct relationship between the density and size of gas-filled inclusions formed during agglomeration and filling of micropores with implanted helium, and the irradiation temperature, the change of which leads to the growth of gas bubbles, was established. In this case, the agglomeration effect due to thermally induced diffusion of implanted helium results in enlargement of the size of gas bubbles, thereby increasing the deformation effect on the crystalline structure of the damaged layer, leading to destabilization and softening.

According to the obtained results, a connection was established between the growth of the density of structural damages, and as a consequence of their evolution, associated with the formation of gas inclusions, and the degree of softening, expressed in a decrease in hardness and bending strength. It was established that the threshold value at which a sharp decrease in strength parameters is observed is about 10 dpa, upon reaching which an acceleration of softening processes is observed, which is most pronounced in the case of irradiation at high temperatures. The established direct relationship between the magnitude of deformation volumetric swelling and the reduction in strength parameters, according to which it can be concluded that the key role in softening is played by gas-filled bubbles, the change in density and size of which leads to more pronounced destabilization and an increase in the softening effect.

The practical significance of the obtained results consists in determination of the role of temperature effects on the rate of structural disorder associated with both the growth of deformation tensile stresses and the increase in the size of gas bubbles, leading to the acceleration of the destabilization of the damaged layer and its softening. The obtained results make it possible to predict in advance the influence of temperature factors and estimate critical doses of accumulated damage in the case of high-temperature irradiation, in which destabilization of the damaged layer is observed. Understanding and studying such effects is very important in the development of structural materials and fuel matrices for new-generation high-temperature nuclear reactors, in which helium acts as a coolant and directly contacts the near-surface layers.

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Conflict of interest

None.

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