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STUDY OF THE EFFECT OF THE FORMATION OF TWO-PHASE CERAMICS BASED ON NEODYMIUM ZIRCONATE DUE TO DOPING WITH MGO AND Y_2O_5 ON THE STABILITY OF STRENGTH AND THERMOPHYSICAL PARAMETERS UNDER IRRADIATION

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Abstract. *The work presents the study results of the determination of the resistance of neodymium zirconate doped with MgO and Y_2O_5 to radiation damage stemming from irradiation with heavy ions akin to nuclear fuel fission fragments. The attraction towards this type of ceramics stems from its potential to raise the operational temperatures within the core of next-generation nuclear reactors. This is owed to its superior thermal conductivity when compared to zirconium dioxide, coupled with the heightened strength parameters that signify the ceramics' resistance against external factors. The main results of this study are to determine the influence of the formation of substitution or interstitial phases when adding magnesium and yttrium oxides to the composition, on increasing the stability of the strength and thermophysical parameters of neodymium zirconate to the radiation defects accumulation in the damaged surface layer. During the studies, it was found that the formation of impurity phases in the form of MgO inclusions (when it is added to the composition) and a substitution phase of the $Y_2Zr_2O_7$ type (with the addition of Y_2O_5) results in an elevation in the hardness and crack resistance stability of neodymium zirconate ceramics, which indicates the positive effect of doping linked to the formation of additional interphase boundaries that prevent strain embrittlement of the damaged layer under high-dose irradiation. During determination of the thermophysical parameters of the studied neodymium zirconate ceramics, it was observed that the formation of interphase boundaries during doping not only enhances thermal conductivity but also mitigates the decline in the thermal conductivity coefficient during irradiation for two-phase ceramics in comparison with undoped neodymium zirconate ceramics.*

Keywords: neodymium zirconate, doping, increasing stability, radiation defects, hardening, thermophysical parameters.

1. Introduction

The main problems in the energy sector today are the need to transition from hydrocarbon raw materials to alternative energy sources, in order to reduce not only harmful emissions from the combustion of hydrocarbons, but also to reduce the production of deposits, leading to the depletion of natural resources [1,2]. Recently, there has been significant focus on advancing nuclear and thermonuclear energy technologies to bolster their contribution to energy production from alternative sources. Specifically, efforts have been directed towards exploring new technological solutions aimed at enhancing the efficiency of nuclear power plants and increasing the burnup rate of nuclear fuel, thereby potentially reducing fuel consumption [3,4]. Simultaneously, a considerable portion of research in this realm is dedicated to devising technological solutions associated with the potential utilization of weapons-grade plutonium as a fissile nuclear material, as well as creation of new fuel elements, which are dispersed ceramic materials, which are assemblies of inert material with fissile nuclear material placed in it [5,6]. The focus on this type of fuel materials (with a dispersed matrix) primarily stems from the potential for containing high neutron densities, which are planned to be used in new types of nuclear reactors (Gen IV generation) [7,8], as well as the possibility of operating this fuel at elevated temperatures (about 700 – 1000 °C). Also, one of the advantages of the transition from the traditional type of fuel rods to dispersed materials is the possibility of creating fuel rods of various shapes and configurations (in the shapes of cylinders, balls, plates, rings, etc.). Also, an unimportant factor when choosing materials for creating inert matrices of dispersed nuclear fuel is the indicator of resistance to radiation damage, in particular, the ability to contain the decay products and fission of fissile nuclear material, which is a necessary condition for increased operating life of nuclear reactors [9]. In most cases, when opting for inert matrices for dispersed nuclear fuel, preference is given to refractory oxide materials [10-12]. The amalgamation of their structural, strength, and thermophysical attributes enables the development of high-strength, radiation-resistant materials capable of withstanding elevated temperatures and substantial radiation doses. Particular attention in this area of research is paid to zirconium-containing materials or zirconates [13-15], the use of which is attributed to high strength characteristics, and low thermal expansion values, which makes it possible to increase the stability of materials during their operation at high temperatures.

The use of ceramics based on zirconates of the $Ln_xZr_2O_7$ type (Ln is a rare earth element) as structural materials in nuclear energy stems from the potential to utilize them as thermal barrier materials, inert matrices of dispersed nuclear fuel, as well as to create containers for disposal and long-term storage of nuclear waste [16-20]. Moreover, the main advantages of these ceramic types (meaning zirconates containing rare earth elements) are high strength indicators (hardness, crack resistance, resistance to mechanical stress), and thermophysical parameters (higher thermal conductivity compared to zirconium dioxide-based ceramics) [15-17]. One of the ways to increase the stability of zirconate-based ceramics is to use methods of alloying or doping with stabilizing additives in the form of oxides of magnesium, cerium or yttrium, the addition of which in certain proportions allows the formation of substitution or interstitial phases in the structure of the ceramics, which in turn results in the appearance of interphase boundaries and an increase in dislocation density. This method of increasing the stability of materials relies on the possibility of creating additional boundary effects linked with the formation of interstitial phases (in the form of simple oxides) or substitution phases (when the main elements are replaced by dopants, resulting in the formation of inclusions in the form of new phases). In this case, the presence of interphase boundaries results in the creation of additional barriers, thereby augmenting resistance to radiation embrittlement, due to the inability of some of the resulting defects to agglomerate into larger inclusions within the damaged layer. These effects of interphase boundaries, along with dimensional factors that determine changes in dislocation density, are one of the most effective ways to increase the radiation damage resistance of ceramics, alongside to increase the service life and stability of strength and thermophysical parameters under high-dose irradiation. However, in most cases, the use of different concentrations of dopants can lead to both positive effects associated with strengthening and increasing the stability of ceramics due to interphase boundaries and dislocation strengthening, and negative effects due to the acceleration of polymorphic or phase transformation processes because of external factors. In this connection, the use of this modification method requires a detailed study and determination of the strengthening mechanisms linked to alterations in ceramic properties during doping. Given the foregoing, the primary objective of this investigation is to examine the impact of adding magnesium and yttrium oxides to the composition of neodymium zirconate ($Nd_2Zr_2O_7$) ceramics to enhance the stability of strength and thermophysical parameters under high-dose irradiation with heavy Xe^{23+} ions, commensurate in energy to fission fragments of nuclear fuel.

2. Materials and research methods

For the synthesis of ceramics, Nd_2O_3, ZrO_2, MgO, Y_2O_5 oxides, acquired from Sigma Aldrich (Sigma, USA) were used. The chemical purity of the oxides used was about 99.95%. The synthesis of $Nd_2Zr_2O_7$ ceramics was carried out using the method of mechanical activation (mechanical grinding), by mixing the initial oxides in a given stoichiometric molar ratio of 1:1 Nd_2O_3 and ZrO_2 oxides. Mechanical activation was carried out using a planetary mill PULVERISETTE 6 (Fritsch, Berlin, Germany) under the following grinding conditions - 250 rpm for 30 minutes. The choice of these conditions was used to obtain powders that were homogeneous in composition, which were subsequently subjected to heat treatment at a temperature of 1200 °C. Doping with oxides of magnesium (MgO) and yttrium (Y_2O_3) was carried out by adding them in a molar ratio of 0.15 M from the total volume of the resulting components Nd_2O_3 and ZrO_2 when mixing them for mechanical activation.

Determination of the effect of variation in the type of dopant on the alteration in the phase composition of the studied $Nd_2Zr_2O_7$ ceramics was conducted using the X-ray phase analysis method, the results in the form of a set of X-ray diffraction patterns of which are presented in Figure 1. The data were obtained using a D8 ADVANCE ECO X-ray diffractometer (Bruker, Karlsruhe, Germany). Diffraction patterns were recorded in the Bragg – Brentano mode in the angular range from 20 to 80°, with a step of 0.03°. The phase composition with variations in the dopant (in the case of magnesium and yttrium oxides) was determined by comparing the positions of the diffraction reflections of experimentally obtained diffraction patterns with the data of reference values from the PDF-2(2016) database. According to X-ray phase analysis of ceramic samples, it was found that the dominant phase in the composition of ceramics is the cubic phase $Nd_2Zr_2O_7$ (PDF-01-074-8769) with a pyrochlore-type structure and crystal lattice parameters a=10.5702 Å. Moreover, the position of the diffraction lines and their intensity indicate the absence of any clearly expressed textural effects that are uncharacteristic of structural alterations linked to phase transformations during high-temperature sintering.

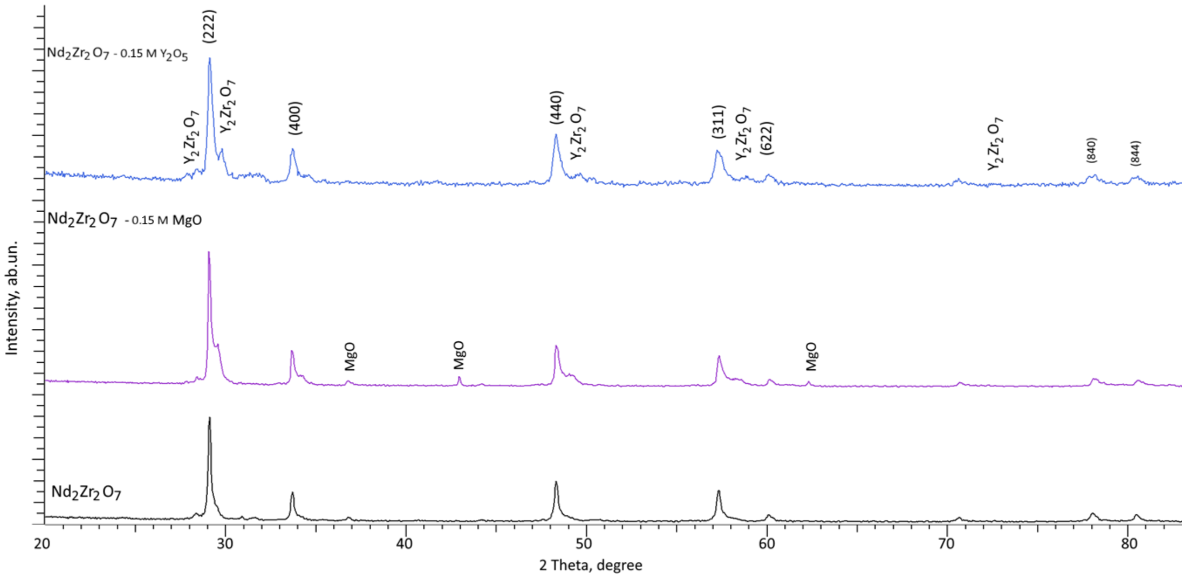

Fig.1. X-ray phase analysis results of the studied $Nd_2Zr_2O_7$ ceramics in the initial state and doped with magnesium and yttrium oxides

In the case of adding MgO to the $Nd_2Zr_2O_7$ ceramics composition in a molar ratio of 0.15 M, according to the obtained X-ray diffraction patterns, the appearance of low-intensity diffraction reflections characteristic of the hexagonal MgO phase (PDF-01-080-4185) with crystal lattice parameters a=3.4785 Å, c=4.2082 Å is observed, the content of which in the ceramic composition is no more than 15 wt. %. Also, an assessment of the structural parameters of the main (dominant) phase of $Nd_2Zr_2O_7$, which amounted to a = 10.6157 Å, indicates an increase in parameters, which is associated with the effects of replacing neodymium or zirconium ions with magnesium ions in the cubic lattice of the main phase. In turn, the formation of the MgO impurity phase causes the effect of strengthening and structural ordering (the crystallinity degree grows approximately by 2 – 3 % for doped ceramics). For samples of $Nd_2Zr_2O_7$ ceramics doped with Y_2O_5, according to X-ray diffraction analysis data, in addition to the main reflection characteristic of the cubic Y_2O_3 phase, the formation of reflections characteristic of the cubic substitution phase $Y_2Zr_2O_7$ is observed, the weight contribution of which is about 12.5 wt. %. The crystal lattice parameters for the $Nd_2Zr_2O_7$ phase were a=10.6604 Å, for the $Y_2Zr_2O_7$ phase a=5.2162 Å.

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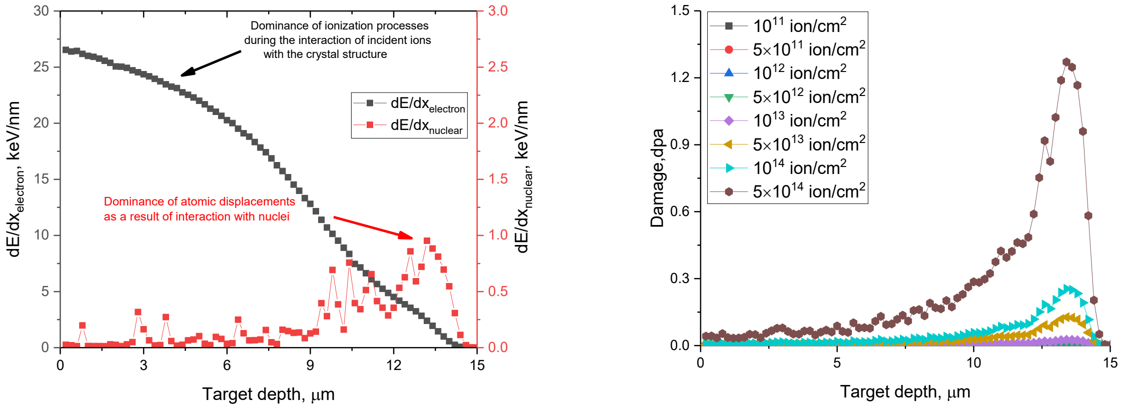
Determination of the effect of modification of $Nd_2Zr_2O_7$ ceramics by doping them with magnesium and yttrium oxides on resistance to radiation damage was carried out by irradiating the ceramic samples under study with heavy Xe^{23+} ions with an energy of 230 MeV (1.75 MeV/nucleon). Irradiation was carried out at the DC-60 heavy ion accelerator (Institute of Nuclear Physics, Astana Branch, Astana, Kazakhstan). Irradiation fluences with Xe^{23+} ions ranged from 10^{11} ion/cm^2 to 5×10^{14} ion/cm^2, the choice of which was determined by the possibility of simulation of the radiation damage accumulation processes, both in the case of the formation of isolated defective areas at low irradiation fluences, and the effects of overlapping defective areas during high-dose irradiation, in which case the effect of the appearance of highly deformed areas with locally altered electron density occurs.

Figure 2 demonstrates the simulation results of the ionization losses of incident Xe^{23+} ions in a near-surface layer with a thickness of about 15 µm of $Nd_2Zr_2O_7$ ceramics, alongside the dependence of the variation in the atomic displacement values (dpa) along the trajectory of ion movement into the ceramics with varying irradiation fluence. The dependence of the change in the values of ionization losses during interaction with electron shells ($dE/dx_{electron}$) and nuclei ($dE/dx_{nuclear}$) reflects the influence of the processes of interaction of incident particles and energy losses along the ion motion trajectory. Based on the provided data, at a depth of 0 to 9 – 10 µm, the dominant role in interaction processes is played by ionization processes during collisions with electrons due to electron–phonon interactions, and as a consequence, the emergence of energy dissipation effects through thermal heating of locally isolated regions along the ion trajectory. Moreover, these processes in the case of dielectric ceramics can compete with the processes of formation of defects, their thermal relaxation and ionization, which subsequently initiates the so-called athermal effects associated with thermally induced deformation processes.

Simultaneously, within a penetration depth of ions ranging from 9 to 15 µm, the primary influence stems from the processes of elastic interaction between ions and nuclei. These interactions result in the generation of atomic displacement effects (dpa), the accumulation of which contributes to amplified deformation distortions within the damaged layer (refer to Figure 2b). Moreover, the obtained relationships depicting alterations in the values of $dE/dx_{electron}$ and $dE/dx_{nuclear}$ closely align with the findings of [21,22], according to which, during irradiation with heavy ions, the main role in changing the properties of materials is played by the interactions of ions with electrons, for which the value of ionization electron losses is an order of magnitude greater than nuclear losses.



a) b)

Fig.2. a) Simulation results of ionization losses along the ion movement trajectory in $Nd_2Zr_2O_7$ ceramics; b) Distribution profile data of atomic displacement values (dpa) along the trajectory of ions in ceramics

The obtained results of changes in the values of atomic displacements (in particular, the maximum values of dpa values), presented in Figure 2b, were subsequently used in assessing the influence of variations in irradiation fluence on changes in strength and thermophysical parameters.

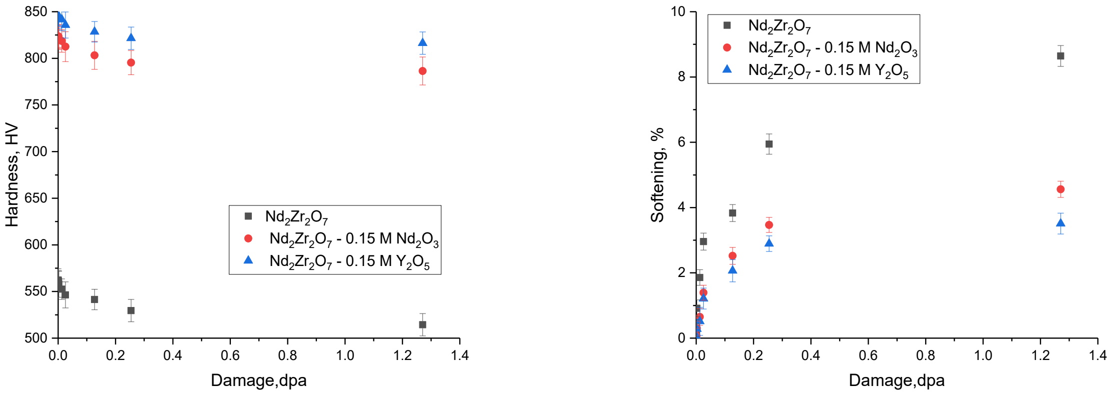
To determine the strength characteristics, the indentation method was used, implemented on a Duroline M1 microhardness tester (Metkon, Bursa, Turkey). The measurements were carried out using a tetrahedral Vickers pyramid with an angle of 136° between opposite faces, by loading the indenter with 100 N for 15 seconds, after which the indent sizes were estimated and the hardness values were determined. Determination of softening factors was carried out by comparative analysis of hardness values in the initial state and after irradiation. The average hardness value was determined by measuring about 10 - 15 surface indentations, followed by determining the hardness values and standard deviation.

Determination of changes in thermophysical parameters depending on the modification type (with doping with magnesium and yttrium oxides), as well as upon irradiation of ceramics with heavy ions, was conducted using the longitudinal heat flow method, which consists in establishing the temperature difference on both sides of the sample when it is heated. The measurements were carried out using a thermal conductivity meter KIT-800 (KB Teplofon, Russia). The stability of thermophysical parameters to radiation damage during irradiation with heavy ions was assessed by comparing data on the thermal conductivity coefficient in the initial state and after irradiation. Moreover, the obtained dependences of heat losses on the atomic displacement values during irradiation were used to describe the mechanisms for reducing the thermophysical parameters of ceramics, alongside to determine the effect of doping with magnesium and yttrium oxides on increasing stability and resistance to radiation degradation.

3. Results and discussion

In the case of choosing materials for creating inert matrices of dispersed nuclear fuel, much attention, as a rule, is paid to the study of their strength and thermophysical parameters, which are one of the key criteria in the selection of materials used in extreme conditions. In this case, a variation in the concentration of accumulated defects during irradiation, especially during high-dose irradiation, can have a significant impact on the change in strength parameters due to softening effects linked to the accumulation of deformation distortions and local defective inclusions. In this case, knowledge of the mechanisms of changes in strength and thermophysical parameters depending on the structural disorder degree or the atomic displacement value is necessary when determining the potential for using new types of ceramics as materials of inert matrices. Thus, Figure 3a demonstrates the results of alterations in the ceramics' surface layer hardness, measured contingent upon the value of atomic displacements caused by irradiation with heavy ions. Also shown in Figure 3b are the comparative analysis results of the alteration in the softening value (ΔHV), which characterizes the decrease in hardness contingent upon the degree of accumulation of atomic displacements.

Based on the evaluation data of the strength parameters of the studied $Nd_2Zr_2O_7$ ceramics, in the case of the original ceramics the hardness value is 563±12 HV, for $Nd_2Zr_2O_7$ ceramics doped with MgO it is 824±15 HV, for $Nd_2Zr_2O_7$ ceramics doped with Y_2O_5 it is 846±11 HV. The strengthening factor for $Nd_2Zr_2O_7$ doped ceramics was about 46 – 50 % compared to undoped ceramics, which indicates a positive effect of the formation of interphase boundaries on increasing the stability and strength of ceramics.



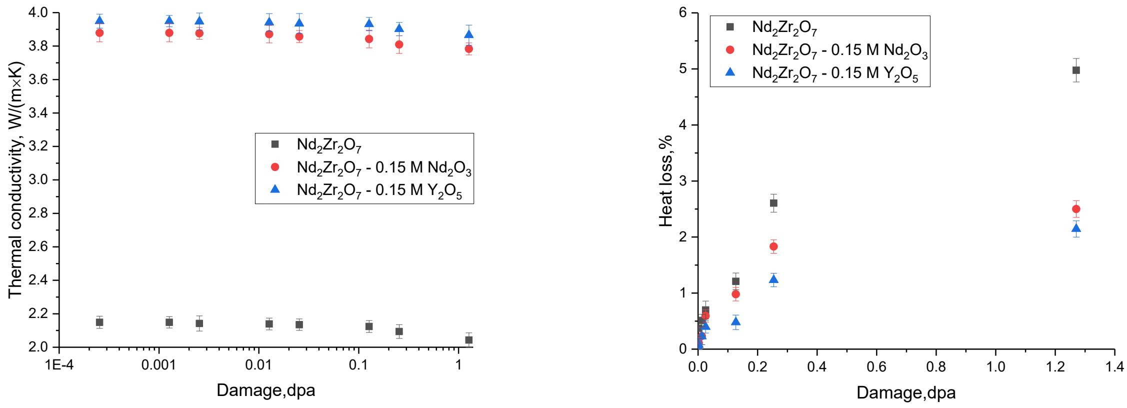
a) b)

Fig.3. a) Results of alterations in ceramic hardness values contingent upon the atomic displacement values; b) Results of alterations in the softening degree

The overall trend observed in the alterations of hardness, contingent on the atomic displacement value, suggests the detrimental effect of accumulated structural changes induced by irradiation on the strength characteristics of the examined $Nd_2Zr_2O_7$ ceramics. Moreover, significant changes in hardness (about 1-2 %) for unmodified $Nd_2Zr_2O_7$ ceramics are observed at irradiation fluences of the order of 5×10^{12} ion/cm^2, while for modified $Nd_2Zr_2O_7$ ceramics similar changes are observed at fluences above 10^{13} ion/cm^2. Such differences in hardness reduction trends are primarily due to the presence of interphase boundaries in $Nd_2Zr_2O_7$ modified ceramics, the presence of which results in an increase not only in the initial hardness values by more than 1.5 times, but also a rise in resistance to embrittlement processes during the accumulation of structurally deformed inclusions in the near-surface damaged layer. In the case of an increase in irradiation fluence above 10^{13} ion/cm^2 for unmodified $Nd_2Zr_2O_7$ ceramics, the reduction in hardness, and as a consequence, the amount of softening is about 6-8.5%, while for modified $Nd_2Zr_2O_7$ ceramics, the maximum decrease in hardness is less than 5 %. It is worth mentioning that the formation of two-phase ceramics of the $Y_2Zr_2O_7/Nd_2Zr_2O_7$ type results in an enhanced resistance to softening in comparison with $MgO/Nd_2Zr_2O_7$ ceramics (the value of the softening factor in this case at the maximum irradiation fluence is about 3.5 %).

The observed effects of strengthening, as well as increasing resistance to embrittlement and softening under high-dose irradiation in the case of modified ceramics, reflect the positive effect of the use of dopants in the form of magnesium and yttrium oxides on increasing resistance to radiation-stimulated softening. Figure 4 demonstrates the results of changes in the thermophysical parameters of the studied $Nd_2Zr_2O_7$ ceramics (in the original state and doped with magnesium and yttrium oxides), reflecting the effect of irradiation on a decrease in thermal conductivity with the accumulation of structural damage (with an irradiation fluence growth). In the case of the initial samples, it is clearly seen that modification of $Nd_2Zr_2O_7$ ceramics by adding dopants in the form of MgO or Y_2O_5 results in the thermal conductivity coefficient growth from 2.15 W/(m×K) to 3.8 – 3.9 W/(m×K), which indicates the positive effect of modification of $Nd_2Zr_2O_7$ ceramics, which was also proven when assessing changes in strength parameters.

According to the presented data on changes in the value of the thermal conductivity coefficient depending on the magnitude of atomic displacements, it can be concluded that the most pronounced changes associated with a decrease in thermophysical parameters appear at irradiation fluences above 10^{13} ion/cm^2, which, according to the data of [23, 24], are characterized by the presence of effects associated with the overlap of local structurally deformed areas, leading to a destructive change in the damaged layer. At lower irradiation fluences (for which the dpa value is less than 0.1), alterations in the thermal conductivity coefficient are less than 0.6 – 0.7 %. Moreover, the most pronounced alterations in the thermal conductivity coefficient are observed for samples of undoped $Nd_2Zr_2O_7$ ceramics, for which the maximum decrease in the thermal conductivity coefficient is about 4.9 – 5.0 %, which is more than 1.5 – 2 times greater than similar changes for doped $Nd_2Zr_2O_7$ ceramics (refer to Figure 4b). It is also worth to note that the most pronounced changes observed with an irradiation fluence growth above 10^{13} ion/cm^2 have a clear trend of accelerating the degradation of thermophysical parameters at large degrees of accumulated atomic displacements, which indicates that the decrease in thermophysical parameters has a clear dependence on the concentration of structurally deformed inclusions in the surface layer.



a) b)

Fig.4. a) Results of a decrease in the thermal conductivity of ceramics contingent upon the irradiation fluence; b) Assessment results of heat loss in samples during the accumulation of structural damage

This change is primarily associated with phonon mechanisms of heat transfer in ceramics, the change of which is influenced by the number of scattering factors linked to the accumulation of deformed regions, leading to an elevation in phonon scattering and thereby reducing the rate of heat transfer. At the same time, analysis of the obtained data on changes in thermophysical parameters indicates that when interphase boundaries associated with impurity inclusions are formed in $Nd_2Zr_2O_7$ ceramics, the observed decrease in thermal conductivity due to increased heat losses is significantly less than in undoped $Nd_2Zr_2O_7$ ceramics. This change in the trend of decreasing thermophysical parameters for modified ceramics can be explained by effects linked to a rise in structural ordering during the formation of interphase boundaries, which leads to heightened resistance to softening and degradation of thermophysical parameters. When using this type of ceramics as inert matrices of dispersed nuclear fuel, the obtained dependences of changes in thermophysical parameters depending on the irradiation dose make it possible to predict the critical service life, as well as the necessary adjustments when using them. Moreover, the data acquired regarding the impact of dopants, specifically magnesium and yttrium oxides, in enhancing resistance against deterioration in thermophysical and strength parameters in irradiated samples, can be valuable for selecting this type of ceramics as inert matrices with high radiation resistance. This is attributed to the presence of interphase boundaries. However, when considering the possibility of using this type of ceramics as materials for creating thermal insulating barriers during the storage of spent nuclear fuel, the observed thermal conductivity reduction patterns of irradiated ceramics allow us to consider these materials as one of the promising thermal insulators for long-term storage of spent fuel. In this case, the spontaneous decay of fissile material of spent fuel during long-term storage is accompanied by the formation of radiation defects, alongside heating of the container material, which in the case of ceramics with low thermal conductivity (which also decreases with the radiation damage accumulation in the irradiated material) allows one to avoid the effect of internal heating of the containers.

4. Conclusion

Analyzing the obtained experimental data related to determining the effect of irradiation with heavy Xe^{23+} ions on the decrease in strength and thermophysical parameters, the following conclusions can be drawn. During the studies, it was observed that for $Nd_2Zr_2O_7$ ceramics, the maximum value of the hardness reduction at fluences over 5×10^{13} ion/cm^2 is more than 6 – 8 %, which in turn indicates that the absence of interphase boundaries characteristic of $Nd_2Zr_2O_7$ doped ceramics results in a more pronounced deterioration in strength properties. In the case of ceramics doped with $Nd_2Zr_2O_7$, the presence of interphase boundaries leads to strengthening by more than 1.5 – 2 times at maximum irradiation fluence, which indicates the softening rate reduction and higher stability of modified $Nd_2Zr_2O_7$ ceramics due to doping. During assessment of changes in the thermophysical parameters of the studied $Nd_2Zr_2O_7$ ceramics depending on the type of modifier used, a decrease in the deterioration trend in the thermal conductivity coefficient under high-dose irradiation was established, which is associated with increased resistance to structural disorder of the modified ceramics.

Conflict of interest statement.

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

CRediT author statement

Kozlovskiy A.L. and Uglov V.V.: Conceptualization, Data Curation, Writing Original Draft; Kabiyev M.B.: Methodology, Investigation; Shlimas D.I.: Writing Review & Editing, Supervision.

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