Journal of Materials Science: Materials in Electronics (2019) 30:15724–15733
https://doi.org/10.1007/s10854-019-01958-x

The use of pulsed beams for increasing radiation resistance of ceramics

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Received: 30 May 2019 / Accepted: 30 July 2019 / Published online: 1 August 2019

Abstract

Paper presents the results of a study of the applicability of pulsed C^+/H^+ (85/15) beams with a high current density for directional modification and hardening of the surface layer of AlN nitride ceramics. The purpose of the modification is to increase the radiation resistance to helium embrittlement and swelling as a result of the accumulation of helium in the surface layer. It has been established that an increase in the number of processing pulses leads to a sharp change in the morphology of surface layer, as well as a change in strength characteristics of ceramics. The sharp deterioration of strength characteristics is due to the presence in the structure of the surface layer of a high density of dislocations and defects, as well as the formation of microcracks as a result of pulsed processing with a large number of pulses. The effect of modification on the change in the structural characteristics of investigated samples has been established. It should be noted that these changes occur in a small surface layer with a thickness of not more than 0.5 µm, which is most susceptible to degradation under irradiation and influence of corrosive media during practical use.

1 Introduction

Today, one of the important problems of nuclear power engineering is the problem of radiation swelling and partial destruction of the near-surface layer of the first wall of nuclear reactors, which can lead to destabilization of the reactor operation and reduction of the working life of structural materials [1–3]. To solve this problem, modern GenIV reactors use new types of structural materials based on carbide or nitride ceramics, which have high thermal conductivity, good insulating properties, etc. [4–6]. The well-studied material among ceramics is silicon carbide SiC, which has established itself as one of the most promising materials for nuclear reactors [7–9]. At the same time, unlike carbide ceramics, aluminum-based nitride ceramics have a higher melting point and thermal conductivity, which makes them a suitable material for materials of the first wall of nuclear reactors operating at high temperatures [10, 11]. However, despite the promising use of ceramic materials in nuclear power and reactor engineering and a large amount of data on the radiation resistance of ceramics to various types of radiation, there is still no single theory of the interaction of ionizing radiation with a crystal structure, as well as the mechanism of radiation swelling of ceramics as a result of the accumulation of high concentrations defects and distortions [12–18]. One of the most common types of radiation defects is helium swelling of the subsurface layer of structural materials, resulting from the accumulation of poorly soluble helium in the structure under the action of irradiation with fission fragments or neutrons, as well as nuclear reactions that occur with the formation of helium [17–20]. With large fluence irradiation (10^{17}–10^{18} ion/cm^2), gas-filled regions of helium can form in the crystal structure. That migrating over the surface layer can lead to the formation of helium bubbles (swelling) with subsequent degradation and an increase in the probability of swelling of structural materials [20–22], which leads to lower performance and thermal conductivity of materials.

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As is well known, one of the ways to increase the radiation resistance of materials is the modification of their structural characteristics in order to change the content of dislocation defects and create a reinforcing thin surface layer that can significantly reduce the radiation impact on the performance of materials. Today, a sufficiently large number of different techniques for creating a hardening layer, such as magnetron sputtering or galvanic deposition of thin films of oxides, nitrides, metal carbides on the surface of the material from 10 to 500 nm thick [23, 24], have been developed, which due to their nanostructural characteristics have significant increase of radiation resistance of materials. Another way to increase the radiation resistance is the addition of various additives to the material during the synthesis process, which lead to a restructuring of the crystal structure and an increase in strength or insulation characteristics [25, 26]. However, the use of these methods is associated with significant energy consumption and complexity of processes of obtaining and controlling structural parameters. One of the developing methods of improving the strength properties of materials in the last 10 years is the use of pulsed beams of high-density ions to modify the surface layer of materials. The basis of this method is the use of pulsed accelerators allowing the creation of nanosecond pulses of light ion beams of gases (50–100 ns) with a high current density from 1 to 2 J/cm^2. When exposed to a single pulse with the structure of the target material, an enormous amount of energy is transferred to it in a short period of time. That leads to the emergence of shock processes of defect formation in the near-surface layer that are comparable with processes of rapid melting of the material and rapid cooling, which leads to the subsequent rearrangement of the crystal structure [27–30]. Such processes are associated with the fact that the heating temperature of a material under the action of a pulse can reach 10^5–10^8 K/s in very short periods of time (1–5 nm) at a small depth of 0.3–0.5 µm, resulting in the structure of the near-surface layer metastable phases or regions containing a high concentration of dislocation defects or distortions that can lead to the formation of a reinforcing layer. Also, by varying the number of impact pulses, it is possible to control change in the surface layer and the change in strength and structural characteristics. Today, the most studied processes of changing the structural and mechanical properties under pulsed beams are refractory materials and stainless steels used in nuclear power engineering and mechanical engineering [31–35]. In most works, the authors point to an increase in strength and mechanical characteristics of materials at a shallow depth of the surface layer. That indicates the creation of a hardening coating as a result of recrystallization processes and the formation of metastable states under the action of pulsed beams. Moreover, the use of these materials for the modification of nitride materials is considered rather weakly [36–42].

Paper considers the effect of pulsed irradiation with an ion beam ($C^{n+}/H^+ = 85/15$), with a high current density on the change in strength and structural properties of the surface layer of nitride ceramics, as well as an assessment of the effect of modification of the surface layer on radiation resistance to helium irradiation.

2 Experimental part

2.1 Investigated sample

The samples are polycrystalline structures with a hexagonal-type lattice, similar to the structure of wurtzite, with a concentration of impurity Al_2O_3 inclusions of not more than 4%. The thickness of the samples was 15 µm. Before irradiation, the samples were polished to obtain a surface roughness of not more than 3–4 nm. The choice in favor of nitride ceramics, in particular, AlN was made due to the fact that these ceramics are one of the most promising candidate materials for shells and covers of the first wall of fission and fusion reactors [43–45]. The potential of their application is due to the low vacancy swelling and creep, a small amount of induced activity, a high rate of radiation resistance to various types of external influences [41–46]. Also, this class of materials is able to withstand temperatures above 1200–1400 °C, which makes them promising materials for GenIV generation reactors [46, 47].

2.2 Modification of ceramics with a pulsed beam

The modification of the near-surface layer in order to create a high dislocation density was carried out by irradiation on a pulsed ion accelerator INURA (Astana, Kazakhstan) with an ion beam consisting of carbon and protons ($C^{n+}/H^+ = 85/15$), with an accelerating voltage of 200 kV, the beam current 10 kA, the time of one pulse was 80 ns (at half-height). Samples were irradiated by 1 pulse at a focal spot of the beam where the beam energy density was 1 J/cm^2 per pulse.

2.3 Methods for the study of structural, strength and optical properties

The study of the effect of modification and irradiation with He^{2+} ions on structural characteristics was performed by X-ray diffraction on a D8 ADVANCE ECO diffractometer (Bruker, Germany) using CuKα radiation. To identify the phases and study the crystal structure, the software BrukerAXSDIFFRAC.EVAv.4.2 and the international database ICDD PDF-2 were used. The conditions for shooting X-ray diffraction patterns: voltage is 40 kV, current is 20 mA, $2\theta = 30°$–$75°$, step 0.03°, time of standing at the point of 1 s.

Thermally stimulated luminescence (TSL) measurements were performed when samples were excited at room temperature for 10 min with an ultraviolet deuterium lamp (50 W). After excitation, the removal of TSL spectra was carried out with linear heating of samples in the range of 300–673 K using an optical multichannel analyzer in the range of 300–800 nm.

A study of the change in samples surface morphology before and after irradiation was carried out using atomic force microscopy on an AIST-NT SPM microscope. AFM scan parameters have been set as in the following: AC-Mode (Non-Contact Mode); scan rate/scan frequency 0.4 Hz, resolution XY 700 × 700, scan size XY 15 × 15 um, Z is automatic, cantilever tip. The radius curvature of the sharp cantilever was < 10 nm.

The study of microhardness was carried out by the method of static indentation with different loads on the indenter. The Vickers pyramid was used as an indenter. Endurance tests were carried out by rolling with a 10% slip at a load of 200 N. Test durations were 20,000 cycles.

2.4 Simulation of helium swelling

The study of the processes of helium swelling of the surface layer of nitride ceramics was carried out by irradiating the initial and modified samples with low-energy 40 keV He^{2+} ions in a heavy ion accelerator DC-60 (Astana, Kazakhstan). The energy loss of ions on electrons is $dE/dx_{elec} = 0.184$ keV/nm, the energy loss on nuclei is $dE/dx_{nuclear} = 0.07$ keV/nm, the displacement threshold value − 25 eV, the energy of the primary knocked out atom is 25 eV, while the binding energy of atoms in the crystal lattice is 3.36 eV. The choice of irradiation fluence with low-energy He^{2+} 3×10^{17} ions ion/cm^2 is due to the critical importance of formation of helium bubbles and the subsequent swelling and destruction of the surface layer according to literature data.

3 Results and discussion

3.1 Selection of optimal conditions for the modification of nitride materials by pulsed beams

One of the important factors affecting the performance of ceramics is a change in the surface morphology as a result of external influences. When irradiated with pulsed beams of high current density, an enormous amount of energy is transferred to a small surface layer with a thickness of no more than 500–800 nm in a short period of time. This energy can lead to athermal melting and processes of phase transformations in structure of the surface layer, with the subsequent formation of regions with a high density of dislocations and defects. Moreover, the use of these materials for the modification of nitride materials is considered rather weakly. To select the optimal conditions for modifying and changing the strength properties of nitride ceramics depending on the number of pulses, the effect of the number of pulses of influence $C^{n+}/H^{+} = 85/15$ ion beams on the properties of ceramics was studied. The number of pulses ranged from 1 to 20. The choice of the maximum number of pulses was due to the destruction of ceramics and the formation of macrocracks on the surface. Figure 1 shows 3D images of changes in the surface morphology of ceramics depending on the number of pulses.

With a single impact on ceramics surfaces, the appearance of fused areas of hexagonal shape, a structure similar to craters, is observed. Similar types of structures on the surface were observed at irradiation of refractory materials and stainless steels. The formation of crater-like inclusions on the surface is caused by a sharp temperature gradient as a result of a single pulse action and subsequent recrystallization and melting processes of the surface layer. An increase in the number of pulses above one leads to the formation of a large number of surface defects and the formation of grain boundaries. When irradiation of 15 and 20 pulses in the surface layer, there is a partial destruction of the surface as a result of delamination and sputtering of part of the modified layer as a result of a large number of distortions and stresses in the structure under the influence of recrystallization processes. An increase in the number of pulses above 20 leads to cracking of the surface and partial degradation of samples. A sharp degradation with a large number of pulses is caused by an increase in the structure of dislocation defects and a change in grain sizes with the subsequent formation of metastable areas and overvoltage areas, which lead to a deterioration of the strength characteristics.

To assess the effect of the number of irradiation pulses on changes in strength characteristics and dislocation density, a nano indentation method was used. The results of the change in hardness in depth from the number of pulses are presented in Fig. 2a.

As can be seen from the presented data, with a small number of irradiation pulses, an increase in the hardness of the surface layer 500–600 nm thick is observed, which made it possible to determine the thickness of the modified layer and the depth of penetration of pulsed ion beam. However, an increase in the number of pulses above 5 leads to a sharp decrease in the hardness of the surface layer or its destruction. The change in hardness is due to the density of dislocation defects in the structure as a result of the action of pulsed beams (Fig. 2b). It is known that changing the density of dislocations and defects can significantly improve the crack resistance and strength of various materials. An increase in the dislocation density above 5×10^{16} leads to a sharp decrease in the strength of the material due to an increase in

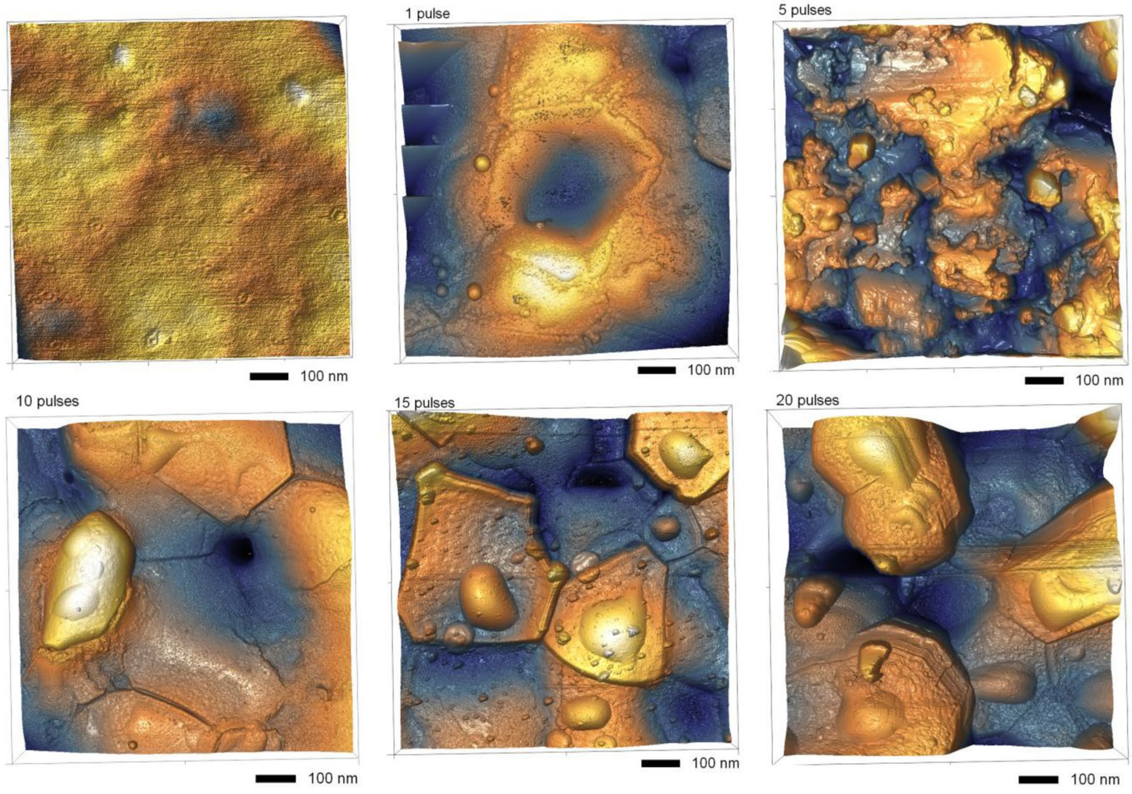


Fig. 1 Dynamics of changes in surface morphology

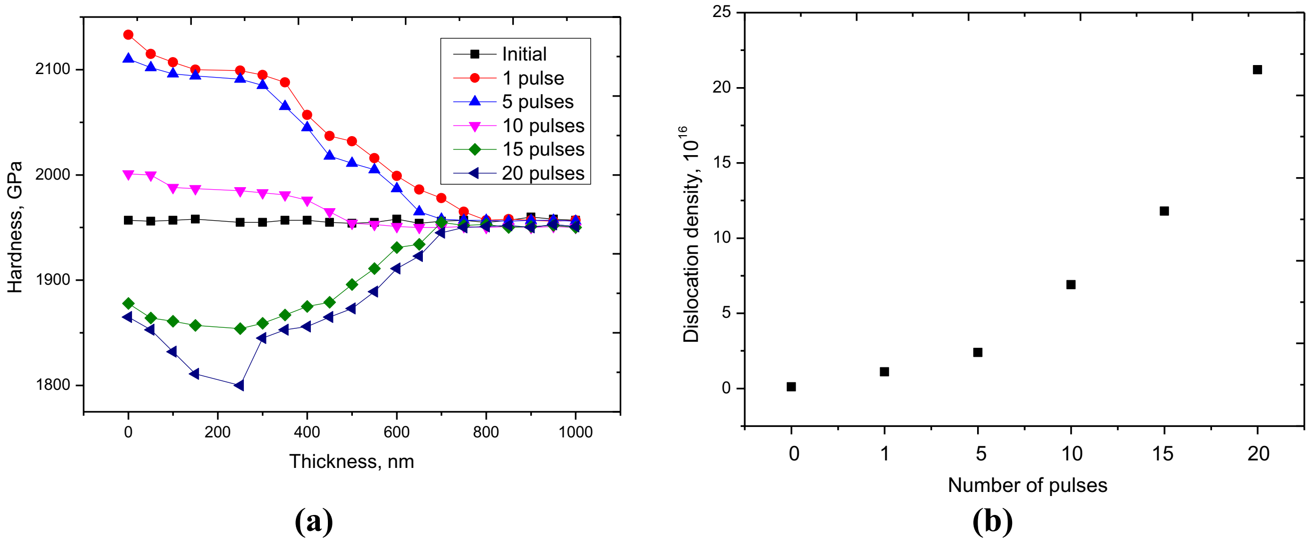


Fig. 2 **a** Dynamics of change in the hardness of the surface layer from the number of pulses; **b** graph of changes in the density of dislocations in the structure as a result of irradiation

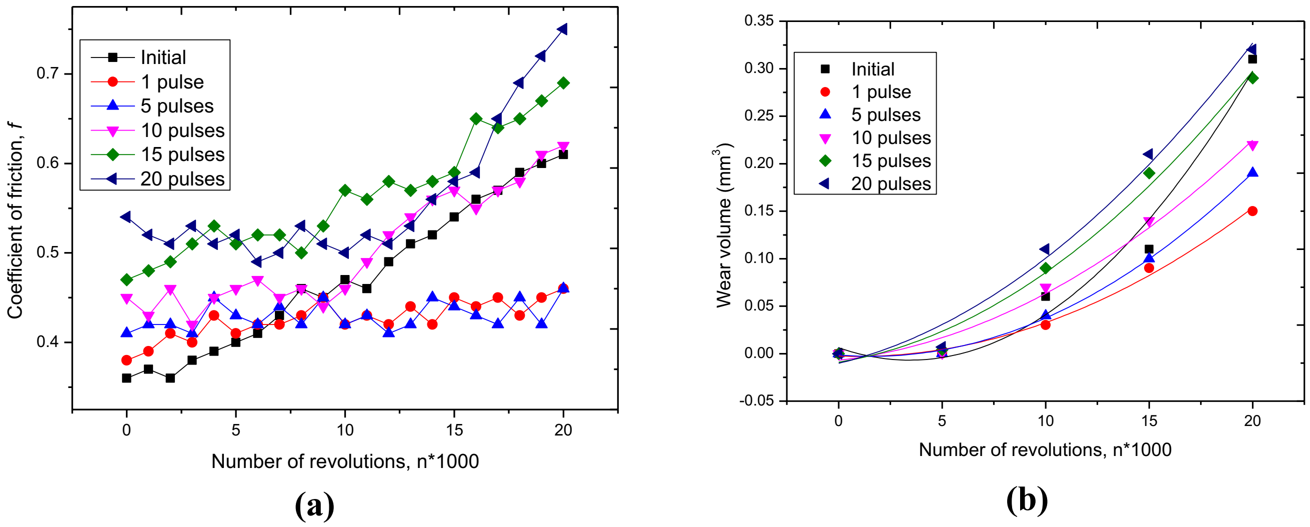


Fig. 3 **a** Changes in the dry friction coefficient after testing; **b** dynamics of change in the amount of wear of ceramics during the test

distortions and deformations in the structure, as well as the formation of overvoltage regions.

Figure 3a shows the dynamics of changes in the coefficient of dry friction for the studied ceramics before and pulsed irradiation.

As can be seen from the presented data, the value of the coefficient of dry friction for the initial sample is 0.35–0.37 and increases after 10,000 test cycles, which indicates the deterioration of the surface during friction. For modified samples by 1 and 5 pulses, there is a slight increase in the value of the coefficient of dry friction, while the magnitude of the coefficient is almost unchanged during testing, which indicates an increase in the strength characteristics as a result of modification of the surface layer. An increase in the number of processing pulses above 10 leads to a sharp increase in the coefficient of dry friction, which is caused by a change in the morphology of the surface of ceramics, as well as by the formation of additional highly defective areas in the surface layer. As a result of life tests, an increase in the coefficient of dry friction for samples irradiated with pulses above 10 is observed, which also indicates a partial degradation of the surface layer during the test. Figure 3b shows the dynamics of changes in the amount of wear after testing. According to the data obtained, the smallest amount of wear is characteristic of samples irradiated with 1 and 5 pulses, while an increase in the number of pulses above 10 leads to a sharp increase in the volume, which indicates a decrease in the strength properties of ceramics. The sharp deterioration of strength characteristics is due to the presence in the structure of the surface layer of a high density of dislocations and defects, as well as the formation of microcracks as a result of pulsed processing with a large number of pulses. An increase in the number of pulses above 10 leads to a sharp degradation of the surface, as evidenced by the results of atomic force microscopy, as well as the results of measuring the strength characteristics.

An important characteristic for nitride ceramics is the change in optical characteristics as a result of external influences. Figure 4 shows the dynamics of changes in the spectral distribution of the main optical maximum arising at 400 K, depending on the number of pulses. The spectral line in the region of 3.2–3.4 eV characterizes the change in the main recombination centers in the AlN structure.

According to the data obtained, an increase in the number of pulses leads to a decrease in the intensity of the spectral maximum and displacement, which is caused by the

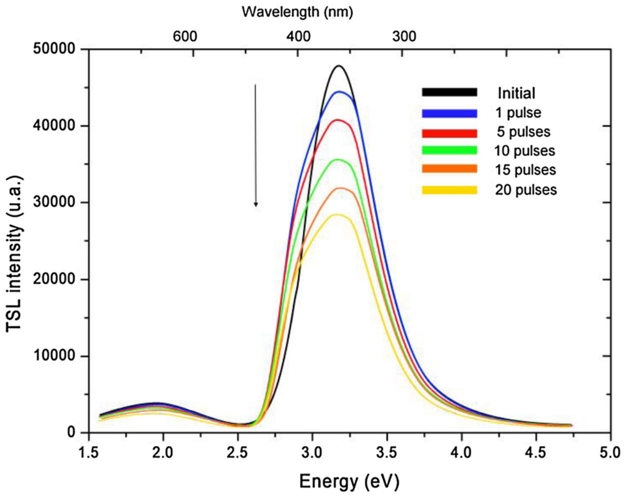


Fig. 4 Dynamics of changes in the spectral distribution of TSL (400 K) depending on the number of pulses

appearance of additional distortions and deformations in the structure of the surface layer, resulting from the modification. An increase in the number of pulses above 10 leads to a decrease in the intensity of the spectral maximum by almost two times, which is due to the high content of dislocation defects and regions of disorder. A decrease in intensity indicates a change in the concentration of absorbing centers in the structure and an increase in the amount of optical absorption, which has a significant effect on the performance characteristics of nitride ceramics.

On the basis of the conducted research, the optimal conditions for the modification of nitride ceramics were determined with the aim of further studying the radiation resistance to helium swelling and embrittlement. Ceramics were chosen as the test samples for radiation resistance before and after modifying 1 and 5 pulses.

3.2 Study of radiation resistance to helium swelling and embrittlement

One of the most acute problems is the accumulation of helium in the structure of the surface layer and the subsequent agglomeration and the formation of gas-filled inclusions. The accumulation of helium occurs as a result of low solubility and high mobility of implanted ions along grain boundaries and defect junctions. In this case, helium accumulation in the structure can occur both as a result of nuclear reactions initiated by neutron fluxes in the structure of ceramics, and when the near-surface layer contacts the coolant and the subsequent introduction of helium into the structure. In this regard, one of the important and urgent tasks of developing new structural materials is to increase the radiation resistance of materials in order to prevent helium accumulation and subsequent swelling and flaking. One of the ways to increase radiation resistance is to create a modified surface layer, which will help reduce the accumulation of helium in the structure and the subsequent agglomeration and partial destruction.

To study the effect of modification on the increase in radiation resistance, samples of ceramics irradiated with by 1 and 5 pulses were selected, as well as an initial sample taken for comparison. Irradiation with low-energy He^{2+} ions was carried out at the DC-60 heavy-ion accelerator, the irradiation fluence was 3.0×10^{17} ions/cm^2, the incident ion energy was 40 keV (20 keV/charge).

Figure 5 shows the dynamics of changes in the surface morphology as a result of irradiation.

As can be seen from the presented data, at irradiation with helium ions on the surface of the initial sample, the formation of spherical inclusions filled with helium is observed. In this case, there is a partial destruction and peeling of sample surface after irradiation, which is associated with the degradation of the surface layer as a result of the swelling processes. In contrast to the original sample, the surface of the modified sample by a single exposure to a pulsed beam was less degraded and the number of helium inclusions was minimal. However, the samples subjected to the fivefold impact of pulsed beams, the degradation of the surface is more pronounced compared with the modified samples by a single exposure. Such a difference in the degradation of the surface may be due to the presence of additional dislocation defects in the structure of the surface layer of modified ceramics, which reduce the rate of helium migration in the structure and also prevent the sintering of helium. However, a high dislocation density and the presence of additional microcracks and areas with a high density of defects characteristic of the repeated effects of pulsed beams leads to an increase in the number of sinks of defects in the structure of the surface layer which serve as agglomeration centers of the introduced helium. Also, the density of helium inclusions for modified samples is orders of magnitude less than for the initial samples, which indicates an increase in the radiation

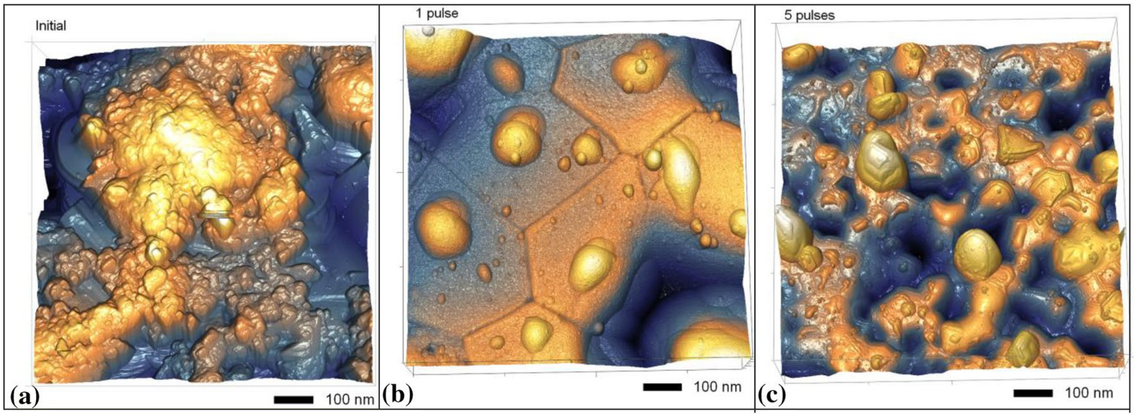


Fig. 5 3D images of changes in surface morphology after irradiation with He^{2+} ions: **a** initial; **b** after modification with 1 pulse; **c** after modification with 5 pulses

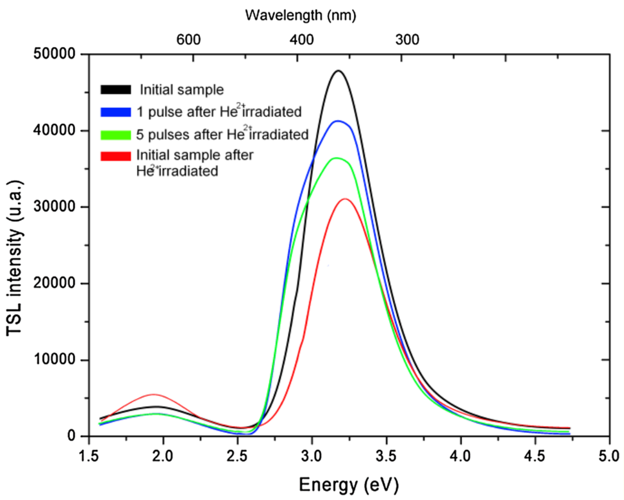


Fig. 6 Dynamics of changes in the spectral distribution of TSL (400 K) after irradiation with He^{2+} ions

resistance of ceramics as a result of preliminary processing by pulsed beams with a high current density.

To study the effect of helium swelling on the optical and structural properties, the methods of thermally stimulated luminescence and X-ray analysis were applied. Figure 6 shows the dynamics of changes in the spectral distribution of the TSL spectrum for samples irradiated with helium ions.

As can be seen from the presented data, the greatest change in the intensity of the spectral line is observed for the unmodified sample after irradiation. The sharp decrease in intensity is due to the presence in the structure of helium inclusions and areas of disorder resulting from the accumulation of helium and subsequent swelling. Also, for modified samples, the decrease in intensity is not as significant as for initial samples, which also indicates an increase in radiation resistance as a result of the modification.

The change in structural characteristics was determined on the basis of changes in X-ray diffraction patterns before and after irradiation with helium ions. Figure 7 shows the dynamics of changes in the diffraction patterns of the samples studied.

According to the data obtained, the initial sample is characterized by a polycrystalline structure with a high degree of crystallinity and three selected textural directions of crystallite orientation (100), (002) and (101), characteristic of the hexagonal type of structures. The presence of low-intensity diffraction peaks characteristic of aluminum oxide Al_2O_3 on diffractograms is due to the technological processes for producing ceramics by sintering from aluminum powders. For unmodified samples (diffractogram No. 3) of helium ions irradiated, a sharp decrease in diffraction peaks and the appearance of asymmetry are observed, which indicates the degradation processes of the crystal structure as a result of irradiation. The appearance of amorphous-like broadening of diffraction maxima is also observed, which is caused by the partial destruction of crystalline and chemical bonds as a result of the accumulation of helium in the structure of the surface layer. The formation of amorphous-like inclusions is due to the implantation of low-energy helium ions with subsequent agglomeration and the formation of gas-filled inclusions, which leads to the degradation of the structure as a result of a large concentration of interplanar distortions.

For samples irradiated with a pulsed beam, there is a slight decrease in the diffraction maxima and a symmetric broadening of diffraction lines, which is caused by a change in the crystallite size and the appearance of dislocation defects and distortions in the structure. In this case, irradiation with helium ions of modified samples has a less destructive effect on the crystal structure, which is expressed in a small change in the intensities and shape of diffraction maxima, as well as a small amount of distortion of the diffraction peaks by amorphous-like inclusions (Fig. 7b). Figure 8a shows the dynamics of changes in the concentration of accumulated helium in the structure of ceramics after irradiation.

According to the data obtained, the highest helium content in the structure is observed for unmodified samples. At the same time, the helium content in the structure is significantly less than the theoretically predicted value for this fluence (helium content in the structure is 0.035–0.037%). The theoretical value was calculated according to the formula for calculating the concentration of helium embedded in the construction materials (1):

$$C_{He} = \sigma\Phi, \tag{1}$$

where σ is the total defect formation cross-section, and Φ is the radiation fluence. This difference may be due to the presence of additional dislocation defects in the structure and a complex crystal structure that prevents the accumulation of helium in the structure. The determination of the concentration of helium in the structure resulting from irradiation was carried out by calculating the concentration of defective areas in the samples under study using formula (2):

$$f_d(\phi t) = \frac{M(\phi t) - M_0}{M_s - M_0} \tag{2}$$

where $M\ (\varphi t)$ is the defect concentration, the measured value as a function of ion fluence, M_o is the concentration of defects before irradiation, M_s is the maximum value of defect concentration at high fluences associated with the formation of amorphous inclusions and disordering regions in the structure resulting from helium accumulation.

Table 1 presents the data on changes in the main crystallographic characteristics of the studied samples after irradiation with helium ions.

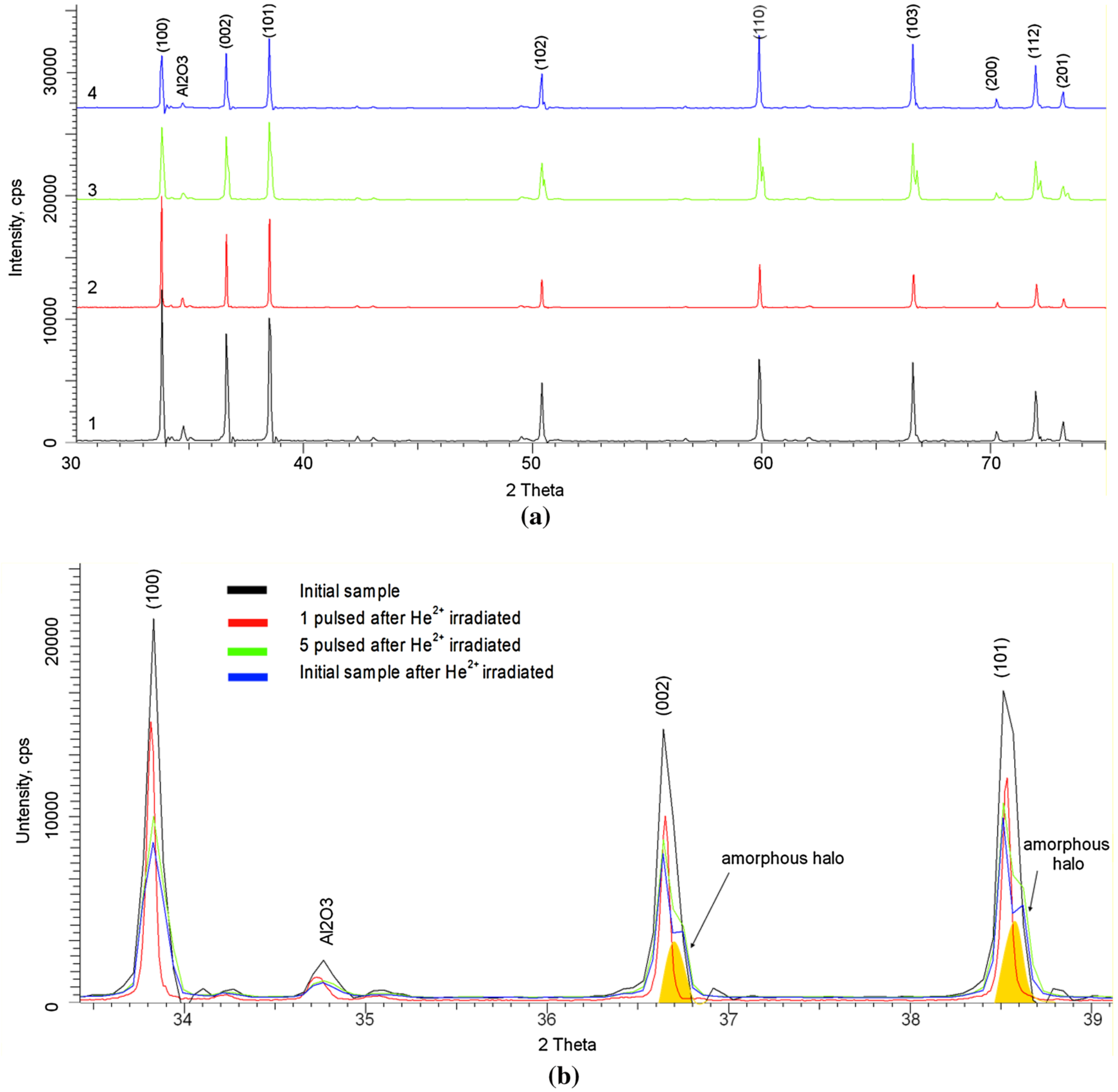
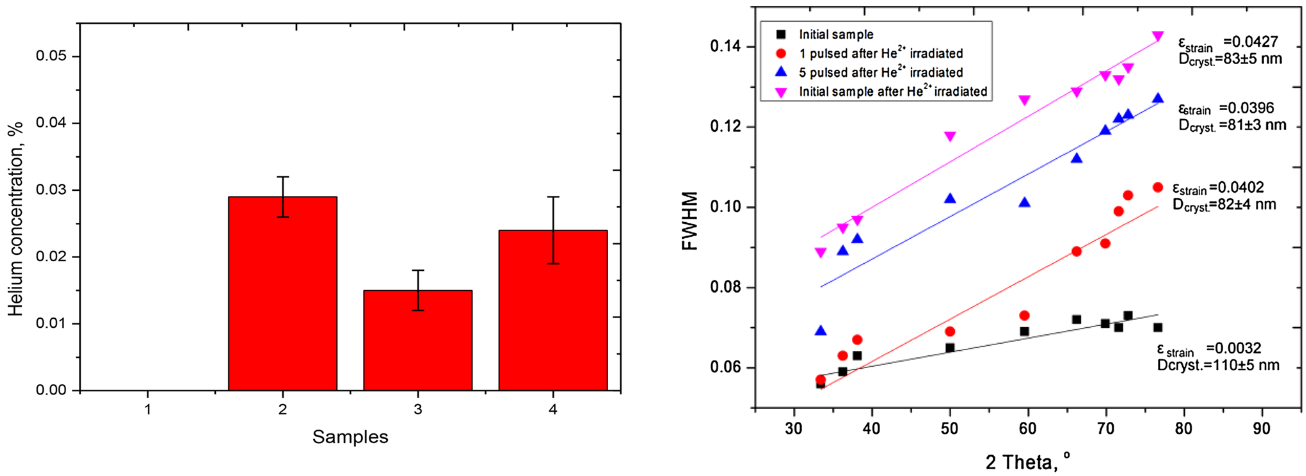


Fig. 7 **a** X-ray diffraction patterns of the studied samples: (1) Initial; (2) irradiated with He^{2+} ions after modification with 1 pulse; (3) irradiated with He^{2+} ions after modification with 5 pulses; (4) unmodified sample irradiated with He^{2+} ions (**b**)

As can be seen from the presented data, the greatest change in crystallographic parameters was observed for unmodified samples. At the same time, an increase in the crystal lattice parameter c/a indicated the appearance of additional distortions and deformations of the interplanar distances in the structure as a result of the introduction of helium ions into the structure. Evaluation of the change in the shape of diffraction peaks due to contributions from resizing and deformation defects was carried out using the Williamson–Hall method. The data of the dependence of the change in the FWHM value on the scattering angle is presented in Fig. 8b.

According to the data obtained, the slope of the angular dependence of FWHM characterizes the magnitude of distortions and deformations in the structure, defined as microstrains, resulting from changes in interplanar distances and deformation of the crystal lattice. As a result of irradiation with helium ions, the deformation factor made the dominant contribution to the change in the shape and intensity of diffraction lines. Also, crystallite sizes for all irradiated samples were reduced by the same amount for both modified and initial samples. The decrease in crystallite size was due to recrystallization processes in the surface layer during irradiation and subsequent accumulation of helium ions in the structure.



(a) **(b)**

Fig. 8 **a** Dynamics of changes in the concentration of implanted helium in the structure of the surface layer: (1) initial sample; (2) unmodified sample after irradiation with He^{2+} ions; (3) irradiated with He^{2+} ions after modification with 1 pulse; (4) irradiated with He^{2+} ions after modification with 5 pulses; **b** Construction of Williamson–Hall for the test samples before and after irradiation

Table 1 Parameters of the crystal structure

Sample	Initial	Initial sample after He^{2+} irradiated	1 Pulsed after He^{2+} irradiated	5 Pulsed after He^{2+} irradiated
Lattice parameter, Å	a = 3.0956, c = 4.9557	a = 3.1076, c = 4.9953	a = 3.0963, c = 4.9694	a = 3.0978, c = 4.9753
The ratio of parameters c/a	1.6008	1.6074	1.6049	1.6061
Density of ceramics (g/cm^3)	3.265			
Integral porosity (%)	0.018	0.202	0.115	0.132
Degree of perfection or degree of ordering of the crystal structure (%)	96.7	84.5	93.4	91.1

4 Conclusion

Therefore, in the course of study, it was found that the modification with a pulsed beam with a high current density and a small amount of impulse is applicable to increase the radiation resistance to helium swelling of nitride ceramics by creating high density of dislocation defects in the surface layer that prevent migration and agglomeration of helium. Also, dependences of the effect of number of irradiation pulses on the change in the strength and optical characteristics were determined. It was established that an increase in the number of irradiation pulses above ten leads to a sharp degradation of the surface layer of ceramics due to the occurrence of a large number of defective areas and a high dislocation density, as well as athermal melting and subsequent formation of microcracks in the surface layer which reduce the strength and hardness of the samples. In this case, the effect of a small number of pulses led to an increase in the strength characteristics and the formation of a near-surface hardened layer with a thickness of no more than 600–650 nm. The presence of this reinforced layer led to a decrease in the rate of migration and agglomeration of helium ions in the structure of the surface layer. It was determined that the main processes affecting the change in structural and optical characteristics are the deformation processes of the crystal structure, as a result of changes in the interplanar distances and lattice parameters.

Acknowledgement This research was funded under NU ORAU project (Nazarbayev University) « Investigation of high-intensity pulsed ion beam neutralization by volumetric plasma ».

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