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INVESTIGATION OF THE EFFECT OF EXPOSURE TO HEAVY XE^{22+} IONS ON THE MECHANICAL PROPERTIES OF CARBIDE CERAMICS

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The paper presents the results of a study of the effect of irradiation with heavy Xe^{22+} ions with an energy of 440 keV and irradiation fluences of 10^{14}, $5x10^{14}$, 10^{15} ion/cm^2 on the properties of ceramics based on silicon carbide (SiC). The choice of the type of irradiation and dose load is due to the possibility of modeling radiation damage to the surface layer with a thickness of 200 nm as a result of the effect of overlapping defective areas. The scientific novelty of the results obtained consists in systematic studies of the stability of the mechanical and strength properties of the surface layer of carbide ceramics to radiation damage. In the course of the studies, it was found that in the case of irradiated ceramics, the damage depth exceeds the estimated ion mean free path by 20-30%, depending on the irradiation fluence. The main mechanism of radiation damage is an increase in the dislocation density of defects and the formation of regions of disordering in the case of large doses. As a result of the simulation of accelerated aging processes, it was found that for irradiated samples the decrease in crack resistance does not exceed 10%. Studies have shown high values of the stability of silicon carbide ceramics to radiation damage to the surface layer.

Keywords: ceramic, mechanical properties, defects, Silicon Carbide, heavy ions, distortion, degradation, radiation resistance

Introduction

The current state of energetics in the world requires cardinal decisions in the field of improving the reliability of nuclear installations, as well as a significant increase in the life of nuclear reactors. One of the solutions in this direction is the use of new classes of structural materials with improved mechanical, strength properties, with a high melting point, radiation and corrosion resistance, etc. [1-4]. The most suitable materials with characteristics corresponding to these requirements are ceramics based on oxides [5-8], nitrides [9-12], carbides [13-15], etc. The interest in this class of materials is due not only to the great potential for practical applications in the nuclear industry, space technology, aircraft manufacturing, microelectronics, but also to the acquisition of new fundamental knowledge in the theory of radiation defects in solids, in particular, in carbide, oxide or nitride ceramics [16 -twenty]. Obtaining new knowledge will allow significant progress in predicting the life of this class of materials in the new generation of GenIV reactors [21-25]. When structural materials are used in nuclear reactors, the surface layers of the first wall of the reactor are exposed to large doses of radiation that can cause amorphization of the crystal structure and subsequent destruction of the surface layer, which can lead to catastrophic consequences. The processes of amorphization and degradation that occur during irradiation are caused by the accumulation of point defects in the structure with the subsequent formation of cluster defects and disordering regions, which contain a large number of stresses and distortions. In the case where the concentration of structural distortions is sufficiently high, peeling and partial delamination processes can be initiated in the surface layer, which leads to a sharp deterioration of not only structural properties, but also mechanical and heat-conducting, which leads to a decrease in the life of materials and its destruction [26, 27]. The use of ceramic materials can significantly increase the service life, due to the high degree of resistance to structural changes that occur during irradiation. Moreover, despite a large number of works on this topic [8-19], interest in studying the processes of radiation damage to surface layers, as well as the effect of irradiation on the mechanical properties and wear resistance of ceramics, is still relevant and requires more and more attention to itself. Among the variety of ceramic materials, silicon carbide and its various structural modifications are considered the most promising, the interest in which is due to structural and mechanical characteristics, as well as its wide range of applications in various industries and technics [28-30]. However, there are few works devoted to the study of mechanical changes in the surface layer as a result of irradiation, despite the huge interest in this topic. Based on the foregoing, the main purpose of this work is to study the influence of the degradation of the surface layer of SiC ceramics on the mechanical and strength properties as a result of the accumulation of defects during irradiation.

1. Experimental part

The study of mechanical properties, including wear resistance and strength depending on the radiation dose, was carried out on samples of commercial polycrystalline silicon carbide (SiC) ceramics with potential applications as the basis for structural materials for nuclear power.

The initial samples were irradiated with a DC-60 heavy ion accelerator with low-energy Xe^{22+} ions with an energy of 440 keV and irradiation fluences of 10^{14}, $5x10^{14}$, 10^{15} ion/cm^2. The choice of radiation doses is due to the modeling of the effects of overlapping cascade defects resulting from elastic and inelastic collisions, the number of which for the selected doses varies from 100 to 1000 multiple overlaps. Figure 1 presents the results of modeling the irradiation effect using the Stopping and Range of Ions in Matter (SRIM) Pro 2013 program code, which clearly shows that in the case of collisions, a large number of secondary defects are observed that can create a branched defective structure.

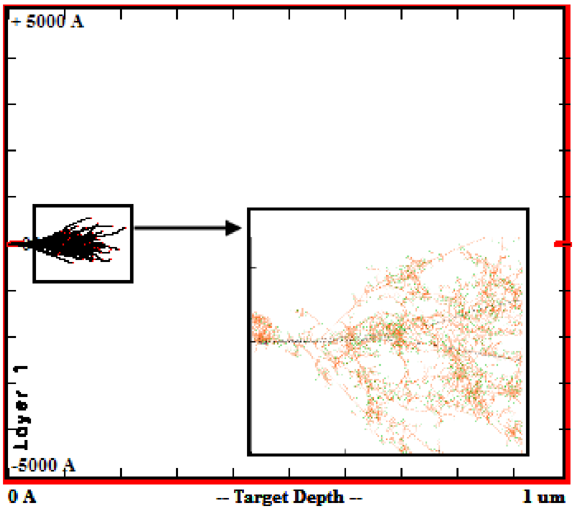

Fig.1. Xe^{22+} ion paths in SiC ceramics

The study of changes in morphological features before and after irradiation was carried out using scanning electron microscopy and atomic force microscopy.

Table 1. SRIM Outputs / Xenon in SiC ceramic

Sample	Projected range, nm	Vacancies /ion	dE/dx_{elect}, 10^3 keV/µm	dE/dx_{nucl}, 10^3 keV/µm
SiC irradiated Xe^{22+}, 440 keV	190±20	5300±100	1.077	2.821

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Strength characteristics, as well as the dynamics of changes in the mechanical properties of nitride ceramics before and after irradiation, were determined using the depth hardness method, as well as tests for wear resistance at a load of 200 N, bending strength, and impact strength before and after irradiation [14]. Assessment of resistance to low-temperature degradation of the surface microstructure, as well as the formation of microcracks, was evaluated by aging tests, under accelerated degradation conditions, obtained by modeling the external effects of water vapor at a temperature of 150°C and a pressure of 2.2-2.3 atm. According to the proposed methodology, 1 hour of testing is 4-4.5 years of aging and degradation under normal conditions.

2. Results and Discussion

Figure 2 shows the results of changes in the morphology of the surface layer of carbide ceramics during irradiation with various fluences obtained using the atomic force microscopy method. According to the data presented, the ceramic surface in its initial state does not contain a large number of structural defects, such as hillocks, elevations or microcracks. The degree of roughness of the ceramic in the initial state does not exceed 3-5 nm. For irradiated samples, the formation of defective regions in the form of sphere-like hillocks is observed, as well as the presence of regions with pronounced elevation differences. In this case, with an increase in the irradiation fluence, the density of these inclusions and their sizes increase, which indicate the occurrence of disordered regions and structural distortions in the surface layer, which leads to partial extrusion of structural defects near grain boundaries with subsequent formation of hillocks. Also, the degradation of the surface layer at a fluence of 10^{15} ion/cm^2 can be due to exfoliation due to the high concentration of defects in the structure due to their accumulation.

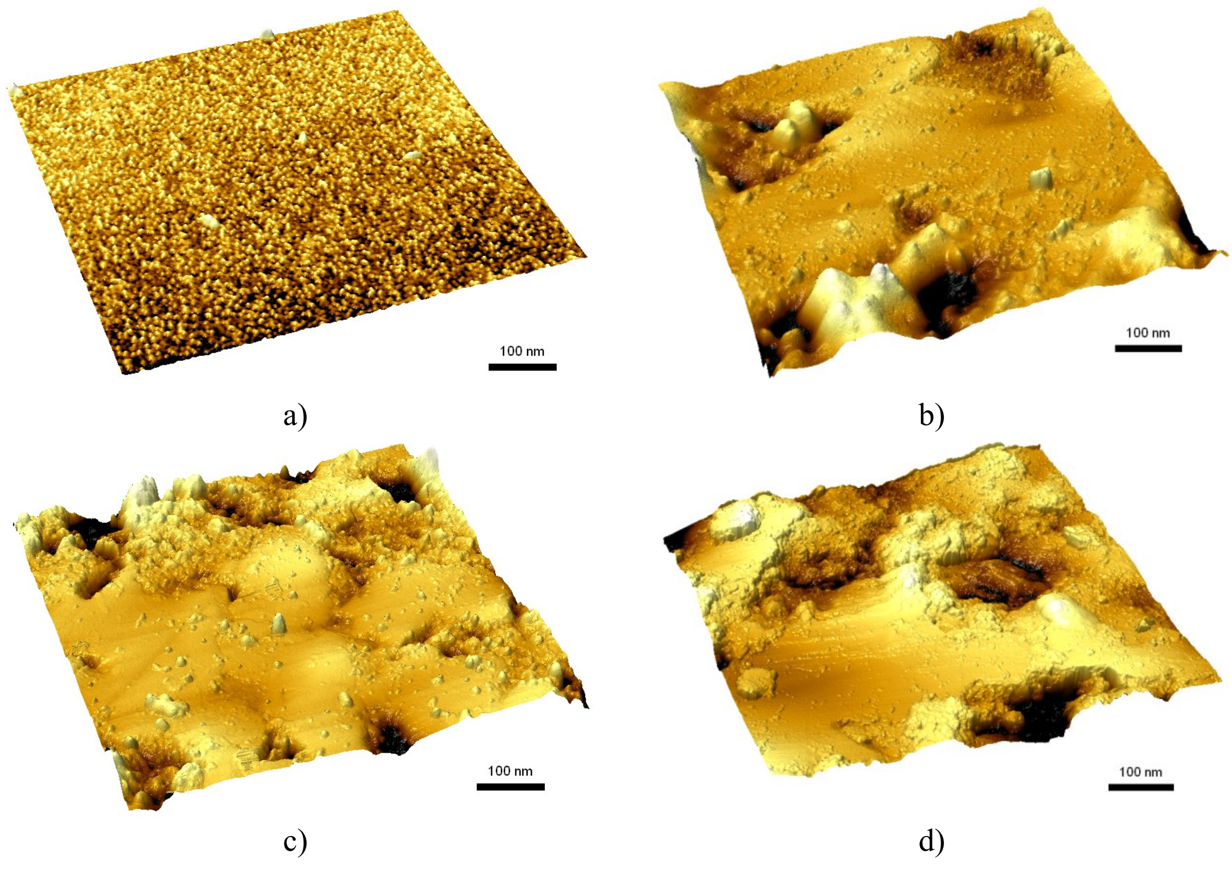

Fig.2. 3D atomic force microscopy (AFM) images of ceramics before and after irradiation: a) Initial sample; b) 10^{14} ion/cm^2; c) $5x10^{14}$ ion/cm^2; d) 10^{15} ion/cm^2

Partial degradation of the surface layer due to the accumulation of defects due to the effect of cascade overlaps can lead to a significant change in the mechanical properties and wear resistance of ceramics, since the formation of anisotropic defect regions near grain boundaries, as well as the partial destruction of crystalline and chemical bonds, followed by the formation of initially knocked out atoms, may lead to distortions and deformations in the structure. Table 2 presents the results of changes in strength characteristics, such as the value of bending strength and impact strength before and after irradiation, as well as a change in the dislocation density, porosity and density of ceramics during irradiation.

Table 2. Data of strength characteristics.

Parameter	Initial sample	10^{14} ion/cm^2	5×10^{14} ion/cm^2	10^{15} ion/cm^2
Three-point bending strength, MPa	178±4	173±4	168±5	143±9
Impact toughness, kJ/mm^2	1.35±0.13	1.31±0.11	1.24±0.12	1.12±0.09
Dislocation density, 10^{15} unit/cm^2	0.14	0.17	0.25	0.62
Porosity, %	0.606	0.656	0.945	2.351
Density, g/cm^3	3.196	3.165	3.121	3.014

According to the data presented, the greatest change in strength characteristics is observed at a fluence of 10^{15} ion/cm^2, which is characterized by the presence of a large number of defects as a result of overlapping cascades of secondary defects, which lead to a strong disordering of the structure with a sharp increase in the dislocation density by more than 4 times compared to the initial sample, and an increase in porous inclusions characterizing defective regions in the crystal lattice. An increase in porosity, as well as dislocation density, caused by grain crushing processes as a result of deformation initiated by irradiation, leads to a decrease in the density of ceramics. A decrease in density indicates a degradation of the ceramic structure during irradiation, and an increase in the dislocation density due to grain crushing can explain the degradation of the surface layer with an increase in the irradiation fluence.

Figure 3 presents the results of changes in the coefficient of dry friction depending on the dose of radiation and the number of tests.

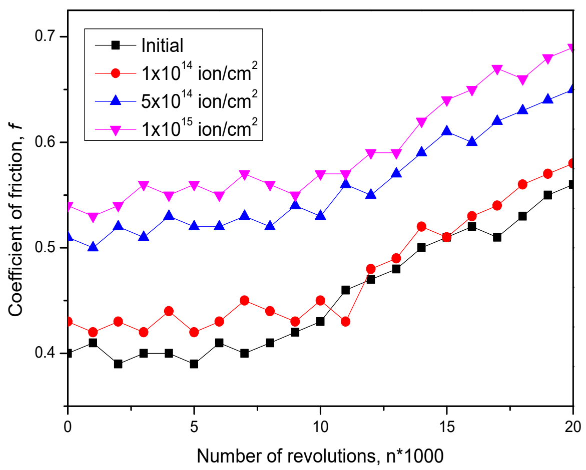

Fig.3. Dependence of the coefficient of dry friction on the dose.

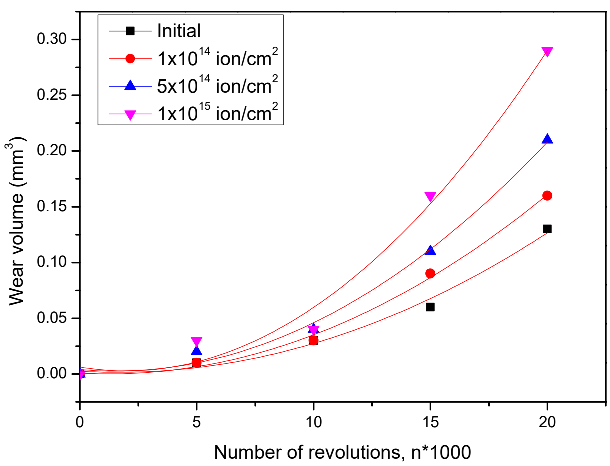

Fig.4. The dynamics of changes in volume loss during wear, depending on the dose.

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As can be seen from the data presented, the dry friction coefficient for the initial sample is 0.4-0.41 and remains for 8000-10000 cycles, which indicates a high degree of wear resistance.

For irradiated samples with a dose of 10^{14} ion/cm^2, a slight increase in the coefficient at the initial stage is observed, while the nature of the change in the value during the tests is comparable with the initial samples. For samples irradiated with doses of $5x10^{14}$ and 10^{15} ion/cm^2, an increase in the dry friction coefficient is observed, which is due to a change in the surface morphology, as well as its partial degradation, which leads to deterioration of friction. It should be noted that the nature of the change in the value of wear resistance during testing is comparable with the original sample. Figure 4 shows the results of changes in the amount of wear during testing, according to which the largest changes in volume loss occur after 10,000 cycles. Moreover, for samples irradiated with a dose of 10^{15} ion/cm^2, the volume loss is maximum and exceeds losses in the initial state by 2.5-2.7 times.

Surface degradation due to irradiation is directly related to a decrease in the hardness of the surface layer. Figure 5 presents the results of a change in the microhardness along the depth of the sample in order to determine the maximum depth of the damaged layer. According to the data presented, for samples irradiated with a dose of 10^{14} ion/cm^2, the decrease in the microhardness is insignificant, with the depth of the damaged layer being 200-250 nm, while the ion path length is not more than 200 nm.

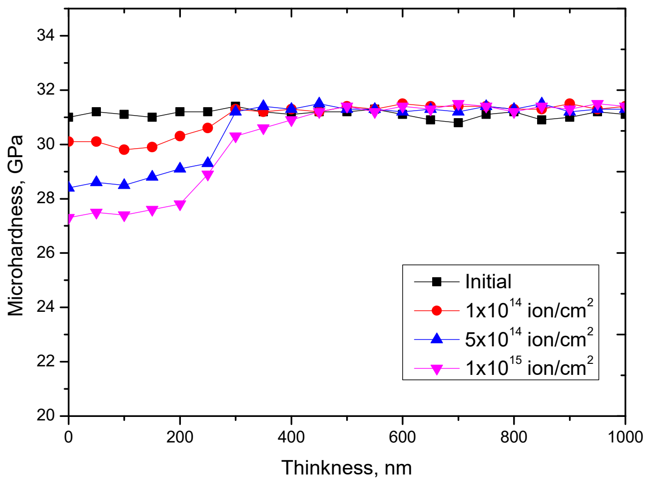

Fig.5. The dependence of changes in microhardness on the dose of radiation.

The increase in the depth of the damaged region is due to the cascading effects of the propagation of defects capable of penetrating to a depth exceeding the maximum mean free path of ions in the ceramic. Also, an increase in the depth of damage may be due to the fact that during irradiation a decrease in the density of ceramics is observed as a result of degradation and the formation of anisotropic porous inclusions, which leads to an increase in the mean free path of ions. In the case of an increase in the radiation dose, not only a decrease in the micro-hardness of the near-surface layer is observed, but also an increase in the depth of the damaged zone, which confirms the previously made assumption about the effect of changes in the density of ceramics on the mean free path of ions.

Figure 6 presents the results of a study of the resistance of ceramics to low-temperature degradation under accelerated aging processes, which are used to simulate temporary aging processes for a long time. As can be seen from the presented data (see Figure 6), for irradiated samples, in contrast to the initial sample, aging occurs linearly, while for the initial samples during the first 10 hours the crack resistance is almost unchanged, which indicates a high resistance of ceramics to cracking . Moreover, according to the data presented in the diagram of Figure 7, the decrease in crack resistance as a result of irradiation does not exceed 2-3% of the initial value, which indicates a high resistance to degradation and crack formation. Moreover, both in the case of the initial sample and in the case of irradiated samples, the decrease in crack resistance after 30 hours of life tests does not exceed 8-10%, which confirms the high resistance to temporary degradation of ceramics.

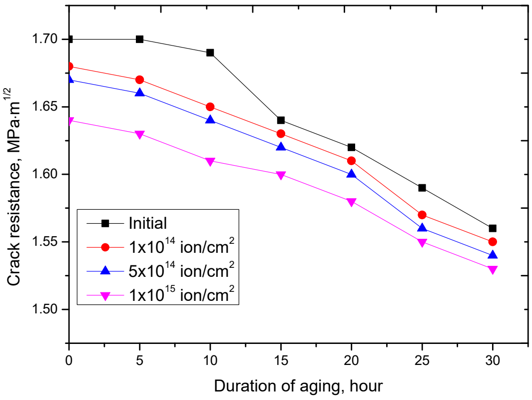

Fig. 6. A graph of the dependence of the crack resistance on the aging time of the samples before and after irradiation.

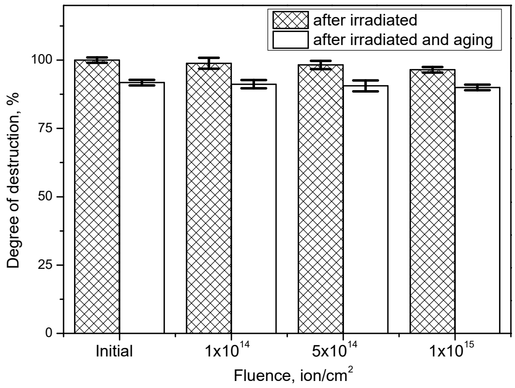

Fig. 7. Diagram of changes in the degree of destruction of ceramics before and after tests.

The data obtained are in good agreement with the previously presented studies of the radiation resistance of ceramics and thin-film coatings [31-34]. For example, the results of mechanical resistance to irradiation have a good correlation with the results of resistance to low-energy irradiation with He ions of thin-film structures based on ZrSiN [31], where it was shown that irradiation with high doses can lead to partial embrittlement and degradation of the surface layer. However, unlike oxide ceramics based on zirconium oxide (ZrO_2) [33] in which phase transformation processes are observed upon irradiation with low-energy Kr ions, no such effects were observed in the case of the studied ceramics.

In total, the presented results of changes in the strength of carbide ceramics as a result of irradiation with Xe^{22+} ions with doses of 10^{14}, $5x10^{14}$, 10^{15} ion/cm^2 can be further used not only from a fundamental point of view, as an addition to the theory of radiation damage to ceramics, but also from the practical side, as the results radiation resistance and modeling the effects of ionizing radiation on structural materials.

Conclusion

The paper presents the results of a study of the mechanical strength, wear and crack resistance of SiC-based ceramics before and after irradiation with heavy Xe^{22+} ions with an energy of 440 keV and irradiation fluences of 10^{14}, $5x10^{14}$, 10^{15} ion/cm^2. The choice of radiation doses is due to the modeling of the effects of overlapping cascade defects resulting from elastic and inelastic collisions, the number of which for the selected doses varies from 100 to 1000 multiple overlaps.

According to changes in mechanical properties, the largest change in strength characteristics is observed at a fluence of 10^{15} ion/cm^2, which is characterized by the presence of a large number of defects as a result of overlapping cascades of secondary defects, which lead to a strong disordering of the structure with a sharp increase in the dislocation density by more than 4 times compared to the initial sample, and an increase in porous inclusions.

During the study of the microhardness of ceramics before and after irradiation, it was found that in the case of irradiated ceramics, the damage depth exceeds the estimated ion path by 20-30%, depending on the irradiation fluence. The increase in the depth of the damaged area is due to a decrease in density as a result of the cascade effects of the propagation of defects that can penetrate to a depth exceeding the maximum mean free path of ions in the ceramic.

It was found that both in the case of the initial sample and in the case of irradiated samples, the decrease in crack resistance after 30 hours of accelerated degradation life tests simulating the aging effect does not exceed 8-10%, which confirms the high resistance to temporary degradation of ceramics. The obtained results will make a significant contribution to the development of the modern theory of radiation damage in ceramic materials applicable in nuclear energy.

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