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Study of destruction processes of mechanical properties of the near-surface layer of SiC ceramics exposed to heavy ion irradiation

Abstract: the work is devoted to the study of the heavy Kr^{15+} ion irradiation effect on the damage degree and changes in the mechanical and strength properties of the near-surface layer of SiC ceramics. The choice of SiC ceramics as an object for research is because these materials are considered as one of the candidate types of materials for nuclear energy, as the basis for materials for the first wall of the core. This choice is due to the combination of structural and strength properties of ceramics, which will allow them to be used under conditions of increased radiation background, as well as high temperatures. Irradiation with heavy Kr^{15+} ions makes it possible to simulate the processes of radiation damage appearance in the form of point defects, as well as to evaluate their evolution and its effect on the change in the resistance of a damaged surface to external influences during the radiation damage dose accumulation. To assess the change in strength properties because of the radiation damage accumulation in the near-surface layer, the methods of indentation, simulation of artificial aging processes, as well as determination of the resistance of ceramics to bending and impact strength, the combination of changes of which makes it possible to assess the material damage resistance degree, as well as to determine the main mechanisms associated with destruction and embrittlement processes. During the studies, it was found that the change in mechanical strength properties has a pronounced dependence on the irradiation fluence, as well as the probability of overlapping of defective regions formed along the trajectory of ions in the material. It has been determined that irradiation with a maximum fluence of $5x10^{13}$ ion/cm^2 leads to a decrease in strength properties by 10-15 %, which is because of radiation damage accumulation and the formation of disorder regions.

Keywords: silicon carbide, radiation damage, surface layer degradation, carbides, heavy ions.

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Introduction. One of the promising areas of research in the field of structural reactor materials science is research related to the study of radiation damage processes and the influence of their consequences on the stability and performance of structural materials [1,2]. At the same time, all research in this direction can be divided into theoretical and practical experimental research. Theoretical studies are related to the possibilities of modeling the radiation effect on the material, as well as the assessment of the consequences based on a priori models or already known similar studies [3-5]. Unlike theoretical studies, experimental work is aimed at obtaining new data on the properties of materials subjected to radiation exposure, as well as determining the combination of changes in material properties and their evolution during the radiation damage accumulation [6,7]. At the same time, due to differences in materials and their properties due to the preparation conditions, differences in structural features and phase compositions, when analyzing experimental data, it is necessary to consider a fairly large number of different factors, which introduces additional restrictions when interpreting the results of experimental work. Also, when analyzing the data obtained, attention should also be paid to the types of external influences and the conditions under which they were committed [8-10]. An important problem in the field of analysis of structural and strength changes in structural materials is the correct assessment of the value of structural damage and their influence on the change in the resistance of materials to the radiation damage accumulation. So, for example, the correct determination of the damage dose and areas with the maximum damage degree allows solving a number of issues related to the interpretation of the observed changes and the determination of the relationship between them [9,10]. One of the promising materials among the known structural materials, the study of the properties of which has been directed in the last few years, is SiC ceramics, which has a number of unique properties, which makes it one of the most suitable candidate materials for high-temperature nuclear reactors, as well as for spent nuclear fuel storage [11-13]. In this regard, the study of the properties of ceramics associated with resistance to radiation damage, which can lead to negative consequences and failure, is receiving increased attention from various research groups. Special attention is paid to the study of the relationship between the radiation damage degree and changes in the mechanical and strength properties of ceramics subjected to irradiation [14,15]. These studies will make it possible to determine the radiation damage resistance degree of ceramics, as well as to determine the level of their application to operation under conditions of an increased radiation background or exposure to direct radiation [16-18]. Based on the foregoing, the purpose of this research is to study the effect of irradiation of heavy Kr15+ ions with an energy of 147 MeV and irradiation fluences of 10^{10} - $5\text{x}10^{13}$ ion/cm^2 on the strength and mechanical properties of SiC ceramics, which will make it possible to determine the radiation damage resistance of the material. Interest in the mechanical and strength properties of SiC ceramics is due to the possibility of their practical application as structural materials for nuclear reactors, as well as to assess the resistance of the near-surface layer to radiation damage and their accumulation.

Materials and methods. Polycrystalline SiC ceramics with a hexagonal type of crystal structure were chosen as the objects of study. The samples were rectangles with a thickness of no more than 30-40 μm and dimensions of 10x10mm. Simulation of radiation damage processes was carried out at the DC - 60 heavy ion accelerator (Institute of Nuclear Physics of the Ministry of Energy of the Republic of Kazakhstan, Nur-Sultan, Kazakhstan). Heavy Kr^{15+} ions with an energy of 147 MeV and irradiation fluences of 10^{10} - $5\text{x}10^{13}$ ion/cm^2 were chosen for simulation.

Determination of the strength properties of the near-surface layer of SiC ceramics was carried out by the indentation method. Measurements were performed using a LECO LM700 microhardness tester (Leco Corporation, USA). A Vickers pyramid was used as an indenter. Tests were carried out by measuring 25 points on the surface, followed by the determination of the average value and measurement error.

The resistance to crack formation during artificial aging and low-temperature degradation of the surface of irradiated SiC ceramics was carried out by modeling the processes of accelerated degradation and exposure to water vapor at a temperature of 150 $^\circ$C and a pressure of 2.2-2.3 atm. The tests were carried out for 30 hours, which makes it possible to simulate aging processes for 3 - 5 years.

Material stability studies for impact strength and three-point bending were carried out on a special pendulum impact tester, according to ASTM D 7264/D7264M-07. These tests were carried out to determine the propensity of irradiated materials to embrittlement when radiation damage accumulates.

Results and discussion. Figure 1 shows the results of changes in the hardness of the near-surface layer and resistance to softening depending on the irradiation fluence, which characterize changes in the strength of ceramics to external mechanical influences. As can be seen from the data presented, a change in the hardness values is observed for irradiation fluences exceeding the value of $10\,11^{11}$ ion/cm^2, which are expressed in a decrease in the hardness value and an increase in the softening degree of the near-surface layer. At the same time, an increase in fluence above 10^{12} ion/cm^2 leads to a sharp decrease in hardness and an increase in the softening degree by more than

15 %, which indicates the disordering effect and partial destruction associated with deformation processes caused by irradiation.

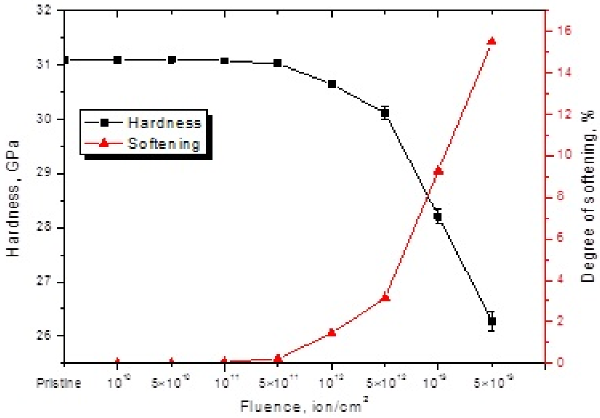

FIGURE 1 – Results of changes in the hardness and softening degree of the damaged near-surface layer depending on the irradiation fluence

Figure 2 shows the results of the change in the crack resistance after artificial aging simulation tests for 30 hours, reflecting the change in the crack and destruction resistance of the surface during aging. The general appearance of changes in the crack resistance, which reflects the resistance of the ceramic surface to degradation processes and the microcrack formation due to a change in the concentration of defects in the near-surface damaged layer, is characterized by two areas with a different trend of changes in this value.

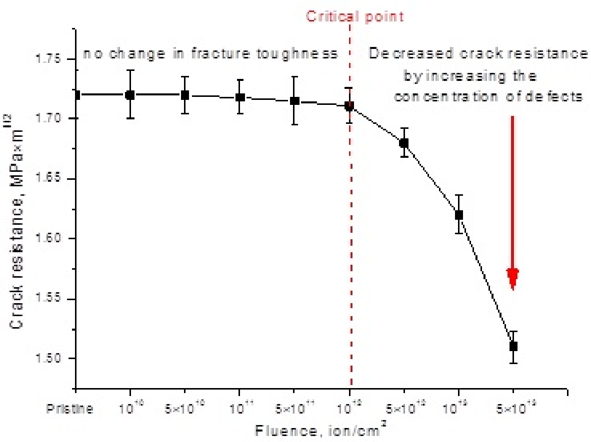

FIGURE 2 – Results of the change in the crack resistance of the near-surface layer of SiC ceramics after testing for artificial aging

The first stage is typical for irradiation fluences of 10^{10} - 10^{12} ion/cm^2 and is characterized by the almost complete absence of changes in the crack resistance in the entire observed range, with a small, no more than 0.1 - 0.5 % deviation with an increase in the irradiation fluence above 10^{11} ion/cm^2 (see the data in Figure 3, which reflect the change in the crack resistance). This behavior of the ceramic surface to changes in crack resistance indicates a high degree of resistance of SiC ceramic materials not only to irradiation with low fluences, but also to their subsequent testing for artificial aging and low-temperature degradation. This behavior of the near-surface layer and its high stability can be explained as follows. At low irradiation fluences of 10^{10} - 10^{11} ion/cm^2, the dominant radiation damage processes are the processes of formation of regions with single point defects, which have a diameter of several nanometers and are quite distant from each other, which indicates their isolation. In this case, due to the nature of the interaction of incident heavy ions with matter, the dominant role is played by the interaction of ions with the electronic subsystem of matter over most of the ion trajectory. Such an interaction of incident ions with a substance leads to a change in the electron density distribution along the ion motion trajectory, which, in the case of low fluences and the isolation of such structurally changed regions, leads to small changes, most of which can relax in very short time intervals. An increase in the irradiation fluence leads to a decrease in the distance between these structurally changed regions, which subsequently affects the probability of the formation of more complex structural defects and dislocation loops. Such an increase in the probability leads to the fact that defective inclusions and additional distortions can appear in the structure, leading to destabilization of the crystal structure of the near-surface damaged layer.

The critical point for the samples under study, upon reaching which the trend of change in the crack resistance changes sharply, is an irradiation fluence of 10^{12} ion/cm^2. When this fluence is reached, the crack resistance decreases sharply, and at an irradiation fluence of 5x10^{10} ion/cm^2, the deterioration in crack resistance is more than 10 %.

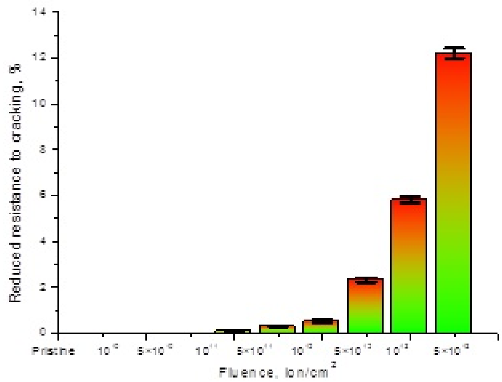

Figure 3 – Evaluation of the change in decrease value of the crack resistance after testing for artificial aging, depending on the irradiation fluence

Such sharp changes in the crack resistance indices of the surface layer of irradiated SiC ceramics after tests for artificial aging can be due to several factors. Firstly, according to estimated calculations, with an irradiation fluence above 10^{12} ion/cm^2, with a radius of the damaged region that occurs along the ion motion trajectory in the material of 5-10 nm, the probability of overlapping of such local regions increases sharply. This leads to the fact that more complex defect compounds can appear in the structure, which can have a significant effect on the structure of the material. Secondly, an increase in changes in the electron density during the occurrence of the overlap effect can lead to the appearance in the structure of metastable states that are strongly deformed and contain many structural distortions and stresses. In this case, the artificial aging processes associated with the creation of additional influences on the damaged layer can lead to accelerated destabilization of these areas, thereby accelerating the formation and propagation of microcracks in the damaged layer. The acceleration of microcrack formation processes due to the increase in distortions and deformations in the damaged layer leads to a deterioration in crack resistance and resistance to low-temperature degradation. Figure 4 shows the results of changes in the resistance of ceramics to three-point bending and impact strength depending on the irradiation fluence, reflecting the resistance of irradiated ceramics to dynamic external influences. The general trends in these values are similar to changes in hardness and crack resistance, however, changes in dynamic effects are also more pronounced at low irradiation fluences, which indicates that the formation of local areas of radiation damage under external bending loads leads to an acceleration of the propagation of longitudinal and transverse deformations, contributing to a sharp deterioration in resistance to external influences.

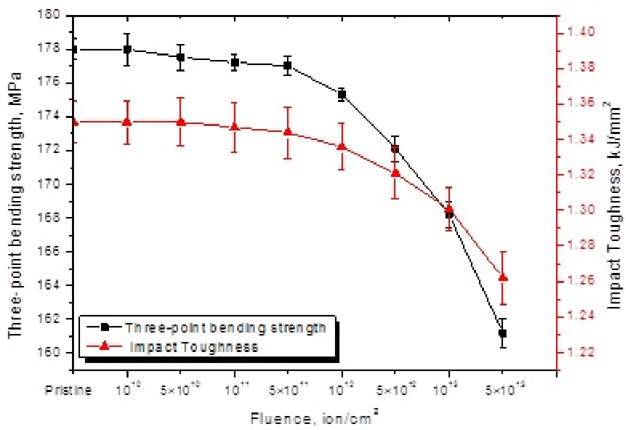

Figure 4 – Results of changes in the resistance of ceramics to three-point bending and impact strength depending on the irradiation fluence.

Figure 5 presents the results of a comparative analysis of changes in the strength characteristics because of external influences depending on irradiation fluence, which make it possible to assess radiation damage resistance of the near-surface layer.

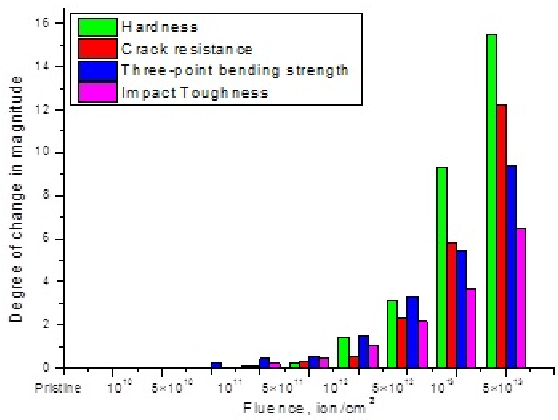

Figure 5 – Results of a comparative analysis of changes in the mechanical properties of ceramics depending on the irradiation fluence.

A general analysis of the change in the stability indicators of the mechanical and strength properties of ceramics to external influences shows that the most pronounced changes are observed for irradiation fluences above 110^{12} ion/cm^2, for which, as shown above, the formation of defect overlap regions with the possibility of the formation of more complex defect compounds, and the formation of dislocation loops is observed. At the same time, ceramics are the least resistant to mechanical impacts on the near-surface layer, the softening degree of which at the maximum fluence was more than 15 %. At the same time, it should be noted that irradiated samples are more resistant to dynamic loads, which indicates an increased resistance to embrittlement and fracture processes.

Conclusion. The paper presents the results of changes in the strength and mechanical properties of SiC ceramics exposed to irradiation with heavy ions Kr^{15+} with an energy of 147 MeV and irradiation fluences of 10^{10} - $5x10^{13}$ ion/cm^2, the choice of which is due to the possibility of radiation damage simulation comparable to uranium nuclei fission fragments in nuclear reactors. Interest in the mechanical and strength properties of SiC ceramics is due to the possibility of their practical application as structural materials for nuclear reactors, as well as to assess the resistance of the near-surface layer to radiation damage and their accumulation. Results of strength tests showed that the main changes in the near-surface layer hardness are observed in the case of an increase in the irradiation fluence above 10^{12} ion/cm^2, which are expressed in a partial softening of the damaged layer and a decrease in resistance to external pressures. During tests for artificial aging of samples, it was found that at irradiation fluences of 10^{10} - 10^{12} ion/cm^2, no changes in crack resistance are observed, which indicates a high resistance of the surface of SiC ceramics to the microcrack formation because of exposure to low-temperature degradation and water vapor, simulating natural aging processes. An increase in the irradiation fluence above 10^{12} ion/cm^2 leads to the formation of defect overlap regions in the structure of the damaged ceramic layer, which leads to a sharp increase in distortions and stresses in the ceramic structure, followed by an acceleration of the deformation and structural degradation processes.

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Ауыр иондармен сәулеленуге ұшыраған SiC керамикасының беткі қабатының механикалық қасиеттерінің бұзылу процестерін зерттеу

Аннотация. Жұмыс ауыр Kr $^{15+}$ иондарымен сәулеленудің SiC керамикасының бетке жақын қабатының зақымдану дәрежесі мен механикалық және беріктік қасиеттерінің өзгеруіне әсерін зерттеуге арналған. Зерттеу объектісі ретінде SiC керамикасының таңдалуы бұл материалдар ядроның бірінші қабырғасы үшін материалдардың негізі ретінде ядролық энергетикаға үміткер материалдар түрлерінің бірі ретінде қарастырылуымен түсіндіріледі. Бұл таңдау керамиканың құрылымдың және беріктік қасиеттерінің үйлесімімен түсіндіріледі, бұл оларды жоғары радиациялық фон жағдайында, сондай-акқ жоғары температурада пайдалануға мүмкіндік береді. Kr $^{15+}$ ауыр иондарымен сәулелену нүктелік ақаулар түрінде радиациялық зақымданулардың пайда болу процестерін модельдеуге, сондай-ақ олардың эволюциясын және радиациялық зақымдану дозасын жинақтай отырып, зақымдалған беттің сыртқы әсерлерге төзімділігінің өзгеруіне әсерін бақалауға мүмкіндік береді. Беткейге жақын қабатта радиациялық зақымданулардың жинақталуы нәтижесінде беріктік қасиеттерінің өзгеруін бағалау үшін шегіну әдістері, жасанды қартаю процестерін модельдеу, сонымен қатар керамиканың иілу және соққыға төзімділігін анықтау, жиынтық материалдың зақымдануға төзімділік дәрежесін бақалауға, сондай-ақ бұзылу және морттану процестерімен байланысты негізгі механизмдерді анықтауға мүмкіндік беретін өзгерістер. Зерттеулер барысында механикалық беріктік қасиеттерінің өзгеруі сәулелену флюенциясына, сондай-ақ материалдағы иондардың траекториясында пайда болған ақаулы аймақтардың қабаттасу ықтималдығына айқын тәуелді болатыны анықталды. Максималды флюменті 5x10 ^{13} ион/см ^{2} сәулелену беріктік қасиеттерінің 10-15%-ға төмендеуіне акелетіні анықталды, бұл радиациялық зақымданулардың жинақталуы әсерінен және ретсіз аймақтардың пайда болуына байланысты.

Түйін сөздер: кремний карбиді, радиациялық зақымдану, беткі қабаттың деградациясы, карбидтер, ауыр иондар.

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Исследование процессов деструкции механических свойств приповерхностного слоя SiC керамик, подверженных облучению тяжелыми ионами

Аннотация. Работа посвящена изучению влияния облучения тяжелыми ионами Kr $^{15+}$ на степень повреждения и изменения механических и прочностных свойств приповерхностного слоя SiC керамик. Выбор в качестве объекта для исследований SiC керамик обусловлен тем, что данные материалы рассматриваются как один из кандидатных типов материалов для ядерной энергетики, в качестве основы для материалов первой стенки активной зоны. Данный выбор обусловлен совокупностью структурных и прочностных свойств керамик, которые позволят использовать их в условиях повышенного радиационного фона, а также высоких температур. Облучение тяжелыми ионами Kr $^{15+}$ позволяет смоделировать процессы возникновения радиационных повреждений в виде точечных дефектов, а также оценить их эволюцию и ее влияние на изменение устойчивости поврежденной поверхности к внешним воздействиям при накоплении дозы радиационных повреждений. Для оценки изменения прочностных свойств в результате накопления радиационных повреждений в приповерхностном слое были применены методы индентирования, моделирования процессов искусственного старения, а также определению устойчивости керамик на изгиб и ударной вязкости, совокупность изменений которых позволяет оценить степень устойчивости материала к повреждениям, а также определить основные механизмы, связанные с деструкцией и процессами охрупчивания. В ходе проведенных исследований было установлено, что изменение механических прочностных свойств имеют выраженную зависимость от флюенса облучения, а также вероятности перекрытия дефектных областей, образующихся вдоль траектории движения ионов в материале. Определено, что облучение максимальным флюенсом 5x10 ^{13} ион/см2 приводит к снижению прочностных свойств на 10-15 %, которое обусловлено эффектами накопления радиационных повреждений и формированием областей разупорядочения.

Ключевые слова: карбид кремния, радиационное повреждение, деградация приповерхностного слоя, карбиды, тяжелые ионы.

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