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STUDY OF CHANGES IN THE MECHANICAL AND THERMAL CONDUCTIVE PROPERTIES OF ALN CERAMICS EXPOSED TO HEAVY ION IRRADIATION

The paper presents the results of study of the effect of irradiation with heavy Ar^{8+}, Kr^{15+} and Xe^{22+} ions with energies of 70, 150 and 230 MeV, respectively, on the stability of mechanical properties, in particular, hardness and wear resistance, as well as thermal conductivity, depending on irradiation fluence. The interest in this research topic is due to the wide prospects for the use of nitride ceramics as the basis for structural materials for nuclear and thermonuclear energy, exposed to ionizing radiation, in particular, particles – fragments of fission of uranium nuclei. During the experiments, dose dependences of changes in strength, mechanical and heat-thermal conductive properties were obtained. It has been established that the decrease in thermal conductivity has a pronounced dependence on both the energy of incident ions and the radiation dose. The obtained dependencies can later be used in the forecasting and design of nuclear power plants, in which it is planned to replace traditional materials with new classes, including ceramics or composite structures.

Key words: nitride ceramics, radiation defects, strength, thermal conductivity, degradation.

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Исследование изменения механических и теплопроводных свойств керамики AlN при облучении тяжелыми ионами

В настоящей работе представлены экспериментальные результаты исследования влияния облучения тяжелыми ионами, такими как, Ar^{8+}, Kr^{15+} и Xe^{22+} с энергиями 70, 150 и 230 МэВ соответственно на стабильность механических свойств, в частности, твердости и износостойкости, а также теплопроводность в зависимости от флюенса облучения. Интерес к данной теме исследований обусловлен широкими перспективами использования нитридной керамики в качестве основы конструкционных материалов для ядерной и термоядерной энергетики, подвергающихся воздействию ионизирующих излучений, в частности частиц – осколков деления ядер урана. Сохранение стабильности и неизменности таких показателей, как твердость, стойкость к трещинам, износостойкость и теплопроводность при длительном радиационном воздействии является основной задачей, которая ставится перед новыми видами конструкционных материалов. В ходе наших экспериментов были получены дозовые зависимости изменения прочностных, механических и теплопроводных свойств. Установлено, что снижение теплопроводности имеет ярко выраженную зависимость как от энергии падающих ионов, так и от дозы облучения. Полученные зависимости в дальнейшем могут быть использованы при прогнозировании и проектировании АЭС, в которых планируется замена традиционных материалов на новые классы, в том числе керамику или композитные конструкции.

Ключевые слова: нитридная керамика, радиационные дефекты, прочность, теплопроводность, деградация.

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Ауыр иондармен сәулелену кезінде AlN керамикасының механикалық және жылу өткізгіштік қасиеттерінің өзгеруін зерттеу

Бұл жұмыста энергиясы 70, 150 және 230 МэВ сәйкесінше ауыр Ar^{8+}, Kr^{15+} және Xe^{22+} иондарымен сәулеленудің механикалық қасиеттердің тұрақтылығына, атап айтқанда қаттылық пен тозуға төзімділігіне, және де сәулелену флюенциясына байланысты жылу өткізгіштігіне әсерін эксперименттік зерттеу нәтижелері берілген. Бұл зерттеу тақырыбына қызығушылық нитридті керамиканың ядролық және термоядролық энергияға арналған құрылымдық материалдардың негізі ретінде иондаушы сәулеленудің әсеріне ұшыраған, атап айтқанда, уранның бөлінуінің бөлшектері – фрагменттері ретінде пайдаланудың кең келешегімен түсіндіріледі. Ұзақ уақыт радиациялық әсер ету кезінде қаттылық, жарықшақтарға төзімділік, тозуға төзімділік және жылу өткізгіштік сияқты көрсеткіштердің тұрақтылығы мен өзгермейтіндігін сақтау – бұл құрылымдық материалдардың жаңа түрлеріне қойылатын негізгі міндеттердің бірі. Біздің жүргізілген тәжірибе барысында беріктік, механикалық және жылу өткізгіштік қасиеттерінің өзгеруінің дозаға тәуелділіктері алынды. Жылу өткізгіштіктің төмендеуі түскен иондардың энергиясына да, сәулелену дозасына да айқын тәуелді болатыны анықталды. Алынған тәуелділіктер кейінірек дәстүрлі материалдарды жаңа сыныптармен, соның ішінде керамика немесе композиттік құрылымдармен ауыстыру жоспарланатын атом электр станцияларын болжау және жобалау кезінде пайдаланылуы мүмкін.

Түйін сөздер: нитридті керамика, радиациялық ақаулар, беріктік, жылу өткізгіштік, деградация.

Introduction

One of the key requirements for new generation structural materials, including ceramics, used in nuclear power engineering in the development of new types of high-temperature nuclear reactors and thermonuclear installations, is to maintain their resistance to radiation damage and the accumulation of radiation-induced defects for quite a long time [1,2]. Maintaining the stability and invariance of such indicators as hardness, crack resistance, wear resistance and thermal conductivity during long-term radiation exposure is the main task set for new types of structural materials [3-5].

As is known, the decrease in the mechanical and strength properties of ceramics occurs due to the accumulation of radiation-induced damage and vacancy defects in the structure of the near-surface damaged layer, the thickness of which can vary from 0.2 μm to 15-30 μm. At the same time, accumulation of defects occurs unevenly in the damaged layer structure and is non-linear with the dose of radiation, since in the case of low doses of radiation, the formed defects and defective areas are isolated from each other and do not have an accumulative effect, which consists in the formation of areas of disorder and amorphization. structures [6-9]. In the case when these areas overlap, distortions and deformations occur in the structure of irradiated near-surface layer, caused both by structural damage associated with swelling and deformation of the crystal lattice, and by gas swelling effects caused by implantation effect and further agglomeration of ions in the crystal structure cavities [10-13]. Both of these factors have a negative effect on the mechanical and wear resistance of ceramics. Radiation defects also make an important contribution to the mechanisms of heat transfer, which consists in creating obstacles to heat removal from ceramics due to the deterioration of the thermal conductivity [14-16]. However, despite the general understanding of the problems associated with the accumulation of radiation damage in the structure of ceramics on mechanical, strength and thermal conductive properties, several issues still need to be clarified and studied in detail [17-20].

The aim of this work is to obtain new data on the kinetics of changes in the mechanical and conductive properties of nitride ceramics exposed to irradiation with heavy ions, which are comparable in mass and energy to uranium fission fragments in a reactor. Interest in this study is due to the prospects of using nitride ceramics as the basis for first wall materials, as well as inert nuclear fuel matrices in new generation reactors, which require the possibility of operating not only in conditions of increased radiation background, but also high temperatures of the core and coolant.

Experimental part

Polycrystalline ceramics, which have a high degree of crystallinity and structure ordering, as well as high hardness and resistance to external influences, were chosen as objects of study. The choice of nitride ceramics as objects of study is due to their thermal conductivity, resistance to mechanical damage, and melting temperature, which classify them as refractory high-temperature ceramics. According to previous studies, these ceramics are highly resistant to radiation swelling processes associated with the accumulation of helium and carbon in the structure during high-dose irradiation.

Simulations of radiation damage by heavy Ar^{8+}, Kr^{15+} and Xe^{22+} ions with energies of 70, 150 and 230 MeV, respectively, was carried out at the DC-60 heavy ion accelerator (Nur-Sultan, Kazakhstan), located on the basis of the Astana branch of the Institute of Nuclear Physics of the Ministry of Energy of the Republic of Kazakhstan. The irradiation doses were 10^{10}-10^{15} ions/cm^2, the ion flux was 10^9 ions/cm^2*s. The exposure dose set was monitored using an integrated particle flux estimation system based on Faraday cups. The temperature of the samples during the entire irradiation process was maintained in the range of 30-50°C using a special water-cooled holder. The control over the irradiation temperature was necessary to avoid the processes of thermal overheating of the samples and partial annealing of radiation-induced defects in the structure.

The study of changes in the strength properties of ceramics before and after irradiation was carried out using the indentation method, where the Vickers pyramid was used as an indenter at a load of 500 N. The surface layer microhardness was determined by 25 successive measurements from different parts of the damaged surface and subsequent determination of the standard deviation of the microhardness value.

Results and discussion

Figure 1 shows the results of measurements of the surface microhardness of ceramics exposed to irradiation with various types of ions, depending on irradiation fluence. In the initial state, ceramics have high strength values (more than 1850 HV), which makes them promising materials for structural materials that are subjected to high mechanical loads.

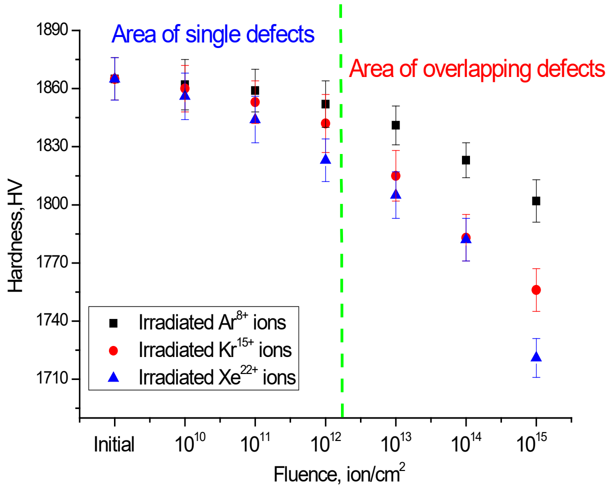

Figure 1 – Results of change in microhardness value depending on type of external effects

The general trend of changes in the hardness of the surface layer subjected to irradiation consists of two characteristic stages, which are associated with the effect of radiation damage and their accumulation. At the same time, it should be noted that a change in the type of irradiation ions leads to a change in the microhardness value, while the general trend of changes for each stage is preserved, which indicates a single mechanism of structural changes affecting the strength properties of ceramics.

The first stage is typical for irradiation doses of 10^{10}-10^{12} ion/cm^2, for which the change in microhardness values is minimal, which indicates a high degree of resistance of ceramics to irradiation with these fluences. Small changes in hardness can be due to the effect of single interactions of incident ions with the crystal structure of ceramics, as well as the isolation of damaged areas caused by irradiation. The isolation effect is explained by the fact that the diameter of the damaged region that appears along the trajectory of ions in the material is, according to calculations, no more than 10-20 nm, and the dimensions of this region have a pronounced dependence on the type of incident ions. The greater the mass and energy of the incident ion, the more destructive effect it has on the properties of materials. In this case, the main changes in the damaged areas are caused by two factors.

The first factor is associated with a change in the electron density along the trajectory of ions, which arises as a result of the passage of ions and elastic interactions of ions with electron shells. At the same time, in the case of ceramics, in contrast to metals that are dielectrics, the change in electron density does not have a return mechanism as in metals. In this connection, the change in the electron density in ceramics is associated with the occurrence of an anisotropic electron distribution along the trajectory of ions in the material, as well as near the defect region formed as a result of the ion passage.

The second factor of changes is associated with inelastic collisions of incident ions with atoms or nuclei, which leads to the appearance of primary knocked-out atoms, as well as vacancy defects in the structure. It should be noted that the formation of primary knocked-out atoms is greatly affected by the energy losses of ions, which directly depend on the initial energy of incident ions. However, it should be clarified that in the case of irradiation with heavy high-energy ions, the ratio of energy losses during interactions with electron shells and nuclei is approximately 1000:1, with great dominance of electronic interactions. As a result, the main contribution in the case of structural changes at low doses of irradiation, when all defective regions are isolated, is made by changes in the electronic structure, as well as by the appearance of electron density anisotropy. At the same time, most of the emerging point defects annihilate as a result of irradiation, and the proportion of surviving point defects is no more than 1-5 %.

As a result of such structural changes caused by isolated defective regions, the microhardness value changes insignificantly, and the decrease is associated with the occurrence of structural deformations and a change in the electron density, which forms disordered regions in the structure.

The second stage of changes in the values of microhardness is typical for irradiation doses of 10^{13} – 10^{15} ion/cm^2 and has a strong dependence of the decrease in microhardness on the dose. Such changes may be due to the effect of overlapping defective regions, as well as defective fractions and disordered regions resulting from such overlapping, leading to strong deformation of the crystal structure and partial destruction of chemical and crystalline bonds. With an increase in the irradiation fluence, the number of defective regions in the structure increases, and the probability of their overlap becomes close to unity at a fluence above 10^{12} ion/cm^2, and in the case of irradiation doses of 10^{14}-10^{15} ion/cm^2, the overlap probability is more than 100 and a deep overlap region is observed. This leads to the fact that previously isolated point defects that have arisen during irradiation are able to interact with each other when defective regions overlap, and in this case the effect of their mutual annihilation is leveled due to the formation of complex defects in the form of cluster defects. The result of such overlaps is the accumulation of a defective fraction in the structure of the damaged layer, swelling and deformation of the crystal lattice due to the knocking out of atoms from the lattice sites, as well as their subsequent migration and the formation of vacancies. In this case, part of the defective volume can be squeezed out from the inner damaged area onto the surface in the form of hillocks or blisters, the occurrence of which leads to embrittlement of the surface layer and a decrease in its strength.

Figure 2 shows the results of change in the near-surface layer softening degree, which reflects the strength degradation degree as a result of external influences. The calculations were made using the calculation formula (1):

$$SD = \left(\frac{H_0 - H}{H_0}\right) \times 100\%, \qquad (1)$$

where H_0 and H are the microhardness values in the initial and irradiated states.

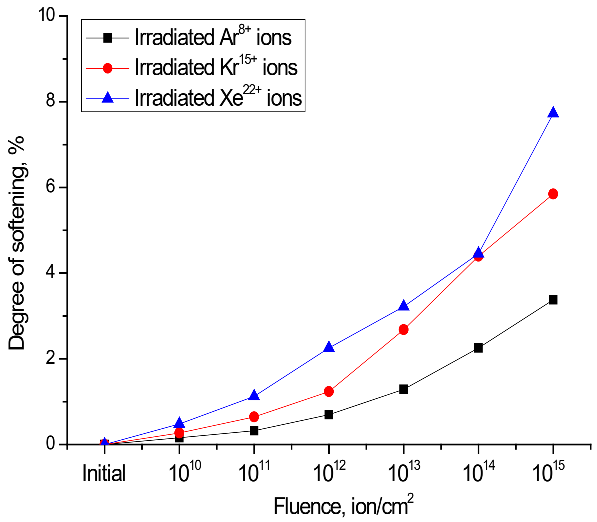

Figure 2 – Results of change in the softening degree depending on irradiation fluence

An analysis of the obtained dependences of softening degree change shows reverse trend of changes in the value of microhardness and reflects the degree of degradation of the strength characteristics of ceramics. As can be seen from the data presented, the greatest changes in softening degree are observed for irradiation fluences above 10^{13} ion/cm^2, which, as mentioned earlier, corresponds to the effect of accumulation of radiation damage of ceramics as a result of increased probability of overlapping damaged areas in the near-surface layer of ceramics. At the same time, it can be seen from the presented dependences that the change in the ion type, and, consequently, the energy and energy losses of incident ions in the material, the surface layer degradation degree increases. It is also worth noting that, in contrast to the samples irradiated with Ar^{8+} and Kr^{15+} ions, for which the change in the softening degree at fluences of 10^{13} – 10^{15} has a dependence close to linear, for samples irradiated with Xe^{22+} ions at an irradiation fluence of 10^{15} ion/cm^2, a sharp increase in surface degradation is observed. This behavior can be explained by a more destructive effect due to an increase in the interactions of incident ions with the electronic and nuclear subsystems.

An important factor determining the scope of ceramics, as well as their service life, is preservation of the stability of thermal conductive properties of irradiated ceramics. At the same time, for materials of inert matrices or walls of nuclear reactors, the values of thermal conductivity play a very important role in determining the efficiency of heat removal from the core, as well as heat transfer from fuel to coolant. Figure 3 shows the results of determining the ceramic thermal conductivity coefficient depending on various influences. The coefficient was determined using formula (2):

$$\lambda = \frac{q\delta}{t_{c1} - t_{c2}}, \quad (2)$$

where q is the heat flux density, W/m^2; t_{c1} and t_{c2} are the temperatures on both sides of the sample, K; δ is the sample thickness, m.

In the case of irradiation with Ar^{8+} heavy ions, the change in thermal conductivity coefficient occurs at fluences above 10^{12} ion/cm^2, while according to the estimate of thermal conductivity coefficient losses shown in Figure 4, the decrease is no more than 0.5 – 3.2 %, depending on irradiation fluence. It should be noted that for the majority of structural materials, the permissible reduction in thermal conductive properties is no more than 10 % of the nominal value. At the same time, irradiation with Ar^{8+} ions, which leads to a decrease by 3.2 % at the maximum irradiation fluence, indicates a weak irradiation effect on thermal conductivity even at high irradiation fluences.

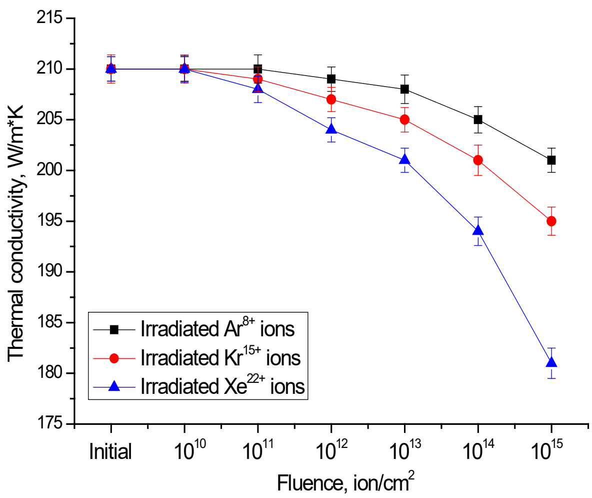

Figure 3 – Results of change in the thermal conductivity coefficient depending on the type of external influences

In the case of irradiation with heavy Kr^{15+} ions, a change in thermal conductivity coefficient is observed at a fluence of 10^{11} ions/cm^2 and higher, which indicates earlier changes in the heat-conducting properties than in the case of irradiation with Ar^{8+} ions. At the same time, the decrease in thermal conductivity coefficient at the maximum irradiation fluence by 7.1 % also falls within the permissible limits, which indicates that ceramics are more resistant to changes in thermal conductivity than mechanical strength and hardness at high irradiation fluences.

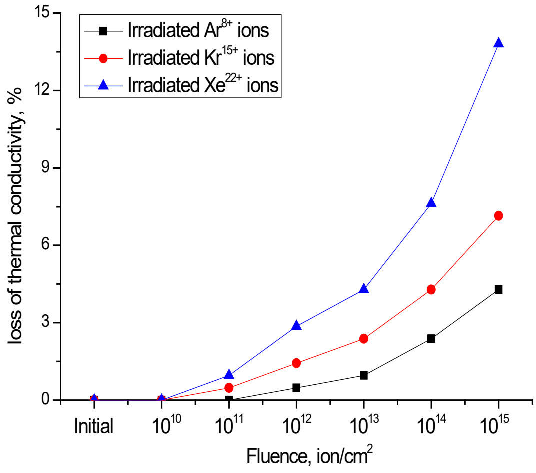

Figure 4 – Results of change in losses of thermal conductivity coefficient

The most pronounced changes in thermal conductivity coefficient are observed for ceramic samples irradiated with Xe^{22+} heavy ions, for which the change in thermal conductivity coefficient depending on irradiation fluence is exponential and reaches a maximum loss of 13.8% at an irradiation fluence of 10^{15} ion/cm^2, which exceeds the permissible limits.

Such behavior of the change in thermal conductivity coefficient, as well as the dependence of the change in thermal conductive properties on irradiation fluence, as well as the ion types, indicates that the main mechanism affecting the change in thermal conductive properties, in addition to the deformation contribution to structural changes, is the anisotropy of electron density. As shown above, a change in the energy and type of incident ions leads to large energy losses during elastic and inelastic interactions, as well as an increase in the diameters of damaged regions [21-23]. At the same time, the dominance of electron energy losses of incident ions, which leads to large changes in electron distribution along the ion trajectory, leads to an increase in anisotropy, which was stated earlier in [24,25]. In the case of irradiation with Xe^{22+} heavy ions, the diameters of the damaged regions are more than 20 nm, which, at high irradiation fluences, leads to overlapping of the defective regions and the formation of an anisotropic change in the electron density, with the formation of regions with a depleted electron density.

Conclusion

The paper presents the results of changes in the mechanical, strength and heat-conducting properties of ceramics depending on the type of external action and the irradiation fluence. Based on the results obtained, the dependences of change in the near-surface layer hardness are established. It has been determined that the main changes in strength have a pronounced dependence on the irradiation fluence, while at low irradiation fluences, changes in the strength properties are due to the effects of changes in electron density, while with an increase in the irradiation fluence, deformation contributions dominate, leading to disordering of the structure and a decrease in the strength of the near-surface layer. It has been established that the decrease in thermal conductivity has a pronounced dependence on both the energy of incident ions and the radiation dose. The obtained dependencies can later be used in the forecasting and design of nuclear power plants, in which it is planned to replace traditional materials with new classes, including ceramics or composite structures.

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