

**The M.N. Mikheev Institute of Metal Physics, Urals Branch of RAS  
The Russian Federal Nuclear Center – Zababakhin All-Russian  
Scientific Research Institute of Technical Physics  
The Scientific Council on Radiation Physics of Solids, RAS**

**The Fourteenth International Ural Seminar**



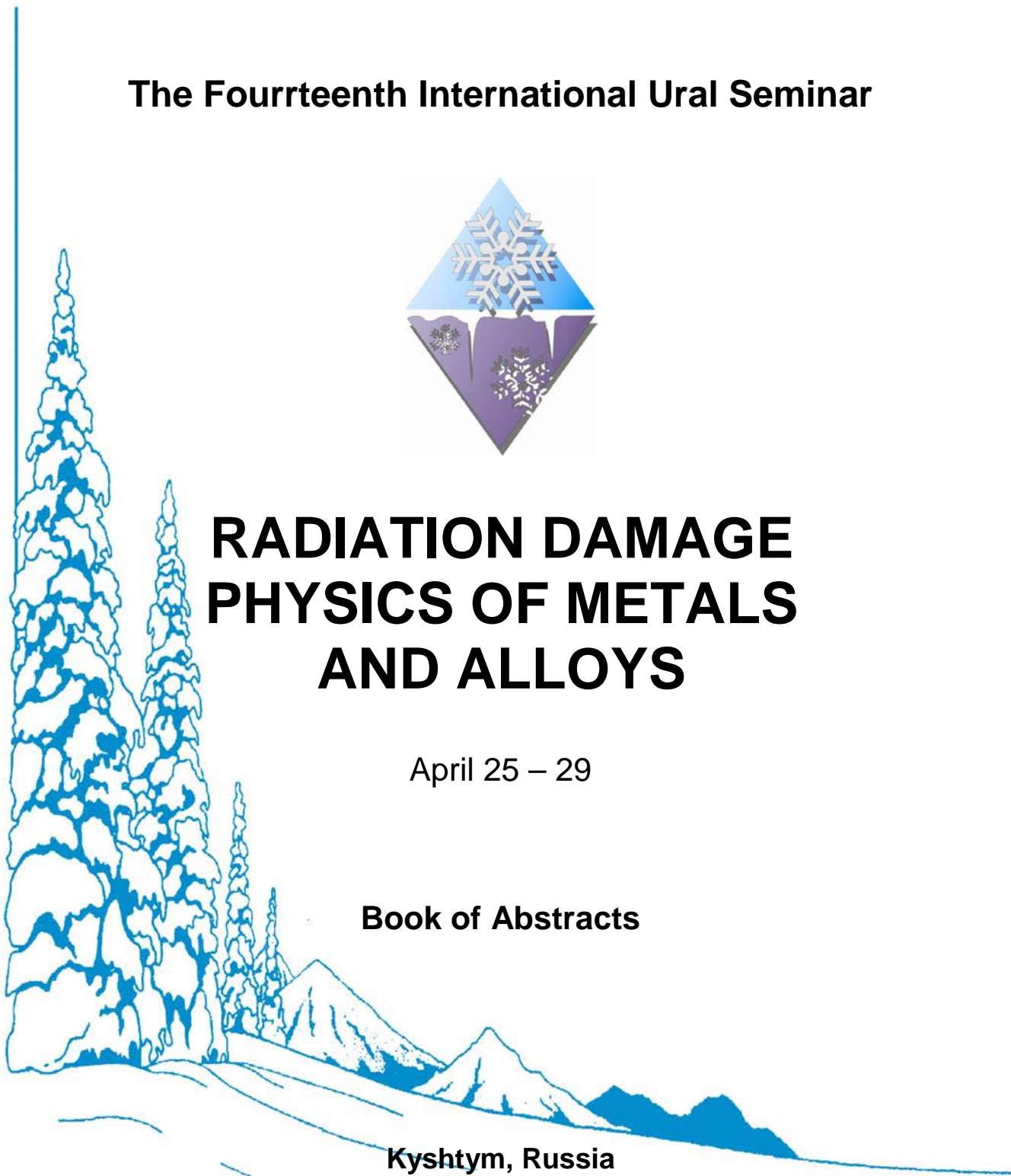
**RADIATION DAMAGE  
PHYSICS OF METALS  
AND ALLOYS**

April 25 – 29

**Book of Abstracts**

**Kyshtym, Russia**

**2022**



**To the memory of Academician of the RAS  
E.N. Avrorin and Corresponding Member of the RAS  
B.N. Goshchitsky**



**The Fourteenth International Ural Seminar  
on  
RADIATION DAMAGE PHYSICS  
OF METALS AND ALLOYS**

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**The Organizing Committee cordially thanks all Sponsors for their support to the Russian science. We hope that our meeting in Snezhinsk will serve to promote and expand further scientific contacts.**

**Compiled by  
Denis Perminov**

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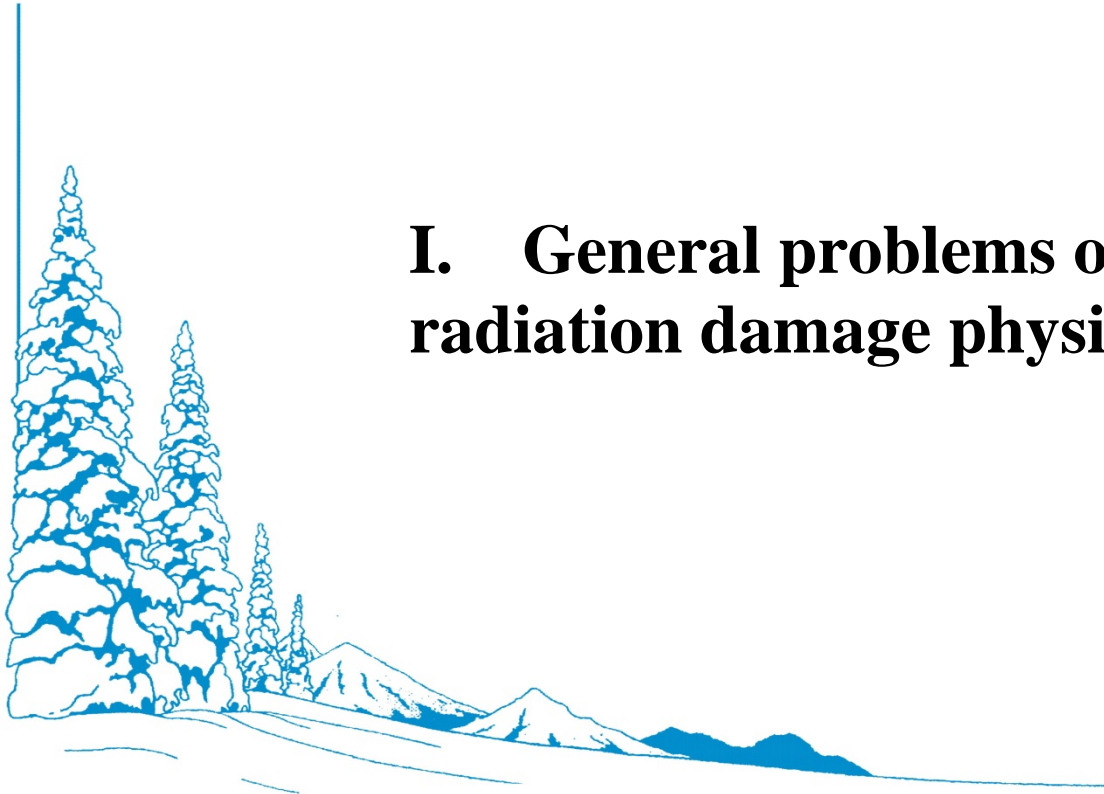
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# I. General problems of radiation damage physics

**This Section is dedicated to most topical, as of now, problems of radiation damage physics of metals and alloys. It includes reports on specific features of the behavior of point defects in various alloys and compounds including Fe-Cr(Ni) systems, which are the basis of many radiation-resistant high-pressure-vessel materials. The program of the Section includes papers dedicated to investigations into specific features of the processes involved in the interaction of radiation- and deformation-induced point defects and their complexes with each other, and with impurity atoms, dislocations, interfaces, and grain boundaries using modern research methods at all stages of the formation of a complicated defect structure in nano- and submicrocrystalline metal systems. Also, the effect of these interactions on deformation- and radiation-induced processes is studied. Much attention is given to multiscale modeling of radiation processes in irradiated materials, analysis of structural and phase transformations, and the behavior of transmuted gas mixtures.**



**ANISOTROPIC DIFFUSION OF RADIATION DEFECTS IN BCC (Fe, V)  
AND FCC (Cu) CRYSTALS: AN MD STUDY**

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To construct physically based models of crystal deformation under damaging irradiation (e.g., irradiation creep), it is necessary to determine the effect of external and internal fields of elastic deformations on the anisotropic diffusion of radiation defects (RDs). This effect one can take into account if one knows the dipole tensor of the saddle-point configuration of the diffusing RD. The dipole tensor is usually calculated by computer simulation methods (molecular statics), since its experimental determination is difficult due to the insufficient accuracy of the available experimental techniques. However, if an RD has many metastable and saddle-point energetically different crystallographic configurations, the problem becomes practically unsolvable due to the need to use many physically unfounded simplifications or assumptions.

In this work, we use a different approach to solving this problem. The molecular dynamics (MD) method is used to calculate the deformation dependences of the RD diffusion tensor for various deformed state types. These dependences are used to determine the RD elastodiffusion tensor, the components of which are the coefficients of the linear terms of the expansions in powers of the deformation of the calculated deformation dependences. The crystal symmetry and the RD diffusion mechanism determine the symmetry of this tensor. It corresponds to the dipole tensor of the effective saddle-point configuration of the RD, which takes into account the contributions of all real saddle-point configurations.

The proposed approach is realized to determine the diffusion characteristics of RDs such as di-interstitials in bcc (Fe, V) and fcc (Cu) crystals. Di-interstitials are one of the most frequently generated types of clusters of self-interstitial atoms under damaging irradiation, they have a large number of different metastable and saddle configurations, on which it is convenient to demonstrate the physical advantages of the approach used. The MD calculations have been performed with use of the interactions potentials, which describe well the experimentally known properties of the crystal bulk and self-point defects. The symmetry of the calculated dipole tensors of the effective saddle configurations is trigonal and orthorhombic for the considered bcc and fcc crystals, respectively. For all the considered types of deformations, crystal lattices, materials, the effect of the external field of elastic deformations on the normalized diffusion tensor (the ratio of the diffusion tensor to its trace) of di-interstitials calculated by the MD method is fully consistent with theoretical expressions, the parameters of which are the components of the dipole tensors, including the range of non-linear dependence of the diffusion tensor on deformations. The results obtained make it possible to simulate the anisotropic diffusion of di-interstitials in external and internal (e.g., dislocation) fields of elastic deformations, to take into account the contribution of di-interstitials to the radiation deformation of crystals.

*This research was funded by National Research Center “Kurchatov Institute” (grant number 2764 from 28.10.2021) and was carried out using computing resources of the federal collective usage center, Complex for Simulation and Data Processing for Mega-science Facilities at NRC “Kurchatov Institute”, (<http://ckp.nrcki.ru/>).*

## ATOMISTIC MODELING OF CROWDION BEHAVIOR IN BEING DEFORMED FCC METAL

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In this work [1], we study the behavior of a crowdion in an fcc metal, for which nickel and copper are taken, in the process of severe deformation. Modeling was carried out by the molecular dynamics (MD) method using the LAMMPS package. The interatomic potentials constructed in the framework of the many-particle embedded atom method are used. After preliminary relaxation annealing, the crystallite was subjected to compression at different temperatures and strain rates. The atomic displacement fields of the crowdion and its average velocity were calculated. The phonon spectrum and density of states were calculated on the basis of MD simulation using the Green's function method (FGMD).

In this work, we used the description of the crowdion in the Frenkel-Kontorova model. The discrete equations of motion describing the crowdion can be represented approximately by the sine-Gordon equation, for which the soliton (crowdion) solution used in the work is known. It is shown that at a deformation of about 10% under the conditions of simulation, a crowdion configuration of the  $\langle 110 \rangle$  type of an intrinsic interstitial atom becomes preferable over a dumbbell configuration in a wide temperature range. The fields of atomic displacements of a moving crowdion, obtained in MD simulation and using the formula of the crowdion solution, are presented. It was shown that a satisfactory description of the MD simulation data is observed at various temperatures and strain rates. It was found that the dimensionless width of a soliton  $N$ , which determines the field of atomic displacements of a moving crowdion, depends on such quantities as Young's modulus and the speed of sound in a metal. The calculation of the phonon density of states as a function of temperature and deformation made it possible to estimate the limiting crowdion velocity corresponding to motion with radiation losses. It is shown that the speed of the crowdion movement under the simulation conditions is significantly lower and decreases with a decrease in the deformation rate, i.e. accompanied by radiation losses. It was found that when the crowdion moves in a wide temperature range, the self-focusing condition is satisfied, which makes it possible for the crowdion to move over significant distances in comparison with the lattice parameter.

The results obtained reveal the important role of crowdions in physical processes occurring during irradiation and severe plastic deformation of fcc metals, especially at room and lower temperatures. In particular, the crowdion mechanism can play an important role in the process of anomalous dissolution (including at cryogenic temperatures up to 77 K) of particles of the intermetallic compound  $\text{Ni}_3\text{Al}$  in the matrix of the Fe–Ni–Al austenitic alloy [2], observed in the study of the process of low-temperature deformation-induced nanostructuring by shear under pressure method.

*The work was carried out within the framework of the state assignment of the Ministry of Education and Science of Russia (topic "Structure", No. AAAA-A18-118020190116-6). The calculations were carried out using "Uran" supercomputer of IMM UB RAS.*

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## DETERMINATION OF THE CHARACTERISTICS OF RADIATION DEFECTS FORMED IN STRUCTURAL MATERIALS UNDER LOW-TEMPERATURE NEUTRON IRRADIATION

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To describe radiation-induced processes occurring in reactor materials under neutron irradiation, it is important to know the kinetics of the formation of point defects in materials and their energy characteristics, in particular, the energy of migration and interaction with sinks. To determine them, calculations are carried out using methods of molecular dynamics and various types of interatomic potentials. The results obtained by different authors differ, the situation becomes much more complicated when they are trying to obtain them for real multicomponent reactor steels or alloys. The development and use of experimental methods to determine the characteristics of point defects generated by irradiation is an urgent task of reactor materials science.

The report presents a comprehensive technique combining low-temperature neutron irradiation, pre-reactor and post-reactor dilatometric measurements and transmission electron microscopy, including high resolution. The analysis of the formation and accumulation of radiation defects is given, both in the form of single Frenkel pairs, the number of which is  $G \cdot \theta$  ( $G$  – is the rate of generation of atomic displacements), and in the form of cascades of displacements in which  $G \cdot \alpha \cdot (1 - \theta)$  vacancies remain after recombination ( $\alpha$  is cascade efficiency). Before and after neutron irradiation at a temperature not exceeding 50 °C, elongation measurements are carried out on the same samples when heated from 50 to 450 °C at a rate of 1 °C/min, after which a difference dilatometric diagram  $\delta\varepsilon(T) = \left(\frac{dl}{l_0}\right)_{unir} - \left(\frac{dL}{L_0}\right)_{ir}$  is constructed, which contains information about the processes of generation and evolution of point defects during irradiation and after reactor heating in the dilatometer. A methodology for analyzing temperature dependence  $\delta\varepsilon(T)$  has been developed.

Using the example of shell austenitic steel CHS68, it is shown that the temperature at which the migration of various components of steel begins in vacancies is not the same for the main replacement elements: Ni, Fe, Cr. Based on the comparison of vacancy flows at different temperatures, a method has been developed for correctly determining the average migration energy of vacancies and it is shown that it depends on temperature. The analysis takes into account the dimensional changes associated with dissociation during heating of vacancy clusters.

The results obtained are illustrated by the TEM data.

## DIFFUSION CHARACTERISTICS OF CLUSTERS OF SELF-INTERSTITIAL ATOMS IN VANADIUM: MOLECULAR DYNAMICS DATA

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Diffusion of radiation defects generated by neutron radiation and their absorption by sinks lead to the evolution of the microstructure of materials and, as a consequence, to changes of their physical and mechanical properties. To construct phenomenological models of the properties of materials under irradiation changes, it is necessary to know the characteristics of radiation defects. It is not only self-point defects but also their clusters that are formed under irradiation. According to the results of molecular dynamics (MD) modelling of atomic collision cascades in bcc metal V, 68% of the surviving self-interstitial atoms (SIAs) after the end of the cascade do not form clusters, while clusters ranging in size from two to five SIAs contain 31% of SIAs [1, 2].

Temperature dependences of the diffusion characteristics of radiation defects, namely, clusters of up to five SIAs, were studied by MD method for V in the temperature range 300—1000 K. The diffusion characteristics included the diffusion coefficient, the tracer correlation factor, the mean distance traveled between changes in the migration direction, the frequency of migration direction changes, etc. The activation energy values for diffusion and changes in the migration direction were determined for the defects of interest for different temperature ranges. The temperature and size dependences of the SIA cluster diffusion mechanism (1D vs 3D) and their potential implications for the microstructure of materials exposed to irradiation are discussed.

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## FRENKEL DEFECTS IMMOBILIZATION IN Fe-13Cr-2Si

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We studied recovery of residual electrical resistivity (RR) after low temperature irradiation with 5MeV electrons and accumulation of radiation-induced defects together with their annealing after room temperature electron irradiation by means of residual resistivity and positron annihilation in Fe-13Cr-1.9Si alloy. A comparison is made with the non-doped alloys and those doped with 0.2 at. % Si and 0.3 at. Sb. According to RR a stage of the onset of long-

range migration in Fe-13Cr-1.9Si is shifted up to 370 K (self-interstitial atoms) and 420 K (vacancies) as compared to 205 K (vacancies) and 220 K (self-interstitial atoms) in the non-doped alloy. A resistivity growth is observed in Fe-13Cr-1.9Si after irradiation at room temperature while a resistivity drop is seen in the other alloys because of the short-range order formation (short-range clustering) enhanced by long-range migration of radiation-induced defects. Positron annihilation indicates the fast accumulation of vacancies at room temperature with saturation of S-parameter at a damage about  $\sim 0.01$  dpa. The latter data confirms the data of RR on immobility of Frenkel pair defects at room temperature in Fe-13Cr-1.9Si.

## NIOBIUM BEHAVIOR UNDER THE INFLUENCE OF PULSED ION AND PLASMA FLUXES DURING IRRADIATION IN DENSE PLASMA FOCUS UNIT

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The damageability of niobium by pulsed fluxes of helium ions (HI) and helium plasma (HP) in the Plasma Focus (PF) setup was studied at a flux power density  $q_i \sim 10^8$  W/cm<sup>2</sup> and  $q_p \sim 10^7$  W/cm<sup>2</sup>, respectively, and pulse duration  $\tau_i \approx 30 - 50$  ns and  $\tau_p \approx 100$  ns.

In the implemented irradiation conditions the erosion of the material is observed associated with the evaporation of the surface layer (SL), which occurs somewhat more intensively in the central part of the irradiation zone under the action of the most high-energy fluxes of HI and HP.

The typical features of the damageability of the niobium SL under the considered irradiation conditions are revealed. These include: melting of SL with the formation of a wavy surface relief and a large number of blisters of two types - gas-filled and with destroyed shells, as well as the presence of microcracks. The appearance of blisters was associated with the formation of complexes based on the combination of implanted helium atoms with vacancies and interstitial impurity atoms (C, O, N, etc.) and their subsequent growth and coagulation in the liquid phase under pulsed action of energy fluxes on the irradiated Nb surface. Some of the microcracks formed in the SL under the action of thermal stresses coincide with the sliding lines of the material that arise under the action of high-speed plastic deformation.

In the irradiated surface layer of niobium, zones of columnar crystals and a cellular microstructure of the surface are found, in which the average cell size is  $\sim 100$  nm. It was shown by the method of numerical simulation that in the indicated zones the process of solidification of the SL proceeded through directional crystallization at a high speed, which reached  $\sim 35$  m/s near the irradiated surface.

*The work was complete according to the state task № 075-00328-21-00*

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## POINT DEFECTS AND SELF-DIFFUSION MECHANISMS IN TRANSITION METAL CARBIDES

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*Ab initio* calculations provide useful data on the energies of atomic defects and defect arrangements, as well as on the activation barriers of defect migration through the lattice [1-5]. This information is essential for modeling the structure and property evolution in materials during manufacturing and service. Systematic *ab initio* studies show that the formation energy of Schottky defects in cubic TM–X compounds decreases along the series X = C, N, O. For example, vacancies are stable defects in TiO, while in TiC the calculated formation energy of a Schottky pair is too high for Ti mono-vacancies to be mediators of Ti self-diffusion. We have considered several possible mediators of metal diffusion in TiC, including clusters of vacancies and interstitial atoms.

Different diffusion mechanisms in TiC will be revisited, in light of the recent finding that a symmetry-broken configuration of a Ti mono-vacancy is almost twice as low in energy as the usually considered symmetric configuration. We search through the configurational space of a Ti mono-vacancy to find several local energy minima. Among them, a planar symmetry-broken configuration with the optimal number, length, and angles of C—C bonds is the global energy minimum. In spite of its superior stability, the symmetry-broken configuration does not form spontaneously upon structural relaxation, because the symmetric configuration is still a local energy minimum. The formation of symmetry-broken configurations is essential for describing metal self-diffusion in transition-metal carbides by the mono-vacancy mechanism.

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## REGULATION OF CTE OF AUSTENITIC FE-NI ALLOYS UNDER RADIATION AND THERMAL EFFECTS

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In composite structures, it is often necessary to connect materials with different values of the coefficient of thermal expansion (CTE). For these purposes, various metals and alloys with different values of CTE are usually used. Considerable interest is the possibility of a significant change in the CTE on the same alloy using heat treatment. The possibility of regulating the CTE in a certain temperature range using radiation or thermal effects was analyzed on austenitic Fe-Ni alloys of the invar range. As follows from the Fe-Ni equilibrium diagram, invar austenitic iron alloys with 31-36 wt.% Ni having low CTE values (less than  $2 \times 10^{-6} \text{ K}^{-1}$ ) can transform into low-nickel and high-nickel components with significantly higher CTE characteristics. In the present work, concentration changes in Ni were determined by the Curie temperature, the field on the  $^{57}\text{Fe}$  and electrical resistance. However, the process of such nickel redistribution at low temperatures is extremely slow (hundreds of hours). It can be accelerated by the introduction of point defects due to irradiation by high-energy particles. The formation of a high-nickel component during electron irradiation of invars was first reported by A. Chamberod et al (*Journal of Magnetism and Materials*, 1979. V. 10, No. 2-3. P. 139-144). We also showed that an increase in the fluence of 5 Mev electrons to  $5 \times 10^{18} \text{ cm}^{-2}$  at 27 °C leads to the Ni-stratification of the Fe-35Ni alloy and an increase in CTE from  $1 \times 10^{-6}$  to  $6.5 \times 10^{-6} \text{ K}^{-1}$  in the temperature range of 20-100 °C. An increase in the irradiation temperature to 150 °C leads to an even greater stratification with an increase in CTE to  $10 \times 10^{-6} \text{ K}^{-1}$ . The disadvantage of radiation exposure by electrons and ions is the small depth of penetration of particles and, consequently, small volumes of modified samples with increased values of CTE. Neutron irradiation eliminates this effect, but modified samples will be radioactive. Therefore, the regulation of CTE by heat treatment was of the greatest interest. An increase in the volume of nickel-enriched  $\gamma$ -crystals can be achieved not only as a result of accelerated diffusion, but also due to the grinding of these crystals to nanoscale sizes comparable to the diffusion paths of nickel. This is observed during the formation of nanocrystalline austenite in the process of reverse  $\alpha$ - $\gamma$  transformation under slow heating in Fe-Ni invar alloys. There is a significant redistribution of nickel between the nanocrystalline  $\gamma$ -phase and the residual  $\alpha$ -martensite in accordance with the Fe-Ni equilibrium diagram. According to the maximum increase in the field at the  $^{57}\text{Fe}$  and the Curie temperature of austenite (from 130 to 500 °C), processing conditions were found for the realization of the greatest nickel redistribution in Fe-(31-32) Ni alloys. This is observed at  $\alpha \rightarrow \gamma$  transformation during slow heating to 470-490 °C followed by a rapid increase in temperature to 600 °C. With the help of various thermal treatments of massive samples of metastable austenitic alloy Fe-31Ni, causing the redistribution of nickel in the process of  $\alpha \rightarrow \gamma$  transformation, the possibility of regulating the CTE of austenite in a wide range (from  $3.7 \times 10^{-6}$  to  $12.2 \times 10^{-6} \text{ K}^{-1}$ ) in the temperature range from -50 to 20 °C is shown

## RESEARCH OF IRRADIATED MATERIALS SCIENCE PACKAGES ELEMENTS FOR ASSESSING THE STATE OF THE PINS OF BOR-60 SMALL ROTARY PLUG

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To assess the condition of the material after prolonged operation, it is necessary to conduct studies of elements and structures that have been operated for a long time at different temperatures in the BOR-60 reactor. However, it is impossible to do without specially designed experiments on samples of various types with the maintenance of sufficiently stable temperature conditions of irradiation that determine a particular radiation phenomenon.

This paper presents some results of recent studies of radiation phenomena, such as radiation swelling, low-temperature radiation hardening and embrittlement of austenitic steels. The influence of microstructure formation features on the physico-mechanical properties of neutron-irradiated steels is also considered.

The studs and nuts of the small rotary plug (MPP) are one of the most critical non-removable elements of the BOR-60 reactor in terms of neutron fluence, affecting the safe operation of the reactor

In the course of work on the justification of the technical re-equipment of the BOR-60 reactor, an analysis of computational and experimental data on the operation of the reactor plant was carried out, which made it possible to clarify the irradiation conditions (fluence, damaging dose and irradiation temperature) of the reactor's SRP pins.

To conduct research to substantiate the condition of the SRP pins of the BOR-60 reactor, guide pipes of the materials science packages TP-03 and TP-05 were used. Samples were cut from the pipes at different heights.

The microstructure and mechanical properties of the elements of materials science packages (guide pipes), irradiated under conditions close to the operating conditions of the MPP pins of the BOR-60 reactor were investigated.

For samples from the TR-05 pipe irradiated under conditions close to the operating conditions of the MPP studs, at all test temperatures, the material has a margin of plasticity (total elongation at test temperatures of 520 and 560 C was above 2.4%) with a sufficiently high level of strength properties. Thus, it can be assumed that there is a margin for the characteristics of mechanical properties up to damaging doses of 32 displacements per atom (dpa).

The acquired data can be used in the course of strength calculations to justify the further operation of SRP pins and nuts at least up to the maximum calculated damaging dose of 29 dpa, which will be achieved on the pins of the small rotary plugs of the BOR-60 reactor in 2025.

## SWELLING IN IRRADIATED COPPER BY ELECTRONS, PROTONS AND NEUTRONS

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Irradiation produces changes in microstructure of materials for nuclear reactors causing size-instability. Swelling is one of the most dangerous phenomena lying behind this instability. Technological limit of swelling for stainless steel is only 5% at dose up to 50 dpa. Big efforts are being made to improve resistance to swelling of the materials at radiation doses up to the hundreds dpa and above. However experimental studies of swelling would be prohibitively time-consuming; therefore theoretical studies are in great demand. In theory, there are two approaches to describing swelling accumulation. One is a model within the framework of the “standard rate theory” (SRT), corresponding physically to the case of electron irradiation with energy  $\sim 1$  MeV, when the dislocation bias gives the only mechanism of swelling. However, it does not explain swelling accumulation during neutron irradiation, since defect production in the case is qualitatively different from that of electron irradiation. Therefore, another approach was developed: the so-called “production bias model” (PBM) [1, 2]. This approach takes into account that in addition to single vacancies and interstitials migrating three-dimensionally, small interstitial loops migrating one-dimensionally are also produced. In the work by B.N. Singh, it was experimentally shown that the results of irradiation with electrons and neutrons are fundamentally different [3]. A numerical analysis of experimental swelling data [3] obtained in irradiated copper with electrons, protons, and neutrons, will be reported in this presentation. Computations performed with newly developed code that takes into account both mechanisms of swelling: dislocation bias and production bias. Results obtained by the calculations are in good agreement with the experiment.

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## THE NATURE OF "STRANGE" RADIATION AT LOW TEMPERATURE NUCLEAR REACTIONS

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During explosions of titanium foils [1] with the help of electric current pulses in chambers filled with water, the appearance of chemical elements (absent in the original samples), as well as "strange" radiation (SR), was recorded. Traditional types of radiation ( $\alpha$ ,  $\beta$ ,  $\gamma$ , n) were not observed. SR was recorded with the help of photodetectors, in the emulsion of which unusual tracks appeared (not only in shape, but also in size - the width of the tracks could be on the order of 10  $\mu\text{m}$ ). It is important that SR was also observed after the explosion from the remains of the foil. According to the experimental data (the symbol \* marks our refinements), SR carriers are electrically neutral (at least before interaction with the photodetector \*). In addition, they have a magnetic moment and, before interacting with a photodetector, have dimensions of  $\leq 10^{-10}$  m (less than or on the order of the size of an atom), which allows them to leave the volume of the metal foil \*. SR carriers can be initiated by nuclear decay processes, are capable of dramatically increasing their size (by five orders of magnitude!\*) when interacting with the detector material, and have the ability to propagate parallel to flat interfaces between media with different dielectric permittivities. In [1], the observed SR features were presumably associated with the Loshak magnetic monopoles whose mass is significantly less than the Dirac monopoles. In [2], the data on SR tracks were confirmed and supplemented. The use of the conclusions of hadronic mechanics [3] allows us to propose a model of an intermediate quasimolecular state (IQS), in which the fusion of the initial nuclei is initiated by electrons contacting into compact (ee) - pairs located in a circular orbit [4]. The transition to a realistic model of IQS with massive (ee) - pairs allows low-temperature synthesis in the presence of a relatively small number of (ee) - pairs. In this case, an annular orbit with (ee) - pairs performs catalytic functions in the general case of fusion of complex nuclei, just as the participation of a muon catalyzes one of the simplest nuclear fusion reactions. In this regard, the catalyzing annular orbit with (ee) - pairs is called CA - activator. The initial state of the SR carrier (with high penetrating power) corresponds to an electrically neutral complex, which includes a CA - activator (or two CA - activators) and an "ionic" component of a pair of converging nuclei (or already merged nuclei) with distorted and incompletely filled electron shells. In the process of collision with the detector, the complex decomposes into a positively charged ionic component, which remains in the detector material, and a free CA activator, which rapidly expands due to repulsion of (ee) - pairs. The formation of SR tracks is due to the movement of precisely CA - activators.

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## TO THE QUESTION OF IMITATION EVALUATION OF THE RADIATION RESISTANCE OF REACTOR MATERIALS

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The theoretical and experimental data determining the similarities and differences between the defect formation processes under the conditions of neutron and imitation ion irradiation are analyzed. The analysis involves such factors as the type and energy of cascade-forming particles, the composition and structure of irradiated materials, and the irradiation temperature.

A number of proven procedures for calculating the concentration of radiation defects and the number of displacements per atom for cascade-forming types of irradiation are revised.

The key factors ensuring the similarity of full-scale and simulation experiments are considered, with the following two of them singled out as the principal factors: the damaging dose accumulation rate (flux) and the temperature, which determine the course of relaxation processes.

Simple methods are proposed for estimating the number of primary knocked-on atoms (PKA) under neutron irradiation and the fraction of elastic energy losses spent on defect formation. The substantiation of a unified fractal structure of atomic displacement cascades in a given target is provided, regardless of the nature and energy of the cascade-forming types of irradiation.

The results of the review are partly presented in [1, 2].

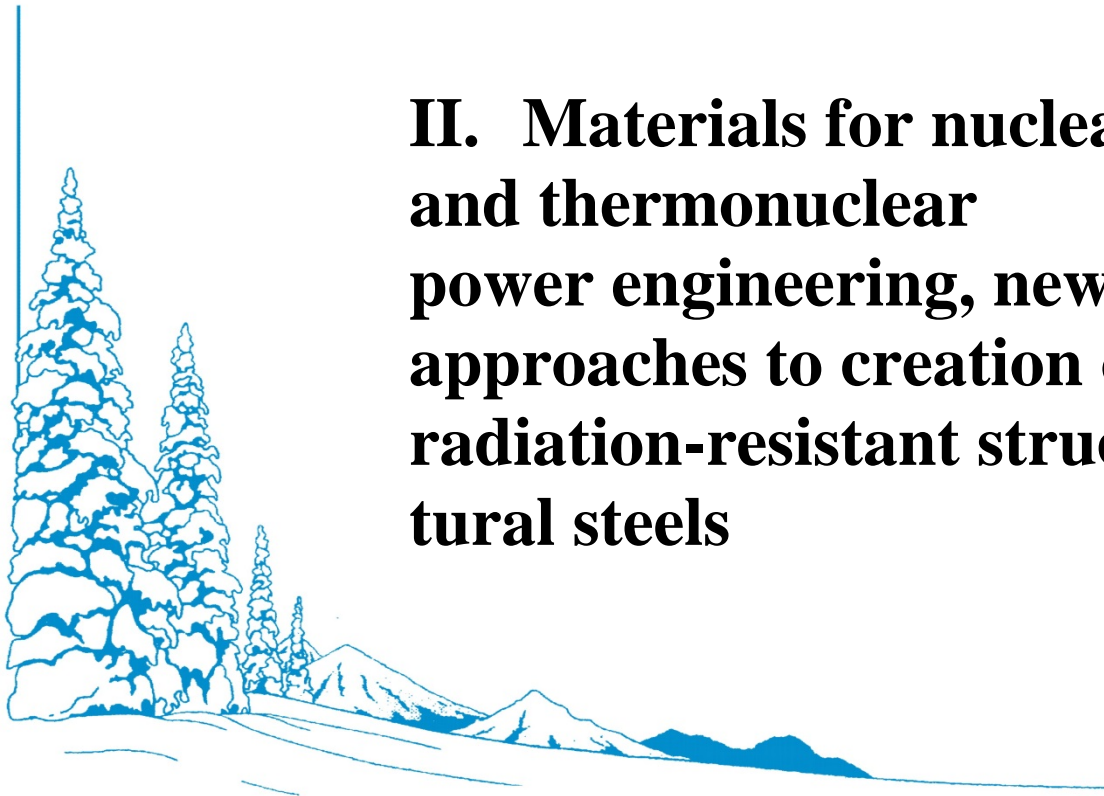
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## **II. Materials for nuclear and thermonuclear power engineering, new approaches to creation of radiation-resistant structural steels**

**The most topical problem of today is development of new metal materials for fusion and fission-type reactors. In particular, the reactors currently under construction (BN-800) and future fast-neutron reactor projects (BN-1800) still expect the constructional materials showing high radiation resistance to withstand the damaging dose of 100-130 dpa, which would ensure the required level of nuclear fuel burnup. The Section includes a great number of material-science presentations on radiation-induced changes in physical and mechanical properties of different high-pressure-vessel materials (those currently in use and showing promise). A consideration will be given to material-science problems of high-temperature creep, swelling of fcc and bcc steels, and the effect of radiation on austenitic high-pressure-vessel steels, including the only "standard" austenitic ChS-68 steel for the BN-600 reactor fuel elements. The results obtained for real high-pressure-vessel materials are analyzed proceeding from the general principles of radiation physics of solids. This Section also includes papers by Russian and foreign investigators reporting the results of studies into the effect of oxide and intermetallic aging on the structure and the mechanical properties of high-alloy constructional steels. Primary emphasis will be on the recently developed steels strengthened with heat-resistant oxides (yttrium, titanium, and thorium).**





## COMPARATIVE ANALYSIS OF THE CREEP RUPTURE STRENGTH OF RUSSIAN REACTOR STEELS WITH BCC LATTICE

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In the work, long-term high-temperature tests of Russian advanced reactor steels with a BCC lattice under the load were carried out. The characteristics of long-term strength, ductility and creep rates at 650, 670 and 700 °C and stresses of 60, 80, 100 and 140 MPa are determined.

The analysis of the data obtained allows us to arrange the studied reactor steels to increase the values of long-term strength (durability) in the following order: 5Cr-5Al-Si-Mo-Nb, 14Cr-4Al-2W-Si-Ti, (EP-823 and EP-900), EK-181, 14Cr-4Al-2W-Si-Ti-ODS, (EP-450 and ChS-139), EP-450-ODS. The reason for the higher thermal stability of "oxide-free" steels during creep testing is the higher content of carbon, carbide-forming and refractory elements.

It was shown that the oxide dispersion strengthened (ODS) stainless steel 14Cr-4Al-2W-Si-Ti-ODS significantly exceeds the matrix steel 14Cr-4Al-2W-Si-Ti in long-term strength. The structure of steel 14Cr-4Al-2W-Si-Ti-ODS represents equiaxially dislocated grains of recrystallized ferrite containing both large oxide particles with a size of 50...150 nm that have not dissolved during mechanical alloying, and hardening nanoxides (Y,Zr,Ti)-O with a predominant size of up to 3-5 nm. Hardening with nanoxides made it possible to increase the long-term strength of the steel 14Cr-4Al-2W-Si-Ti-ODS noticeably more than in the reactor steels EP-823 and EP-900, which have significantly lower corrosion resistance. It should be noted that the steel 14Cr-4Al-2W-Si-Ti-ODS does not yet have an optimal structure and is inferior in plasticity and creep resistance to the Al-free steel EP-450-ODS at 700 °C and a voltage of 100 MPa, EP-450-ODS steel samples withstand more than 28000 hours without destruction, which is 2 orders of magnitude longer than the time before the destruction of all "oxide-free" steels with a BCC lattice.

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## COMPLEMENTARY ANALYSIS OF RADIATION EFFECTS IN MATERIALS

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Prediction of radiation resistance of structural materials under irradiation is inextricably linked with understanding the microstructure evolution (the formation of radiation defects, pre-

precipitates and precipitation of phases, redistribution of impurities and alloying elements). It is well known that significant macroscopic effects, such as swelling, embrittlement, etc., are directly related to processes occurring at the atomic or nanoscale level. However, the experimental study of processes on such a scale in steels and other multicomponent materials is difficult. The study of structural rearrangements of such materials requires the use of a number of experimental techniques.

Transmission electron microscopy (TEM) is a universal method for analyzing the microstructure of materials, which makes it possible to analyze the grain structure, phase state, and various inclusions. The smallest inclusions and clusters can be detected using the small-angle neutron scattering (SANS) or atomic probe tomography (APT). SANS makes it possible to determine with high accuracy the number density of inclusions and nanoclusters, as well as to obtain their size distribution. The chemical composition and spatial distribution of these clusters can be studied in detail using atomic probe tomography. APT studies show that the composition of nanosized inclusions often differs from the composition of large stoichiometric phase inclusions.

This work presents the results of an analysis of the nanostructure of various reactor steels. The most attention is oxide dispersion hardened steels containing a high density of oxide inclusions and nanoclusters enriched in Y, O and other alloying elements. The results of a comprehensive analysis using the methods of transmission electron microscopy, atom probe tomography, small angle X-ray scattering and small-angle neutron scattering are presented.

## **CORROSION STUDIES OF NICKEL-BASED STRUCTURE MATERIALS IN FLUORIDE MELT UNDER NON-ISOTHERMAL CONDITIONS**

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The selection of fuel solvent composition and structure materials that meet the requirements and regimes of reactor operation is the key task to validate the concept of molten salt reactor for burning minor actinides. Currently, the works have been initiated in terms of creating a research reactor to refine molten salt technologies for minor actinide transmutations. In this reactor the fluoride melt of 73LiF-27BeF<sub>2</sub> (% mol.) composition is proposed to be used as the solvent of fuel additives while nickel-based alloys act as the structure materials.

In 2021, the corrosion tests were performed by RFNC-VNIITF using the samples made of chromium-molybdenum-nickel alloys CrNi80MoTi, CrNi80MoTiAl, and CrNi80MoTiAlNb in the melt of the molecular composition of 99.5(73LiF-27BeF<sub>2</sub>)-0.5(UF<sub>4</sub>+UF<sub>3</sub>) (% mol.) and with the addition of 0.1 % wt. Te. The samples were exposed in the melt circulating at the velocity of ~ 5 cm/c, maximum temperature of 690 °C, and temperature gradient along the circulation circuit of ~70 °C for 200 and 600 hours under in-situ controlled oxidation-reduction potential of the melt. During the tests, the data were obtained on the melt behavior, character and scales of corrosion damage of the structure materials under investigation.

The corrosion tests were carried out on the corrosion bench based on the thermal convection facility developed by NRC "Kurchatov Institute". During the tests, the device created by IHTE UB RAS was used to control the oxidation-reduction potential of the melt.

The work was performed according to the statement of work and under the financial support of JSC “SSC RIAR” in accordance with the Unified Industry Standard of State Corporation Rosatom in the area of Research and Development “Selection and justification of materials for closing nuclear fuel cycle in molten salt nuclear reactor using minor actinides to reprocess SNF from thermal reactors. Stage 2019-2021”.

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## **EFFECT OF HIGH-TEMPERATURE THERMOMECHANICAL TREATMENT ON REGULARITIES OF LOW-TEMPERATURE EMBRITTLEMENT OF HEAT-RESISTANT LOW-ACTIVATION FERRITIC-MARTENSITIC STEEL EK-181**

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Low-activation 9-12 % chromium ferritic-martensitic steels are currently considered as promising structural materials for cores and internals of nuclear and thermonuclear reactors. One of the main problems of ferritic-martensitic steels is the tendency to low-temperature embrittlement – the transition to a brittle state with decreasing temperature. In addition, radiation exposure can lead to a significant increase in their ductile-brittle transition temperature.

In this work, we investigated the effect of high-temperature thermomechanical treatment (HTMT) with deformation in the austenitic region on the regularities of low-temperature embrittlement of low-activation ferritic-martensitic steel EK-181 during tensile and impact tests in the temperature range from -196 to 100 °C, in comparison with its traditional heat treatment (THT: quenching + high tempering).

HTMT changes the shape and position of the stress-strain curves of EK-181 steel. It provides an increase (by  $\approx 20\%$ ), in comparison with traditional heat treatment, of the steel yield strength in the investigated temperature range while maintaining a satisfactory level of plasticity; increases the degree of its work hardening at the initial stage of deformation.

A decrease in the tensile test temperature in the range from 20 to -196 °C leads to an increase in the strength properties of EK-181 steel after both investigated treatments. A significant decrease in plasticity (up to  $\approx 2-3\%$ ) was found only at -196 °C.

Impact bending tests of Charpy-type specimens of EK-181 steel showed that HTMT provides an increase, relative to THT, its impact toughness practically at all studied temperatures (from 20 to -100 °C). The temperature of ductile-brittle transition of steel after THT is  $T_{db} \approx -7\text{ °C}$ , after HTMT  $T_{db} \approx -15\text{ °C}$ , which corresponds to the values of impact toughness  $KCV \approx 28\text{ J/cm}^2$  and  $KCV \approx 33\text{ J/cm}^2$ .

A fractographic study of specimen fractures after impact tests revealed significant differences in the type of fracture depending on the steel treatment mode. A distinctive feature of specimen fractures after HTMT, fractured in the temperature region of the ductile-brittle transition, is the formation of separations in planes parallel to the rolling plane.

Thus, high-temperature thermomechanical treatment of EK-181 steel leads to both an increase in strength properties and an increase in fracture toughness, without significantly affecting the

position of the temperature of its ductile-brittle transition. The main factors of steel strengthening after HTMT are refinement of the tempered martensitic structure as a result of plastic deformation in the austenitic region and subsequent quenching, an increase in the dislocation density, volume fraction and dispersion of nanosized vanadium carbonitride particles, which fix the defective substructure of the material. These factors, along with the separations effect of specimens in the Charpy test, have a positive effect on its toughness.

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## **EFFECT OF HYDRIDES ON MECHANICAL PROPERTIES AND FRACTURE OF IRRADIATED Zr-2.5% Nb ALLOY**

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The justification of the operability of the RBMK-1000 reactors CPS channels, which operate beyond the project service life, is one of the integral tasks for extending the service life of power units.

In accordance with the requirements of regulatory control, periodic post-reactor studies of channel sections in shielded boxes are carried out. In our works [1-4], the main results obtained from post-reactor studies of CPS channels, which are made of Zr-2.5% Nb alloy, are presented. The fact of an increase in the hydrogen content in the material of pipes and nipples of the CPS channels adapters after operation has been revealed. The increased hydrogen content is accompanied by the appearance of blister-type hydride clusters on the inner surface of local areas.

Investigations of the operating time influence, the accumulated damaging neutron fluence, the structure of the zirconium alloy and the size of the blisters on the short-term mechanical properties have been carried out. It was found that the strongest hydrogenation occurs in the areas of the CPS channel with an annealed structure. Hydrogenation leads to the formation of hydrides, which form local surface accumulations in the form of blisters. Destruction along the blisters occurs with brittle cleavage, with the formation of a lath relief, due to the destruction of hydrides along the needles. The degree of embrittlement and drop in strength properties depends on the size of the blisters and hydride chains, the structure of the zirconium alloy, and the damaging fast neutron fluence.

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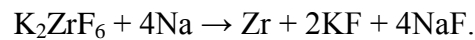
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## EFFECT OF PREPARATION OF INITIAL REAGENTS ON THE QUALITY OF SODIUM-REDUCED ZIRCONIUM POWDER

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Zirconium powder is produced by sodium reduction of a complex salt of zirconium, potassium hexafluorozirconate (PHFZ), according to the chemical equation



The process flowchart for the production of the powder includes preparation of the initial reagents, making-up of the charge mixture, reduction, leaching of the reaction mass, size grading and drying of the finished product.

The requirements imposed on the zirconium powder are based on the narrow range of values established for pyrophorosity (flash point), granulometric composition, and content of the base material and of a number of impurities.

Quality of the finished product is determined, to a large extent, by the properties of the initial reagents. For example, possible reason for rejection of zirconium powder in terms of such parameters as hydrogen mass fraction and carbon mass fraction is that storage medium of the initial sodium metal is kerosene. Kerosene, due to its high penetration capability, is deeply incorporated into the structure of the sodium metal ingot (into cracks and pores of the ingot; kerosene dissolves neither in sodium nor in sodium metal melt) and cannot be removed at the stage of the reaction charge mixture preparation.

There is a method [1] of sodium metal purification from organic components based on its remelting under the layer of liquid, sodium does not interact with, such as kerosene or petrolatum oil.

The objective of this research was to study the effect of metal sodium purification by means of remelting under the layer of petrolatum oil on the quality of the produced finely dispersed zirconium powder.

In the course of sodium melting under the oil layer, the contaminants were displaced into oil after destruction of an oxide layer on the surface of the sodium melt due to better oil wettability of solid organic contaminants present in the sodium, capability of organic contaminants (kerosene) to dissolve in oil and density difference of the sodium melt and the contaminants.

As a result of this research

– batches of zirconium powder were produced using sodium remelted preliminarily. The laboratory analysis carried out for all the produced batches revealed that hydrogen and carbon content did not exceed the established requirements;

– the effect of additional sodium purification on the properties of the produced zirconium powder was assessed;

– sequence of operations was recommended for preliminary purification of sodium metal during manufacture of the target product under industrial conditions.

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**FEATURES OF CORROSION INTERACTION OF 12 % CHROMIUM FERRITIC-MARTENSITIC STEEL EP-823 WITH A FLOWING LEAD COOLANT UNDER CONDITIONS OF A REDUCED CONCENTRATION OF DISSOLVED OXYGEN**

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In this work, using transmission and scanning electron microscopy, X-ray diffraction analysis, a detailed study of the microstructure and phase composition of the Russian 12 % Cr ferritic-martensitic steel EP-823 (Fe-12Cr-Mo-W-Si-VW-Nb) after exposure (during 2500 h) of flowing oxygen-containing lead coolant at 540 °C (in the range of operating temperatures of fuel element cladding) under conditions of deviation from the normal oxygen regime of the BREST-OD-300 reactor – at a reduced concentration of O in Pb ( $c_o = (4-8) \times 10^{-7}$  wt. %) were investigated.

Scanning electron microscopy revealed light gray “islands” with uneven edges on the surface of the samples. Elemental microanalysis showed that they are adhered residues of lead coolant. In areas free from the visible lead layer, the surface of the steel has a developed relief. The method of energy dispersive analysis showed that the content of O and some elements of steel, such as Cr, Si, Mn, etc. in this areas was increased, in comparison with the initial steel state. The Fe content, on the contrary, is reduced relative to the nominal composition of steel. This is evidence of the formation of oxide layers on the steel surface, enriched with its main alloying elements.

In the study of cross section, the presence of oxide scale was found on the surface of the samples, which heterogeneous covered the material. In some parts of the surface, its thickness is only a few tens of nanometers, in other places, centers of corrosion with a depth of  $\approx 7-10$  microns are found. The scale consists of two layers – the outer layer is enriched in iron, the inner one – in chromium and manganese. They have an increased oxygen content in comparison with the matrix. In this case, depletion in chromium occurs in the near-surface matrix layer (about 10  $\mu\text{m}$  thick) due to its intense diffusion to the interface “sample-oxygen-containing lead melt” with the formation of chromium oxides. X-ray diffraction analysis also reveals traces of a lead coolant on the surface of the samples, peaks from oxide phases corresponding to magnetite ( $\text{Fe}_3\text{O}_4$ ).

Analysis of the maps of the distribution of elements over the area and along the line near the surface of the samples cut out by a focused ion beam revealed a depth-nonuniform change in the content of such elements as O, Fe, Cr, Mn, and Si. It also confirms the presence of a two-layer oxide scale on the surface of the samples. Electron diffraction analysis showed that it consists of oxides  $\text{Cr}_2\text{O}_3$  and  $\text{Fe}_3\text{O}_4$ .

Apparently, the considered oxygen concentration in Pb at 540 °C (under flow conditions) is insufficient for the formation of continuous protective oxide layers on the surface of EP-823 steel. Thus, despite the absence of traces of intense dissolution of the steel surface, the formation of plumboferrites, or the penetration of liquid lead into the corrosive layers after 2500 hours of testing, the implemented mode of corrosion interaction can be dangerous after longer exposures.

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ISPMS SB RAS, project No. FWRW-2021-0008 and with the support of the RFBR grant No. 19-48-700020 r\_a and the Administration of the Tomsk region.

## FORMATION OF FERRITE IN THE STRUCTURE OF AUSTENITIC STAINLESS STEELS AFTER HIGH-DOSE NEUTRON IRRADIATION

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Austenitic stainless steels are widely used as the main structural material for the internals of pressurized water reactors (PWR) and sodium cooled fast reactors (SFR). Their main competitive advantages are the combination of high strength and ductility, corrosion resistance combined with a relatively low tendency to radiation swelling. Irradiation of austenitic steels with high-energy particles lead to formation of various radiation defects such as dislocation loops and voids, as well as the precipitation of secondary phases.

There were some investigations in recent decades that were reported on the radiation-induced phase transformation of fcc austenite to bcc ferrite (martensite) observed in austenitic steels. A short time ago the formation of ferrite during long-term neutron irradiation was considered as a second-order problem in the operation of a nuclear reactor. However, the planned service life extension of existing PWR from 40 to 80 years raises certain concerns that some degradation second-order effects, such as the formation of bcc phases, may become of primary importance. Radiation-induced ferrite, precipitates at the grain boundaries, reduces the resistance of the material to cracking and corrosion in the coolant water. All this negatively affects the mechanical and corrosive properties of the irradiated steel, which can lead to a limitation of the service life of the entire reactor facility.

In this presentation, we present the results of a study of specimens of 12Cr18Ni10Ti stainless steel cut from the hexagonal wrapper tubes of the fuel assemblies of the BN-350 reactor. During normal operation, the material was irradiated to a maximum damaging dose of 60 dpa at temperatures from 280 to 420°C and dose rate of 0.25-60.2 dpa/year. The presence of a magnetic bcc phase (ferrite) formed as a result of neutron irradiation was confirmed by magnetometric and X-ray diffraction analyzes. It was found by scanning and transmission electron microscopy in combination with energy dispersive X-ray spectroscopy that a new radiation-induced ferrite was formed in the material as a result of the redistribution of alloying elements during long neutron irradiation and had a complex intermetallic composition. It was shown that ferrite precipitates formed a specific structure, located along the grain boundaries and had very low corrosion resistance.

The results of simulation experiments of 12Cr18Ni10Ti steel samples after ion irradiation ( $\text{Fe}^{2+}$ , 2.3 MeV, irradiation temperature 40-450°C) are also presented, where the formation of the bcc phase was also observed.

The obtained data could be relevant for justifying the service life extension of present PWRs.

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## HYDRIDES IN Zr-2.5% Nb ALLOY AFTER PROLONGED LOW-TEMPERATURE NEUTRON IRRADIATION

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The main reason for the CPS channels of RBMK reactors, which are made of a Zr-2.5% Nb alloy, during operation is hydrogen saturation [1, 2]. Hydrogen forms hydrides, which spread over the entire thickness of the channel wall, and also leads to the formation on the inner surface of areas with a high density of hydrides - blisters [1, 2].

Post-irradiation studies of the morphology of blisters and hydrides, their phase composition in different sections of the CPS channel after 32 years of operation have been carried out.

The studies were carried out using optical and electron microscopy: metallography, X-ray structural analysis, scanning electron microscopy using SE (secondary electrons), BSE (reflected electrons), EBSD (reflected electron diffraction) detectors [3].

The effect of the structure of the zirconium alloy on the size of blisters and the morphology of hydrides is revealed, the structure and phase composition of blisters and hydride chains are estimated.

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## INFLUENCE OF RADIATION DEFECTS ON STRESS-STRAIN STATE CHARACTERISTICS OF IRRADIATED FUEL CLADDING SPECIMENS DURING MECHANICAL TESTING

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The main reasons limiting the service life of fuel elements in the core of fast neutron reactors are radiation swelling and embrittlement of the fuel cladding material. During operation, the material accumulates a large number of various radiation defects changing its physical and

mechanical properties. In particular, radiation hardening and reduction of plasticity occur. In some cases, the plasticity of the fuel cladding material can reach very low values, down to "zero". A conventional method of expanding circular specimens to assess their residual plasticity has limited applicability due to peculiarities of its test procedure. A promising method of loading circular specimens to assess the mechanical characteristics of the cladding materials with "zero" plasticity is radial compression of a circular specimen between two flat dies. For example, this method of specimen loading has been applied in assessing the residual plasticity of fuel claddings from zirconium alloys in PWR reactors under emergency conditions, namely, during high-temperature oxidation of zirconium fuel cladding due to loss of coolant (LOCA type accident), resulting in embrittlement of the fuel cladding material [1, 2].

Currently, a relevant objective is to increase the service life of fuel elements in fast neutron reactors by using advanced austenitic steel as the fuel cladding material, as well as heat-resistant ferritic-martensitic steel grades. To validate the operability of the candidate materials, examination of fuel elements is being conducted after trial operation or irradiation of their claddings in the BN-600 material science assemblies to large doses (up to 140 dpa), including radial compression mechanical tests.

The paper presents some experimental results of the fuel cladding materials examination after irradiation in the BN-600 reactor, obtained through the comprehensive procedure of circular specimens mechanical testing that combines ovalization of circular specimens under radial compression with their subsequent expansion. At the same time, the stress-strain state of circular specimens during testing has been analysed, taking into account changes in their stress-strain state characteristics (in particular, the level of damage) caused by formation of radiation defects.

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## **INVESTIGATION OF THE FORMATION PROCESS OF SOLID SOLUTIONS IN A TWO-COMPONENT Rh-Cu SYSTEM BY VACUUM-THERMAL EVAPORATION**

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The UN Climate Group presented a report on global warming and Earth's climate change in August 2021. The climate is undergoing accelerated change due to significant CO<sub>2</sub> emissions and environmental pollution from the use of fossil fuels by industry and humans. It is expected that the work on reducing CO<sub>2</sub> emissions by the countries participating in the Paris Agreement (2020) will contribute to the additional development of the global nuclear industry, which is considered one of the "cleanest" in terms of CO<sub>2</sub> emissions per capita. The development of the industry will contribute to the creation of new types of reactors, improvement of technological processes and motivation to search for new radiation-resistant structural materials that can improve energy efficiency and safety at all stages of the nuclear fuel cycle – from obtaining

nuclear fuel to storing radioactive waste.

One of the most chemically resistant materials is rhodium. This metal has a unique set of properties: high corrosion resistance and melting point, good catalytic and mechanical properties, etc. Due to this, Rh and alloys based on it have found wide application in such areas as the automotive, glass and chemical industries. In nuclear reactors, rhodium is used in thermocouples and as emitters for detecting neutron flux sensors [1]. The large value of the neutron-capture cross section for Rh gives a high sensitivity of such sensors, but leads to a faster burnout of the emitters. It is worth noting that the high cost of Rh also limits its applications in nuclear power. The use of Rh-based alloys can be a potential solution to the problem. For example, plastic and cheaper copper, which has a low activation barrier to diffusion, can increase the efficiency of healing radiation defects. Rh and Cu form a continuous series of solid solutions upon mixing, which excludes the formation of a second phase, thereby allowing one to expect a high stability of the crystal lattice. Thus, the presence of Cu is capable of endowing the alloy with the necessary mechanical properties to increase the radiation resistance.

This paper presents an approach to the formation of thin-film coatings of the  $Rh_xCu_{1-x}$  alloy. Rh-Cu nanoparticles with different Rh:Cu ratios (25:75, 50:50, 75:25 at.%) were formed by vacuum-thermal evaporation of Cu and Rh and vacuum annealing at 350 °C. The structure and composition of nanoparticles were studied by transmission and high-resolution electron microscopy (JEOL JEM-2100 Plus).

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## MÖSSBAUER SPECTROSCOPY INVESTIGATION OF STRUCTURAL PHASE TRANSITIONS IN THE EP823 STEEL UNDER NEUTRON IRRADIATION IN THE BN600 REACTOR

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The study of radiation-induced structural-phase transitions in EP823 industrial steel under high-dose irradiation in a BN-600 fast neutron reactor is carried out using the methods of transmission Mossbauer spectroscopy with resonance detection. It is shown that in ferritic-martensitic steel, after irradiation with doses above 50 dpa at temperatures of 570-660 °C, decomposition of a solid solution with a BCC structure occurs with the release of doping elements (chromium, molybdenum and carbon) from the metal matrix. This leads to a decrease in the effective chromium concentration in the ferritic matrix and the formation of an intermetallic  $\chi$ -phase and carbides of the  $Me_{23}C_6$  type. It is established that the degree of chromium escape from steel metal matrix and the volume of formed intermetallic  $\chi$ -phase are determined by the dose of irradiation.

A comparison of results of an irradiation in a fast-neutron reactor and those of long-term

thermal annealing of the EP823 steel testifies to the realization of considerably more intense radiation-induced processes of solid solution decomposition.

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## SILVER TRANSPORT THROUGH SiC LAYERS OF TRISO FUEL PARTICLES

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Gas-cooled reactors are considered not only for electricity generation but also for the supply of process heat and steam and the management of CO<sub>2</sub> induced climate change. The tristructural-isotropic (TRISO) fuel particle is the heart of a number of past and current high temperature gas-cooled reactors (HTGRs) and consists of a fissionable kernel which is coated with various layers in a fluidized bed CVD reactor. A key design feature of these particles is the ability to contain all radioactive fission products (FPs) inside the fuel structure to avoid contamination of the coolant. It has been reported by [1] that certain metallic fission products such as <sup>110m</sup>Ag are released by seemingly intact particles at high temperatures. The exact mechanism of Ag release from TRISO fuel particles has eluded researchers over the past thirty years and has largely been attributed to grain boundary diffusion leading to a requirement of small crystal size in the SiC coatings. Previous work [1-3] has shown that Ag alone is not mobile in SiC even along grain boundaries, while [4] proposed that Ag migration in SiC is only possible in the presence of Pd or similar fission products.

In this work, various out of reactor experiments were performed in order to investigate the Pd assisted transport mechanism of Ag in SiC. Results were compared to recently irradiated TRISO particles from the AGR1 experiment. For out of reactor experiments, both 3C and 6H SiC from NovaSiC (France), 3C SiC from Rohm and Haas (USA), as well as surrogate TRISO particles with ZrC kernels (PBMR South Africa) were subjected to various heat treatments in the presence of Pd and Ag. Specimens were then examined by SEM using a JEOL 7001F equipped with an Oxford instruments EDS detector as well as TEM using a JEOL 2100 and ARM 200F both equipped with Oxford Instruments EDS detectors.

Results showed clear evidence of Pd induces SiC decomposition with subsequent epitaxial reformation. Ag dissolves into liberated Si forming mobile nodules that migrate along Pd corroded paths. At the same time C is transported through the nodule and new SiC is epitaxially precipitated at the back of the nodule.

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## STRUCTURAL - PHASE STATE OF AUSTENITIC STEELS WITH DIFFERENT NICKEL CONTENT UNDER NEUTRON IRRADIATION

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Austenitic corrosion-resistant steels are the main material used for the manufacture of core elements of VVER type reactors. During operation, the core elements are exposed to significant neutron irradiation, and therefore, the study of the interrelationships of radiation-induced changes in the structure and mechanical properties of austenitic steels during operation play an important role in justifying the design life of the core elements and the choice of materials for them. The degradation of the structure of austenitic steels under the action of neutron irradiation largely depends on the concentration of nickel.

Nickel, along with its influence on the swelling of steels, is also the main element of radiation-induced phases formed in austenitic steels: G- and  $\gamma'$ -phases. In this regard, a change in the nickel concentration in steel can lead to significant changes in the density and volume fraction of the phases formed during irradiation and, as a result, has a significant effect on the mechanical properties of the material.

In the work, complex microstructural studies were carried out using high-resolution transmission and scanning electron microscopy, as well as atomic probe tomography, it was shown that, other things being equal, an increase in nickel concentration leads to:

- a decrease in the tendency to form radiation-stimulated titanium carbides under irradiation and a significant increase in the density and volume fraction of the radiation-induced G-phase;

- a significant increase in the level of radiation-induced nickel segregation with a slight decrease in chromium along the grain boundaries and radiation defects with the formation of G-phase release chains for these defects;

- the dependence of swelling on the Ni content in the studied samples is complex. However, there is a tendency to decrease the level of swelling with its heterogeneous distribution.

## STRUCTURAL AND PHASE STATE OF CERAMICS BASED ON SILICON CARBIDE IRRADIATED WITH LOW-ENERGY KRYPTON IONS

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Due to its excellent high-temperature strength, high thermal conductivity, chemical inertia and small neutron capture cross-section, silicon carbide (SiC) is suitable for use as a structural element in thermonuclear reactors or as a sealing material for nuclear fuel in light water, fission reactors and gas-cooled fission reactors, as well as in radioactive nuclear waste burial sites.

Ceramic SiC samples were obtained at IHMT NAS RB by binding two fractions of SiC powders M5 and M50 (grain size 5 microns and 50 microns, respectively) using a thermoplastic binder based on paraffin P-2 [1].

The samples were irradiated with low-energy krypton ions (280 keV) at the DC-60 heavy ion accelerator (Institute of Nuclear Physics, Nur-Sultan, Kazakhstan). Irradiation with krypton ions was carried out with fluencies  $1 \cdot 10^{13}$ ,  $1 \cdot 10^{14}$ ,  $5 \cdot 10^{15}$  cm<sup>-2</sup>. The structural and phase state of the initial and irradiated silicon carbide samples was studied by X-ray diffraction analysis (XRD) and Raman scattering.

According to the XRD data, we can conclude, that the initial samples are a multiphase system: SiC-6H – hexagonal (P63mc) crystal system, Si – cubic (Fd-3m) crystal system and SiC-15R – trigonal (R3m) crystal system. The main phase is SiC-6H (about 80%), the content of the SiC-15R phase is about 20%, Si is less than 5%.

Four peaks of the first order of Si-C bonds of oscillations (700-1000 cm<sup>-1</sup>) corresponding to the optical modes E<sub>2</sub> (TO), E<sub>2</sub> (TO), E<sub>1</sub> (TO) and A<sub>1</sub> (LO) are observed on the Raman spectrum. During an increase in the irradiation doses from  $1 \cdot 10^{13}$  to  $1 \cdot 10^{14}$  cm<sup>-2</sup>, a decrease in the intensity and broadening of the peaks is observed, due to the disordering of the crystal structure, the formation and accumulation of radiation defects in SiC. At a dose of  $5 \cdot 10^{15}$  cm<sup>-2</sup>, there are no peaks of the first order of oscillations, so we can conclude the amorphization of the SiC surface layer, which is also confirmed by electron microscopic studies.

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## **STRUCTURAL AND PHASE STATES OF EQUIATOMIC ALLOYS BASED ON V-Ti-Nb-Ta IRRADIATED WITH HELIUM IONS**

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One of the promising areas of research in modern materials science is the study of properties and methods for producing high-entropy alloys [1]. It is believed that maximizing the configuration entropy of high-entropy alloys promotes the formation of a single-phase disordered solid solution instead of the formation of complex intermetallic or second phases, as a result, the alloy has a simple microstructure with improved properties compared to traditional alloys. Numerous studies have shown that high-entropy alloys have a high elastic limit, wear resistance, creep resistance, thermal resistance and radiation resistance [2].

Multicomponent solid solutions based on V-Ti-Nb-Ta were synthesized using high-purity metals (>99.9%) by arc melting followed by homogenization. Then annealing was carried out for 24h and 72h at a temperature of 1150°C with cold rolling up to 85 % reduction in thickness.

The X-ray diffraction analysis showed that, regardless of the number of elements in the alloy, a single-phase solid solution with a BCC lattice is formed in all initial samples. The lattice

parameter for samples V, Nb, VNbTa, VNbTaTi was 0.3027 nm, 0.3177 nm, 0.3227 nm, 0.3234 nm, respectively. The distortion values decrease with an increase in the number of elements and amounted to 4.48%, 4.17%, 3.80%. The size of the coherent scattering regions and the magnitude of the micro deformation calculated by the Holder-Wagner method were 21-23 nm, 0.15-0.19%; 16-29 nm, 0.38-0.54%; 20-34 nm, 0.58-0.68%; 14-17 nm, 0.22-0.33%. Stress relaxation in the VNbTiTa alloy is mainly related to the smaller size of atoms and the difference in melting temperatures of Ti compared to Nb and Ta. The results of scanning electron microscopy confirm the formation of homogeneous equiatomic multicomponent solid solutions, the grain size in the VNbTiTa alloy was 100-200 nm.

When irradiated with helium ions with an energy of 40 keV and a fluence of  $2 \times 10^{15} \text{ cm}^{-2}$ , the uniformity of the distribution of elements and the phase composition of a multi-component solid solution based on V-Nb-Ti-Ta did not change, which is confirmed by the results of scanning electron microscopy and X-ray diffraction analysis. The values of macro stresses for V, Nb, V Nb Ta and Nb Ta Ti were 0.162 GPa, 0.392 GPa, 1.80 GPa, 2.42 GPa, respectively.

The work discusses the mechanisms of radiation resistance of the V-Nb-Ti-Ta system.

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## STUDY OF NANOSTRUCTURE CHANGES IN 9-13% Cr ODS STEELS ALLOYED WITH V, Ti, AND Al UNDER Fe ION IRRADIATION

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Creation of new fission and fusion reactors largely depends on the development of core materials, which have to satisfy high requirements for radiation and heat resistance. The requirements for new structural materials are close to technological limits of their manufacture, and the efforts of the developers of such materials are aimed at further optimization of elemental compositions (including further minimization of impurity elements), microstructures, structural-phase states, modes of thermomechanical processing of materials and structural elements for innovative nuclear reactors. Operational characteristics of new materials should be considerably better in comparison with the existing ones. Radiation resistance is expected to be up to 200 dpa, mechanical properties must be stable at high temperatures ( $> 700^\circ\text{C}$ ), and new materials also have to be corrosion resistant in coolant. Prospective candidates to meet these requirements are oxide dispersion strengthened (ODS) ferritic-martensitic steels. Mechanical properties of ODS steels significantly depend on the nanostructure of the material: size and spatial distribution of dispersed inclusions (oxide particles and clusters). It is known that Ti, V and Zr affect the formation of nanoscale particles by reducing their size and increasing the number density. In this context, it is important to study ODS steels with different alloying systems. This approach allows a deeper level of understanding the processes of nanostructure formation of ODS materials, depending on the initial composition. Moreover, study of inclusion stability behavior under irradiation, including a variety of simulated impacts, such as heavy ion irradiation is important

issue, because of ODS steels application in nuclear reactors.

In this work a complex study of initial state of ODS steels with different alloying systems (Zr, Al, V and Ti) was carried out by transmission electron microscopy (TEM) and atom probe tomography (APT). In addition, TEM and APT study of some ODS steels were carried out after Fe ions irradiation up to 30 dpa at 350°C.

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## STUDY OF THE EFFECT OF Fe ION IRRADIATION ON THE NANOSTRUCTURE OF 10Cr ODS OXIDE DISPERSION-STRENGTHENED STEEL THROUGH ULTRAMICROSCOPY METHODS

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Oxide dispersion-strengthened steels are potential core materials for the next generation of fast-neutron reactors. They are expected to withstand radiation loads up to 200 dpa at high temperatures of 400-700 °C. Such a load can be withstood by a material whose structure includes oxide particles. It is known that the presence of a large number of small oxide particles in a material leads to improved mechanical characteristics of ODS steels.

To analyze the evolution of the distribution of oxide inclusions in the material during irradiation, simulation experiments were carried out on the 10Cr ODS steel samples with 5.6 MeV Fe<sup>2+</sup> ions to doses of 3, 6 and 30 dpa at 350 °C. The study was carried out using modern methods of ultramicroscopy: transmission electron microscopy (TEM) and atomic probe tomography (APT).

With the use of APT a dependence of changes in size and density of Ti-Y-O-V-Cr clusters on irradiation dose was observed. The average cluster diameter decreased from (4±1) nm in the initial state to (2±1) nm at irradiation up to the maximum dose of 30 dpa. At the same time, the cluster density decreased from (9±1 x 10<sup>22</sup> m<sup>-3</sup>) to (4±2 x 10<sup>22</sup> m<sup>-3</sup>). By TEM method, oxide particles with Y<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub> or Y<sub>2</sub>TiO<sub>5</sub> stoichiometry were detected. The average size of oxide particles did not actually change during irradiation, but the density of particles decreased from (13±2 x 10<sup>22</sup> m<sup>-3</sup>) to (4±1 x 10<sup>22</sup> m<sup>-3</sup>) when the maximum dose of 30 dpa was reached.

It was found that ion irradiation causes partial dissolution of clusters and movement of elements into the matrix. Analysis of cluster chemical composition showed a sharp decrease of Cr and V amount with the increase of the irradiation dose, while the concentration of Ti, Y, O remains within error range, which indicates the stability of the Ti-Y-O type clusters.



**SURFACE EROSION IN MULTILAYER nc-ZrN/a-Si<sub>3</sub>N<sub>4</sub> AND nc-ZrN/a-ZrCu FILMS IRRADIATED WITH HELIUM IONS**

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Currently, the development of new radiation-resistant materials is an important problem, which is especially relevant for the nuclear/thermonuclear industry, aerospace industry, etc., where objects are exposed to strong irradiation by ions, neutrons, electrons. For this, it is necessary to create materials with a large amount of sinks for point defects, such as dislocations, grain boundaries, and interphase boundaries [1]. Multilayer systems are promising for research, since interlayer boundaries can affect the elimination of radiation defects [2]. They have significant interfacial regions that can act as stable defect absorbers.

Multilayer films nc-ZrN/a-Si<sub>3</sub>N<sub>4</sub> and nc-ZrN/a-ZrCu were formed at 500°C by reactive magnetron sputtering in a high vacuum chamber (base pressure <math>10^{-5}</math> Pa) equipped with three confocal targets and a cryogenic pump (max. 500 l/s). Ion implantation of multilayer films was carried out with He<sup>2+</sup> ions with energy of 40 keV on a DC-60 heavy ion accelerator at fluences from  $3.0 \times 10^{17}$  to  $1.1 \times 10^{18}$  cm<sup>-2</sup>.

Transmission electron microscopy studies of multilayer films after deposition have shown that the films consist of alternating layers of nanocrystalline ZrN and amorphous a-Si<sub>3</sub>N<sub>4</sub> or a-ZrCu. The formation of horizontal continuous layers with flat and sharp interfaces was revealed.

Microscopic (SEM, AFM) studies of multilayer films irradiated with He ions showed that the radiation erosion of the surface in nanolayer ZrN/a-Si<sub>3</sub>N<sub>4</sub> systems is significantly lower than in ZrN mononitride. It was found that the degree of radiation erosion of multilayer films strongly depends on the ratio of the thicknesses of the crystalline and amorphous layers ( $f_{\text{ZrN}}$ ) and is minimal at  $f_{\text{ZrN}}$  equal to 0.29. The most resistant to radiation erosion are multilayer systems with an amorphous layer thicker than that of a crystalline layer.

It was revealed that the main mechanism of radiation erosion of multilayer nc-ZrN/a-ZrCu films is flaking. The helium-vacancy complexes and their clusters diffuse into the amorphous layers and dissolve in them with the formation of helium bubbles. This results in swelling of the multilayer film without surface erosion. With an increase in the concentration of copper and an increase in the thickness of the amorphous layer, the critical dose of flaking increases. The phase composition showed that irradiation leads to destruction of the amorphous layer with the formation of metallic copper. For nc-ZrN/a-Zr<sub>26.2</sub>Cu<sub>73.8</sub> samples, copper crystallization occurs at a dose of  $9 \times 10^{17}$  cm<sup>-2</sup>. The rest remain stable at all irradiation fluences.

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## THE EFFECT OF NEUTRON IRRADIATION ON THE PHYSICOMECHANICAL PROPERTIES OF REACTOR STEELS

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Austenitic steels are used as structural materials in almost all power nuclear reactors. The problem of operating fast neutron installations is the high level of radiation damage of structural materials, in particular, fuel cladding materials.

In metals and alloys exposed to neutron irradiation, an increased concentration of point defects (vacancies and interstitials) is established. The prolonged effect of neutron irradiation leads to the evolution of this system, which manifests itself in the redistribution of elements (diffusion, formation of depleted zones, phase transformations, creep under irradiation), changes in chemical composition due to transmutation of elements (nuclear transformations due to neutron capture) and conglomeration of vacancies (clusters, gas-filled bubbles, vacancy pores).

With an increase in the duration of irradiation for austenitic steels, the problem of swelling comes to the fore, which leads to a sharp decrease in physical and mechanical properties (loss of strength and plasticity, shape change).

Currently, an urgent task is to increase the service life of fuel elements in fast neutron reactors, to solve which it is necessary to identify the effect of radiation-induced changes in the microstructure on the physical and mechanical properties of reactor steels, such as Young's modulus, shear modulus and Poisson's ratio.

The paper presents experimental results of the study of fuel cladding materials after irradiation in the BN-600 reactor, including those obtained by determining the elasticity characteristics by ultrasonic dynamic resonance methods. The influence of swelling and other microstructural changes of the studied samples on the elasticity characteristics of reactor steels is analyzed. It is shown that for austenitic steel cladding, swelling has a dominant effect on the change in physical and mechanical properties.

## THE USE OF THE FINAL STAGE UNSTEADY SWELLING MODEL TO PREDICT THE SWELLING OF AUSTENITIC STEEL FUEL ROD CLADDINGS

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Fuel cladding swelling after operation in sodium-cooled fast reactors is one of the main negative factors limiting fuel burnup of fuel assemblies. Fuel element shells, particularly those made of austenitic steel, under the influence of neutron irradiation become damaged in the crystal lattice, which leads to the appearance of an excessive number of point defects (PD), which leads to radiation swelling [1]. Traditionally, when describing the process of radiation swelling, three stages are distinguished. Incubation stage, non-stationary stage and stationary

stage. The peculiarity of the final stage of nonstationary growth consists in the fact that due to the shift of the value of critical diameter towards larger sizes pore germs cannot pass into the class of pores and their number in the formed ensemble does not increase, but only decreases due to coalescence. Pore growth leads to an increase in the integral area of their surface, and coalescence leads to a decrease in the number of pores. As long as pore growth has a stronger effect on the integral area than coalescence, the rate of swelling will increase. When both factors are equalized, the swelling rate will become constant [2].

The model of the final stage of the unsteady swelling stage [3] of austenitic steel fuel rod claddings includes changes in the following porosity characteristics: average pore diameter and concentration, porosity and damage dose. Each change in the porosity characteristics, in turn, affects the quasi-stationary value of the PD concentration in the matrix, which is based on the model of PD migration presented in [4]. In turn, changes in the concentration of PD in the matrix and their distribution on the effluents affect the fluxes of PD into and out of the effluents, including the flux of PD into the pores.

Experimental data to be used as initial values in the calculation model were obtained on samples of austenitic steel fuel rod cladding that were operated in the BN-600 reactor. The material structure, pore size distribution and pore concentration were analyzed using SEM and TEM methods. From the obtained data the characteristics of porosity were calculated and used as initial parameters for calculations.

To avoid a difficult calculation procedure in [3], the model used semi-empirical dependencies of pore diameter change on integral pore area as an approximation, which negatively affected the accuracy of the results.

The purpose of this work is to calculate, using software tools, the whole cycle of changes in the material, both the porosity characteristics and the quasi-stationary concentrations of point defects in the matrix and in the drains (dislocations, grain boundaries, twin boundaries, pores). On this basis, the time dependence of swelling at the final stage of the nonstationary swelling stage is calculated for the studied materials. The calculations performed to predict the residual service life of fuel assemblies are used to justify the safety of extending pilot operation.

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# THEORETICAL STUDY THE EFFECT OF RESIDUAL GASES ON SWELLING ACCUMULATION IN STRUCTURE REACTOR MATERIALS AT HIGH NEUTRON DOSES

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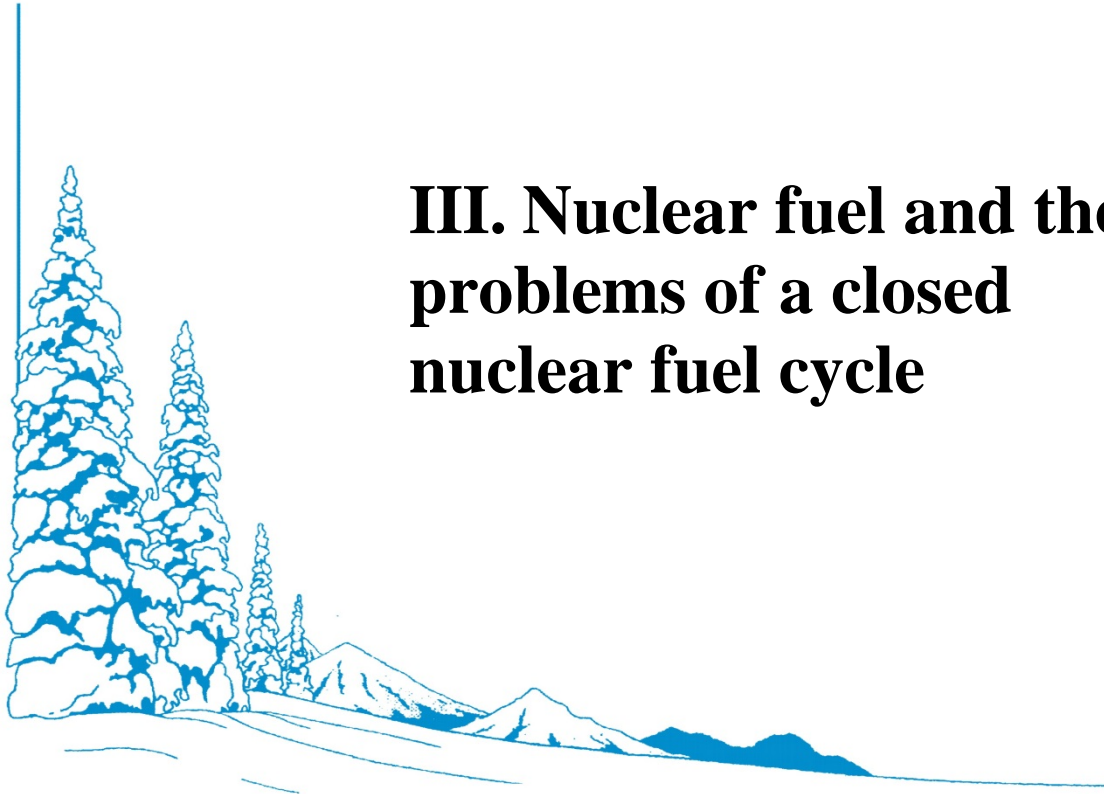
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The effect of residual gases on void nucleation has already been reported in the literature, see e.g. [1-3], although it has never been considered as a way for increasing swelling resistance. However, there is a common belief that, e.g., in austenitic stainless steels, He atoms from  $^{59}\text{Ni}$  ( $n, \alpha$ ) reactions are mainly responsible for stabilization of void nucleus, so He accumulation soon or later will provide high void nucleation regardless of a residual gas concentration. However, such a view is in contradiction with the observations that the terminal void density in the case of neutron irradiation is established at doses of the order  $\sim 1$  dpa, or even less, i.e., when the concentration of He may be rather small. Hence, residual gas may play a certain role in void nucleation hence may also affect swelling accumulation as well. However, experimental study of the effect of residual gas on swelling accumulation at practically important doses, i.e., of the order of tens of dpa, is not possible since it would require an extremely large time. Fortunately, it could be done via calculations by using code RIME (Radiation-Induced Macrostructure Evolution) [4], which is based on Production Bias Model (PBM) [5,6]. Calculation results on swelling accumulation in 316 stainless steels irradiated with neutrons to doses 150 dpa in temperature interval 400-650°C at different residual gas concentrations are discussed.

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### **III. Nuclear fuel and the problems of a closed nuclear fuel cycle**

**The universal processes for the reprocessing of spent nuclear fuel of various types are hydrometallurgical and gas fluoride ones, which make it possible to obtain refabricated fuel with a high purification rate from fission products. For promising fuels (metal, nitride, carbide), alternative pyrochemical processes for the processing of spent nuclear fuel in molten salts have been developed: electrochemical (electrolysis, electrorefining), reductive extraction, deposition of oxides, and others.**

**The field of application of pyrochemical methods is the reprocessing of spent nuclear fuel with a short holding time, when high cleaning coefficients of refurbished fuel from PD are not required. These developments have not yet emerged from the stage of pilot plants, however, interest in their industrial implementation is still high due to the urgency of creating large-scale atomic energy with fast neutron reactors.**



## **BEHAVIOR OF MOLYBDENUM ELECTRODES OF ELECTRIC FURNACES EP AT OPERATING TEMPERATURES IN AIR ATMOSPHERE**

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The Mayak PA has been reprocessing liquid high-level waste in industrial electric furnaces of direct electric heating since 1987. The vitrification process allows converting liquid HLW to solid state, i.e. aluminophosphate glass, for long-term safe storage and subsequent final disposal. During the above-mentioned period five furnaces had been in operation. Currently all of them have been brought into a discontinued load mode.

The design of the vitrification furnaces was based on invariable concept and specific design solutions. One of such specific solutions was rejection of the use of an overflow area and a water-cooled baffle at one of the electric furnaces to ensure maximum complete emptying of the electric furnace at completion of its operation. As a consequence, there was a risk of discharge of large amount of finished and/or not completely finished glass product from the entire space of the electric furnace in case of failures in operation of the discharge unit. Besides, at significant lowering of the melt level, inevitable exposure of a part of the surface of molybdenum electrodes applied for the melt heating was observed.

The electrode was designed as a molybdenum cylinder connected with a current lead. Several such electrodes make an electrode combs. The electrode combs, in their turn, make comb electrode pairs, between which electric current flows through the melt. In case of electrode exposure molybdenum starts interacting with oxygen, which in the course of time results in decrease of effective area of the electrode and, consequently, in redistribution of the electric field.

The objective of this research was to study variation of the weight of molybdenum pellets due to oxidation with atmospheric oxygen as a function of temperature to determine its reduction rate per unit of time in relation to the surface area of the specimens. This research used molybdenum pellets as electrode simulators.

Preliminary thermal analysis of the molybdenum specimens was carried out using thermal analyzer STA 449 F3 Jupiter to determine temperature of peak rate of molybdenum oxidation that was 770 °C.

The molybdenum pellets in crucibles were placed in the laboratory electric furnace. The temperature range was from 550 °C to 1,000 °C. Duration of testing was 6 hours. The specimen exposed to a temperature of 1,000 °C, was tested for 5 hours. Besides, half of the pellet was covered with glass.

The research determined the rate of the specimen weight loss. The calculations carried out on the basis of the obtained data demonstrated that the service life of new molybdenum electrodes in an industrial electric furnace in case of decreased melt level would not exceed 130 hours.

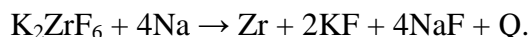
The research findings suggest that accelerated molybdenum oxidation starts at a temperature of more than 700 °C. Maximum oxidation intensity is observed at a temperature of 760 °C. In the industrial electric furnace, molybdenum oxidation due to the melt level decline starts at a higher temperature and will run faster than in the laboratory experiments as a result of molybdenum self-heating and the effect of corrosive environment. In practice, this will inevitably result in fast loss of practically the entire part of the electrodes protruding from the melt.



## EFFECT OF REDUCTION REACTION CONDITIONS ON THE QUALITY OF FINELY DISPERSED ZIRCONIUM POWDER

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Sodium-reduced zirconium powder is produced by reduction of a complex salt of zirconium, potassium hexafluorozirconate (PHFZ), with sodium metal. The reaction is described with the equation



The reaction takes place at layer-by-layer loading of PHFZ and pieces of sodium metal. As the released heat (1,100 kJ per a kilogram of charge mixture [1]) is insufficient to ensure full progress of the reaction, the charge mixture is subject to additional heating. The reaction is carried out in controlled nitrogen-oxygen atmosphere. The reduction progress is determined on the basis of readings of the thermocouple, the tip of which is placed into the reaction mass (RM). When the RM temperature variation does not exceed 10 °C during 1 min, the reduction reaction is considered to be complete.

It is known [2, 3] that the properties of the produced powder (mass fraction of the basic material, ignition temperature and granulometric composition), may be affected, among other things, by the presence of comparatively small quantities of inert (fluxing) additive in the charge mixture.

The objective of this research was to study:

- the effect of the fluxing agent of sodium chloride (NaCl) taken in a weight ratio of 1 : 0.05 as related to PHFZ;
- the time of additional high-temperature treatment of the RM upon completion of the reduction reaction.

The use of the fluxing agent NaCl:

- increased the duration of the reduction reaction, which may be related to partial consumption of thermal energy for NaCl melting;
- resulted in decrease in mass fraction of fine fraction and increase in the fraction of coarse fraction as compared with zirconium powder charges produced without adding NaCl into PHFZ. Increase in dispersivity of the finished zirconium powder can be attributed to longer exposure of the RM to higher temperature (due to longer duration of the reaction) and the resulting particle aggregation.

Increase in the reaction duration resulted in:

- decrease in the mass fraction of total zirconium, which can be explained by longer contact of the reaction mass with the nitrogen-oxygen mixture;
- decrease in ignition temperature (in spite of reduction of dispersivity and mass fraction of zirconium in powder), which may be attributed to dissolution of oxide film generated on the surface of the powder particles, in the bulk of the metal.

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## HANDLING OF POTENTIALLY PYROPHORIC MATERIALS DURING SEGMENTATION AND DISSOLUTION OF URANIUM-ZIRCONIUM SPENT NUCLEAR FUEL

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Radiochemical facilities are potentially dangerous because of fire or explosion hazards. Therefore, issues related to ensuring safe operation of such facilities are the main priority.

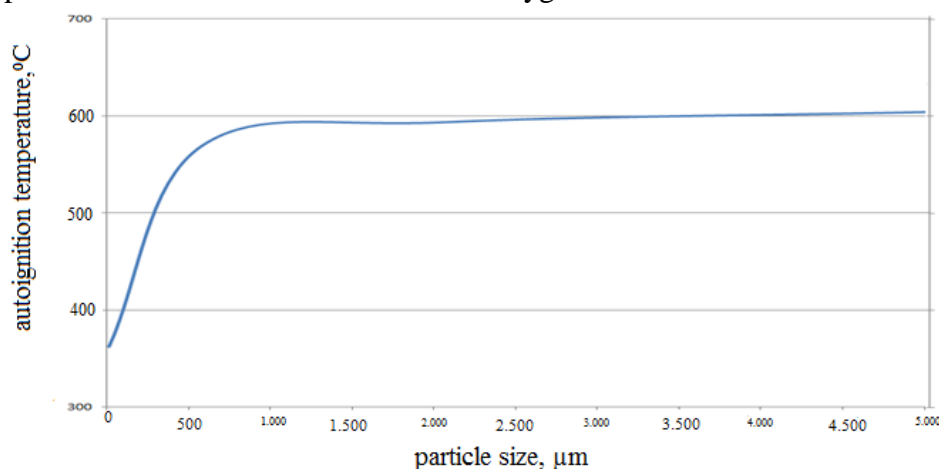
Nowadays, the RT-1 Plant carries out chemical dissolution of uranium-zirconium SNF with the help of fuel segmentation. Segmentation of SFAs that contain pyrophoric materials with a uranium-zirconium-alloy-based fuel composition is carried out using a nitrogen atmosphere with an oxygen volume concentration of not more than 2.5% in accordance with the available statements of safety. The shielding atmosphere serves to prevent ignition and spread of flames via uranium-zirconium fuel particles in the working volume of the cutting unit and the dissolver prior to the fuel loading completion. Maintenance of the required volume concentration of oxygen in the inert atmosphere is always rather expensive.

The work was aimed at assessing routine process conditions, optimizing technological operations and ensuring fire and explosion safety during segmentation and dissolution of uranium-zirconium SNF, while regrading this fuel as potentially pyrophoric.

The paper studies the dependence of autoignition temperature of the uranium-zirconium alloy on particle sizes in a nitrogen atmosphere with a volume concentration of oxygen of not more than 4%, determines the overall gas release rate and the hydrogen release rate in the presence of fluoride ion that is a complexing agent.

As for experimental part of the research, the experiment was carried out using the STA 449 F3 Jupiter synchronous thermal analyzer, whereas sample geometry was identified with the help of the Olympus SZX16 microscope. In addition to that, the overall gas release rate, as well as the release rate of hydrogen that is an explosive gas was found under the most adverse conditions, i.e. at dissolution of the uranium-zirconium alloy in the presence of fluoride ion that is a complexing agent.

Experimental dependence of autoignition temperature on particle sizes in a nitrogen atmosphere with a volume concentration of oxygen of not more than 4% was determined.



The obtained experimental data demonstrate that methods that are currently used at the Mayak PA for assessing fire safety parameters allow evaluating safety of the procedure established for

implementation of the technological process, optimizing conditions for carrying out process operations and minimizing explosion and fire hazards.

## STUDY OF CORROSION EFFECT OF BOROSILICATE GLASS MELTS ON XH45IO AND XH70IO ALLOYS

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The vitrification method solidifies liquid HLW in glass to ensure their safe long-term storage. Nowadays, there are industrial-scale HLW vitrification facilities operating in Russia, the United States, France, Great Britain, Germany and Japan. Vitrification is carried out there using two technologies, i.e. furnaces of direct electric heating and induction furnaces. The first technology is a more widespread, well-proven and efficient option.

Commissioning of a new Mayak PA vitrification complex that will be versatile in terms of composition of LRW to be solidified is tentatively scheduled for 2027. According to the developed concept, the new vitrification complex is planned to have two removable melters of direct electric heating for aluminophosphate glass and a removable small-scale melter of direct electric heating for borosilicate glass [1].

Borosilicate glass is characterized by a number of advantages: on the one hand, more fission products may be incorporated by this glass, and chemical stability and radiation resistance of this glass are better; on the other side, this glass has a higher melting temperature. This glass is highly corrosive. Service lifetime of melters is usually limited not by an overall unsatisfactory condition of the entire refractory brickwork, but rather by some damage (often accident-related) of a certain number of structural elements of the melting chamber and gas space in the zones associated with melting and maximum temperatures. Not only refractories of the furnace brickwork are in a direct contact with the melt, but also other structural materials, including electrode metal, materials of auxiliary systems (thermocouple pockets, mixing devices, etc.).

The paper presents results related to the loss in weight and materials science studies of rolled steel metal samples taken after dynamic tests that were carried out during redox melting of borosilicate glass. The tests lasted for 100 hours at a temperature of 960 °C, bar rotation speed was 60 rpm, glass melt viscosity was 15 dPa.

The following steel and alloy grades: 12X18H10T (a reference sample), XH45IO, XH70IO were analyzed in the course of the materials science studies. The above-mentioned samples were examined with the help of the OLYMPUS SZX-16 optical microscope and the Phenom XL scanning electronic microscope with an optionally integrated system of energy-dispersive spectroscopy. The weight loss in samples was also determined within the context of this work.

On the basis of the conducted studies, a conclusion can be drawn that all samples that were taken from the top part, which had been in contact with the furnace hot air, had corrosion damages associated with the high temperature of the process and an unimpeded access of oxygen. A XH70IO alloy sample turned out to be the most corrosion-resistant material under the given conditions.

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## STUDY OF THERMOPHYSICAL PROPERTIES OF THE MATERIAL OF THE ACTIVE PART OF CERIUM-144 SOURCE

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The Mayak PA experts have carried out a wide range of computational and experimental studies and research works focused on the potential suitability of cerium-144 oxide for the manufacture of high power beta-particle source. One of the main criteria of ensuring safety of this source design is determination of the maximum permissible temperature of the structural elements and the radioactive material.

The research objective:

Studying thermophysical properties of cerium dioxide at high temperatures to determine safe process parameters of the source design.

The main tasks:

Obtaining computational and experimental data on thermal conductivity of cerium dioxide. Thermal conductivity is numerically equal to a product of a thermal diffusivity coefficient of the material and its specific heat capacity (at constant pressure) and density. Thus, in order to determine thermal conductivity of cerium dioxide it was necessary to specify variation of the specific heat capacity and of the thermal diffusivity coefficient of cerium dioxide at high temperatures.

The experimental procedure:

Variation of heat capacity of the cerium dioxide powder produced at the Mayak PA, in the temperature range of 50 °C to 550 °C was determined using differential scanning calorimetry (DSC). The DSC technique measures heat flux related to variations in the material structure as a function of time and temperature in the controlled environment that are accompanied with absorption or release of heat. The method for determination of heat capacity was based on the comparison of thermal fluxes of the analyzed and reference specimens (we used sapphire in our experiments). The equipment used in the research was thermal analyzer STA 449 F3 Jupiter.

Specific heat capacity of cerium dioxide at normal conditions is 0.358 J/g·K. However in the temperature range under study, the specific heat capacity varied from 0.35 to 0.47 J/g·K. Calculated thermal conductivity of cerium dioxide at high temperatures and different densities in compressed form is provided in the table.

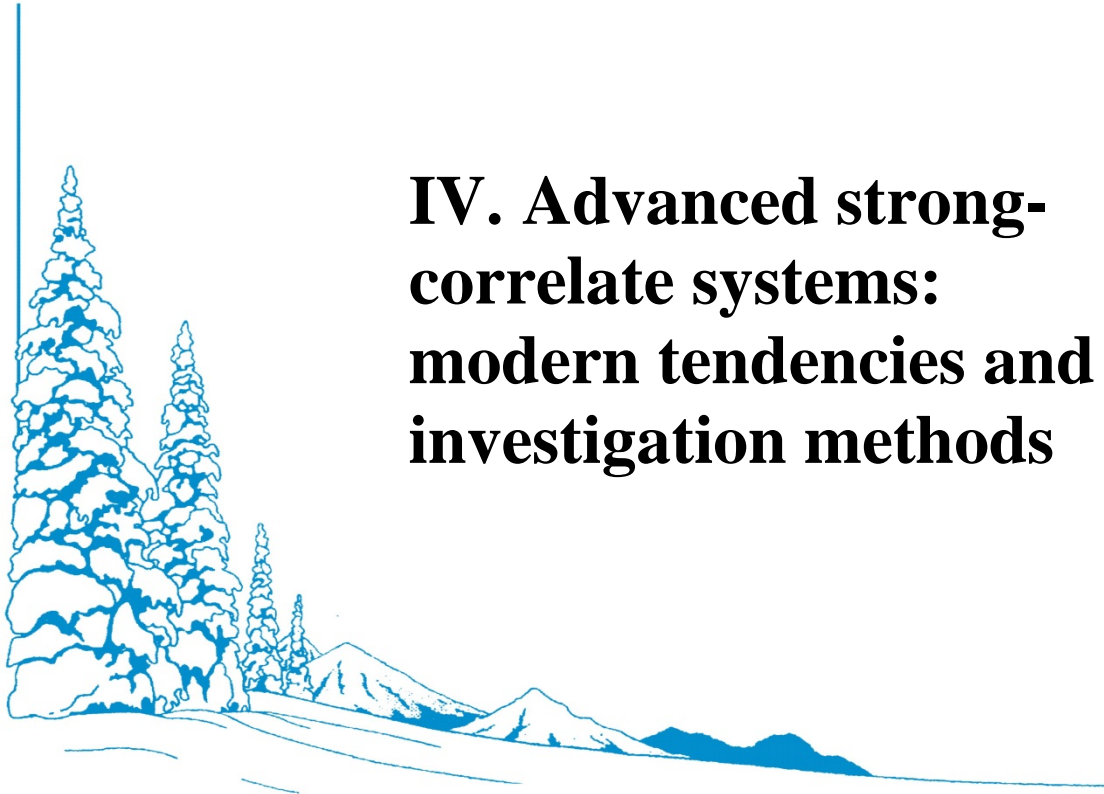
Density, g/cm <sup>3</sup>	Specific heat capacity, J/g·K	Thermal diffusivity, cm <sup>2</sup> /s · 10 <sup>-3</sup>	Calculated thermal conductivity, W/m·K
3.6	0.47	2.5	0.428
4.0	0.48	3.5	0.672
5.0	0.49	5.8	1.421

The obtained experimental data are in good agreement with the data published by foreign experts.

Conclusions:

It is proposed to use the obtained data on specific heat capacity, thermal diffusivity and thermal conductivity of cerium dioxide for calculation of safe technological design parameters of the cerium-144 source.





## **IV. Advanced strong-correlate systems: modern tendencies and investigation methods**

**The development of advanced technologies, including new generation nuclear techniques, make a strict requirement to structural and functional materials on the basis of which the element base for progressive computer, information and monitoring systems will be created. From this point of view, the perspective materials for it are materials based on d- and f-elements, having unique physical properties and known as systems with strong electron correlations. The spectroscopic investigations by neutron and X-ray scattering methods play the outstanding role in studying of physical properties of these materials. For this reason, in the Seminars' Programme is included reports, devoted to examination of perspective materials - new generation superconductors, frustrated magnets, valence-unstable systems, ferroelectrics, quantum magnets and hybrid nanostructures - by mean of inelastic neutron scattering, X-ray spectroscopy and measurements of microscopic parameters.**



## ATOMIC AND SPIN DYNAMICS IN CONDENSED MATTER BY NEUTRON SCATTERING WITH CRYSTAL SPECTROMETERS

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The crystal neutron spectrometers have strongly profited from now routinely used two-dimensionally focusing monochromators and analysers with variable and remotely controlled curvatures. The brightness of a measured ( $Q$ ,  $\phi$ ) position may be improved by mechanical devices by a factor of more than 100. Several multi-analyser (multi-pixel) schemes have been proposed and designed with a varied progress. Promising results have been achieved in studies of polycrystalline materials using large focusing crystal surfaces that permit registration of scattered neutrons in considerable solid angle. Several examples of recently collected data will be presented. Further efforts on the optimisation and multiplexing of neutron crystal spectrometers will be outlined.

## CHANGE IN THE CHEMICAL BOND AND ELECTRONIC STRUCTURE OF THE MAX-PHASE AS A RESULT OF HIGH-ENERGY IMPACT

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Recently, due to the unique combination of such properties as high radiation and corrosion resistance, stability in wide temperature ranges [1], new materials consisting of MAX phases have proven themselves well. MAX-phases are compounds which describe formula  $M(n+1)AX_n$  ( $n=1,2,3$ ), where M – 3d transition metal (Ti, V, Cr, etc.), A is an element of subgroup IIIA or IVA (Al, Si, etc.), X – C and / or N [2, 3]. Materials of this kind are demanded in nuclear power engineering. They can be used in the core of a nuclear reactor, which is an aggressive environment for materials due to high radiation densities, chemically aggressive coolant and high temperatures.

One of the perspective compounds is  $Ti_2AlC$ , since it has a number of high mechanical, electrical and thermal properties, which makes it one of the candidates for use in nuclear power plants. Previously, studies of the radiation resistance of the  $Ti_2AlC$  phase due to irradiation with heavy ions, light ions, neutrons and electrons were carried out; recent [2] studies have shown a high radiation resistance of the MAX phases. However, the question of their chemical stability, as well as changes in the electronic structure as a result of irradiation, remains largely unexplored.

In this regard, the aim of this work was to study the chemical bond and electronic structure of  $Ti_2AlC$  powder obtained by mechanical activation method followed by treatment with argon ions.

The object of the study was powders of the  $Ti_2AlC$  composition, with particle sizes from 1 to 10  $\mu m$  before and after heat treatment in an argon atmosphere at 1000 °C for 1 were prepared by mechanical activation in a ball mill using precursors in the form of titanium, aluminum, and carbon powders.



Irradiation was carried out in an ion-beam setup based on an ultra-high-vacuum station USU-4 with a Pion-1M ion source in a pulse-periodic mode with a pulse duration of 1 ms and a repetition rate of 100 Hz, a current density in a pulse of  $100 \mu\text{A} / \text{cm}^2$ , a fluence of  $5 \cdot 10^{17}$  ion /  $\text{cm}^2$ , the energy of argon ions was 30 keV.

The study of changes in the chemical bond and elemental composition was carried out by the methods of X-ray photoelectron spectroscopy and energy dispersive spectroscopy. Investigations of changes in the electronic structure of the MAX-phase of  $\text{Ti}_2\text{AlC}$  as a result of thermal and radiation exposure were carried out by analyzing the spectra of electron energy losses in the mode of backscattering from surface sample. It is shown that both radiation and thermal treatments have a significant effect on the electronic structure and chemical state of  $\text{Ti}_2\text{AlC}$  powders.

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## COOPERATIVE AND LOCAL FEATURES OF THE SPIN GAP FORMATION IN THE KONDO INSULATORS $\text{YbB}_{12}$ AND $\text{CeFe}_2\text{Al}_{10}$

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The formation of the excitation spectra of the ground state for Kondo insulators like  $\text{YbB}_{12}$ ,  $\text{CeFe}_2\text{Al}_{10}$  is studied and analyzed focusing on the correspondence between cooperative and local effects responsible for its character. Experimental results of the introduction of different types of defects into the rare earth (RE) and d-metal sublattices are discussed. It is demonstrated how the transformation from Kondo insulator to heavy fermion regime has an effect on the f-electron spectra for  $\text{CeFe}_2\text{Al}_{10}$ .

It is suggested that the gap in the electron excitation spectrum of the Kondo insulator is formed due to the setting the coherence (at low temperatures  $T < T^*$ ) in Kondo scattering processes of the itinerant band electrons on the array of localized rare-earth magnetic moments. This coherence appears to be destroyed by the introduction in this Kondo lattice of magnetic moments defects even in the relatively small quantity of the order of 10 %.

The influence of the nonmagnetic and magnetic impurities on the f-electron spectral function characterized by the resonance mode at low temperature for the  $\text{YbB}_{12}$  based systems is qualitatively reproduced in the frames of spin-exciton model [1] by the variation of the exchange parameter in the RE sublattice keeping hybridization parameter unchanged.

Decreasing of the hybridization strength experimentally controlled by the width of the quasielastic line in magnetic neutron scattering spectra, has been analyzed on the example of the Kondo insulator  $\text{CeFe}_2\text{Al}_{10}$  [2]. For this system it is realized basing on two physical mechanisms.

One is the change in the band structure (produced by the replacement in the surrounding of the rare-earth Kondo-ions). Also, the hybridization is modified by the changing of the interatomic distances. The decrease of the hybridization gives rise to the continuous transition from the Kondo insulator (spin gap, resonance mode) to the heavy fermion regime (quasielastic excitation) accompanied by spectral changes - the suppression of the gap and corresponding decrease of the resonance mode energy.

The analysis carried out allows to define the conditions for the formation of the Kondo insulator ground state, with essential factors as the interactions in the RE-sublattice as well as RE-ion interaction with near neighbors surroundings as well.

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## MICROSCOPIC PROPERTIES OF OXIDE COMPOUNDS CONTAINING RARE-EARTH IONS

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The investigation of rare-earth compounds is of great interest due to their unique properties that observed in such compounds. One of these compounds are orthoferrites, structurally distorted perovskites with the general chemical formula  $R\text{FeO}_3$ , where  $R$  is a rare-earth ion. This oxide magnetic materials, which interesting properties such as spontaneous spin-reorientation transition [1], laser-pulse induced ultrafast spin rotation [2], anisotropic magnetic entropy evolution [3], the occurrence of soliton lattices [4] and more. In the compounds there are two magnetic sublattices represented by  $\text{Fe}^{3+}$  and  $R^{3+}$  ions. The ordering of the  $\text{Fe}^{3+}$  lattice occurs at temperatures  $T_N \approx 600\text{-}700$  K, and the rare-earth subsystem at  $T \sim 10$  K. It is generally accepted that the unique magnetic properties of orthoferrites are the consequence of 3d-4f interaction, however, the formulation of a universal microscopic model encounters some difficulties.

In our work, we try to describe these systems independently in view of the large difference in excitation energies. The monocrystalline samples under study were obtained by optical floating zone method and investigated using inelastic neutron scattering. The study of spin dynamics at two different energy scales:  $\text{Fe}^{3+}$  ( $\sim 60$  meV) and the rare earth subsystem  $\text{Tm}^{3+}$  ( $\sim 4.5$  meV) [5, 6] and  $\text{Tb}^{3+}$  ( $\sim 15$  meV) were provided at Oak Ridge National Laboratory.

The study of the iron subsystem in the compounds  $\text{TbFeO}_3$  and  $\text{TmFeO}_3$  showed pronounced anisotropy "in" and "out" of plane. The investigation of the rare-earth subsystem revealed CEF transitions located in the low energy region for  $\text{Tm}^{3+}$  (2 and 5 meV) [7] and  $\text{Tb}^{3+}$  (17, 27 and 35 meV), the description of these dispersions was carried out using a point charge model showing that these levels belong to  $R$ -ions, and their behavior depends on the splitting of their ground state.

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## NEUTRON SPEROSCOPY OF ACTINIDE MATERIALS

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We present an overview of the experimental results for uranium (U), plutonium (Pu), and some U-and Pu-based compounds studied by inelastic neutron scattering technique (based on materials published in the open press). Actinide materials, widely used in civil and defense applications, have unique physical properties due to their *5f* shells. Metallic uranium is the first representative of the class of actinides, the properties of which - from the point of view of solid state physics - became the object of decades of study, and showed that during neutron irradiation the anisotropic thermal expansion and consequent dimensional instability is an intrinsic property of this metal [1]. As a result, metallic U as a nuclear fuel was abandoned in favor of the oxides. This surely represents one of the most rapid changes of technology driven by basic research. Inelastic neutron scattering played a decisive role in decrypting the nature of the anomaly in the  $\alpha$ -U elastic moduli near 40 K, showing that the anomaly is associated with formation of a charge density wave [2], and that the softening of the  $\alpha$ -U phonon spectrum with increasing temperature is explained not by ordinary anharmonicity, but caused by changes in the electronic structure [3].

The most common nuclear fuel, UO<sub>2</sub>, is a Mott insulator, i.e. a system with strong electronic correlations. At low temperatures, UO<sub>2</sub> transforms into an antiferromagnetic state, and at  $T < T_N = 30.8$  K demonstrates a completely unique dynamics due to the interaction of vibrational, magnetic, and quadrupole excitations [4].

Without the results of inelastic neutron scattering, it was impossible to solve the long-term problem of the magnetic state of metallic plutonium [5]. These experiments not only confirmed the fluctuating character of the Pu *5f* electrons, but allowed to understand the nature of the anomalies in the elastic properties of the Pu  $\delta$ -phase [6].

Uranium nitride UN, considered as promising nuclear fuel, also exhibits non-trivial physics. The prevailing point of view is that the *5f* electrons in UN are of an itinerant nature. However, recent NMR experiments indicate more complex UN nature [7].

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## SPECIFIC IMPACT OF INTERMEDIATE-VALENCE IMPURITY ON INDUCED MAGNETISM IN SINGLET GROUND STATE SYSTEM PrNi

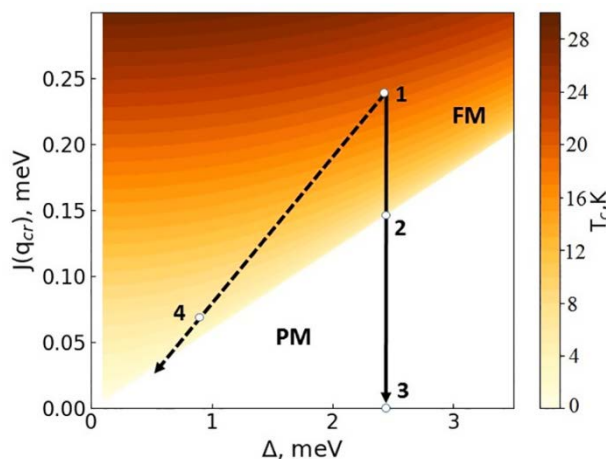
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The features of the phenomenon of "induced" magnetic ordering in the intermetallic rare-earth compound PrNi are considered. The influence of defects of various natures (nonmagnetic impurity La - Pr<sub>1-x</sub>La<sub>x</sub>Ni[1] and intermediate valence Ce - Pr<sub>1-x</sub>Ce<sub>x</sub>Ni[2]) in the rare-earth sublattice on the conditions for the formation of long-range magnetic order (LRMO) in PrNi-based systems has been studied in detail. A microscopic approach to structural modeling of the effect of defects on the LRMO is proposed and implemented.

The results of experiments on the substitution of a nonmagnetic impurity for La demonstrate the presence of a critical parameter that determines the transition temperature. It was found that the critical parameter is formed as a result of competing interactions: crystal field effects and exchange interaction. The transition to the ferromagnetic phase upon substitution by La is observed up to the La concentration x = 0.5 (solid line in Fig. 1).

The substitution of Pr for the IV Ce ion leads to an increase in the region of existence of the LRMO in the phase diagram (dashed line in Fig. 1), in comparison with the substitution of a nonmagnetic impurity. The transition to an ordered state in Pr<sub>1-x</sub>Ce<sub>x</sub>Ni is observed up to x = 0.9. It has been experimentally established that the magnetic moment on the Ce ion in the IV state does not manifest itself in any way, apparently due to its suppression by fast spin fluctuations. It

has been found that the renormalization of the CEF splitting for the Pr<sub>1-x</sub>Ce<sub>x</sub>Ni is the main origin of the T<sub>c</sub>(x) dependence.



**Fig.1.** The ground state diagram for systems based on PrNi. The interrelation of the magnetic ordering temperature ( $T_c$ ), exchange parameter  $J(q)$  and splitting between CEF singlets ( $\Delta$ ) is shown. The solid line corresponds to the change in  $T_c$  with increasing the La concentration in the Pr<sub>1-x</sub>La<sub>x</sub>Ni, the dashed line corresponds to the change in  $T_c$  with increasing the Ce

concentration in the  $\text{Pr}_{1-x}\text{Ce}_x\text{Ni}$ . The marked points correspond to the compounds: 1. PrNi, 2.  $\text{Pr}_{0.5}\text{La}_{0.5}\text{Ni}$ , 3. LaNi, 4.  $\text{Pr}_{0.25}\text{Ce}_{0.75}\text{Ni}$ .

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## STUDY OF STRONGLY CORRELATED ELECTRON SYSTEMS USING DIFFUSE NEUTRON SCATTERING WITH XYZ POLARIZATION ANALYSIS

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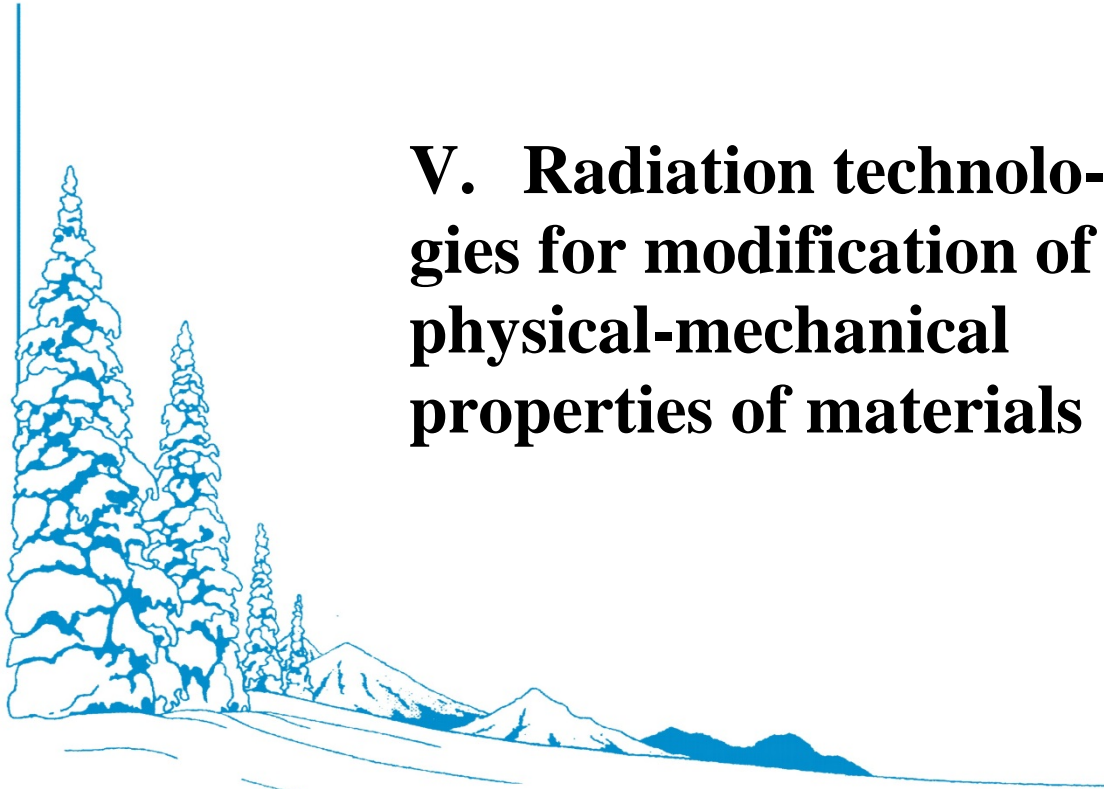
Diffuse neutron scattering with polarization analysis is a powerful instrument for studying interatomic correlations in condensed matter. In this talk I discuss basics of the formalism and practical realization of the method at DNS spectrometer [1,2] at Heinz Maier-Leibnitz Zentrum (research reactor FRM II, Garching, Germany). Large position-sensitive detector in combination with high flux ensures the DNS to collect the information from a large area of the reciprocal space. While the polarization analysis allows to separate the magnetic, nuclear coherent and spin incoherent components of the measured signal.

I present examples of the method application for the study of various groups of compounds with strong electron correlations, such as frustrated magnets, iron-based superconductors and multiferroics. Depending on the peculiarities of the studied system, diffuse neutron scattering with polarization analysis provides information not only on the character, but in certain cases also on the strengths of the interaction. It is especially efficient for the systems with short-range correlations and pronounced anisotropy.

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## V. Radiation technologies for modification of physical-mechanical properties of materials

This section is introduced in the Seminar's Program in order to exchange information about the latest achievements in the field of radiation material science associated with the development of physical bases and the use of electron- and ion-beam and ion-plasma methods for modifying the structure and properties of materials at the macro-, micro-, meso- and nanoscale level. The section program includes presentations devoted to the consideration of the fundamental aspects of the impact of accelerated ion beams with matter, that are determined their corpuscular nature and are general for both continuous and pulsed ion beams.

The powerful pulsed beams of electrons, ions, plasma flows and laser irradiation ( $P > 10^7$  W /  $\text{cm}^2$ ) cause instant melting and even evaporation of surface layer material. Modification of the properties at the same time is a result of the formation and propagation of powerful thermo-elastic waves. The combination of ion implantation with other methods, such as ion-beam mixing of films deposited previously on the surface of the target (Ion Mixing) or ion-assisted deposition of elements in vapor or plasma (Ion Beam Assisted Deposition) in order to increase the depth of exposure, which at normal conditions is only a percentage of a micron, leads to considerable complication and rise in price of the process. In connection with this, the intensively investigated recently long-range effects occur during ion bombardment, which allow to significantly increase the depth of the modified zone, become urgency, that is particularly important to develop methods for the modification of surface properties of construction materials.

The most promising is currently studying of nanoscale dynamic effects caused by corpuscular irradiation. Nanoscale regions of dense cascades of atomic displacements, warmed up for about  $10^{12}$  seconds to temperatures of 3000-5000 K and higher, are zones of explosive energy release and the source of the post-cascade solitary shock waves, which can rebuild the metastable environment. The rate of energy release is comparable to that for a nuclear explosion. Radiation-dynamic effects, which are not taken into account in the classical radiation physics of condensed matter, play an important role also under neutron irradiation and self-irradiation of fissile materials. They should be considered in connection with the nuclear safety issue in the development of new materials for use in the internals of nuclear power plants, as well as materials for use in outer space.



## **ANALYSIS OF CARBON FILMS ON GLASS OBTAINED BY MAGNETRON SPUTTERING**

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In this work, thin carbon films were deposited on a glass surface by magnetron sputtering of a carbon target in an Ar working gas medium, followed by bombardment with nitrogen and argon ions. In the process, the radiation dose was varied. The thickness of the films was verified by AFM by measuring the scratches on the film. At the level of measurement error on the original film, the thickness values correlate with the calculated data during deposition.

The analysis of the chemical composition of the carbon target was carried out using a Quattro S electron microscope. It is shown that there are no uncontrolled impurities in the target. Oxygen impurities of less than 1% are detected at various points of the analysis. This is due to the presence of the porous target in air.

The results of measurements of electrical conductivity showed that irradiation with nitrogen ions leads to an increase in the initial electrical resistance by 10-15 times, after irradiation with argon ions in the same dose - more than 30 times. There is a correlation between changes in the electrical resistance of carbon films before and after irradiation with AFM data on the average size of topography elements.

In this regard, an attempt was made to determine the differences in composition and structure by Raman spectroscopy and XPS.

According to XPS data, there are no significant differences in the films. The satellite structure in the region of  $\pi$  and  $\sigma$  plasmons, as well as an increase in the width of the main C1s peaks, indicate that we are initially dealing with films of predominantly disordered carbon (not graphite). It is also seen from the survey XPS spectra that the ion bombardment of the films with argon leads to the "diffusion" of sodium from the glass into the film. The larger the dose, the greater the intensity of the sodium spectrum. Silicon and its oxides are not observed, i.e. the films are generally continuous, and glass cannot be seen through the film.

## **EFFECT OF ACCELERATED $\text{Ar}^+$ AND $\text{Xe}^+$ IONS OF DIFFERENT ATOM MASS ON THE MAGNETORESISTANCE OF SUPERLATTICES OF $\text{Co}_{90}\text{Fe}_{10}/\text{Cu}$**

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The literature contains mainly data on the effect of ion irradiation on Fe/Cr superlattices [1, 2], which are currently model objects. It is of interest to study the effect of ion irradiation on the magnetoresistive properties of improved superlattices, which are promising from the point of



view of practical applications, in particular, on the structure and properties of  $\text{Co}_{1-x}\text{Fe}_x/\text{Cu}$  superlattices.

The magnetoresistive characteristics of magnetic metallic superlattices  $\text{Co}_{90}\text{Fe}_{10}/\text{Cu}$  (on glass,  $\text{Al}_2\text{O}_3$ , and Si substrates) exposed to beams of accelerated  $\text{Ar}^+$  and  $\text{Xe}^+$  ions of various atomic masses with an energy of 10 keV have been studied. It was found that irradiation of  $\text{Ar}^+$  and  $\text{Xe}^+$  ions with fluences  $1.25 \cdot 10^{15}$  and  $1 \cdot 10^{16} \text{ cm}^{-2}$  leads to a decrease in the magnetoresistance of  $\text{Co}_{90}\text{Fe}_{10}/\text{Cu}$  superlattices. Irradiation with  $\text{Ar}^+$  ions causes a more significant drop in magnetoresistance than irradiation with  $\text{Xe}^+$  ions, which is associated with an increased average projective range of argon ( $R_p = 5-6 \text{ nm}$ ) compared to  $\text{Xe}^+$  ( $R_p = 3.3-4.3 \text{ nm}$ ) and, accordingly, a greater depth of the atomic mixing zone  $\sim (2-3) \times R_p$  when ions move from the upper layers of the superlattice towards the substrate.

It is shown that the main contribution to the observed changes in the magnetoresistance is made not by heating, but by ion irradiation, which causes a partial mixing of atoms in layers of different compositions over the depth of the superlattices and affects the state of interlayer boundaries.

*This work was supported by the Russian Scientific Foundation, project no. 19-79-20173.*

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## **EFFECT OF $\text{N}^+$ IMPLANTATION ION ON SURFACE MORPHOLOGY, CHEMICAL COMPOSITION AND ATOMIC STRUCTURE OF SURFACE LAYERS OF STAINLESS STEEL AND TITANIUM ALLOY**

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The formation of surface layers of metallic materials with improved mechanical and operational properties through the introduction of accelerated ions into them belongs to one of the promising areas of modern science and technology [1-4]. Despite research in this direction, the influence of metal alloy components on the formation of surface morphology, elemental and structural-phase composition of surface layers as a result of ion irradiation remains unclear. Therefore, the purpose of this work is to study the morphology of the surface, nitrogen accumulation, the formation of chemical compounds and the structural-phase state of the surface layers of stainless steel 316 and titanium alloy Ti-6Al-4V during implantation of  $\text{N}^+$  ions. The choice of these metal materials is due to the different chemical activity of their components to nitrogen. The chemical activity of the components increases from iron to titanium. In addition, these alloys are widely used as structural materials in the chemical industry, aircraft and shipbuilding. Steel 316 is non-hardening, therefore nitriding of this type of steel is one of the ways to increase hardness, wear resistance and corrosion resistance [5].

The work revealed that irradiation with  $\text{N}^+$  ions leads to the accumulation of nitrogen in stainless steel up to 17 at.%, the formation of chemical compounds  $\text{CrN}$ ,  $\text{Cr}_2\text{N}$ ,  $\text{MoN}$ ,  $\text{TiN}$  and  $\text{Fe}_4\text{N}$  in the form of clusters of small sizes and crater-like surface morphology. In the case of a

titanium alloy, nitrogen accumulation is carried out up to 32 at.% and is accompanied by the formation of titanium nitrides, in particular TiN, in the form of phase inclusions over the entire surface. It is shown that the formation of the morphology of the surface of stainless steel is determined by the processes of sputtering, and the formation of a titanium alloy by the formation of many inclusions of titanium nitrides.

*The work was carried out within the framework of the state task of the Ministry of Science and Higher Education of the Russian Federation GZ N 121030100002-0. The research was carried out using the equipment of the Center for Physical and Physico-Chemical Methods of Analysis, Research of Properties and Characteristics of Surfaces, Nanostructures, Materials and Products of the UdmFIC Ural Branch of the Russian Academy of Sciences.*

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## **FORMATION OF HIGH-MANGANESE-AUSTENITE IN THE Fe–6.35 at.% Mn ALLOY AT TEMPERATURES 300-450°C IN THE ABSENCE OF THERMALLY ACTIVATED DIFFUSION PROCESSES AS A RESULT OF SHORT-TERM “RADIATION SHAKING” BY AR<sup>+</sup> IONS (*E* = 15 keV)**

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Using the methods of Mössbauer spectroscopy and transmission electron microscopy, it was found that short-term (for 4 seconds) irradiation of cold-rolled foils of the Fe-6.35 at.% Mn composition with a thickness of 25 microns with Ar<sup>+</sup> ions (*E* = 15 keV) with heating to 300-450°C causes an  $\alpha \rightarrow \gamma$  phase transformation with the formation of the repeatedly enriched with manganese austenite (from 23.8 to 38.0 at.%). The estimates made do not allow us to explain these processes by the phenomena of thermal and radiation-enhanced diffusion. As an explanation, we used a model of nanoscale dynamic effects associated with shaking the medium by powerful post-cascade solitary waves. Such waves, becoming undamped in metastable media and playing, in a certain sense, the role of temperature, cause a short-term giant increase in the mobility of atoms and the transition of these media to a state with minimum free energy. The concentrations of Mn  $c_1$  and  $c_2$  in the  $\alpha$ - and  $\gamma$ -phases, calculated on the basis of decoding the Mössbauer spectra for irradiation temperatures 311, 378 and 449°C, correspond quite well to the results of extrapolation of the lines of the equilibrium phase diagram to the low temperature region. The data obtained are compared with the results of earlier work [1, 2].

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## **GIANT RADIATION-DYNAMIC EFFECTS UNDER CORPUSCULAR IRRADIATION**

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Nanoscale dynamic effects and processes occurring in cascade-forming types of learning of condensed matter by heavy ions, neutrons, and fission fragments are considered. The role of these processes is outside the field of vision of classical radiation physics of condensed matter. They were first considered in publications by the author and his colleagues and confirmed by joint work with German colleagues (Helmholtz-Zentrum Dresden-Rossendorf, Germany). The sources of the recorded effects are the regions of passage of dense cascades of atomic displacements, thermalized in times of the order of one trillionth of a second (thermal spikes), with giant temperatures and thermal pressures in these regions ( $T = 3000-6000$  K,  $P = 5-40$  GPa). For this reason, it is necessary to take into account shock-wave effects in cascade-forming types of irradiation, together with purely migration processes with the participation of radiation defects, which are taken into account by classical radiation physics.

A theory of self-propagating (theoretically over unlimited distances) structural-phase transformations in metastable media initiated by ion bombardment is developed. In practice, this provides: 1) an increase by at least 3-5 orders of magnitude of the linear scale of the effect of ionizing radiation on materials (an increase in the depth of exposure for ions); 2) the temperature of the transformations initiated by irradiation decreases by tens and hundreds of degrees in comparison with similar thermally activated processes; and 3) their rate increases by several orders of magnitude in comparison with thermally and radiation-stimulated migration processes.

A lot of examples of radiation-dynamic (RD) processes in metals and alloys (intrapphase and phase transformations) with changes in their electrical, magnetic, mechanical, resource and other characteristics are considered. Specific applications concern the modification of the properties of functional materials (resistive, magnetic, mechanical, resource) by beams of accelerated ions. Simulation studies of the radiation resistance of materials have also been carried out.

The results of the survey are partially presented in papers [1-4].

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## **INVESTIGATION OF DIFFUSION PROCESSES IN METALLIC MATERIALS UNDER ION IRRADIATION**

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A comparative analysis of samples in amorphous and crystalline states, irradiated with boron ions, has been carried out. In a number of works, it was previously shown that ion treatment of crystalline materials in an initial nonequilibrium state (for example, amorphous or rolled metal foils) leads to a number of effects that differ from materials initially in an equilibrium state, such as anomalously deep mass transfer, phase formation etc. One of the discussed issues of the migration paths of atoms is the presence of a highly developed defect structure. A comparative analysis of annealed samples shows that the defect structure is the driving force behind the migration of atoms under the action of ion irradiation, but is not a sufficient condition.

In this work, we study the features of segregation processes in the surface layers of amorphous rapidly quenched and polycrystalline multicomponent alloys under conditions of ion irradiation. The samples were irradiated under the same conditions. Boron ions were implanted with energies  $E = 20, 30, 40$  keV and a dose of  $10^{17}$  cm<sup>-2</sup> using an ILU-3 setup. The ion current densities were chosen so that at each ion energy the power density released on the samples was the same, and for the indicated energies were 3, 2, and 1.5  $\mu\text{A} / \text{cm}^2$ , respectively.

A component analysis of the surface layer of the samples showed that, as a result of irradiation, segregation processes occur in the surface layer under the influence of the ion flux. The nature of the observed segregation processes fits into generally accepted models. It can be seen that the nature of the distribution of the components on the irradiated side and the side opposite to the irradiation indicates the wave nature of the driving force, namely, the release of elastic waves to the surface. At the same time, one of the main results of the work is the experimentally established absence of boron atoms on the side opposite to irradiation on amorphous samples, while the presence of boron on the opposite side is noted on crystalline samples.

The main goal of this work was to determine the mechanism of migration of atoms in a radiation field. Comparing the results of X-ray structural analysis and data on the component composition of the near-surface region, it can be assumed that as a result of irradiation, metastable phases are actively formed in the surface layers, the composition of which changes depending on the irradiation parameters. The absence of implantable ions (boron) on the reverse side of the samples indicates that the diffusion of atoms occurs along the grain boundaries - and in amorphous materials these mechanisms are not realized due to the absence of grain boundaries, in contrast to polycrystalline materials in which the presence of implanted atoms was noted on the opposite side samples. The driving force of diffusion processes is the process of propagation of elastic waves arising from the development of microscopic processes of atomic-atomic collisions.

## INVESTIGATION OF THE CHEMICAL COMPOSITION AND PHYSICO-MECHANICAL PROPERTIES OF Ti/Al MULTILAYER FILMS AFTER ION-BEAM MIXING

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Methods of ion-beam and ion-plasma treatment, having a number of fundamental advantages over traditional methods of chemical-thermal treatment, have been actively developed in the field of modification of surface layers of metals and alloys in order to increase their strength properties [1, 2]. In addition to the classic advantages of ion treatment (the possibility of exceeding the solubility limit, control of the depth of impurity distribution, the possibility of selective processing of parts, etc.), in the last decade, it has been possible to add completely new methods of influencing the surface layers of materials. In particular, by forming on the surface of the target, one or several layers of other materials of a nanometer thickness range, and their subsequent ion treatment with high-energy particles, it was possible to form new compounds and phases in the surface layers [3, 4].

In this work, the methods of X-ray photoelectron spectroscopy, atomic force microscopy and nanohardness measurement were used to study the morphology of the surface, the formation of the chemical composition and changes in the physical and mechanical characteristics of the surface layers of Ti/Al multilayer films on the surface of titanium VT1-00 depending on the dose of argon ion irradiation ( $5 \cdot 10^{15}$  ion/cm<sup>2</sup>,  $10^{16}$  ion/cm<sup>2</sup>,  $5 \cdot 10^{16}$  ion/cm<sup>2</sup> and  $10^{17}$  ion/cm<sup>2</sup>).

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*The work was performed with the equipment of the Centre for Shared Usage "Center of physical and physical-chemical methods of analysis and research of properties and characteristics of surfaces, nanostructures, materials, and samples" of the UdmFRC UB RAS.*

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## **INVESTIGATION THE FORMATION PROCESSES OF Ni-Al COMPOUND UNDER THE LASER RADIATION**

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The paper considers the results of studying the effect of focused pulsed laser radiation on a nickel matrix with an aluminum layer deposited on its surface, depending on the power density of the laser radiation.

The aim of the work is to study the processes occurring in the material as a result of irradiation and to provide the necessary modes of laser action on the Ni-Al system for the purposeful modification of the surface layers with intermetallic compounds.

The formation of intermetallic compounds based on nickel and aluminum under thermal laser action are characterized by different values of their activation energy. Depending on the amount of energy transferred to the material, the type of intermetallic compound is formed, the formation of which is thermodynamically most favorable. Consequently, the phase composition of the intermetallic interlayer formed in the Ni-Al system will largely be determined by the amount of energy transferred to the sample. In this case, the rate of heating-cooling of the material also has a very significant effect on the mechanism of obtaining an intermetallic compound by a laser. If it is high, then the heat loss in the system is small, therefore, it is possible to overheat the system and quickly “freeze” the resulting state not to obtain the required connection. If it is small, then a slow increase in temperature can be observed and, therefore, inhibited interaction between the elements of the system, which also does not contribute to the production of the required intermetallic compound.

A pulsed fiber-optic ytterbium laser “Ldesigner F1” was used as a source of laser action. The samples were irradiated with a focused laser beam in an argon atmosphere. The exposure was single-pulse, and the variable parameter during irradiation was the power density of the laser radiation. The irradiation area was  $10 \times 10$  mm in size.

The analysis of the state of the surface layers of the samples before and after laser exposure was carried out using the methods of atomic force microscopy (“SOLVER-47 PRO”), X-ray photoelectron spectroscopy (“ES-2401”), and microhardness measurements (“PMT-3”).

As a result of the studies carried out, it was found that under the selected modes of laser action, the samples were subjected to strong thermal action. Strong surface melting is observed on the surface of the samples in the irradiation zone. According to the obtained XPS data, it was found that after laser treatment, in all cases of irradiation, the formation of any intermetallic compounds is not observed in the surface layers. The spectra obtained for the samples under study showed the presence of a line with  $E_b = 74.0$  eV, which corresponds to aluminum in the composition of nickel aluminates  $NiAl_2O_4$ . According to the literature, the  $NiAl_2O_4$  compound is characterized as a complex oxide of the spinel type, which can be formed during high-temperature oxidation (above 1000 °C) as a result of the interaction of aluminum and nickel oxides.

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## MODES SELECTION AND ANALYSIS OF RESULTS OF DEFORMED Ni – 13.9 wt. % W ALLOY ANNEALING USING BEAMS OF ACCELERATED ARGON IONS

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The effect of Ar<sup>+</sup> ions with an energy of 15-20 keV (at ion current densities of 100-300 μA/cm<sup>2</sup>) on the microstructure, the level of internal micro stresses, and the texture of cold-rolled ribbons of Ni-13.9 wt. % W alloy was studied. The alloy after deformation and subsequent recrystallization annealing ( $T = 1000^{\circ}\text{C}$ , 1 h) acquires an especially sharp texture and is a promising material for the manufacture of substrates for HTSC cables.

Irradiation with continuous beams of Ar<sup>+</sup> ions was carried out on an ILM-1 implanter equipped with a PULSAR-1M ion source based on a low-pressure glow discharge with a cold hollow cathode. In this case, we experimented with irradiation modes to achieve different heating and holding of specimens at specified temperatures.

An X-ray structural study of the strips in the initial state and after ion-beam treatment was carried out using the Rietveld method using the FullProf program. X-ray diffraction patterns were recorded on a DRON-4 diffractometer.

It was found that short-term irradiation of 80 μm thick ribbons with a fluence of  $3.1 \cdot 10^{16} \text{ cm}^{-2}$  (for 50 s) at temperatures  $T \leq 370^{\circ}\text{C}$  and  $T = 630^{\circ}\text{C}$  leads to a decrease in micro stresses in their entire volume, but at the same time, the original texture is retained. With an increase in the fluence to  $9.7 \cdot 10^{17} \text{ cm}^{-2}$  at  $T = 630^{\circ}\text{C}$ , the texture also changes from (220) to (200). Changes in microstresses and texture on the irradiated and unirradiated sides of 80-μm-thick ribbons are comparable to each other, even though the projective range of Ar<sup>+</sup> ions with an energy of 15-20 keV in the alloy is only ~7 nm. It is known that annealing in an oven (700°C, 30 min) of such ribbons does not lead to their recrystallization.

Irradiation at a temperature of target 850°C only at fluence  $3.2 \cdot 10^{16} \text{ cm}^{-2}$  (for 17 s) leads to the microstress relaxation in the volume of the deformed strip and a radical change in the texture from (220) to (200). The texture change also occurs during similar annealing in a furnace, however, in this case, the stress relaxation effect is 3 times lower than in process of ion irradiation.

Therefore, in the alloy understudy during irradiation Ar<sup>+</sup> ions the occurrence of recrystallization processes at a temperature lower than the temperature of the onset of thermally activated recrystallization has been observed. Besides a higher rate of micro stresses drop (and to lower values) in the course of irradiation than during furnace annealing. This indicates a significant role of nanoscale radiation-dynamic effects at the cascade-forming irradiation of metastable media [1].

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## **MOLECULAR DYNAMICS STUDY OF THE ATOMIC STRUCTURE FORMATION UNDER ARGON IONS IRRADIATION ON THE METAL SURFACE**

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This work considers the change in the near-field structure of a metal surface under argon ions irradiation with an energy of 30 keV. Modeling was performed using the LAMMPS software package for molecular dynamics. The processes of formation of the short-range order atomic structure of surface layers of iron under ion irradiation with argon are investigated. The iron sample was argon ions irradiated with an initial energy of 30 keV to record changes in the structure. Paired correlation functions are constructed at equal time intervals. Sputtering of atoms from the surface and the formation of point defects in the simulated region are observed.

The simulation data were compared with the results of experimental studies of the local atomic structure within the first coordination sphere. The experimental data were obtained from the iron surface after irradiation with argon ions. The spectra of energy losses of electrons were obtained in the geometry of backscattering of secondary electrons from the sample surface on a JAMP-10S Auger microanalyzer (JEOL). The analysis of the obtained experimental data was carried out by the method of solving the inverse problem.

Thus, the results of modeling with the used potential and given irradiation parameters describe well the experiment on the radiation effect on the iron surface.

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## **ON THE EFFECT OF ION-BEAM MODIFICATION OF THE FATIGUE RESISTANCE OF PRESSED PROFILES FROM ALUMINUM ALLOYS**

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The study of the effect of irradiation with Ar<sup>+</sup> ions on the fatigue resistance of hot-pressed profiles 6 mm thick made of alloy D16 (Al-Cu-Mg) and V95 (Al-Zn-Mg-Cu) after quenching and natural aging has been carried out.

The samples were irradiated using an ILM-1 ion-beam treatment facility equipped with a PULSAR-1M ion source based on a glow discharge with a cold hollow cathode. The following irradiation parameters were used:  $E = 20$  keV,  $j = 200$   $\mu\text{A}/\text{cm}^2$ ,  $F = 2 \cdot 10^{15}$  and  $1 \cdot 10^{16}$   $\text{cm}^{-2}$ . The maximum temperature to which the samples were heated during irradiation did not exceed 40°C.



The specimens were tested on an INSTRON 8801 servo-hydraulic test rig. To determine the Weller fatigue curve, cyclic fatigue tests were performed in a sinusoidal cycle with a loading frequency of 3 Hz. The cycle unbalance factor (asymmetry coefficient) is  $-1$ .

It was found that short-term double-sided irradiation with  $\text{Ar}^+$  ions with an energy of 20 keV with the indicated fluences in the absence of heating of the samples leads to a significant increase in the number of cycles to failure under conditions of reduced load amplitudes  $\sigma/\sigma_u = 0.3$ . For alloy D16, the maximum increase in 2.4 times is observed at an ion fluence  $F = 2 \cdot 10^{15} \text{ cm}^{-2}$ . The fatigue resistance of V95 increases 5 times after  $\text{Ar}^+$  ions irradiation with a fluence  $F = 1 \cdot 10^{16} \text{ cm}^{-2}$ .

This result may be associated with the fact that the surface that under conditions of high-cycle fatigue, only small elastic deformations take place. In addition, the surface and volume of the irradiated sample undergo "instantaneous" (within a few seconds) radiation annealing with an ion beam, as a result of which both the surface and the volume are practically freed from deformation defects. Because of this, destruction begins from the surface of the sample much later, since the processes of damage accumulation and cracking develop much more slowly.

The data obtained indicate the possibility of using ion-beam treatment at the finishing stages of the production of promising aluminum alloys.

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## **SWIFT HEAVY ION IRRADIATION EFFECTS IN ODS STEELS: TEM EXAMINATION AND MICRO-PILLAR COMPRESSION TESTS**

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Development of Gen IV nuclear reactors demands integration of novel class of constructive materials with enhanced functional characteristics corresponding to extreme operating conditions. Oxide dispersive strengthened (ODS) steels are perspective materials for fuel cladding which high working properties are due to nanosized Y-Ti and Y-Al oxide particles embedded in ferrite matrix [1].

According to the numerous studies, nanoparticles remain crystalline after neutron and low energy ion irradiation up to high (tens of dpa) radiation damage levels. By now swift heavy ion (SHI) irradiation with the energies  $>1 \text{ MeV/amu}$  is the only known irradiation type that can produce considerable structure degradation of nanooxides up to their complete amorphization. In the present work ODS steels were irradiated by high energy xenon ions with fluences corresponding to individual latent track formation in nanooxides and track overlapping irradiation regimes. The aim of the work is study of radiation-induced changes of the mechanical properties of ODS steels containing oxide particles in different (crystalline and amorphous) crystalline state.

It was shown that 167 MeV xenon ion irradiation leads to individual amorphous track formation in  $Y_2Ti_2O_7$ . The latent tracks start overlapping with ion fluence  $\sim 2 \cdot 10^{12} \text{ sm}^{-2}$  and with the following fluence increasing nanooxides undergo complete amorphization [2]. Threshold stopping power for latent track formation in pyrochlore is in the range of 7.4-9.7 keV/nm, that corresponds to depth of  $\sim 7 \mu\text{m}$  in damage depth profile. In order to evaluate the impact of SHI irradiation on mechanical properties of ODS steels, nanoindentation and FIB fabricated micro-pillar compression tests were carried out. Micro-pillar height was less than the target depth where latent track were formed. Compression tests of micro-pillar with completely amorphized nanoparticles have never been made before.

The comparison of mechanical tests results obtained by different methods were done and correlation between the crystallinity of the nanoparticles and mechanical properties changes of SHI irradiated ODS steels are discussed.

*This work was partially performed using the equipment of the Advanced Imaging Core Facility of Skolkovo Institute of Science and Technology.*

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## **THE CONCEPT OF QUASINEUTRONS AND CHARACTERISTIC RELATIONS OF THE ISOTOPE CONTENT OF THE SYNTHESIZED ELEMENTS**

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The concept of quasineutrons was proposed in [1]. The essence of the quasineutron concept is simple. Since there are limiting bound states of an electron and a proton, on the one hand, in the form of a hydrogen atom (with a size of  $\sim 10^{-10} \text{ m}$ ), and on the other hand, in the form of a neutron [2] (with a size of  $\sim 10^{-15} \text{ m}$ ), then naturally admit the possibility of intermediate quasineutron states, when the electron carries out the "escort" of the proton, screening its positive charge. Obviously, with respect to the positive nucleus, a quasineutron can be considered as a neutral particle if the size of the quasineutron itself is small compared to the distance from the quasineutron to the nucleus. As shown in [3], as the radius of the internucleon strong interaction, one can take  $R_s^* \approx 10^2 \phi_M = 10^{-13} \text{ m}$ . Consequently, for the capture of a proton by the initial nucleus  ${}^A_Z X$  with the formation of a nucleus  ${}^{A+1}_{Z+1} Y$  an electronic "escort" of the proton to the distances  $R_s^*$  is sufficient. In experiments [4], the synthesis of zinc with the participation of copper was observed, which is consistent with the concept of quasineutrons. In this regard, the problem naturally arises of determining the ratios of the synthesized isotopes  $f(A_2^*) / f(A_1^*)_{\text{exp}}$ , which should be specified by the natural ratio of the initial isotopes  $f(A_1) / f(A_2)_{\text{nat}}$ . Here  $A_{1,2}$  are the mass numbers of the initial isotopes,  $A_{1,2}^* = A_{1,2} + 1$ . As a result, the ratio of the synthesized isotopes may differ markedly from their natural ratio  $f(A_2^*) / f(A_1^*)_{\text{nat}}$ , as follows from the data in

Table 1 for the synthesized isotopes of zinc and gallium, where it is taken into account that  $f(A_2^*)/f(A_1^*)_{\text{exp}} = f(A_1) / f(A_2)_{\text{nat}}$ .

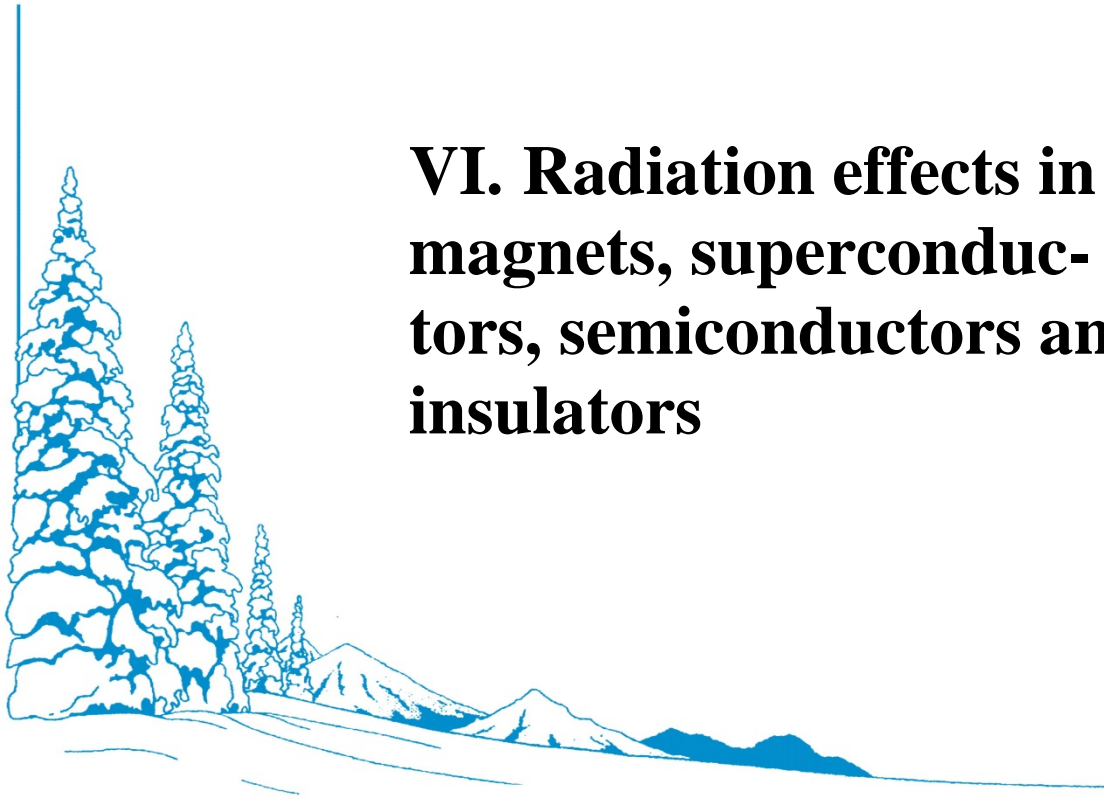
Table 1. The difference between the expected ratios of isotopes (exp) synthesized during the capture of protons by isotopes of copper and zinc from the ratios in nature (nat)

Z	Z +1	Z, A <sub>1</sub> , A <sub>2</sub>	Z +1, A <sub>1</sub> <sup>*</sup> , A <sub>2</sub> <sup>*</sup>	$f(A_2^*)/f(A_1^*)_{\text{exp}}$	$f(A_2^*)/f(A_1^*)_{\text{nat}}$
29 (Cu)	30 (Zn)	Cu, 63, 65	Zn, 64, 66	0.446	0.56
30 (Zn)	31 (Ga)	Zn, 68, 70	Ga, 69, 71	0.03306	0.66367

The results of mass spectrometry for the synthesized isotopes of zinc (when electrolysis of water using copper electrodes) and gallium (using zinc electrodes) are generally consistent with the expected results. The observed deviations from the expected values are due to the contribution of reactions additional to the capture of protons.

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## **VI. Radiation effects in magnets, superconductors, semiconductors and insulators**

**The subject of this Section is traditionally formulated with a view to introduce the Seminar attendees (mainly metal physicists) to the results of the latest research into radiation effects in superconductors, semiconductors and dielectrics (magnetic dielectrics including). In the case of the first two materials, their physical properties change significantly upon exposure even to rather low fluences of high-energy particles. Therefore, investigation of the causes of damage and the impairment of the physical and mechanical properties of the materials of this group has always been - and is today - a topical task. The Seminar Program includes papers on physics of radiation effects in semiconductors and insulators. The behavior of radiation defects and changes in the physical and mechanical properties of materials such as manganites, oxides, etc. are analyzed. The amorphization of silicon upon exposure to ion beams, the dielectric effect in HTSC ceramics, principles underlying the radiation modification of semiconductors and dielectrics, and the influence of radiation-induced disordering on semiconductor radiation detectors are discussed.**



## CALCULATION OF DEFECT FORMATION IN HTSC TAPE COMPOSITES UNDER ION IRRADIATION

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The report presents the results of a numerical analysis of radiation damage in high-temperature superconducting (HTSC) tape composites. We have prepared and adapted software products for numerical simulation of defect formation processes in HTSC material. An algorithm is formulated for an express experimental technique that makes it possible to estimate the inhomogeneity of the critical current of a superconducting layer over the film thickness, which arose as a result of irradiation of a superconductor with fast charged particles (hydrogen and helium). The SRIM / TRIM package was used to calculate the profiles of radiation damage by these particles in the HTSC layer (see figure). This technique can be used to study the inhomogeneity of the superconducting properties of an HTSC layer in external magnetic fields. It is shown that for the studied sample of the superconducting layer GdBaCuO-123, the technique can be applied at the value of the external magnetic field induction  $B_{ext} = 0.1$  T and higher. The calculation of the number of displacements per atom (dpa) for various irradiation fluences is carried out. The following feature of the radiation degradation of the superconducting properties of the HTSC film was discovered. After irradiation of samples of a superconducting composite with different coatings, the inhomogeneity of the critical current over the thickness significantly depended on the nature of the coating, but did not depend on the type of irradiation. In the future, it is planned to extend the calculations to fast ions of heavier elements.

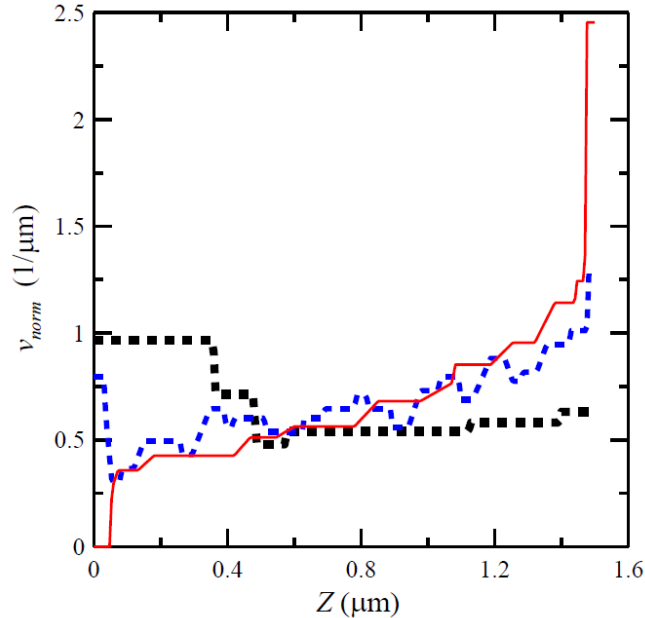


Fig. Normalized density of radiation vacancies upon irradiation with helium ions (Bold dashed black line sample with protective layers of copper and silver; middle broken blue line sample with a protective layer of silver only; thin solid red line - sample without protective layers)

*The reported study was funded and by RFBR and ROSATOM according to the research Project No. 20-21-00085*

## EFFECT OF ELECTRON IRRADIATION ON THE ELECTRIC PROPERTIES OF HETEROEPITAXIAL n-InSb-i-GaAs STRUCTURES

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The narrow-gap semiconductor of the  $A^3B^5$  group, indium antimonide, has significant practical application in solid-state microelectronics and semiconductor instrument making. Highly sensitive photocells, Hall sensors, optical filters, etc. are made on the basis of indium antimonide. Such microdevices are highly energy efficient, allowing to significantly reduce energy, material and operational costs for the reliable operation of vehicles, aviation and space technology.

Spacecraft in flight are exposed to radiation. This leads to deterioration in operational parameters and failures in the operation of materials and equipment elements. The occurrence of failures is mainly associated with certain radiation effects caused by the action of electrons and ions on materials, the energies of which lie in a very wide range:  $\sim 10^3 - 10^{20}$  eV [1].

The n-InSb-i-GaAs structures were obtained by explosive thermal evaporation of a single-crystal InSb powder onto GaAs (100) plates [2]. The thermocouple-controlled temperature of the substrate-holder with the GaAs wafer was  $(375.0 \pm 2.5)$  °C. Studies have shown that heteroepitaxial InSb films are formed at this temperature [3]. Thickness was measured using a MahrMarSurf M400 profilometer. The measured InSb film thickness was  $(2.00 \pm 0.05)$   $\mu\text{m}$ .

To simulate the conditions of radiation exposure in the near-earth orbit, n-InSb-i-GaAs structures were irradiated with an electron beam on a linear Y-003 accelerator with an energy of 5.5 MeV and a dose of up to  $2.5 \cdot 10^{14}$  e/cm<sup>2</sup>.

Table shows the electrical properties of n-InSb-i-GaAs structures before and after electron beam irradiation.

Table – Electrical properties of n-InSb-i-GaAs structures before and after electron beam irradiation

Dose, e/cm <sup>2</sup>	n, cm <sup>-3</sup>	$\mu$ , cm <sup>2</sup> /(V×s)
Initial	$(5.4 \pm 0.5) \times 10^{16}$	$(21 \pm 2) \times 10^3$
$2.5 \times 10^{13}$	$(1.7 \pm 0.1) \times 10^{19}$	$(11.1 \pm 0.8) \times 10^3$
$2.5 \times 10^{14}$	$(1.82 \pm 0.13) \times 10^{19}$	$(10.2 \pm 0.7) \times 10^3$

As a result of the study, it was found that the concentration of charge carriers increases with irradiation. It is assumed that there is a transition of valence electrons to the conduction band due to internal ionization caused by irradiation. In this case, the mobility of charge carriers upon irradiation with an electron beam with a dose of  $2.5 \cdot 10^{13}$  e/cm<sup>2</sup> decreases by a factor of 2. An increase in the irradiation dose of n-InSb-i-GaAs structures to  $2.5 \cdot 10^{14}$  e/cm<sup>2</sup> does not lead to a change in the concentration and mobility of charge carriers.

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## INFLUENCE OF ION IRRADIATION ON THE CRITICAL CHARACTERISTICS OF THE HTSC TAPE COMPOSITES

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One of the practical applications of second generation HTSC tapes (or CC-coated conductors) is accelerator magnets. During operation, the superconducting winding will inevitably be exposed to ionizing radiation with the formation of radiation defects in the HTSC layer. This process can lead to gradual degradation of both the critical characteristics and the structure of the HTSC. The report presents the results demonstrating the change in the critical temperature and critical current of HTSC tapes of the second generation under irradiation of the HTSC layer with Cu ions  $E = 6.3$  MeV in a wide range of fluencies up to complete loss of superconducting properties. The critical temperature was measured by a four-contact method. The critical current was calculated from the magnetization curves, which were measured in the temperature range from 5 to 77 K and magnetic fields up to 8 T. It was demonstrated that critical current is more sensitive to radiation defects than the critical temperature (see Fig.). This means that HTSC tapes will lose their current carrying characteristics even at low operating temperatures in the ranges of relatively low fluencies, when the critical temperature is still high. The data obtained will be used for resource forecasting of the operation of HTSC elements in real radiation fields.

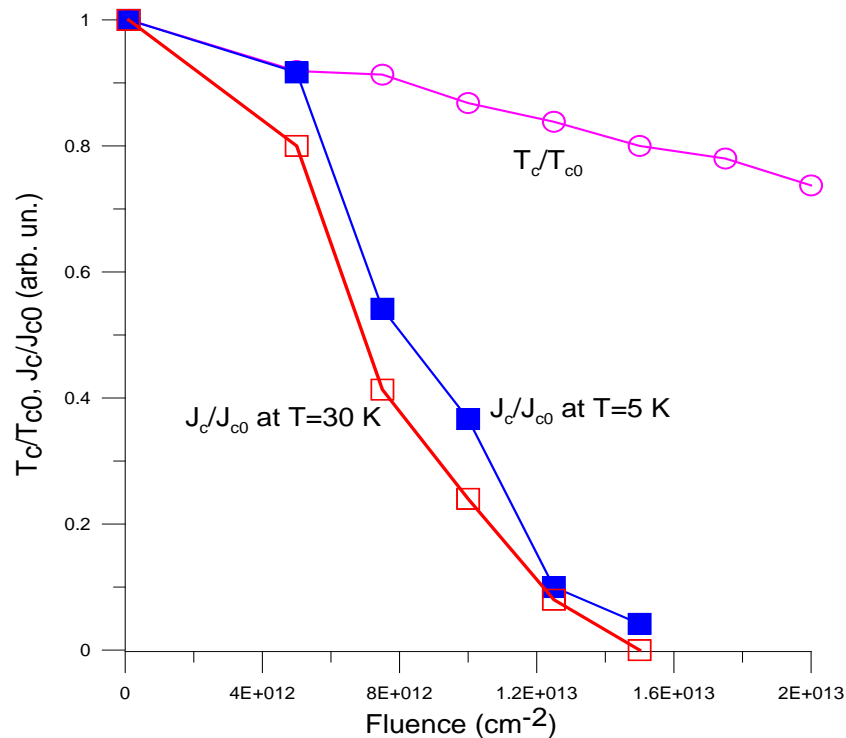


Fig. Dependencies of normalized critical temperature and normalized critical current at 5 K and 30 K on fluence of ion irradiation

*The reported study was funded and by RFBR and ROSATOM according to the research Project No. 20-21-00085*



## LATENT TRACKS IN NANOCRYSTALLINE $Y_4Al_2O_9$ IRRADIATED WITH HIGH ENERGY HEAVY IONS

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This work focuses on the investigation of structural changes in  $Y_4Al_2O_9$  (nc-YAM) irradiated with swift heavy ions in a range of electronic stopping powers 6÷35 keV/nm using high resolution TEM techniques. Bi and Xe ions were found to form latent tracks in studied nanooxides (Fig.1) that contrasts with their extremely high radiation stability in a metallic matrix [1].

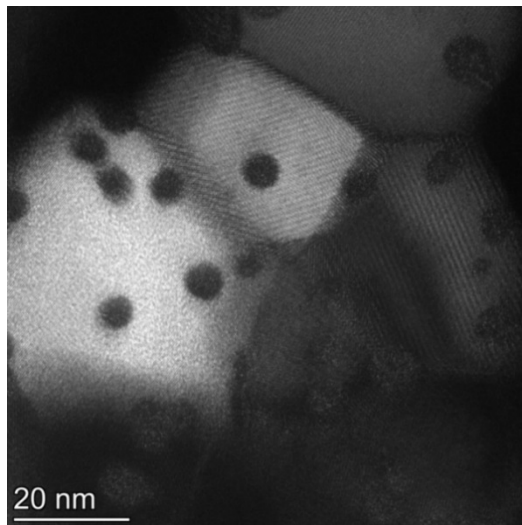


Fig. 1. DF TEM image of nc-YAM irradiated with Xe with energy of 0.2 MeV/u

Based on the analysis of the TEM data threshold conditions to the formation as of continuous so discontinuous tracks in nc-YAM were estimated. No effect of grain size on the ion track parameters was observed for all specific ionizing energy losses used in experiments.

*Acknowledgement. This research was funded by the Ministry of Education and Science of the Republic of Kazakhstan (grant No AP09058081 “Radiation stability of dielectric nanoparticles in oxide dispersion-strengthened alloys against irradiation with heavy ions of fission fragment energy”). Authors would like to thank the IC-100, U-400 and DC-60 accelerator staff of the FLNR JINR and INP.*

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## MECHANICAL STRESSES IN Si<sub>3</sub>N<sub>4</sub> IRRADIATED WITH HIGH ENERGY HEAVY IONS

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Irradiation with heavy ions of 1-3 MeV/nucleon energies is characterized by pronounced inhomogeneous ionization and nuclear stopping profiles. As a result, the level of energy losses varies over a very wide range, which in own turn leads to a inhomogeneous spatial distribution of radiation damage and associated mechanical stresses. The range of ions with the above energies, depending on the density of the material, does not exceed several tens of microns. For energies of ~ 1 MeV/nucleon, which are of the greatest interest from a practical point of view for simulation of the fission fragments impact, this value is in the range from several microns to ~ 10 microns. Therefore, to get reliable information about stress profiles, it is necessary to use experimental methods with a spatial resolution of ~ 1 micron. Such accuracy can be achieved in techniques based on the use of the piezospectroscopic effect), which connect the spectral shift in optical absorption, luminescence, or Raman scattering spectra with the level of mechanical stresses [1,2].

In this work depth-resolved Raman spectroscopy technique was used to study the residual stress profiles in polycrystalline silicon nitride irradiated with Xe (167 MeV,  $1 \times 10^{11} \text{ cm}^{-2} \div 4.87 \times 10^{13} \text{ cm}^{-2}$ ) and Bi (710 MeV,  $1 \times 10^{11} \text{ cm}^{-2} \div 1 \times 10^{13} \text{ cm}^{-2}$ ) ions. It was shown that both compressive and tensile stress fields are formed in the irradiated specimen, separated by a buffer zone located at a depth coinciding with the thickness of layer, amorphized due to multiple overlapping of the track regions. Compressive stresses are registered in subsurface region, while at a greater depth, the tensile stresses are recorded and their level of reaches the maximum value in the end of ion range. The size of the amorphous layer was evaluated from the dose dependence of the FWHM of the dominant  $204 \text{ cm}^{-1}$  line in Raman spectra and scanning electron microscopy.

*The work was supported by grant of the Ministry of Education and Science of the Republic of Kazakhstan AP 08856368 «Radiation resistance of nitrides and carbides based ceramics against of impact of heavy ions with fission fragments energies».*

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- crystal

## MECHANISMS RESPONSIBLE FOR THE DEPTH DEPENDENT CRYSTAL ROTATION OBSERVED IN NiO EXPOSED TO SWIFT HEAVY ION IRRADIATION

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Exposure to swift heavy ion irradiation (SHI) is known to induce material specific changes in the microstructure. Various models are used to predict damage formation and explain these microstructural changes. Certain materials (such as NiO) have been seen to exhibit significant crystal rotation when exposed to SHI irradiation at an off normal incidence. To date, very little published data is available regarding SHI induced rotation, with only bulk sampling techniques such as x-ray diffraction spectra and surface shifts having been employed [1, 2]. Without electron microscopy investigations, nothing can be definitively said about the microstructural changes facilitating this effect.

In this study, the microstructural changes in single crystal NiO were studied. These crystals were irradiated with Au ions at 45° with energies of 593 MeV and 940 MeV. Fluences ranged from  $1.4 \times 10^{14}$  to  $9.6 \times 10^{14}$  ions/cm<sup>2</sup>. A depth dependent investigation of the crystal rotation was performed using electron backscatter diffraction (EBSD). In-plane rotation varied from 20.2° – 51.6°. High-resolution transmission electron microscopy (HRTEM) was utilized to interrogate the microstructural changes facilitating bulk rotation. No amorphization was observed and the specimen remained single crystalline despite the depth dependent rotation. The crystal rotation seen thus needs to be facilitated by an advanced network of dislocation cells.

TEM imaging revealed a dense network of dislocations along the primary slip directions of the material. The dislocations at high fluences ( $\sim 10^{14}$  ions/cm<sup>2</sup>) are seen to form cells of approximately 30 nm diameter, which seems to resemble mosaic Low Energy Dislocation Structures (LEDS). A different mechanism of ion hammering is thus considered.

*The authors would like to acknowledge Siegfried Klaumünzer for the provision of the NiO specimens used in this study as well as all his valuable discussions.*

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## MICROSTRUCTURAL RESPONSE OF SILICON NITRIDE TO FISSION FRAGMENTS

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$\text{Si}_3\text{N}_4$  is not only an important semiconductor material but is also under consideration for use as candidate-inert-matrix-fuel-hosts (IMs) for the burn-up of plutonium and minor actinides [1,2]. IMs are envisioned as one possible means to close the nuclear fuel cycle in an effort to reduce the amount of high-level waste materials which require long-term storage [3]. The physical properties of this material make it well suited to reactor conditions [2]. However, to prove the viability for nuclear applications its' radiation stability must be tested. In this investigation swift heavy ions are therefore used to simulate the effects of fission fragments on microstructure of  $\text{Si}_3\text{N}_4$ .

To assess the behaviour of different phases of  $\text{Si}_3\text{N}_4$  under irradiation, polycrystalline (Al doped) bulk samples, amorphous thin films and nanoparticles were irradiated with ions of varying stopping power. The samples were irradiated with Xe (167, 220, 480 MeV) and 710 MeV Bi ions. Selected samples were also irradiated with 220 MeV Xe ions at temperatures ranging from LNT to 1000 K. The microstructural effects of swift heavy ions on these materials were analysed using transmission electron microscopy techniques.

The effect of fission fragments on the microstructure of  $\text{Si}_3\text{N}_4$ , through electronic energy deposition processes, and the formation of latent tracks in crystalline, amorphous and radiation-induced-amorphous phases of silicon nitride will be presented.

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## **VII. Facilities and techniques of experiment**

**The Seminar Program traditionally includes a methodological section. Its purpose is to acquaint the attendees with the latest methodological developments in the sphere of radiation physics and radiation material science, and inform them about new radiation sources and application of the new methods for condensed matter investigation.**



## FORMULATION OF RADIATION MATERIALS SCIENCE PROBLEMS FOR AUTOMATED EXPERIMENTS ON THE WORLD'S FIRST SATELLITE PROBE MICROSCOPE

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The development of modern space systems faces the need to develop new experimental methods for assessing the radiation resistance of structural materials directly in space, since the exposure of individual samples of materials in space [1] for their subsequent study on Earth cannot provide continuous monitoring and cannot provide sufficient data due to the complexity and high cost of accurately returning samples from space. In 2020, we developed a new method – the production of experimental equipment in the form of Earth satellites. This was a new worldwide, patentable and surprisingly inexpensive solution due to the launch of mass production and the cost at the same time of 2-3 million rubles of the basic part of the satellites, in which we plan to place both samples of materials and various devices that study them, sending data to Earth for many years of work in orbits. Vacuum and various radiation fields of outer space are a pleasantly free natural application here.

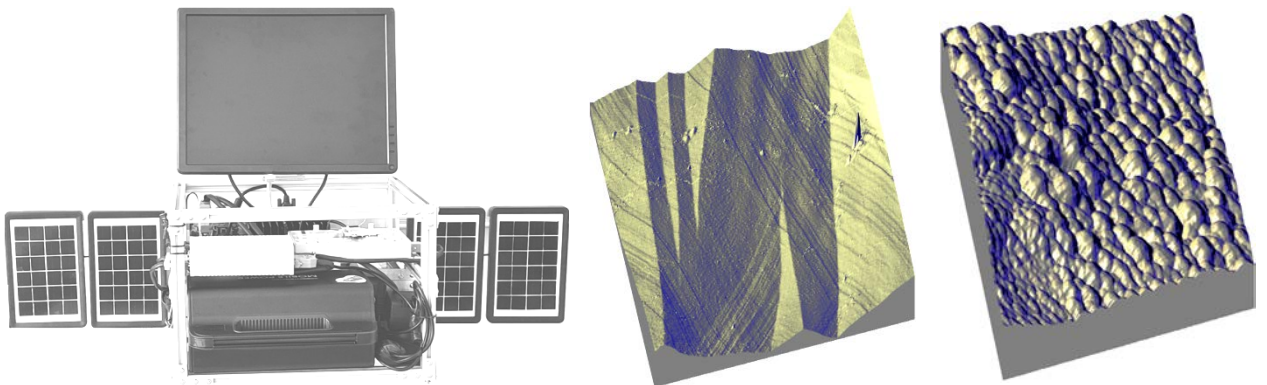


Fig.1. A working prototype of the world's first probe microscope-satellite of the Earth (left) and the obtained scans of 8x8 microns of the bismuth surface before (center) and after (right) impacts with solar wind parameters of ion flow velocities up to 200 km/s..

More than 30 years of experience [2] in the design of radiation-resistant probe microscopes with a resolution of up to 1 nm allowed us to create the first such laboratory (Fig.1) – a satellite in the form of the world's first probe microscope, the launch of which is planned for the summer of 2022. A number of organizations, including the military Academy of Communications named after budennogo, several universities and institutes of the Russian Academy of Sciences have begun to prepare actual samples [3], for example, graphene [4], bismuth and piezoceramics, but it seems significant that organizations of the Russian nuclear industry that have advanced groundwork in radiation materials science also participate in this.

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## **GLOVEBOXES AND GLOVEBOX LINES «SPECS GB»: MADE IN RUSSIA LABORATORY EQUIPMENT FOR LITHIUM BATTERY RESEARCH, DEVELOPMENT & PRODUCTION**

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Spectroscopic system's highly qualified, experienced and certified engineers deliver complex and diverse inertgas system solutions. Our combined process experience and proven solution capabilities enable our Russian customer base to meet the demands of today's emerging technologies.

Most commonly the presence of moisture and oxygen are the principal air components which shall be excluded from the process environment for lithium battery research, development & production. Therefore technologies are required which allow running processes under oxygen and moisture free conditions. Two predominant techniques have been established to address this specific problem – vacuum technology and inertgas technology. Whilst vacuum bases on the principle of creating nearly gas free environments by evacuating rigid structures to pressures less than 10<sup>-9</sup> mbar, the inertgas technology selectively removes harmful components from the air resulting in a completely oxygen and moisture free working environment at ambient pressure. The main benefit between both approaches is that inertgas technology is compatible with all kinds of equipment, processes, and system enclosure sizes offering full auto and manual access at a fraction of the costs of normal vacuum systems. In case of the vacuum technology many tools are not vacuum proof, solvent containing materials cannot be processed.

Since its foundation in 2004 JSC «Spectroscopic systems» has been able to offer its comprehensive product portfolio of interdisciplinary systems which combine vacuum technology as well as inertgas technology. The core element of a well-designed inertgas-system is the «SPECS GB» gas purifier. This unit is a closed-loop system with integrated, fully regenerable scrubber units which selectively remove moisture, oxygen and solvents from an inertgas stream. Most commonly nitrogen, argon or helium is used as the process gas.



Specs GB systems are primarily used for the standard research and development of lithium ion battery technologies and our custom enclosures are used for critical dry production environments necessary for battery manufacturing. Boxes can help in maintaining a minimum-humidity atmosphere that will neither harm operators nor be disrupted by them. Our glovebox systems and custom enclosures also allow the ability to record and trace the production environment for enhanced quality control. Connected to the purifier is a gas-tight, hermetically sealed enclosure called a glovebox. These system come either in standardized sizes mainly used

for research and development or in customized designs for industrial use.

## PROFILOMETRY AND CONTUROGRAPHY OF NONLINEAR SURFACES OF NUCLEAR AND THERMONUCLEAR EQUIPMENT ELEMENTS

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The modern development of nuclear power requires more and more precise manufacturing of plant parts, as well as control of changes in the shape and structure of the surface of parts during operation. Monitoring of exposed surfaces of materials allows you to identify coatings and materials with the longest service life. A well-developed and standardized method for determining the quality of the surfaces of a part and studying the structure of surfaces is the profilometry method, which consists in "feeling" the surface with a diamond probe with a tip radius of about 1 mkm along the line, followed by displaying the profile with an accuracy of 1nm vertically and calculating the roughness parameters. However, profilometers manufactured in the world are not adapted to roughness measurements of concave and curved surfaces specific to the nuclear industry. Being the only manufacturers of profilometers in Russia (model 130) [1], our organization at the request of the RFNC-VNIITF (Snezhinsk) in 2021 developed and tested for this purpose fundamentally new designs of vertical type sensors for such tasks, which makes it possible to significantly expand the scope of profilometers for nuclear industry tasks.

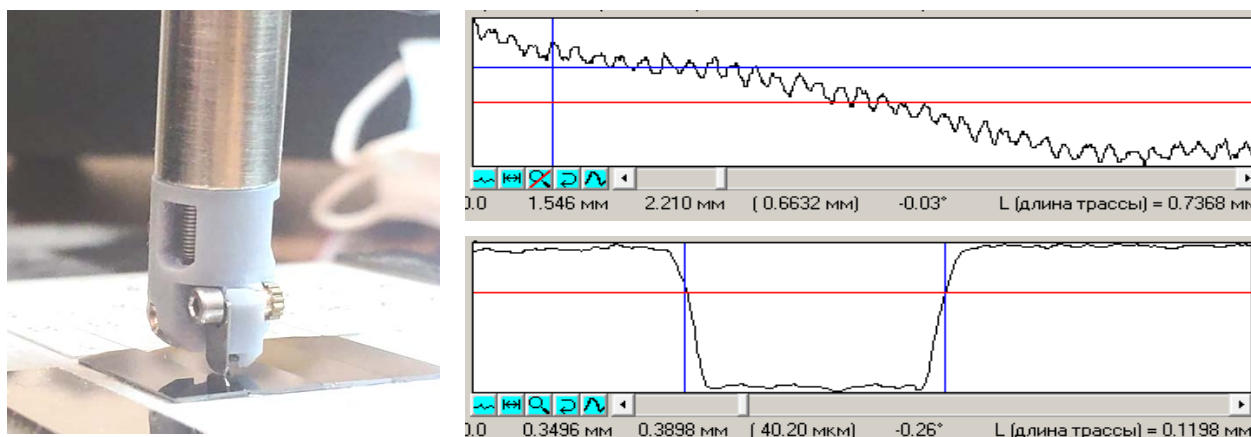


Рис. 1. A working prototype of the world's first vertical sensor for measuring surface roughness (left), as well as the resulting profiles with the possibility of digitizing each surface defect with the calculation of their geometry.

To control the geometry of parts with an accuracy of up to 1 mkm with a size of parts up to 1 meter, especially with their complex shape, we have also developed and patented [2] Russia's first conturograph (model 220) - a device for measuring the contour of the surface and digitizing geometric dimensions [3] - edge radii, angles, step heights, thread parameters and other dimensions. This device has been successfully tested in almost 20 organizations of various industries, including «Tochmash» (Vladimir) and «Mayak» (Ozersk), and has the potential for use in the nuclear industry.

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**PROMICES IN STUDIES OF STRUCTURAL REACTOR MATERIALS ON  
THE PREMICES OF NEUTRON MATERIAL SCIENCE CENTER  
IN IMP UB RAS**

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The scope of radiation-induced phenomena in structural reactor materials that scale from the crystal lattice parameter to macroscopic sample's dimensions covers formation of nanoclusters, precipitates, dislocations, grids of internal stresses, voids, as well as changes in the grain structure and radiation-induced swelling and creep of material. Hence, rather a complete research must be multi-scale and based on a combined usage of different experimental techniques that take their "ecological niches". On a nanoscale, important results are gained by electron microscopy, Messbauer, atom-probe, and positron-annihilation spectroscopies, and low-angle neutron scattering. To reveal diffraction effects, neutron and X-ray analyses are to be employed. Of use can be magnetic measurements. Going further, macroscopic properties will be under study.

Using neutron diffraction, one can first of all determine crystallographic parameters of materials, such as crystal system and symmetry group, lattice parameters, occupancies of crystallographic sites. Moreover, such information can be supplemented by important peculiarities of microstructural state reflecting imperfection of crystal lattice that manifest themselves in the diffraction spectrum. Modern techniques of neutron diffraction analysis make it possible to determine from the experimental data the presence and parameters of crystallographic texture, size of regions of coherent scattering, and value and anisotropy of internal lattice microstrains given rise by microstresses generating near both dislocations and precipitates. Certainly, neutron diffraction patterns present evidence of changes in the phase content of material, providing possibility of analyzing microstructural state of individual phases. Such an analysis is performed using well-elaborated program packages, which are under continues development.

Neutron materials science center of IMP UB RAS (NMC IMP) possesses facilities and unique experience of working with highly irradiated structural materials, including those after real operation in industrial fast neuron reactors. The NMC IMP includes three neutron diffractometer, vibrating sample magnetometer, X-ray diffractometer, and Mossbauer spectrometer designed for studies of irradiated samples..

The most promising way of improving further studies of the materials specified, in our opinion, is development of scientific cooperation with the JSC "Institute of reactor materials" to consolidate the experimental facilities for solving the tasks of radiation physics.

*The work is performed within State assignment of the Ministry of Education and Science of the Russian Federation, (topic «Flux»).*

## REVEALING HIDDEN DEFECTS THROUGH STORED ENERGY MEASUREMENTS OF RADIATION DAMAGE

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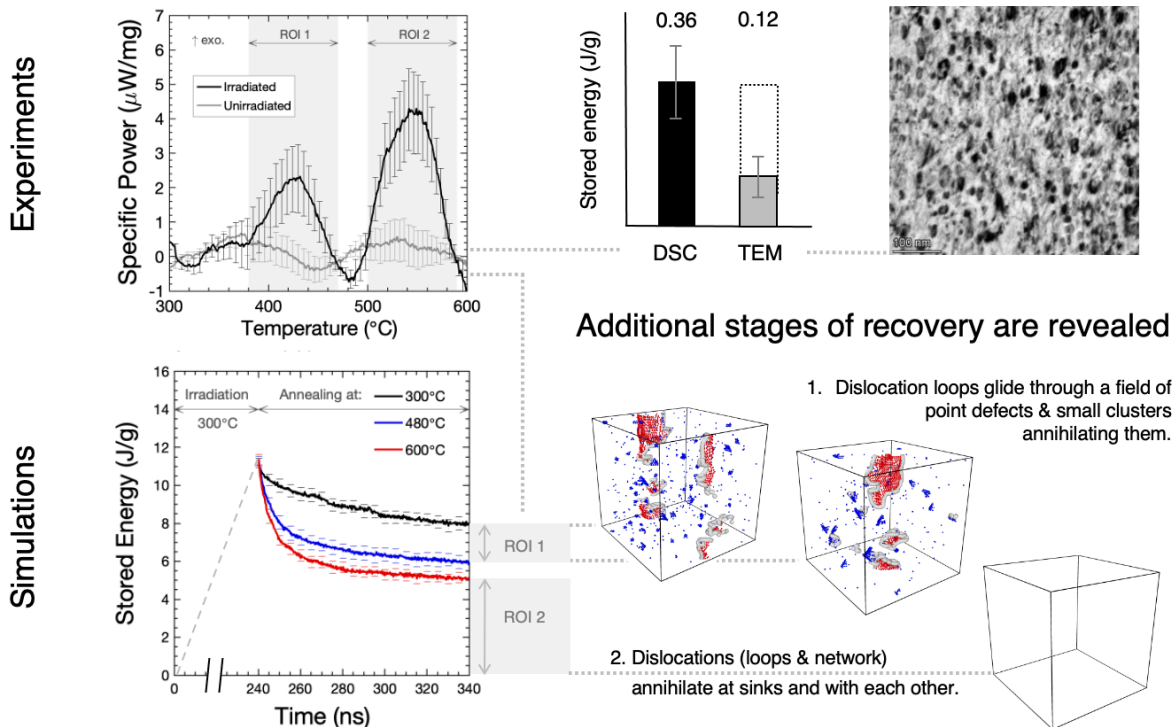
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With full knowledge of a material's atomistic structure, it is possible to predict any macroscopic property of interest. In practice, this is hindered by limitations of the chosen characterization techniques. For example, electron microscopy is unable to detect the smallest and most numerous defects in irradiated materials. Instead of spatial characterization, we propose to detect and quantify defects through their excess energy. Differential scanning calorimetry (DSC) of irradiated Ti measures defect densities 3 times greater than those determined using transmission electron microscopy. Our experiments also reveal two energetically-distinct processes where the established annealing model predicts one. Molecular dynamics (MD) simulations discover the defects responsible and inform a new mechanism for the recovery of irradiation-induced defects. The combination of annealing experiments and simulations can reveal defects hidden to other characterization techniques, and it has the potential to uncover new mechanisms behind the evolution of defects in materials.

### Annealing radiation damage

### Stored energy measures hidden defects



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