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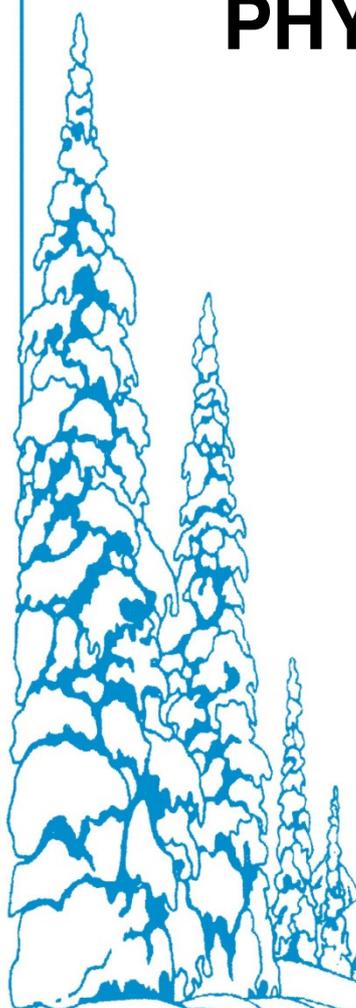
Тезисы докладов

The Eleventh International Ural Seminar

RADIATION DAMAGE PHYSICS OF METALS AND ALLOYS

February 23 – March 1

Abstracts



Кыштым
Россия

Kyshtym
Russia

2015

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OF METALS AND ALLOYS**

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**Kyshtym, Russia
2015**

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**Compiled by
Denis Perminov**

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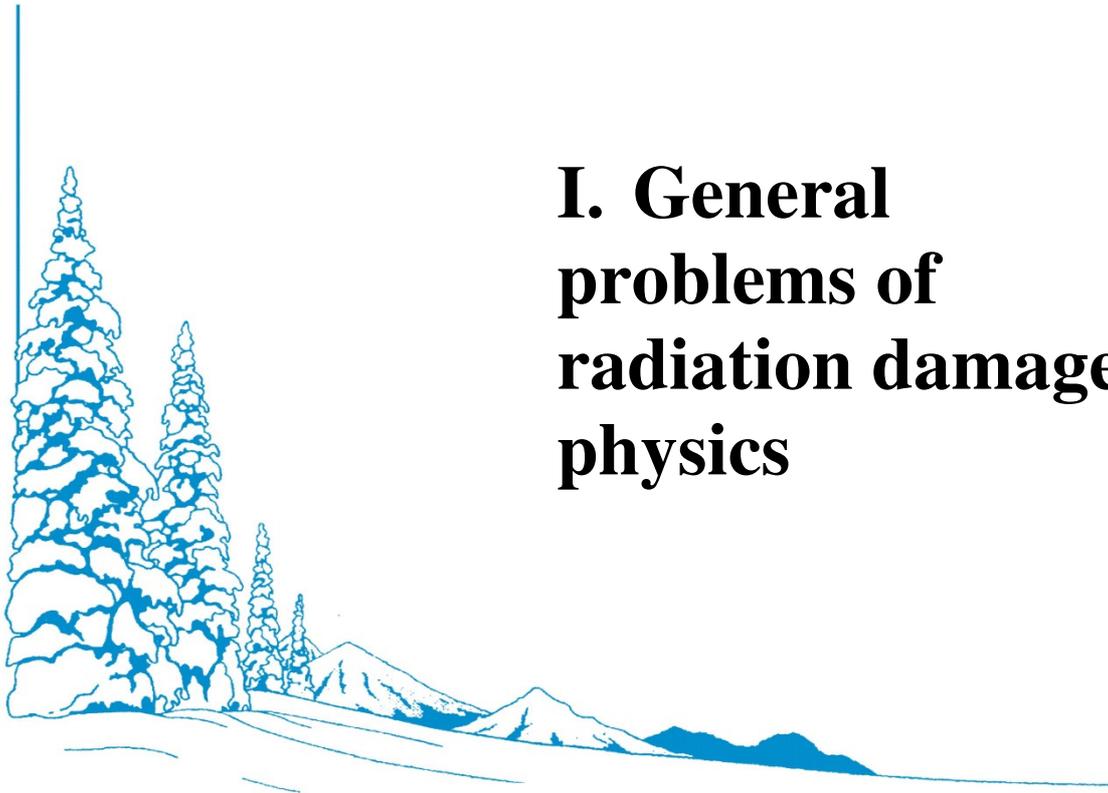
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I. General problems of radiation damage physics

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.

INTEFACES IN NANOMATERIALS AS EFFECTIVE SINKS FOR IRRADIATION DEFECTS

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By now there are numerous results which are evidence of the better irradiation stability of nanomaterials-based metals, alloys and compounds, as compared with their coarse-grained those. These results relate both irradiation by ions at accelerators and reactor tests (those are still not so large in number). The results obtained by the ionic irradiation of the multilayered films, such as Cu/V, Cu/Nb, Cu/W, W/ZrO₂, Ta/Ti, Ag/Ni, CrN/AlTiN, and others, are the most representative. Changing the layers number at constant film thickness, one can easily study the interface numbers (i.e. sinks) influence of the irradiated films characteristics such as hardening, swelling and reservation of lamination.

The nanotwinned boundary effect in the irradiation defect mowing away has been observed in FCC metals using high-resolution transmission electron microscopy. In this connection some technology methods in the nanotwinned materials fabrication are discussed. The data of the irradiation defect behavior simulation using molecular dynamic are also generalized.

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DEFORMATION MICROSTRUCTURE AND SEPARATION OF RADIATION DEFECTS IN NICKEL

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Structural steel work of nuclear reactors over a wide temperature range of 300-1000 K and at high doses damaging places high demands on their resistance. Nickel is a model material for the construction of austenitic metals and alloys.

Previously we have experimentally observed the effect of the separation of radiation defects in nickel under electron and neutron irradiation, which is that a significant portion of generated by irradiation self-interstitials in deformed nickel are annihilated when migrating to the dislocation sinks and does not participate in recombination with vacancies [1]. As a result, there is an accumulation of vacancies, the concentration of which exceeds substantially in deformed nickel in comparison with the quenched nickel.

We used pure, annealed at 1173 K nickel deformed by rolling. The degree of deformation ranged 10-90%. To remove deformation vacancies samples were annealed at 450 K. The 5 MeV electron irradiation in an atmosphere of purified helium were carried out at a temperature of about 320 K, when vacancies are immobile and interstitial free to migrate to the sinks.

In a deformed nickel it is formed the subgrain structure. Cell size varies depending on the degree of deformation, from 700 nm to 200 nm. With increasing degree of deformation of the

cell walls become more dense, more narrow, the density of dislocations therein varies from 2 to $10 \times 10^{10} \text{ cm}^{-2}$, increases the misorientation between cells and microstresses. The dislocation density in the cells is within $(1-4) \times 10^{10} \text{ cm}^{-2}$. It is shown that the increase in electrical resistivity when irradiated in the deformed nickel compared to quenched nickel depend on the degree of deformation of non-monotonic. Maximum growth is observed at a degree of deformation of about 40%. Depending on the electrical resistivity growth with increasing doses approaching the quasistationary level.

In deformed and annealed at 450 K nickel it is formed two types of point defect sinks - subgrain boundaries and dislocation structure in the body of subgrain. Kinetics of post-irradiation annealing of vacancies is determined, as well as for interstitial atoms during irradiation, sinks of point defects. When analyzing the changes in the electrical resistivity curves for isochronal annealing is shown that there is a dependence of the peak position of annealing on the degree of deformation, which corresponds to the length of the free migration of vacancies to sinks with increasing degree of deformation. The correlation between the power of point defect sinks and effect of separation of radiation defects is discussed. Regularities observed separation of radiation defects on the degree of deformation indicate that this phenomenon must be considered when predicting radiation damage existing and future structural materials for nuclear energy.

This work was performed within the framework of the “Spin” Theme of the Russian Academy of Sciences, no. 01201463330. It was partially supported by the Ural Branch of the Russian Academy of Sciences (project no. 13_2_007 YaTs), Russian Foundation for Basic Research (project nos. 13-02-00321 and 14-02-00150).

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THE EFFECTS OF IMPURITIES ON KINETIC CHARACTERISTICS OF RADIATION DAMAGE IN IRON AND STEELS

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Using of previously developed methodology for characteristics of radiation defects (RD) and radiation – enhanced diffusion (RED) calculation in metals with impurities and grain boundaries [1, 2] the effects of impurities trapping on kinetic characteristics of RD and RED as well as on parameters of void nucleation and growth processes have been examined

For iron and austenitic steels the next some approximate estimates have been carried out: the temperature dependences of RD concentration for materials with variable binding energies of impurities complexes; the effective recombination coefficients or RD for materials with impurities complexes; the dependences of defect recombining fraction on dose rate; the effects of impurity trapping on the free energy of void nucleations and void growth rates in volume and grain boundaries [3]. The possibility the effect of diversification of impurities – defects complexes characteristics on the suppression of radiation swelling are analyzed.

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EFFECT OF PHOSPHORUS ON VACANCY-TYPE DEFECT EVOLUTION IN ELECTRON-IRRADIATED NI STUDIED BY POSITRON ANNIHILATION

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Very dilute Ni-P system (containing 50-240 appm phosphorus) irradiated by 5 MeV electrons at various temperatures (270-543 K) was studied by positron annihilation spectroscopy (PAS) and the electrical resistivity measurements [1]. Under irradiation at 270 K (below stage III in Ni), the accumulation of the monovacancies in the Ni-P system is 1.5-2.0 times greater than that in pure Ni irradiated in the same condition. This fact attests to the strong interaction between P atoms and self-interstitial atoms (SIAs). As a result of the non-mobile SIA-P complexes formation, the mutual recombination of point defects is suppressed and the vacancy accumulation is, respectively, enhanced. During post-irradiation annealing, the vacancy migration induces transport process of the phosphorus atoms and leads to the formation of the vacancy clusters decorated with P atoms. The annealing behaviour of the defect structures in Ni-P systems after irradiation at enhanced temperatures was also studied. The influence of phosphorus on the formation and further evolution of the vacancy aggregates decrease with increasing of the irradiation temperature.

This work was done within programme "Spin" (No 012001463330) with partial support of Russian Foundation for Basic Research (Project No 13-02-00321-a)

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THE INFLUENCE OF IRRADIATION AND THERMAL TREATMENT ON (SnSe)_{1-x}(PrSe)_x SOLID SOLUTIONS

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We studied the influence of irradiation and heat treatment after irradiation on several kinetic coefficients of solid solutions (SnSe)_{0.75}(PrSe)_{0.25}. Prior to irradiation, the resistivity of the sample $\rho(T)$ slightly decreases depending on T in the temperature range studied (77-120 K), passing through an extremum at $T=100$ K. After irradiation with γ - quanta with the dose $D = 35$ kGr, $\rho(T)$ remarkably increases. The relative change of the resistivity, thermo emf, and heat conductivity makes up at $T=100$ K 35, 60, and 13 %, respectively, while at 120 K, 80, 80, and 25 %. After heat treatment at 620⁰ C for three days and nights in the samples there take place recovering processes and $\rho(T)$ approaches the value $\rho_0(T)$. After the heat treatment of the crystal at $T=100$ K, a small residual thermo emf of 20 % appears, whereas, at $T= 120$ K it amounts to 25 %.

OCCURRENCE OF DEFECTS IN SOLID ALLOYS Tb_xSn_{1-x}Se IN THE γ -IRRADIATION

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Several electrophysical properties of single crystals of the alloy system (SnSe)_{1-x}(TbSe)_x have been studied in a wide temperature range (250-520 K) prior and after irradiation, and analysis of the impact of γ - irradiation on these properties has been performed. The samples were irradiated with γ -rays 1,25 Mev (⁶⁰Co) in energy and $D=65$ Mrad. Analysis of the temperature dependences of Hall mobility of charge carriers showed that in the sample Tb_{0,01}Sn_{0,99} the mobility of the charge carriers prior to irradiation increases with temperature by the law $\mu \propto T^{1.5}$, while after irradiation, - $\mu \propto T^{2.0}$. This means that the main mechanism of scattering the charge carriers is scattering on the charged impurity centers, which after irradiation enhances. In the sample Tb_{0,05}Sn_{0,95}Se, where the terbium content is enhanced, the dependence $\mu(T)$ is the same prior and after irradiation and obeys the law $\mu \propto T^{0.8}$, which corresponds to the scattering on weakly charged impurity centers.

MOLECULAR DYNAMICS SIMULATION OF ATOMIC DISPLACEMENT CASCADES NEAR SYMMETRICAL TILTED GRAIN BOUNDARIES IN ZIRCONIUM

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Zirconium has a good mix of nuclear-physical characteristics and mechanical properties. Zirconium alloys are widely used as structural material for elements of fuel assemblies and fuel-element cladding in nuclear power plants. This work considers atomistic models of symmetrical twin grain boundaries (GBs) in HCP-Zirconium and molecular dynamics simulation of atomic displacement cascades near these borders.

Many-body interatomic potential proposed by Mendeleev and Ackland [1] is used in calculations. Twin boundaries $\Sigma 32(23 \bar{4}6 23 27)$ and $\Sigma 32(\bar{1} 2 \bar{1} 3)$ with the axis of rotation $[\bar{1} 0 \bar{1} 0]$ and boundaries $\Sigma 14(4 \bar{5} 1 0)$ and $\Sigma 14(2 1 \bar{3} 0)$ with the axis of rotation $[0 0 0 1]$ are considered. Periodic boundary conditions perpendicular to the GBs plane are used during the simulation. The boundary which is parallel to the GBs plane is free. The grain boundary width, free and grain boundary specific energy estimations are obtained.

A simulation of atomic displacement cascades near GBs is performed. The initial temperature of the bicrystal model is 300 K, the primary knock-on atom (PKA) energy is 10 keV, the PKA primary momentum is always directed to GBs along the normal to the boundary plane, the distance between PKA and GBs is about 20-25 Å. Eight cascades were modeled for each boundary type. Qualitative analysis of the simulation results allows us to conclude that point defects (primarily self-interstitial atoms) tend to accumulate near GBs. A similar effect for metals with BCC and FCC lattices was previously described in works of other researchers. Quantitative estimates of HCP-Zirconium crystal structure damage are obtained.

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EQUILIBRIUM THERMODYNAMICS OF HELIUM IN δ -PHASE Pu–Ga ALLOYS

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The paper presents a theoretical study of the response of δ -phase Pu–Ga alloys to self-irradiation. We investigate the long-term behaviour of He atoms in the face-centred cubic (fcc) lattice under ambient conditions by means of classical molecular dynamics. Because of the relatively low diffusive mobility of He atoms in the lattice their dynamics cannot be tracked directly in molecular dynamics simulations. Instead, we use the Helmholtz free energy function to investigate the equilibrium thermodynamics of metastable microconfigurations of perfect crystals with artificially introduced He atoms in different configurations. The Helmholtz free energy of the microconfigurations are calculated using the thermodynamic integration method. Based on the free energy evaluation, inferences about the relative thermodynamic stability of various microconfigurations under ambient conditions are drawn. Estimates of the equilibrium parameters of He bubbles in the lattice and the distribution of He bubble radii are calculated and compared to those observed experimentally.

KINETIC MODEL OF RADIATION-INDUCED AGING IN ALLOYSV.A. Khlebnikov¹, A.Yu. Kuksin¹, A.V. Yanilkin¹¹ *All-Russia Research Institute of Automatics, Moscow, Russian Federation*
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We develop a physical model of point-like defect diffusion in multi-component alloy with pair interactions between atoms and apply it into free distributed parallel kinetic Monte-Carlo simulator on rigid lattice named SPPARKS. Thermal activated transitions of atoms to vacant sites (both for on-lattice and interstitial atoms) as well as spontaneous formation of Frenkel pairs due to external radioactivity are possible classes of elementary events. The activation energy of a particular thermal process (and thus its probability/rate) depends on the atoms neighborhood. The precipitation dynamics, the pores and dislocation loops growth can be described in the framework of our model. The pair interaction energies are expressed through such quantities as the cohesive energy, the mixing energy and different binding energies which are gathered from ab initio calculations or partially are taken from experiments. Numerical simulations allow to reveal the impact of different parameters (energies, temperature, impurities concentration and irradiation intensity) on the diffusion process, the inhomogeneities growth, the variations in the local alloy composition. Working in parallel mode, the program reproduces the results of lengthy (few hours) experiments on the steels annealing, describes the accumulation of radiation damage up to hundredths of dpa.

ATOMIC SCALE INVESTIGATION OF PHASE DECOMPOSITION OF Fe-22% Cr DURING THERMAL AGING AND SUBSEQUENT HEAVY ION IRRADIATION

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Understanding of damaging and degradation processes along with correlation between atom-scale alteration and macroscopic properties of solid state materials is of very importance being a key topic in modern material science. One of the focuses in this field is binary systems based on iron, namely Fe-Cr alloy. Its phase diagram is still a field of ongoing debates, which originates from the affinity of iron and chromium and magnetic nature of both of them. In case of Cr concentration higher than 10% decomposition of solid solution occurs.

This work is devoted to understanding of the kinetics of such a process in a Fe-22%wt.Cr alloy by means of atom probe tomography. The latter was used to quantitatively describe the nucleation and growth processes at atomic scale as well as stability of formed phases under cascade forming irradiation. Material was heat treated up to 1200 h at 500 °C and some of the samples were afterwards irradiated by heavy ions up to 1 dpa. It was shown that the formation of α' phase as well as behavior of oversaturated solid solution defies the classical law of coalescence theory of Lifshitz-Slezov.

**ATOMISTIC SIMULATIONS OF SUBSTITUTIONAL ELEMENTS
SEGREGATION AT GRAIN BOUNDARIES OF VARIOUS TYPES**

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Segregation of alloying elements at grain boundaries (GBs) has a significant impact on physical and mechanical properties of alloys, phase transitions, and on the formation of a microstructure. Although this problem is examined theoretically and experimentally for many years, the mechanisms of interaction of alloying elements with GBs and segregations formation remain subjects of debate. In particular, you can not select one or more parameters, depending on the characteristics of GBs (misorientation angle, free atomic volume, the energy of the GB), or on the type of impurity atom (ion radius, the energy of dissolution), which determine essentially the segregation effect on GBs.

We examined the mechanism of segregations formation at GBs of different types, using the approaches of different scale levels: the calculation of segregation energy within density functional theory (PAW-VASP), molecular dynamics (MD) method, and Monte Carlo (MC) method of thermodynamic simulation. As the object of the study, Al alloys, alloyed with Mg, Si or Ti, were used. The results obtained give an idea not only of the magnitude of the segregation energy, which determines the driving force of the process, but also on the width of the boundary area L_{GB} , which is enriched by impurity elements.

The calculations results revealed the role of the various contributions to the interaction impurity-GB. It is shown that for *sp* elements, arranged in the periodic table to the right of Al, the presence of additional valence electrons leads to increased chemical bonding in areas with broken atomic coordination, which makes the dominant contribution to the interaction with the GB. For other alloying elements, deformation interaction is dominant. Wherein substitution of Al at the GB by the atom with a smaller radius (Ti) leads to a loss in energy, while moving on the GB larger atom (Mg) is energetically favorable. It is shown that for the general type GB, containing structural defects, the width L_{GB} of the region of intensive impurity-GB interaction is significantly greater than in the case of symmetric special GB. Since the segregation energy shows strong oscillations along the GB, it is proposed to describe the interaction impurity-GB with effective quantity which characterizes the ability of GB segregation in general.

To study the kinetics of segregation formation at GBs of various types, we conducted atomistic simulations, using an approach that combines Monte Carlo and molecular dynamics (MD + MK). For Al-Mg system we found that the enrichment and distribution profile of the GB concentrations are in good agreement with experiment. Thus, to obtain a correct description of segregations formation, one must consider not only the interaction of dissolved elements with the GB, but also among themselves. The results help to explain the observed features of GBs enrichment in alloys subjected to severe plastic deformation.

This work was supported by RNF (project 14-12-00673).

PROCESSES OF ATOMIC REDISTRIBUTION UNDER IRRADIATION- AND DEFORMATION INDUCED DISSOLUTION OF INTERMETALICS IN Fe-Ni-Si ALLOY

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Method of Mossbauer spectroscopy was used to study dissolution of disperse intermetallics Ni₃Si in the FCC matrix of the aging alloy FeNi₃₄Si₃ at 340 K in the displacement cascades of neutrons, and a comparison was made with the dissolution of intermetallics upon cold megaplastic deformation (with different rate and degree). The neutron-induced dissolution of intermetallics Ni₃Si is found to be different from the separation in binary FCC Fe-Ni alloys, radiation-accelerated with electrons. A weakening is established of the kinetics of the process of formation of intermetallics, which is accelerated by high-energy particles, in the alloy Fe-Ni-Si compared to the alloys Fe-Ni-Ti(Al). A similar lowering of the relaxation component of aging upon irradiation with neutrons raceable to a low diffusion mobility of silicon atoms causes intense deformation and radiation-induced dissolution of intermetallics Ni₃Si.

KINETICS OF PHASE TRANSFORMATION DURING PLASTIC DEFORMATION OF 12Cr18Ni10Ti AND AISI 304 NEUTRON-IRRADIATED METASTABLE STEELS.

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Deformation induced martensitic $\gamma \rightarrow \alpha'$ -transformation effects the considerable influence on parameters of strength and plasticity of metastable austenitic steels. It is that, under the influence of stress in the course of plastic deformation fcc lattice of austenite is transformed to bcc lattice of α' -martensite. Formation of martensitic phase can increase plasticity and power consumption of material because martensitic phase is stronger than austenite phase.

Cylindrical stainless steel samples were austenitized (1050°C, 30 minutes) and irradiated in WWR-K nuclear research reactor core at <80°C. Fluencies were up to $1.3 \cdot 10^{20}$ n/cm². Mechanical tensile tests were conducted with strain rate 0.5 mm/min at room temperature. During plastic deformation amount of martensitic α' -phase, induced in the sample and sample's geometry changes were registered.

As a result of experiments mechanical and power characteristics were received, values of the stacking fault energy (SFE) were calculated for each material. Charts of martensitic α' -phases accumulation in coordinates "martensitic α' -phase – "true" stress", "martensitic α' -phase — "true" local strain" and "martensitic α' -phase – density of energy of deformation" were constructed.

With use of the equations received in [1, 2] approximation of charts of martensitic α' -phases accumulation were made. The parameters characterizing kinetics of martensitic transformation were determined. The interrelation of stacking fault energy and kinetics of martensitic transformation is analyzed.

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EVOLUTION OF SURFACE RELIEF UNDER DIFFUSIVE FLUX OF POINT DEFECTS

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In metals and alloys point defects are formed under irradiation, it can affect properties of materials significantly, causing swelling, embrittlement and creep [1]. Besides that, diffusion of point defects to the free surface can alter its relief. Taking this effect into account can be important for detailed prediction of evolution of materials properties under irradiation, which could be important for some technological applications.

By the means of kinetic Monte-Carlo, coupled with classical molecular dynamics, we simulate evolution of the free surface relief under diffusive flux of interstitials.

In the first part of our work separately interstitial and vacancy fluxes were considered. It is shown, that in the case of interstitials flux domination surface serration tends to flatten. On the other hand, dominance of the vacancy flux to the surface leads to increasing serration. At different vacancy generation rates different regimes of jogs formation were observed: from dendrite like structures to pits.

Swelling of the material under irradiation leads to appearance of strain gradients near the surface serrations. As far as stress affects point defects diffusion coefficients [2], in the second part of the work we developed our model to consider the influence of stress field on diffusion properties of vacancies and interstitials.

For this purpose we calculated pressure dependence of point defects formation enthalpy by the means of classical molecular dynamics. Based on this data defect migration energy is corrected with respect to the local elastic field. The proposed model allows simulate spatial dependence of diffusion coefficients. It is shown, that taking into account the effect of stress can significantly affect the surface modification regime.

The results, obtained in the work, can be used for qualitative estimation of the free surface relief evolution of metallic materials under irradiation.

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COMPARATIVE STUDIES OF ION BOMBARDMENT AND SHOCK-WAVE LOADING EFFECT ON THE STRUCTURE OF ALUMINUM ALLOYS

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Dynamic long-range effects occurring under the particle bombardment of condensed matter can be explained by the hypothesis on emission and subsequent propagation of post-cascade shock waves in material [1]. In order to confirm the hypothesis, we have performed comparative experiments to reveal the effects of ion-beam exposure and mechanical shock loading on the properties of the alloys of the Al–Cu–Mg system (using experimental stands at Russian Federal Nuclear Center – Zababakhin All-Russian Scientific Research Institute of Technical Physics). Cold-worked samples made of aluminum alloys VD1 and D16, which were 3 mm thick, were irradiated with continuous Ar⁺ ion beams emitted by a PULSAR-1M ion source on an ILM-1 ion beam implanter. The implanter was developed at the Institute of Electrophysics, UB RAS [2]. The ion energy was varied from 20 to 40 keV under irradiation. The ion current density was from 50 to 400 $\mu\text{A}/\text{cm}^2$, and the fluence was from 10^{15} to $7.5 \cdot 10^{17} \text{ cm}^{-2}$.

The temperature of the samples was controlled in the course of ion-beam exposure. The monitoring of the sample temperature, which was performed with the help of a thin chromel-alumel thermocouple welded to an identical test sample, indicated that at low fluences (10^{15} – 10^{16} cm^{-2}) the samples were heated from 40 to 130 °C, while at higher fluences ($\geq 10^{17} \text{ cm}^{-2}$) the heating temperature did not exceed 400 °C.

Similar cold-worked samples of the aluminum alloys were subjected to mechanical shock loading. The speed of a striker was about 400 m/s. The heating temperature of the samples under inelastic interaction did not exceed 300 °C, which is below the furnace annealing temperature of those alloys (~400°C).

The electron-microscopic investigations of the cold-worked VD1 and D16 alloys showed a dislocation cellular structure with the cell boundaries in the form of dense dislocation tangles. Both shock loading and ion bombardment (at fluences 10^{15} – 10^{16} cm^{-2}) transform the *cellular structure* into a subgrain one. In both cases, the products' composition of solid solution decomposition changes. Particles of a metastable phase S'(Al₂CuMg) were revealed to form in the VD1 alloy, and a phase, the composition of which is close to Al₅Cu₆Mg₂, formed in the alloy D16 instead of a metastable phase θ'' that was observed in the cold-worked state.

Summarizing, the direct relation was found between the microstructure change in the VD1 and D16 alloys under mechanical shock loading and accelerated cold (at abnormally low temperatures) radiation annealing with Ar⁺ ion beams (E = 20–40 keV). The results of the study can serve as an indirect proof of the shock-wave nature of structural and phase transformations initiated in the initial stage of irradiation.

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MODELING OF DIFFUSION MASS TRANSPORT IN ALLOYS UNDER ION IRRADIATION

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Irradiation of alloys with metallic ions leads to significant changes in local alloy composition along the projected range. Their reasons are radiation-induced segregation (RIS) revealing near the sample surface and near the region of high non-uniformity of point defect (PD) generation rate, and also radiation-enhanced diffusion of implanted ions.

In this work the characteristics of damage dose: "standard" (NRT) and "effective" displacements per atom (dpa) rates, energy spectra of primary knocked atoms (PKA) are calculated in fcc Fe-Cr-Ni and bcc Fe-Cr-Si alloys along the projected range under irradiation with 7 MeV Ni⁺² and 1.8 MeV Cr⁺³ ions. PKA energy spectra are different under ion and neutron irradiation in the cores of thermal and fast reactors with the mean PKA energy is considerably less under ion irradiation.

A system of diffusion equations for PD and alloy elements under ion irradiation is formulated accounting for accumulation of implanted ions. Modeling is performed of changes in alloys composition accounting for PD recombination and simple approximations of PD sink strengths. It is shown that an increase in sink strength above 10^{14} m^{-2} leads to slowing down of mass transport along the projected range.

The approach proposed is also applied for an analyses of published data on irradiation of F82H ferritic-martensitic steel with 4 MeV Ni ions at 450°C up to 100 dpa. Calculations with account of data on PD sink strengths agreed satisfactory with measured profiles of Ni ions implanted.

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POSITRON ANNIHILATION STUDIES OF THE EVOLUTION OF SULFUR NANOCCLUSERS IN AGEING Ni-S SYSTEM AT IRRADIATION AND THERMAL TREATMENT

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Sulphur (S) is a widespread 3sp-impurity in nickel-base alloys. It is known that sulphur has a low solubility in nickel. Thus, the decomposition of the Ni-S solid solution may occur during ageing heat treatment even when the sulphur concentration in the solid solution is low (tens of at.ppm). The radiation-induced solid solution decomposition may also take place during irradiation that is due to interaction of sulphur with point defects and formation of mobile sulphur-point defects complexes.

Ageing processes in materials are commonly studied using residual electrical resistivity measurements, small-angle X-ray scattering, electron microscopy and other techniques. Unfortunately, the direct methods, such as transmission electron microscopy are not efficient in this case because of the extremely small sizes of precipitates. It is clear that (sub)-nanoscale S-rich clusters (SRCs) will form in Ni-S at earlier stages of ageing.

Positron is a sensitive probe for vacancy-type defects. Moreover, the positron is also sensitive to ultrafine precipitates embedded in materials, even if they have no defect, due to affinity-induced positron confinement. PAS methods, such as Coincidence Doppler broadening (CDB) and angular correlation of annihilation radiation (ACAR), enables us to identify the chemical element whose electrons annihilate with the positron by measuring the electron momentum distribution in the high-momentum region. It can identify the chemical environment of the annihilation site. Thus, those methods can to probe the formation of the vacancy-solute complexes. In addition, due to elemental selectivity, CDB and ACAR enable to detect small impurity clusters or precipitates embedded in a matrix.

In this work, the Ni-S system (contained 50-350 at.ppm S), thermally aged or irradiated by 5 MeV electrons at 270 K with subsequent step-wise annealing, are studied by means of ACAR. The formation of ultrafine S-rich precipitates (clusters) is observed during thermal ageing at about 650 K. These clusters do not contain vacancy-type defects and they are coherent to the Ni matrix. The positrons localize in these SRCs that is caused by enhanced local electron density in clusters with compare to it in Ni bulk. The enhancement in electron density is connected with sulphur draw of the electrons from the Ni-Ni metal bond. In irradiated Ni-S system, the mobile vacancy-S atom complexes are formed at post-irradiation annealing above 350 K (stage III). These complexes form the three-dimensional vacancy clusters (nanovoids) decorated with S atoms. Above 550 K, the dissociation of vacancies from these nanovoids leads to the formation of the defect free S-rich clusters.

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THE EFFECT OF TEMPERATURE ON PHASE COMPOSITION AND MECHANICAL PROPERTIES OF AUSTENITIC Cr-Ni STAINLESS STEELS IRRADIATED WITH NEUTRONS IN WWR-K REACTOR

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Metastable high-alloyed austenitic steels are characterized by their tendency to direct $\gamma \rightarrow \alpha'$ martensitic transformations induced by the strain and temperature gradients. Development of bcc-martensitic inclusions in the steel leads to fragmentation and strengthening of grain structure. The formation of new phase results in embrittlement of austenitic matrix and it affects physical and mechanical properties. It is also known that martensitic α' -phase in the steels is metastable and post-deformation annealing leads to reversed $\alpha' \rightarrow \gamma$ transformations.

To investigate the nature of direct $\gamma \rightarrow \alpha'$ martensitic transformations in austenitic steels, the samples (300 microns in thickness) of 12Cr18Ni9Ti and 12Cr18Ni10Ti steels were machined and irradiated with neutrons to the fluencies of 10^{18} - 10^{20} n/cm². Non-irradiated and irradiated steel samples were deformed at various temperatures (20°C and -100°C). As a result, α' -phase was formed and the $M_f(\epsilon)$ relationships between magnetic phase and strain were obtained.

To study reversed martensitic transformations a number of deformed samples were annealed at 300-800°C for 30 minutes. Afterwards the subsequent measurements of magnetic phase and microhardness were performed.

It has been shown that a decrease in temperature leads to an increase of σ_{02} and σ_B values compared to those at a room temperature and it makes the flow of direct $\gamma \rightarrow \alpha'$ transformations easier at deformation. At the same testing temperature a preliminary neutron irradiation results in an additional strengthening of the steel.

It has been found that neutron irradiation in deformed at sub-zero temperatures Cr-Ni steel samples resulted in a decrease of intensity of martensitic α' -phase formation compared to that in the non-irradiated material. An increase in fluence slows down $\gamma \rightarrow \alpha'$ transformations at low strains and partly impede the kinetics of martensite formation in the steel. This effect will be observed in austenitic steels if the $M_f(\epsilon)$ relationship changes from exponential to the S-like form and, at the same time, M_f value increases. The final product of the deformation-induced transformations is martensite with areas of residual austenite. In the samples with such microstructure, post-deformation annealing hardening takes place with a relatively low increase of ferromagnetic phase. Temperature interval for annealing-induced hardening is 300-600°C (for 12Cr18Ni10Ti steel) and its intensity does not depend on preliminary irradiation.

INTENSIFICATION OF DIFFUSION PHASE TRANSFORMATIONS IN Fe-BASED ALLOYS UNDER DEFORMATION GENERATION OF POINT DEFECTS AT CRYOGENIC TEMPERATURES

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It is shown that deformation of the aged austenite alloy Fe-37Ni-3Ti upon lowering temperature from 573 to 77 K results in an increase in the average hyperfine field at the ^{57}Fe nucleus and, consequently, to a growth of concentration of nickel in the austenite matrix, which testifies to an intensification of the process of dissolution of γ' - phase Ni_3Ti upon interaction with dislocations under cryogenic deformation. The result obtained can be explained by a difficulty of the development of an alternative process of the precipitation of Ni_3Ti particles that is initiated by point defects at cryogenic temperatures.

MD SIMULATION OF RADIATION DAMAGE OF Fe-Cr BINARY ALLOY WITH TWIN GRAIN BOUNDARIES UNDER UNIAXIAL STRESS

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The grain boundaries (GB) are commonly considered as sinks for point defects and impurity atoms. However, the mechanism of interaction of boundaries with the defects has not been studied enough. Besides, such important phenomena of phase instability as Cr segregation and depletion at GBs are observed in high-chromium ferritic–martensitic steels under irradiation. Therefore, the computer simulations of GB structure and properties at the atomic level have attracted much attention in recent years.

This work is devoted to radiation damage simulation of Fe-Cr binary alloy with twin GBs by molecular dynamics method. Two GBs with [001] tilt axis and two GBs with [110] tilt axis were considered. All GBs were simulated to be under uniaxial stress of both signs and several values. The load was always perpendicular to GB. Interatomic potentials of G. J. Ackland, M. I. Mendeleev et al. [1] and P. Olsson, J. Wallenius et al. [2] are chosen for iron and chromium, respectively. Modified version of model potentials proposed for Fe-Cr system by A. Caro, D. A. Crowson, and M. Caro [3] were used.

The specific energy of GBs, sizes of corresponding GB regions, binding energies of the point defects and substitutional Cr atom to the GB region were estimated for several stress values. The features of displacement cascade evolution in the presence of loaded GBs are studied by means of MD simulation. Simulation is performed at an initial temperature of 300 K for primary knocked-on atom (PKA) energy of 10 keV.

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RECOMBINATION AND CLUSTERING OF POINT DEFECTS DURING CASCADE EVOLUTION

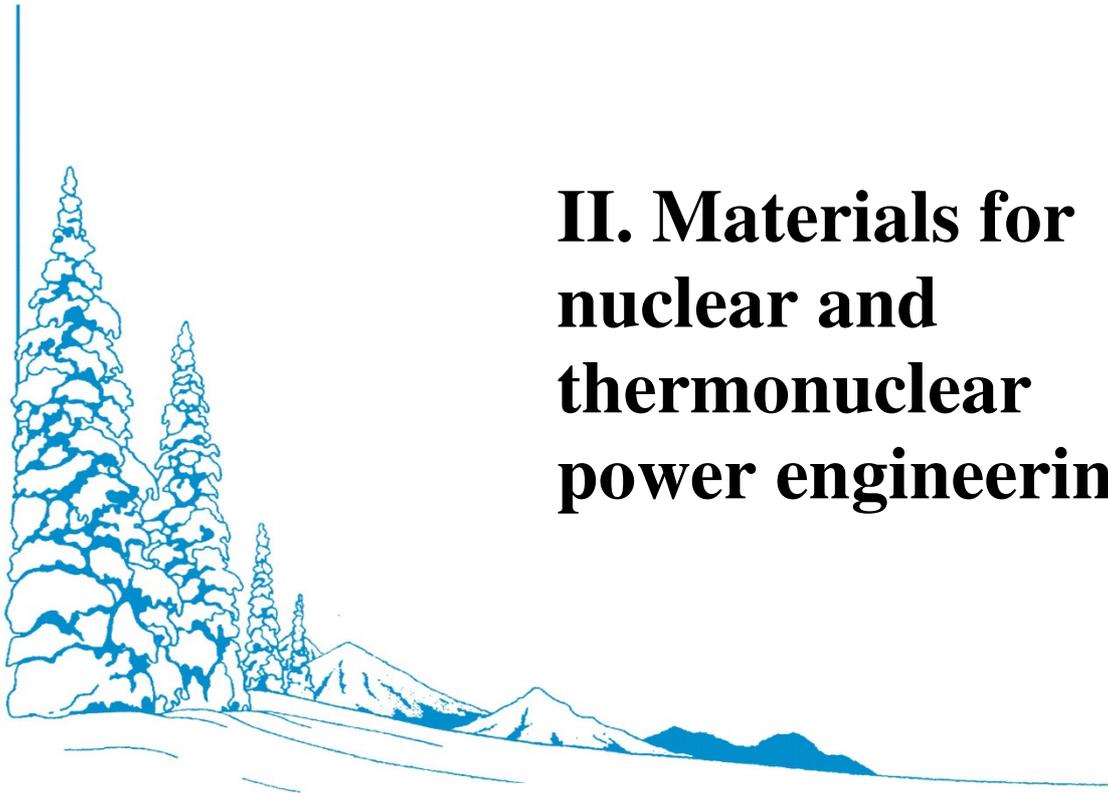
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Modeling of defect evolution in materials under irradiation is started from the calculation of defect formation in displacement cascade, which is successfully carried out by means of molecular dynamics simulation. Based on such calculations the number of defects, size distribution and cascade localization are obtained. But the method of molecular dynamics simulation is restricted by time order of nanoseconds. In order to expand the time and spatial scales the results of MD simulations are used in kinetic modeling in terms of mean field theory, when the defect concentration is averaged over large volume. This allows us to simplify the calculation of defect evolution under irradiation and model the macroscopic volumes and large timescales. But there is a quality discrepancy between this two steps: on one hand the cascade is localized and typical defect concentration is about 10^{-3} - 10^{-2} , on the other hand the kinetic rate theory is based on the average concentration, which is typically much more smaller than in cascades. The discrepancy can result to difference in kinetics of recombination and clustering.

By means of MD simulations the cascade blur is not possible to calculate due to large timescales of such process. In this work the kinetic Monte-Carlo is used to simulate the cascade evolution after the initial cascade stage. Initial stage is simulated by means of MD. Molybdenum is considered as the main candidate for alloys, operated under high temperatures. The PKA energies are 5, 10, 20 keV, and temperatures are 300, 1000 and 1500 K.



II. Materials for nuclear and thermonuclear power engineering

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).

COMPARISON OF NANOSTRUCTURAL STATE OF ODS STEEL EUROFER 97 UPON IRRADIATION WITH HEAVY Fe AND Cr IONS TO FLUENCE 10^{15} cm^{-2}

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In the work presented, irradiation of plates made of ferrite/martensite steel ODS Eurofer 97 was performed with ions of Fe and Cr with an energy of 101 keV/nucleon and fluence of 10^{15} cm^{-2} (~3 d.p.a.). Tomographic atomic-probe data from different areas are given. A comparison was made of different damages for the specified geometry and for the needle-shaped samples investigated earlier.

ACCUMULATION AND ANNEALING OF RADIATION DEFECTS IN AUSTENITIC STEEL 16Cr15Ni3Mo1Ti AT LOW TEMPERATURE NEUTRON AND ELECTRON IRRADIATION

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Currently, reactor internal elements most widely used austenitic stainless steel. Their radiation resistance is determined by the evolution of the microstructure. Primary radiation damage is realized in the form of clusters of interstitial defects or vacancy-type formed in cascades of atomic displacements, or individual point defects, with the ability to freely migrate. This paper presents experimental results on the effect of low-temperature neutron and electron irradiation on physical and mechanical properties of austenitic stainless steel.

It was taken for research austenitic stainless steel Cr16Ni15Mo3Ti1. Neutron irradiation with energy 0.1 MeV was performed in the IVV-2M reactor at 77 K to a fluence in the range $(1,5-15) \times 10^{18} \text{ cm}^{-2}$. Irradiation by 5 MeV electrons was performed on linear electron accelerator LUE-5 at 77 K to a fluence $(1-4) \times 10^{18} \text{ cm}^{-2}$.

Irradiation at 77 K, significantly increases the yield strength of steel, as at the neutron and electron irradiation. As with electron and neutron irradiation, there is almost a linear relationship between the increase in the yield strength and the square root of the growth of the residual resistivity. However, a single linear relationship was not observed. This is due to the inhomogeneous distribution of defects in displacement cascades and a uniform distribution in electron irradiation.

Displacement cascades overlap at neutron irradiation begins at a fluence greater $1,5 \times 10^{18} \text{ cm}^{-2}$. At lower fluences contribution of radiation defects in residual resistivity it is not depends of defects distributed homogeneously, or they are offset in the cascades. However, the heterogeneously distributed point defects and overlapping of displacement cascades have a strong influence on the increase of the yield strength of steel. In the process of annealing at 250-600 K, the free migration of vacancies, vacancy clusters formation, modification of vacancy clusters formed in displacement cascades, and the dissociation of these clusters is occurs. These processes lead to a decrease in yield, and at cascadeless electron irradiation it is completely

restored to its original state at 400 K

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METALLOGRAPHIC EXAMINATION OF MERIDIONAL SECTION OF A SHELL MADE FROM URANIUM ALLOYED WITH IRON AND GERMANIUM AFTER EXPLOSIVE LOADING

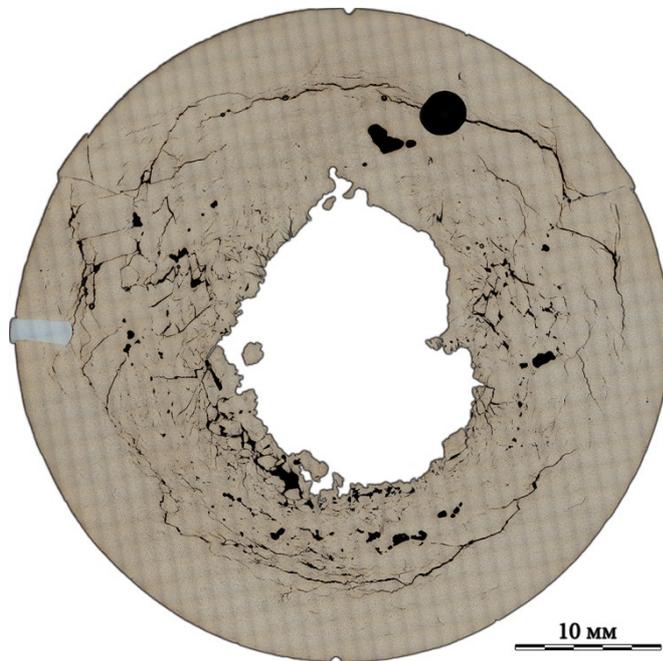
D.A. Belyaev, A.S. Aleksandrov, Yu.N. Zuev, E.A. Kozlov, S.A. Lekomtsev, A.S. Nedosviti, I.L. Svyatov, E.A. Levi
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Along with technical applications, shock waves are widely used in fundamental scientific research as they are currently a basic experimental technique used to study thermodynamic properties of the matter in the area of high-, and super-high pressures and also rheological behavior in conditions of high strain rate deformation. Significantly greater amplitudes of shock waves can be generated with the help of explosive systems that use the principle of detonation wave convergence. In the spherical geometry, pressure at the detonation wave front is continuously growing as far as waves move to the center. Therefore, experiments on the explosive spherical compaction are a good tool to study materials under super-high pressures [1].

This paper presents results of metallographic examination of a thick-wall spherical shell from uranium alloyed with iron and germanium. This shell is recovered after low-level explosive loading [2].

Light microscopy and hardness measurement were used to investigate the meridional section of the test shell as this section most completely exhibits the whole variety of structural features associated with explosive loading of the material (figure) [3].

Concentrically arranged zones were observed to have different structural conditions, hardness and microhardness levels, different fracture extent, concentrations, and sizes of nonmetallics.



Macrostructure of meridional section of the shell from uranium alloyed with uranium and germanium

Our experimental data were analyzed and this analysis revealed structural-changes regularities due to the formation and recompaction of spall fractures caused by shock-wave interactions in the material. This analysis served as the basis for certain conclusions on the details of loading-induced physical processes that resulted in local structural changes and also localization of both

fractures and associated structurally sensitive parameters of the material: hardness, microhardness, distribution of metallurgical inclusions.

Processing, presentation, and analysis of experimental data on volumetric distribution of studied physical quantities were performed with the help of digital panning and color mapping similarly to [4 — 6].

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TRANSMISSION ELECTRON MICROSCOPIC STUDY OF THE U-1.5Mo ALLOY SHELL RECOVERED AFTER SHOCK-WAVE LOADING

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Studying behavior of materials under explosive loading provides information on their properties under extreme conditions of super high pressures and temperatures unattainable with other methods. Spherical geometry of the experiment makes it possible to obtain different states, i.e. either with maximum amount of accumulated damages or with full range of phase transformations up to material melting, through simple variation of HE amount [1, 2].

This paper studies the fine structure of a thin-wall spherical shell from U-1.5%Mo alloy, recovered after explosive loading with converging spherical shock waves.

In the recovered shell, four zones having different-type structures were observed to form after loading. Two zones turn out to be spherically symmetric and the other two ones are available only in the “north” sector of the shell. The previous metallographic examination demonstrated that the structure of the external spherical zone remained practically undeformed and turned out to be an eutectoid mixture of the α -phase of uranium with the γ -phase enriched in Mo where colonies are 30 – 70 μm in size. The second spherical zone has a deformed structure. The other two spherical non-symmetric zones have the structure unresolvable in the optical microscope [3].

Electron microscopic study of samples cut out normally to the radius of the shell at different distances from its center was performed in order to have better understanding of the structural nature of zones available in the recovered shell. Zone 4, nearest to the shell center, is stated to have a highly dispersed structure formed, probably, due to melting and fast crystallization of the metal. Here, colonies of eutectoid plates are 0.5 – 1.5 μm in size and this is almost 2 orders of magnitude less compared to the undeformed external zone.

Zone 3 observed in small areas at the distance of 6.5 – 9.5 mm from the external surface of the shell also has a dispersed structure that probably resulted from material recrystallization due to heating in the course of loading.

Phase and crystallographic analyses are further planned based on interpreted electron diffraction patterns.

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RADIATION SWELLING OF THE NEW GENERATION AUSTENITIC STEELS – BN600 FAST REACTOR FUEL CLADDING MATERIALS AT HIGH DAMAGE DOSES. 1. EXPERIENCE AND METHODOLOGY TO ENHANCE THE LIFETIME CHARACTERISTICS OF THE REACTOR SUB-ASSEMBLIES

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The report presents the methodology of the post-irradiation examination of the fuel sub-assembly serviceability, as developed on the basis of the experience accumulated during technological investigations of the main (non-irradiated) condition of the structural materials and post-irradiation investigations of the condition of the irradiated components of the reactor sub-assemblies accumulated extra effective full power days. With the help of this methodology the immediate goal, i. e. the extension of the lifetime of the bulk of the standard fuel sub-assemblies

of BN600 core up to 592 efpd with the uranium oxide fuel burnup of 11.7 % of h. a. at the maximum damage dose of the cladding manufactured of the cold-worked ChS68-ID steel of the new generation as high as 87 dpa, has been achieved. The next stages, i. e. the increase of the peak fuel burnup values up to about 14 to 16 or 19 % of h. a. and the damage dose up to about 104 to 116 or 140 dpa in the fuel sub-assemblies with EP-450 steel wrapper and the cladding of the improved advanced cold-worked EK164-ID steel, are being carried out.

MEASUREMENT OF VOID SWELLING IN THICK NON-UNIFORMLY IRRADIATED 304 STAINLESS STEEL BLOCKS USING NONDESTRUCTIVE ULTRASONIC TECHNIQUES

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Void swelling is of potential importance in PWR austenitic internals, especially in components that will see higher doses during plant lives beyond 40 years. Proactive surveillance of void swelling is required to identify its emergence before swelling reaches levels that cause high levels of embrittlement and distortion. Non-destructive measurements of ultrasonic velocity can measure swelling at fractions of a percent. To demonstrate the feasibility of this technique for PWR application we have investigated five blocks of 304 stainless steel that were irradiated in the EBR-II fast reactor. These blocks were of hexagonal cross-section, with thickness of ~50 mm and lengths of ~218-245 mm. They were subjected to significant axial and radial gradients in gamma heating, temperature and dpa rate, producing complex internal distributions of swelling, reaching ~3.5% maximum at an off-center mid-core position.

Swelling decreases both the density and elastic moduli, thereby impacting the ultrasonic velocity. Concurrently, carbide precipitates form, producing increases in density and decreases in elastic moduli. Using blocks from both low and high dpa levels it was possible to separate the ultrasonic contributions of voids and carbides. Time-of-flight ultrasonic measurements were used to non-destructively measure the internal distribution of void swelling. These distributions were confirmed using non-destructive profilometry followed by destructive cutting to provide density change and electron microscopy data. It was demonstrated that the four measurement types produce remarkably consistent results. Therefore ultrasonic measurements offer great promise for *in-situ* surveillance of voids in PWR core internals.

MICROSTRUCTURAL CHARACTERIZATION OF AISI 316L TENSILE SPECIMENS FROM THE SECOND OPERATIONAL TARGET MODULE AT THE SPALLATION NEUTRON SOURCE

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The Spallation Neutron Source (SNS) at Oak Ridge National Laboratory (ORNL) is a megawatt class accelerator-based neutron source that generates intense neutron pulses for neutron scattering based research. Neutrons are produced by bombarding a target module containing flowing liquid mercury with 1 GeV protons at a frequency of 60 Hz. The SNS target module is composed of AISI 316L stainless steel and consists of an inner mercury target vessel surrounded by a water-cooled shroud. The target module operates in a harsh environment (dual proton and neutron irradiation, cavitation, vibration, contact with liquid metal) and the analysis of the material property degradation effects is of high interest.

Tensile testing was previously performed on material removed from the first and second operational target module at the SNS. During these characterizations a tensile specimen from the second operational target, irradiated to 5.4 dpa, was tested at room temperature to approximately 57% total elongation and exhibited significantly more elongation compared to the other specimens tested. The tested tensile specimen was examined using electron-backscatter diffraction to characterize the post-deformation microstructure.

Deformation twinning and strain-induced martensite were observed and analyzed in detail. Examination revealed an elevated volume fraction of martensite in the deformed gauge section, and abnormally large grains in both the specimen head and gauge section. Results of the microstructural characterization suggests the specimen deformed plastically via the “deformation wave” process observed in a number of recent studies on 300 series steels irradiated in other reactors. This wave phenomenon always causes a substantial increase in the measured total elongation. It was shown that in the SNS specimen the combined effect of large grain size and phase transformation appears to serve as the cause of high ductility and wave formation.

PHASE INSTABILITY DURING PLASTIC DEFORMATION OF AISI 304L STEEL IRRADIATED IN LIGHT-WATER AND FAST REACTORS

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Many austenitic stainless steels are metastable under straining and are prone to deformation twinning and formation of alpha (α , bcc) and epsilon (ϵ , hcp) martensite. However, the question of how irradiation impacts the phase and structural stability has not been answered in detail. Some recent papers show that neutron irradiation tends to accelerate strain-induced austenite instability, and this could be an issue during long-term operation or life extension of nuclear power plants.

In the present work, martensitic transformation during plastic deformation has been studied for commercial purity austenitic 304L and 316L steels and also model austenitic alloys irradiated

in both a light water power reactor and the BOR-60 fast reactor. To investigate phase and structure transformations (α - and ε - martensite formation), scanning-transmission electron microscopy (STEM) and electron backscattering diffraction (EBSD) have been employed. Finite-element analysis (commercial COMSOL v.4.3. FEA software) has been used to evaluate stress and strain distributions in the deformed specimens.

For both non-irradiated and irradiated materials, the critical stress and critical strain required to produce martensite were studied as a function of damage dose and material starting condition. It was shown that in the irradiated steels martensite formed at a lower strain level; however, the critical stress increased compared to that of non-irradiated material. The morphology of martensite as function of irradiation, strain, stress value and stress state were examined using STEM and EBSD. The role of grain orientation is analyzed in detail; martensite formed in grains oriented along the [001]-[111] direction, but not in [001] or [101]- oriented grains.

RELATIONSHIP OF VOID SWELLING AND DISPERSOID STABILITY IN VARIOUS ODS FERRITIC-MARTENSITIC ALLOYS IRRADIATED WITH SELF-IONS TO VERY HIGH DPA LEVELS

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Recently published studies conducted in the Ukraine have shown that ferritic and ferritic-martensitic alloys irradiated with 1.8 MeV Cr-ions resist rapid swelling for hundreds of dpa before accelerating to ~0.2%/dpa thereafter, with the transition highly dependent on composition and grain structure, varying from 100-500 dpa. More recent studies now focus on oxide-dispersion-strengthened (ODS) alloys irradiated with 1.8 MeV Cr-ions and/or 3.5 MeV Fe-ions, the latter at Texas A&M University. Results are presented here for an EP-450 ODS variant, MA956, MA957, and 14YWT, these alloys having dispersoids in a ferrite matrix, and also a unique 9Cr duplex ODS alloy with dispersoids in both ferrite and tempered martensite phases.

A number of major questions are addressed. Is void swelling delayed by dispersoids? Are the dispersoids stable under irradiation? Are the two phenomena directly or indirectly related? Does the dispersoid-suppressed swelling arise from their role as sinks for point defects or more from their pinning of the nano-grain microstructure to resist radiation-induced recrystallization?

In MA956 dispersoids were not used to produce nano-grains and the overwhelming majority of voids nucleate at relatively low dose directly on the surfaces of the dispersoids. The dispersoids in MA956 are also unstable during irradiation, first becoming amorphous and then dissolving. In other alloys such as MA957 and 14YWT the dispersoids are much more stable, and the transient regime of swelling persists to 400-500 dpa. The voids in highly nano-structured alloys appear to be aligned in internal patterns associated with the deformation texture. It also appears that the role of dispersoids in delaying accelerated swelling arises primarily from their action to pin and maintain the nano-grain structure such that grain boundary denuding is a

significant contribution. Swelling observed in this study appears to be dependent not only on composition but also on grain structure, most easily observed in duplex alloys.

CHANGES IN CHARACTERISTICS OF FINE STRUCTURE OF STEEL ЧС-68 UNDER NEUTRON IRRADIATION

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The austenitic steel ЧС-68 is used as a cladding material of fuel elements of the fast neutron reactor BN-600 and is to be used as fuel element claddings of the reactor BN-800 in its starting period of operation. The optimization of a chemical composition and structure of the claddings made of this steel [1] permits to increase both burn up (from 10 to ~12.5%), and a damage dose (from 75 to 87 d.p.a.) at an operational reliability of the core. However, the main problem limiting an application of this material at high burn ups remains swelling.

The main factors that restrict swelling are [2]: rising stability of solid solution, phase stability of precipitating particles MeC and γ' - phase, stability of dislocation structure; increasing a boron content in solid solution, as well as a cold deformation (by 20-25%), that forms a cellular structure, that includes micro-twins of deformation. At the same time it is considered that a swelling controlling structural factor is the solid- solubility factor [3], that is determined by a concentration of a matrix of alloying elements and impurity elements that form complexes “point defect – impurity” in solid solution.

Thus, operational properties of a material depends on its structure condition. Changes in the structure influence a crystal lattice parameter, its distortion and such characteristics of fine structure as packing defects, point defects and dislocation loops.

The results of X-ray structure and electron microscopy studies of the steel ЧС68 after neutron irradiation in a temperature range of 370÷600°C to different damage doses up to ~ 84d.p.a. are given in this work. The authors made an attempt to differentiate the effects of temperature and a damage dose of neutron irradiation on the changes in characteristics of fine structure.

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IRRADIATION EFFECT ON INTERACTION IN THE SYSTEM U-Mo-Al

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Most research reactors use a dispersion nuclear fuel, where aluminum or its alloys are employed as a matrix and uranium oxides (UO_2), silicides (U_3Si) and aluminides (UAl_4) are applied as a fissile material. The research nuclear reactors have to preserve high their neutron flux density in a transfer from a HEU^{235} to LEU^{235} fuel, for that a fuel of higher density and higher loading on U^{235} is needed as compared to a traditional one. Uranium–molybdenum alloys as the high – density fuels can be among the possible candidate materials. However, these alloys can interact with aluminum to form uranium aluminides of the type UAl_x , whose thermal conductivity is significantly lower than that of aluminum and that may be a limiting factor for using U-Mo in a capacity of a highly dense fuel. Thus, a stability of U-Mo-Al system under in-pile irradiation conditions is quite a pressing problem.

In this work an influence of in–pile irradiation on a rate of interaction of U-Mo alloy and a commercially pure Al, on changes in phase and elemental compositions of the interaction layer were investigated. Four IVV-2M reactor fuel elements of a standard configuration were experimentally tested in-pile. The tests were conducted in standard operation conditions in reactor IVV-2M (each of 2 fuel assemblies contained 2 fuel elements, they were irradiated to reach 2 different levels of average fuel burn up).

Complex post-irradiation materials-science studies were conducted by using the methods of optical metallographic observations, X -ray structure phase analysis, neutron diffraction and X-ray spectrum micro-analysis (XSMA).

It is found that a width of the interaction layer and its volume fraction increase with time of testing, temperature, fission rate and burn up of the fuel. The analytical expression is obtained to determine an interaction layer width against time of testing, temperature and fission rate of the fuel. It is shown that in-pile irradiation increases a rate of interaction of U-Mo alloy with a commercially pure Al by 1 to 1.5 orders of magnitude as opposed to thermal out-of-pile tests and the fuel fission rate makes a significant effect on that.

It is revealed that the layer of interaction between the U-Mo alloy and Al, that is forming under irradiation conditions, is a roentgen – amorphous compound because there are no lines corresponding to phases UAl_4 , UAl_3 UAl_2 on roentgenograms and neutronograms, though these phases are detected in the conditions of the thermal out-of-pile tests.

The interaction layer consists of uranium, aluminum, molybdenum with a content of solid and gas fission products of the fuel; and the quantitative ratio of Mo/(U+Mo) content, typical for fuel particles with a relevant burn up, keeps stable in the interaction layer. The formula for an interaction layer can be presented in the form $(\text{U},\text{Mo})\text{Al}_x$, where x , from the XSMA data is within the limits of 1.3 to 1.6.

MICROSTRUCTURE AND MECHANICAL PROPERTIES OF LOW-ACTIVATION VANADIUM ALLOYS AFTER THERMOMECHANICAL TREATMENTS

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The results of comparative study of thermomechanical treatment effect on the parameters of the structural-phase states and characteristics of short-term strength and ductility of low-activated vanadium alloys of V-4Ti-4Cr, V-2.4Zr-0.25C, V-1.2Zr-8.8Cr and V-1.7Zr-4.2Cr-7.6W systems are presented.

A universal regime of modification of heterophase structural states of vanadium alloys during thermomechanical treatment was developed. It has been established that the application of this treatment regime results in the transformation of initial coarse-grained state of these alloys to fine-crystalline state, including polygonal structures. It has been shown that realized thermodynamic conditions allow the transformation of the initial coarse (more than 1 μm) particles of metastable carbides to uniformly distributed nanoscale (3-10 nm) particles of stable non-metallic interstitial elements-based phase.

The result of the modification of heterophase structural state of vanadium alloys is a significant increase in short-term high-temperature strength, while maintaining a significant reserve of low-temperature ductility.

The nature of high thermal stability of heterophase structure states, formed as a result of thermomechanical processing of the investigated alloys by the regime developed, is discussed.

The contribution to the increase in yield strength of various (solid solution, disperse, grain boundary, substructural) strengthening mechanisms is considered. The efficiency of dispersion strengthening of the alloys mentioned above on realization of Orowan-type strengthening mechanism was analyzed. According to the estimations, to increase the alloys strength ($\Delta\sigma$ up to ≈ 100 MPa) at least 25-50 % of the initial volume fraction of coarse particles should be transformed into fine-dispersed state with a uniform volume distribution.

Investigations were carried out using the equipment of the Tomsk Regional Center for collective use of Tomsk state university.

PLASTIC DEFORMATION AND FRACTURE OF FINE-CRYSTALLINE V-4Ti-4Cr-SYSTEM ALLOY

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Comprehensive investigation of the features of plastic deformation and fracture of fine-crystalline V-4Ti-4Cr alloy after mechanical tension tests at temperatures of 20 °C and 800 °C was carried out by transmission and scanning electron microscopy.

It was found that plastic deformation at room temperature generally takes place in grain volume. Thus this leads to the fragmentation of large crystallites into fine grains and to the formation of high-defect microstructure with elements of submicrocrystalline and nanocrystalline structural state.

During tension at elevated (800 °C) temperature, as a result of grain boundaries softening, the plastic deformation is effected mainly on these boundaries.

It is assumed that the activation of localization of plastic flow is the result of the formation of a powerful stress concentrators in high-strength states and new high-energy carriers of cooperative modes of deformation – the interconnected ensembles of dislocations and point defects interacting with ultrafine particles of the second phase.

To explain the structure and current instability of plastic flow in the zones of deformation localization in the framework of dislocation-vacancy model [1, 2], as a mechanism of plastic deformation is considered the mechanism of motion of edge dislocations, whose mobility is determined by the processes of their climb in overcoming second-phase particles in conditions of high concentration of deformation point defects.

It is shown that the interrelation of microstructure parameters and level of strength properties of the alloy under study is largely determined by acting mechanisms of plastic deformation and reorientation of the crystal lattice, the activation of which, in turn, depends on the conditions of external influence, and on the characteristics of the local elastic-stressed state.

Investigations were carried out using the equipment of the Tomsk Regional Center for collective use of Tomsk state university.

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INVESTIGATION OF THE EVOLUTION OF THE MICROSTRUCTURE OF Fe ION IRRADIATED ChS-139 FERRITIC-MARTENSITIC STEEL

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Heat resistant ferritic martensitic steels are the most promising structural materials for new generation of fusion and fast breeder power reactors. Well known examples of such steels developed in Russia are 12%-Cr steels EK-181 and ChS-139 [1].

Mechanical properties degradation of reactor materials is caused by reactor irradiation and high temperature. Whereas structure-phase state play a key role in mechanical properties alteration, information about behavior of the microstructure of these materials under irradiation is necessary. The aim of the present work is to study of the evolution of the structure-phase state of irradiated ChS-139 steel by atom probe tomography and transmission electron microscopy. In this work irradiation by iron ions beams were used. It allowed to simulate cascade formation of defects, consequently, to simulate influence reactor irradiation on the fine structure of the material. Irradiation had been performed at the linear accelerator facility TIPr-1 (ITEP). TEM samples were irradiated at room temperature by iron (Fe^{+2}) ions beam up to damage dose of ~ 1 dpa. Large number density of dislocation loops $\sim 5 \times 10^{10}$ defect/cm² (average size this loops is 14 ± 3 nm) was observed in irradiated samples. It was shown by atom probe tomography that initial state of ChS-139 steel contains high number density of nanoscale clusters enriched in Cr V, Nb, and N. Similar clusters were found in irradiated up to damage dose of 10 ± 3 dpa samples.

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ORIENTATION RELATIONSHIPS BETWEEN THE STRUCTURAL COMPONENTS OF THE EUTECTOID ALLOY U-1.5% Mo

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In the alloy U-1.5%Mo, using electron microscopy, orientational relations (OS_E) were determined between the structure components of an eutectoid consisting of alternating plates of depleted-of-Mn Mo α -phase (α -U) and ordered γ -phase (U_2Mo) in accordance with the phase diagram. The eutectoid structure, unlike the structure of martensite, was produced by quenching of the samples from 1000°C into water with insufficiently rapid. The relations obtained OS_E : $(100)_\alpha \parallel (113)_\gamma$, $(010)_\alpha \parallel (11\bar{6})_\gamma$, $(001)_\alpha \parallel (\bar{1}10)_\gamma$ turned out analogous to the martensite orientational relations (OS_M): $(100)_{\alpha'} \parallel (111)_\gamma$, $(010)_{\alpha'} \parallel (11\bar{2})_\gamma$, $(001)_{\alpha'} \parallel (\bar{1}10)_\gamma$, that were determined in the present work and earlier [2,3] in a number of alloys of U. For both types of relations, OS matrix were calculated that allow a transition from the coordinates of one of the

structure components to another one.

CHANGES IN STRUCTURE AND HEAT RESISTANCE OF FERRITE-MARTENSITE STEELS AND THEIR ODS MODIFICATIONS

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In the work, the most heat-resistant reactor steels dispersion-strengthened with oxides (ODS) have been studied. Yttrium oxides 2-4 nm in size, which were inserted in the steel matrix via mechanical alloying, do not dissolve at high temperatures and thus favor the retention of high long-term strength. Structure and heat resistance of the BCC steel EP-450-ODS are analyzed in comparison with this steel EP-450 without strengthening oxides at 650 – 700°C and strengths 100 and 140 MPa. The creep velocity at 700°C and 140 MPa in the steel EP-450-ODS is by the order of magnitude less than in the oxideless steels, while the period before fracture is 10 - 50 times as long. This is due to a high thermal stability of the strengthening Y-Ti oxides.

TOMOGRAPHIC ATOM PROBE STUDY OF ODS STEEL 12Cr-1.1W-0.2V-0.3Ti-0.3Y₂O₃

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One of the important problems for advanced fission and fusion power plants is the development of structural materials for the reactor core. Operation 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°C), and new materials also must have corrosion resistance in coolant, and so on. Prospective candidates to meet these requirements are oxide dispersion strengthened (ODS) ferritic-martensitic steels. The development of such materials is currently underway in the world research centers (ORNL, KIT, KAERI, VNIINM etc.). 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 and V affect the formation of nanoscale particles by reducing their size and increasing the number density [1, 2]. In this context, it is important to study ODS steels with different alloying systems [3, 4]. In this work ferritic-martensitic ODS steel 12Cr-1.1W-0.2V-0.3Ti-0.3Y₂O₃ produced in KAERI (Korea) was investigated by tomographic atom probe. The 3D-distribution of chemical elements in the bulk of the material has been obtained. Clusters enriched in Ti, O, V, and Y were found. The average number density of clusters is ~ 10²³ m⁻³ with the size about 2-4 nm.

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A RELATION BETWEEN AN ENERGY OF VACANCY MIGRATION IN AUSTENITIC STEELS AND THEIR RADIATION SWELLING RESISTANCE

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Radiation swelling resistance of fuel claddings is one of the main phenomena that limit operation lifetime of fuel assemblies of fast neutron reactors. Revealing the factors that influence the radiation swelling resistance is one of the important tasks of the radiation failure physics. This work was aimed to find a relation between energy of vacancy migration in austenitic steels and a radiation swelling resistance.

For this investigation the fuel claddings made of austenitic steels ЧС68 and ЭК164 were irradiated to different damage doses in BN-600 reactor. Radiation swelling in the claddings was measured by the hydrostatic weighing method and the characteristics of radiation porosity were determined by the transmission electron microscopy. It was found that swelling of steel ЭК164 was less than that of ЧС68 under close irradiation conditions. Besides, a swelling maximum in ЧС68 claddings is located in the reactor at a distance of 350... 480 mm from the bottom of the core and that in ЭК164 claddings is at a distance of 200...350 mm. Radiation swelling is a complicated process that depends on many factors, including an intrinsic point defects mobility, on which a saturation degree of point defects concentration depends under neutron irradiation.

For revealing a relation between the point defect migration energy and the radiation swelling resistance the experiments were carried out to determine the vacancy migration energy in steels ЧС68 and ЭК164. For that tubular specimens made out of unirradiated fuel claddings of these steels were exposed to different damage doses at a temperature of ~30°C in reactor IVV-2M. After that dilatometric measurements with heating the specimens to 600°C were made. A comparison of the data obtained with dilatometric measurement results on the unirradiated specimens permits to find kinetics of defect annealing and determine the energy of vacancy migration [1]. The vacancy migration energy values measured were $(1,10 \pm 0,02)$ eV for ЧС-68 and $(1,00 \pm 0,02)$ eV for ЭК-164. These values were used to calculate stationary concentrations of vacancies in both steels under irradiation in BN-600 reactor. It has been shown that a difference in migration energy values should lead to a temperature shift of the ЭК164 swelling maximum by 10...15°C lower as compared to that of ЧС68. Because of that the dose in the region that is the most beneficial for swelling is less for steel ЭК164 than that for steel ЧС68; that leads to a smaller swelling and the observed shift of a swelling maximum in fuel with claddings

made of ЭК164 as compared to the cladding made of steel ЧС68.

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MECHANOSYNTHESIS OF IRON DISPERSION-STRENGTHENED WITH OXIDES WITH PRELIMINARY SURFACE OXIDATION

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It is shown that mechanoactivation results in the dissolution of the surface oxides and formation of solid solution of oxygen in the iron matrix. Subsequent annealing favors precipitation of secondary strengthening oxides.

SHORT-RANGE ATOMIC SEPARATION IN BCC Fe-Cr AND Fe-Mn ALLOYS UNDER MEGAPLASTIC DEFORMATION IN BALL MILL

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In the work presented, Mossbauer spectroscopy was employed to ascertain in the single-phase alloys Fe-Cr_x (x = 5...15) and Fe-Mn_y (y = 4, 7, 9) the processes of deformation-induced atomic redistribution in a ball mill of manganese and chromium by the mode of short-range separation.

RADIATION ANNEALING OF RADIATION EMBRITTLEMENT OF THE REACTOR PRESSURE VESSEL STEEL

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Influence of neutron irradiation on RPV steel degradation are examined with reference to the possible reasons of the substantial experimental data scatter and furthermore – nonstandard (non-monotonous) and oscillatory embrittlement behavior. In our glance this phenomenon may be explained by presence of the wavelike component in the embrittlement kinetics.

We suppose that the main factor affecting steel anomalous embrittlement is fast neutron intensity (dose rate or flux), flux effect manifestation depends on state-of-the-art fluence level. At low fluencies radiation degradation has to exceed normative value, then approaches to normative meaning and finally became sub normative. In our opinion controversy in the

estimation on neutron flux on radiation degradation impact may be explained by presence of the wavelike component in the embrittlement kinetics. Therefore flux effect manifestation depends on fluence level. At low fluencies radiation degradation has to exceed normative value, then approaches to normative meaning and finally became sub normative.

Moreover as a hypothesis we suppose that at some stages of irradiation damaged metal have to be partially restored by irradiation i.e. neutron bombardment. Nascent during irradiation structure undergo occurring once or periodically transformation in a direction both degradation and recovery of the initial properties. According to our hypothesis at some stage(s) of metal structure degradation neutron bombardment became recovering factor. As a result oscillation arise that in turn lead to enhanced data scatter. In this case we have to consider irradiation as a recovery factor.

Foregoing hypothetical assumptions on “low-dose effects” in terms “radiation embrittlement contains oscillatory component” and “radiation annealing of the radiation embrittlement” is questionable and needs additional experimental verification and profound scientific study.

DESIGN OF MATERIALS FOR RADIOACTIVE WASTES ISOLATION DURING THE SNF REPROCESSING

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The authors have proposed a modification of existing reprocessing scheme of spent nuclear fuel (SNF) to reduce the volume and activity of liquid radioactive waste (RW) [1]. It involves extracting waste components from SNF solutions by ion exchange into the crystal matrix, which can be used to immobilize the waste after a thermochemical treatment. Inclusion of additional ion-exchange materials into SNF streams during a stage of separation of uranium and plutonium to absorb the most dangerous components of waste - strontium, cesium, cobalt - would reduce the activity and toxicity of the waste liquid and simplify the scheme. The mixed oxides of antimony, antimonates have been chosen for this process. The report describes details of the process, computational and experimental methods of search of materials for its implementation, the existing results.

Simulation algorithm for the process includes the following steps: extraction of waste components from solution of spent nuclear fuel, the transformation and immobilization. The entire analysis is based on calculations of the energy of the crystal lattices. For the extraction it is necessary to select materials with high selectivity toward target waste components. Selectivity is estimated by comparison of the binding energy for antimonate-based compound containing target components with the binding energy of the compounds containing the other components of SNF solution. At the next step the stability of the systems obtained at the first step at elevated temperatures is analyzed by comparing the binding energies of these compounds with binding energies of simple oxides. If the instability is determined, the doping component is searched, which providing resistance at high temperatures. Finally, the stability of selected systems at high concentrations of point defects is evaluated. The result is a technological process chain, which are subject to experimental verification. This algorithm has been implemented using the program GULP [2] and the IDE developed by the authors.

The results of the modelling and experimental studies suggested materials and process flow for Sr isolation, as well as the directions of search for systems for Cs and Co. Expected reduction in the number of process steps of waste management due to new process could decrease the

volumes of liquid waste, reduce environmental hazard of wastes generated during reprocessing, facilitate the tasks of accounting and control.

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NOVEL TECHNOLOGY OF HIGH TEMPERATURE MELT TREATMENT FOR NUCLEAR ENERGY MATERIAL PRODUCTION

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Nuclear power production requires radiation-resistant materials having good mechanical and performance properties [1]. These materials include a number of alloyed steels with high requirements regarding impurity contamination and nonmetallic inclusions. [2]. One of the main methods to purify steel from non-metallic inclusions and gas contaminants is the procedure of ladle degassing the melt with simultaneous flushing with inert gas through a porous bottom insert. Thereafter, a modifier is added to the melt and the melt is sent to the casting in continuous casting machine or mold [3]. This is a conventional technology which allows, in most cases, to obtain satisfactory mechanical properties and a predetermined chemical composition. However, in the smelting of highly alloyed steels high percentage of manufacturing defects is identified by ultrasonic analysis of the finished ingots. One of the main drawbacks of this technology is the contamination of the melt by the materials that are part of the alloying and modifying additives. Besides, it is worth noting non-optimal temperature of modifier's input due to a decrease in temperature of the melt during the ladle degassing [4].

In the present report, considering steels St45, 17G1S, and 40CrMo4 as examples, we discuss the results of implementation of the novel technology for high temperature melt treatment by silicocalcium compositions with the rare earth (Ce and La) additives before ladle degassing, as well as perspectives of the application of this technology for high quality alloyed steel's smelting for the needs of nuclear industry.

Also, we compare the effects of modifier preparation technology on the Fe-C melt supercooling degree. The silicocalcium alloy with Ba, Ce, and La additives was chosen for this study. We used commercially available modifiers produced by a standard technology as well as rapidly cooled alloy, both of an identical chemical composition. The modifiers were analyzed with respect to the fraction, phase and chemical compositions. Degree of the melt supercooling was estimated by means of high-temperature viscosity measurements.

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ANALYSIS OF THE NANOSTRUCTURE OF METAL OF ANTI-CORROSIVE COVERING WELDED TO ENERGY REACTOR VESSELS BY NEUTRON SCATTERING METHOD

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Presently in world and Russian power industry the problem of prolongation of reactor exploitation period remains still very actual for working nuclear power plants. The method applied usually to solve this problem were based on the thermal treatment of critical zones in reactor vessels WWER-1000 (annealing at the temperatures $560\pm 15^\circ\text{C}$). The anti-corrosive covering welded and the material of inner gadgets (base composition of materials H20H10G2B and H18N10T respectively) belong to the reactor materials to be treated for recreation by this annealing. The existing experience is related to reactors of series WWER-440 the vessels of which were treated at the temperatures not higher 470°C . However, the functional properties of their anti-corrosive covering welded and inside gadgets have not yet studied. First in detail the mechanical the mechanical, physical and chemical tests were began by the authors [1].

The character of the processes of structural transformations in these materials and the action of them on mechanical, corrosive, radiating embrittlement still remain not clear. The problem of study of welded materials is connected with their usually metastable and non uniform structure due to the conditions of materials preparation (high gradients of temperature, chemical composition, different annealing rates in material volume etc.).

In paper are presented results investigations of nanostructures of fresh-prepared (non-irradiated) constructional austenite stainless steels H20H10G2B and H18N10T and of the samples after thermal treatment (475°C – 680°C) using thermal neutrons scattering and Fourier-analysis data to find the dependencies between structural transformations and the processes of degradation of mechanical properties.

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MATERIAL-SCIENCE BASES FOR NANOSTRUCTURING FRICTION TREATMENTS OF METASTABLE AUSTENITE STEELS

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Nanostructuring treatments with sliding indenters provide a significant growth of wear resistance and decrease of the friction coefficient of austenite steels under conditions of boundary friction (in tests for sliding friction with lubricant) and adhesion resistance (upon dry sliding friction). This is due to elimination after the friction treatment of the break-in period when the most intense wearing takes place and slowing of the material fracture in the process of stationary wearing.

MECHANICAL PROPERTIES AND STRUCTURE OF Fe-18Cr-10Ni-Ti STEEL AFTER LONG-TERM IRRADIATION IN REACTOR BOR-60 AS COMPONENTS OF BLANKET ASSEMBLIES

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Structural materials of any reactor operating in a sufficiently stressful conditions that promote their damage and deterioration of physical and mechanical properties. To ensure reliable operation of the reactor designs and irremovable reactor generally uses data obtained from special irradiation experiments the samples and data from studies of actual products and structures, operated in the reactor for a long time.

The results of materials research 18Cr10NiTi steel samples cut from the blanket element assemblies BOR-60 irradiated to damage doses exceeding 50 dpa at low temperature irradiation. New experimental results on swelling, physical and mechanical properties and structure of the samples Fe-18Cr-10Ni-Ti steel - material internals BOR-60 reactor and VVER reactors. The data obtained can be used to supplement the data base on the radiation resistance of Fe-18Cr-10Ni-Ti steel needed to justify extending the life of internals BOR-60 reactor and power reactors of the VVER type. Experimental data can be used to justify the extension of the lifetime of the BOR-60 non-removable components.

STRUCTURAL CHANGES IN Fe-0,12C-18Cr-10Ni-Ti STEEL AS A RESULT OF IRRADIATION AFTER 41 YEARS IN BOR-60 REACTOR

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Structural materials of any reactor work in hard conditions, when coupled with a high level of neutron damage dose existing stresses of different types, allowing them to damage and degradation of physical and mechanical properties. To guarantee reliable operation of the permanent structures of the reactor and the reactor are generally used data obtained from special experiments on irradiation of samples and data from studies of real elements and structures, used in the reactor for a long time.

The paper presents the results of microstructural studies of samples of Fe-0,12C-18Cr-10Ni-Ti steel cut from elements of the reflector assembly of the BOR-60 reactor, irradiated up to a damaging dose of 150 dpa at temperatures from 330°C to 380°C. New experimental results on the microstructure of the samples, swelling, physic-mechanical properties of Fe-18Cr-10Ni type steel - material internals BOR-60 and VVER reactors were presented. When the TEM-samples study found three types of radiation-induced phases that formed in the material in the processes of redistribution of alloying elements during irradiation.

The obtained data can be used to justify the criterion gamma-alpha transition in materials internals of VVER reactors in continuous operation up to high damage doses. These data will also be used to update the database on radiation resistance of Fe-18Cr-10Ni steel necessary to justify the extension of the service life of the internals of the reactor BOR-60 and VVER type power reactors.

DEFORMATION OF ZIRCONIUM-NIOBIUM ALLOY E635 IN SUB-MICROSECOND SHOCK WAVES

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Strength characteristics of zirconium - niobium alloy E635 were measured under shock-wave loading conditions at normal and elevated temperatures and results of these measurements are presented. Measurements were taken in conditions when samples were impacted by plane shock waves with the pressure up to 13 GPa and duration from ~0.05 μ s up to 1 μ s. Free-surface velocity profiles were recorded with the help of VISAR and PDV laser doppler velocimeters having nanosecond time resolution.

Evolution of elastic precursors with samples thickness varying from 0.5 up to 8 mm is also considered. Temperature effect on the value of dynamic elastic limit and spall strength at normal and elevated temperatures is studied.

This work is implemented with the support of the State Atomic Energy Corporation "Rosatom" under State Contract H.4x.44.90.13.1111.

STRUCTURE AND PHASE COMPOSITION OF THE TRANSITION AREA IN 3-LAYER MATERIAL "STEEL/VANADIUM ALLOY/STEEL" AFTER DEFORMATION-HEAT TREATMENT

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One of the advanced structural materials for energy applications are vanadium alloys like V-(5-10)%Ti-(4-6)%Cr. They have various advantages over austenitic and ferritic-martensitic steels, first of all - high short- and long-term strength at temperatures up to 800 °C and high radiation stability.

However, limiting factor of using vanadium alloys is their embrittlement upon interaction with oxygen and nitrogen, which have high solubility in vanadium at operating temperatures up to 400 °C and available at any coolant during tests, in the atmosphere and technology stages.

Therefore, it is required to protect the surface by corrosion-resistant materials, for example, via creation of multi-layer composites. Multi-layer structural material, which is a high-temperature vanadium alloy, protected from the surface by corrosion-resistant ferritic steel is proposed in this work. Vanadium alloy provides high long-term strength of the material, and steel protective layer - high corrosion resistance in various media (liquid metals, water, steam). In this case, the creation of monolithic material is provided by the formation of iron-vanadium solid solution in a wide range concentration that assures obtaining durable connection between dissimilar metals in the "transition area" material/coating.

In this paper, we have investigated the transition area of three-layer material samples based on alloy V-10Ti-5Cr, protected from the surface by ferritic steels. The three layer samples "steel / vanadium alloy / steel" were produced by two different methods: hot-pressing of billet using

Gleeble System 3800 at temperature 1080 °C with a maximum stress of compression 95 MPa; torsion under hydrostatic pressure by Bridgman anvil at temperatures 20, 200, 400 °C and pressure of 6 GPa (5 rpm). The effect of different deformation-heat treatment and parameters (deformation rate and temperature, type and magnitude of the stress and et.) on the structure and phase composition of the transition area of three-layer material was investigated.

Variable chemical and structure-phase composition forms in the transition area "steel/vanadium alloy" during each of the deformation-heat treatment. Using optical microscopy, scanning electron microscopy and X-ray mapping have been established that the transition area is a diffusion layer of a solid solution of the components (V, Ti, Cr, and Fe) with monotonically changing chemical composition. The thickness of the diffusion area was determined as the total thickness of a region with variable chemical composition on both sides of the section "Steel / vanadium alloy". The average thickness of the diffusion area was 12 - 20 µm. Annealing at 1000 °C for 2 hours lead to increase in the average thickness of the diffusion area until 40 - 60 µm, but it significantly increased the steel grain size (30 - 50%).

The work was supported by the Russian Ministry of Education (Contract №14.A12.31.0001).

RADIATION SWELLING OF THE NEW GENERATION AUSTENITIC STEELS – BN600 FAST REACTOR FUEL CLADDING MATERIALS AT HIGH DAMAGE DOSES. 2. STANDARD COLD-WORKED CHS68-ID STEEL

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The results of radiation swelling investigation of the cold-worked ChS68-ID steel irradiated as a standard fuel cladding material in the second and the third modification cores of BN600 fast reactor at the maximum damage doses within the interval from 50 to 94 dpa and at the temperatures from 360 to 710°C are presented. Based on the example of the increase of the radiation resistance of this steel the important role of the comprehensive process inspection of the structure and service properties of the fuel cladding throughout all the stages of its manufacturing starting from metal melting to ready tubes and their post-irradiation condition investigations is demonstrated. The results of the residual lifetime evaluation of the fuel pins with the cladding made of this material plating uranium oxide and mixed uranium-plutonium fuels of two types are presented.

RADIATION PHENOMENA IN IRRADIATED AUSTENITE STEELS AFTER LONG IRRADIATION IN REACTOR BOR-60

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To estimate the state of material after long-term performance, it is necessary to perform investigation of elements and constructions that have operated for a long time at different temperatures in the reactor BOR-60. However, special experiments on the samples of different

constructions have to be designed to pick out certain important service conditions that control different radiation phenomena.

In the work, a current state is presented of the novel researches that pay attention, first of all, to the influence of the irradiation duration (the rate of dose increase, the surface of interaction with the reactor coolant) on the properties and structure of irradiated steels. свойства и структуру облученных сталей. The problems of interrelation of swelling and creep are considered, as well as the influence of peculiarities of the microstructure formation on the physico-mechanical properties of the neutron-irradiated steels.

NEUTRON DOSE RATE INFLUENCE ON RADIATION EMBRITTEMENT OF THE REACTOR PRESSURE VESSEL STEEL

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For the purpose of the clarification of the dose rate effect on the radiation embrittlement of the reactor pressure vessel steel some factors such as elemental composition and technology were advisedly excluded.

This approach allows finding radiation embrittlement dependence on flux level however for virtual steel not being in reality. Data consist of 4 groups with mean intensities of 560, 31, 7 и $0,8 \times 10^{11} \text{sm}^{-2} \text{s}^{-1}$ ($E \geq 0,5 \text{ МэВ}$).

In such a way from the ductile to brittle transition temperature dependence on fluence (F) coefficient of the radiation embrittlement A_F as a function of the flux was determined as an equation $A_F = 17,8 + 9,5/\varphi$, where φ is a dose rate in $10^{11} \text{sm}^{-2} \text{s}^{-1}$ units.

This method allows estimating radiation embrittlement of the RPV materials at low fluxes by means of extrapolation.

FEATURES OF STRUCTURAL PHASE TRANSFORMATIONS IN FERRITIC-MARTENSITIC STEELS UNDER HIGH TEMPERATURE NEUTRON IRRADIATION

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At present time prospects of an atomic energy advancement in many aspects are associated with a development of fast neutron reactors, that makes metal physicists to search for new alloys capable to operate in more severe environments of new power engines. For cost effectiveness of new reactors it is necessary to achieve an essential increase of fuel burn up and that leads to an increase in appropriate dose and temperature burdens on structural materials.

For a solution of specified tasks the most performance potential materials are low swelling heat –resisting ferritic–martensitic steels. Currently at the JSC «All – Russian Scientific Research Institute for Inorganic Materials» the steels EK181, ChS139 and also EP450 hardened

with dispersive oxides (ODS) of yttrium and aluminum are considered as candidate materials. Therefore it is of interest to study behavior of the candidate materials in the conditions of higher temperatures and damage doses.

The electron –microscopy studies of a structure–phase stability of candidate ferrite – martensitic steels under neutron irradiation at temperatures to 1000 °C and higher are given in this work. All the materials studied, that were irradiated to damage doses of ~80 d.p.a., demonstrated a high swelling resistance. At irradiation temperature less than 1000 °C a partial recrystallization of alloys is observed; as a result an initial martensitic structure disappears and grains grow. At the same time, there takes place an intensive coagulation of a carbide phase of the type $M_{23}C_6$ that precipitates mainly at the boundaries of ferrite grains, and a carbide size can attain several microns. An average size of grains in ODS steels is almost one order of magnitude less than in steels ЭК181 and ЧС139. After irradiation at temperatures higher than 1000 °C in the structure of steels EK181 and ChS139 coarse equiaxed ferrite grains and martensitic structure grains (their portion reaching 15 to 30 %) are observed; and second phase interlayers are observed at the ferrite grain boundaries. After irradiation in ODS steels a small grain ferrite structure remains intact, alternating with strongly fragmented regions of an annealed martensitic structure. In the ODS steels a laminate texture preserves itself close to the initial one in the hardening oxides even after irradiation at peak temperatures. Thus, the role of the refractory oxides is decisive for the stabilization of the structural state of the ferrite – martensitic ODS steels even at irradiation temperatures higher than 1000 °C.

SUBATOMIC STRUCTURE OF FERRITE-MARTENSITE STEELS ChS139 AND EK181 IRRADIATED IN REACTOR BN-600

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Study was performed on two samples of each steel (one in the form of cylinder $\varnothing 3 \times 30$ mm, the other in the form of tube $\varnothing 6.9 \times 0.4 \times 30$ mm) in the initial state and after irradiation in a Reactor BN-600. Conditions of the irradiations were as follows: cylinders – fluence 21 *dpa*, $T_{irr} = 390$ °C; tubes – fluence 13 *dpa*, $T_{irr} = 850-900$ °C. Subatomic structure of the steels under study is characterized by the presence of precipitates of two essentially different sizes: small (1,2-1,4 nm) – vacancy clusters and large (6,1-7,2 nm) – precipitates of a carbide phase. In the cylindrical samples, the size of vacancy clusters is almost the same in both steels prior and after irradiation, whereas their number after irradiation is essentially larger. The volume fraction increases by a factor of 3-5 and makes up 2 % and the density, 4 times, reaching in the irradiated samples the value $1.7 \times 10^{25} \text{ m}^{-3}$. In the tube samples the absence of vacancy clusters should be noted both in the initial state and after irradiation. As for the irradiated samples, this peculiarity can be explained by the temperature impact (recall that for tubes $T_{irr} = 850-900$ °C). Pores and vacancies became annealed. In the nonirradiated samples, some part is taken probably by their sizes. The thickness of the tube walls and the diameter of the cylindrical sample differ by a factor of 7.5. The average size of large particles is virtually equal for all the samples under study. Irradiation, changes only their number. The volume fraction and density of large particles in the irradiated samples decrease 5 to 10 times and make up 0.1-0.3 % and $(1.5-3) \times 10^{21} \text{ m}^{-3}$, respectively. In our opinion, this indicates that under radiation field and temperature the carbide phases dissolve. Note that in earlier studies of the samples of steels ChS139 and EK181 irradiated in the reactor IVV-2M to a fluence of 0.07 dp, we did not observe such an effect, which can be most probably

explained by a low fluence value. One more circumstance is worth noting. Polarization of the neutron beam after passing through a sample is in the majority of cases high – 0.8-0.9, whereas for the irradiated cylindrical samples it equals only 0.15-0.2. This may well be due to the formation in the latter case of nonferromagnetic inclusions 0.1-1 μm in size. Near a nonmagnetic particle magnetic flux lines in the sample become distorted, which causes a partial depolarization of the beam

DYNAMIC PROPERTIES OF ZIRCONIUM ALLOY E110 UNDER SUB-MICROSECOND SHOCK-WAVE LOADING CONDITIONS

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Zirconium alloy E110 samples with the 0.5 – 8 mm thickness were sock-loaded to measure stress waves at normal and elevated temperatures; results of these measurements are presented. Duration of shock loading pulses varied from $\sim 0.05 \mu\text{s}$ up to $1 \mu\text{s}$ with the amplitude from 3.4 up to 23 GPa. Free-surface velocity profiles were recorded with the help of VISAR and PDV laser doppler velocimeters having nanosecond time resolution. Measured attenuation of the elastic precursor was used to determine plastic strain rate behind the precursor front; with plastic strain propagation its rate decreases from 10^6 s^{-1} at the 0.46-mm distance to $2 \cdot 10^4 \text{ s}^{-1}$ at the 8-mm distance. Spall strength is measured at normal and elevated temperature, the relationship how spall strength depends on strain rate in the range from 10^5 s^{-1} to 10^6 s^{-1} is constructed. Under shock compression above 10.6 GPa, shock wave was recorded to have the three-wave configuration due to polymorphous $\alpha \rightarrow \omega$ transformation.

This work is implemented with the support of the State Atomic Energy Corporation “Rosatom” under State Contract H.4x.44.90.13.1111.

INTERACTION OF BOTH FUEL COMPOSITION AND A LIQUID METAL COOLANT WITH EK164 STEEL FUEL PIN CLADDINGS AFTER THEIR IRRADIATION IN THE LOW-ENRICHMENT FUEL ZONE OF THE BN-600 REACTOR AT 540...620°C

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One of the factors that limits operation lifetime of fuel assemblies of fast neutron reactors is an occurrence of failure of internal and external surfaces of fuel claddings. The most corrosion sensitive are the high- temperature regions of a cladding, where radiation swelling is not practically observed. The objective of this work was to obtain experimental data on fuel cladding failure from the side of a fuel composition and sodium coolant as dependent on irradiation temperature.

The investigations were made with a scanning electron microscope (SEM) TESCAN Mira 3LMU equipped with the system of energy dispersion analysis of characteristic X-ray

radiation X-Act 6 (Oxford Instruments), that permits along with a surface topography study to analyze an elemental composition of the regions of a matrix, grain boundaries and precipitations. This paper presents the results of investigations of cladding parts of the ЭК164 steel fuel element assemblies after their service life in a BN -600 reactor low – enrichment zone that reached a maximum damage dose rate of 84 d.p.a. at irradiation temperatures of 540...630 °C. It has been shown that the interaction of a fuel cladding material with a fuel composition takes place at the internal side of the cladding and that with the sodium coolant proceeds at its external side. From the internal side of the fuel cladding, at irradiation temperature higher than 540 °C a palladium grain boundary diffusion into the steel is observed to form particles in the boundaries; their composition is being changed. Cracking and exfoliation of the grain boundaries are observed. At the same time there are corrosion attacks on a cladding material grain body from the side of a fuel composition. In the temperature range under study the interaction of steel ЭК164 with a sodium coolant from the external side of the cladding leads to a formation of an oxide layer and the layer that is poor in alloying elements: chromium and nickel. An intensity of all the interactions from both the internal and external sides increases with the irradiation temperature. A similar manner of changes was observed for ЭК164 fuel cladding specimens after a service life of fuel assemblies in the BN -600 reactor high enrichment zone that reached a maximum damage dose of 96 d.p.a [1].

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DETAILED ANALYSIS OF DEFORMATION WAVES IN NON-IRRADIATED AND IRRADIATED METASTABLE Cr18-Ni10-Ti STEEL

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Recently it was observed that under some PWR-relevant irradiation conditions, low-nickel steels such as austenitic AISI 304 and 316 steels used in pressure vessel internals can develop a unique strain mechanism characterized by a "deformation wave" when exposures greater than ~20 dpa are reached [1]. This wave phenomenon is analogous to the TRIP effect in non-irradiated steels but in the irradiated case involves a strong martensite instability at the wave front. On the one hand, we may speculate that the initial radiation-induced ductility loss might be recovered via the deformation wave at higher exposure for deformation temperatures characteristic of zero-power shutdown conditions. On the other hand, the microstructural alteration accompanying the wave raises the question of loss of stability with respect to cracking and corrosion especially, and this may be an issue during life extension of light-water reactors.

At the moment, a number of questions related to wave formation and phase instability remain to be investigated. For instance, how much martensite is required to initiate and form a wave, and what is the geometry and strain distribution along the travelling wave. One of the most intriguing aspects is the influence of grain orientation on martensite formation and the kinetics of

phase transformation.

In the present work, the deformation wave was simulated using non-irradiated Cr18-Ni10-Ti steel by varying the annealing temperature (800-1050°C) and test temperature (-100°C to RT) of cold rolled (~ 40%) specimens. Also, some specimens were irradiated in the WWR-K test reactor to 1.9×10^{19} and 1.1×10^{20} neutrons / cm². A special low-temperature test chamber was constructed that allows for specimen observation and non-contact optical strain measurement. The parameters of the deformation wave (relative velocity, local flow stress, local strain, etc.) and the kinetics of magnetic phase accumulation were studied in detail.

Also, structural analysis of the specimens was performed using SEM-EBSD allowing for observation of martensite particle distribution and size. The amount of martensite and its distribution along the specimen gauge was defined and analyzed. It was shown that the wave front had a width of ~0.3 mm and that grain orientation significantly influenced the onset and amount of phase transformation. The largest martensite amount was observed in the [111]-oriented grains, while the softest grains with largest Schmid factor; the [001]- and [011]-grains were found to be much less transformed.

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EFFECT OF TENSILE TESTS TEMPERATURE ON FEATURES OF PLASTIC DEFORMATION AND FRACTURE OF INTERNALLY OXIDIZED V-Cr-Zr-W ALLOY

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The results of investigation of the effect of temperature on features of plastic deformation and fracture of V-4,23Cr-1,69Zr-7,56W (wt.%) alloy after complex thermomechanical plus chemical-heat treatment are presented. Mechanical tests of samples were conducted by active tension at temperatures of 20 °C, 800 °C, 900 °C and 1000 °C.

It was found that at room temperature plastic deformation is characterized by brittle and viscous type of fracture. At high temperatures (800 - 1000 °C) ductile fracture prevails and a distinctive feature is the formation of cracks, that viscous open in the direction of stretching.

During analysis of the orientation maps, obtained using the method of electron backscattered diffraction, it was found that the microstructure of the undeformed part of the samples is represented by elongated in the rolling direction large grains (length up to 200 μm and width of 20 μm) separated by layers of small (length of 2 - 20 μm and width of 2 - 6 μm) crystallites. Volume fractions of large and small grains are approximately equal. Microstructure in uniform elongation areas of the samples after the test at 20 °C, 800 °C and 900 °C is qualitatively similar to the microstructure of undeformed part. Increase of deformation temperature up to 1000 °C leads to a decrease of volume fraction of large grains in the uniform elongation areas to 30%.

In the deformation localization area, subject to the test temperature, the following features of grain structure fragmentation were revealed:

- At 20 °C and 800 °C partial preservation of the initial coarse grains is observed, about 70% of the material volume is occupied by the fine grains (length of 10 μm and width of 1 - 4 μm), inside the most part of both large and small grains the high density of small angle boundaries with misorientations of continuous and discrete type is formed;

- When increasing test temperature to 900 °C, the volume fraction of the initial coarse grains is reduced to 20% and the most material volume is presented by fine grains (length of 10 - 20 μm and width up to 5 μm), practically no formation of low-angle boundaries is observed in crystallites volume;

- Increasing the temperature to 1000 °C results in only a reduction in the volume fraction of large grains to 7%.

Investigations were carried out using the equipment of the Tomsk Regional Center for collective use of TSU.

NEUTRON DIFFRACTION STUDY OF FUEL ELEMENT CLADDINGS IN REACTOR BN-600

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For a successful and safe operation of the fast-neutron reactors it is important to develop materials that are radiation-resistant up to maximal damage doses of 120-140 dpa. At present, the most promising material is cold-worked austenitic steel EK-164. A significant resource of operational characteristics of austenite steels is realized via optimization of composition and structure of materials upon casting and subsequent pipe processing. Changes in the technological routines that are inserted via fitting require estimation of the state of the ready fuel cladding tubes both in the initial (unirradiated) and irradiated states. The application of methods of structural neutron diffraction allows more extensive information to be gained, in comparison with traditional methods of reactor material science, on the massive samples about the presence of defects, including dislocations..

In the work, samples of fuel cladding tubes made of cold-worked steel EK-164 were investigated prior and after exploitation of fuel-element subassembly (FES) in the fast neutron reactor BN-600 under different conditions (temperature, dose, density of neutron flux). The anisotropy of microstresses and its changes in the fuel element claddings during the reactor service life was determined. An attempt was made to explain the observed anisotropy by the presence of dislocations and changes in their type. A significant impact of the irradiation temperature on the structure state of the FE claddings has been revealed.

The work was done at IVV-2M Neutron Material Science Complex within the State Task "Potok" № 01201463334 with partial support of UB RAS grant No 13-2-047-BN.

RESISTANCE TO LOCAL CORROSION OF 12Cr18Ni10Ti AUSTENITIC STAINLESS STEEL AFTER IRRADIATION AND TENSILE TESTING

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Stainless austenitic steels under neutron exposure undergo significant structural and phase changes at certain doses which lead not only to a significant decline in the mechanical properties of the material, but also to a loss of corrosion resistance. In most cases, the local types of corrosion appear, in particular, stress corrosion cracking, intergranular and pitting corrosion.

This paper presents the results of studies related to the effect of plastic deformation and martensitic α -phase on susceptibility to localized corrosion in 12Cr18Ni10Ti reactor steel irradiated with neutrons and deformed at subzero temperatures.

Flat samples of 10.0x5.0x0.3 mm after austenitization (777 K for 30 minutes) were irradiated in the WWR-K reactor to neutron fluences of $4 \cdot 10^{18}$ and $1.9 \cdot 10^{19}$ n/cm² ($E > 0.1$ MeV). Unirradiated and irradiated samples were uniaxially deformed at strain rate of 0.5 mm/min to different strains at room and low (-20, -40, -60°C) temperatures using “Instron-1195” tensile testing machine. During deformation the changes in working sample sizes and the amount of the ferromagnetic α -phase were controlled. Weight measurements were performed with KERN-700 electronic analytical scales. Steel microstructure after deformation and corrosion tests was investigated using optical (MEF-2, Neophot-2) and electron (JEM 100CX) microscopes. Corrosion studies have shown that the austenized and non-deformed non-irradiated material resists least to pitting. It has been observed that large pits appear close to deformation bands, where α -martensitic phase (bcc) is mainly formed and accumulated. The steel deformed to 30 %, in addition to structural heterogeneity, is characterized by phase heterogeneity, which defines its increased susceptibility to localized corrosion. Corrosion studies of neutron-irradiated steel established its good resistance to pitting corrosion. Corrosion rate (mm/year) in irradiated samples has decreased in two orders of magnitude compared to those in unirradiated samples. An increase of fluence from 10^{18} to 10^{19} n/cm² resulted in resistance to corrosion in nearly an order of magnitude.

Deformation of the irradiated samples at low temperatures resulted in formation of a large amount of α -magnetic phase. Irradiated samples after tensile testing at -60°C contained ~ 40 ÷ 45% martensite, while the value of α -phase after deformation at room temperature did not exceed 24%. At the same fluence the irradiated samples (the content of martensite is higher) showed an increased corrosion rate, which confirms the negative impact of the martensitic phase on pitting corrosion resistance of 12Cr18Ni10Ti stainless steel.

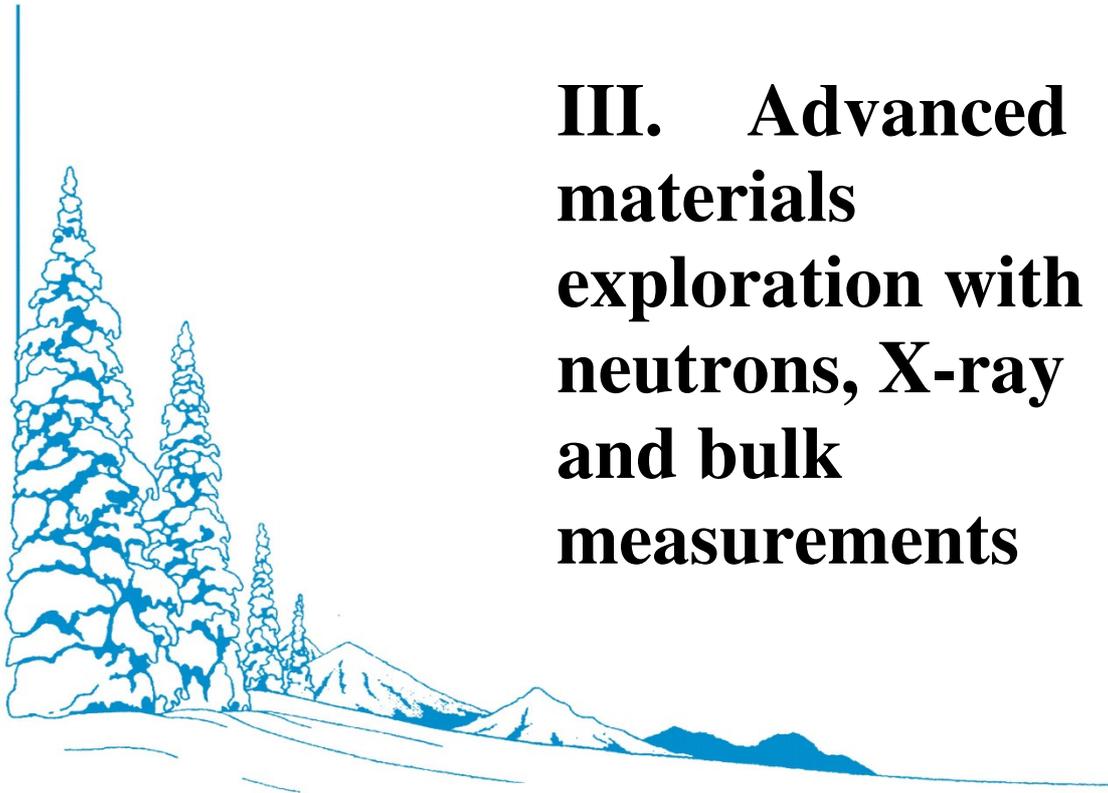
Structural differences defining features for development of pitting corrosion on the irradiated and deformed samples at subzero temperatures were established. The current studies of irradiated and deformed samples showed a decrease in resistance of 12Cr18Ni10Ti stainless steel to localized corrosion due to the formation and growth of deformation-induced α -martensite.

**RADIATION SWELLING OF THE NEW GENERATION AUSTENITIC
STEELS – BN600 FAST REACTOR FUEL CLADDING MATERIALS AT
HIGH DAMAGE DOSES. 3. ADVANCED COLD-WORKED EK164-ID
STEEL**

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The results of radiation swelling investigation of the advanced cold-worked EK164-ID steel irradiated as a fuel cladding material in the third modification core of BN600 fast reactor at the maximum damage doses within the interval from 74 to 96 dpa and at the temperatures from 360 to 700°C are presented. The results of the residual lifetime evaluation of the fuel pins with the cladding made of this material plating the standard uranium oxide pelletized fuel are presented.



III. Advanced materials exploration with neutrons, X-ray and bulk measurements

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.

NEUTRON SCATTERING STUDIES OF THE FEATURES OF THE SYSTEMS WITH STRONGLY CORRELATED ELECTRONS: KONDO-UNDERCOMPENSATION EFFECT, SPIN FLUCTUATIONS VERSUS MAGNETIC ORDERING

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Rare earth based strongly correlated electron systems (SCES) possesses by a wide range of the different types of ground state. Its extend from trivial paramagnetic originating from crystal field splitting of f-electron multiplet up to highly exotic ones. The latter could be as Kondo-insulators with combination of charge-, spin-gap with valence instability; long range magnetic order in initially singlet ground state system; coexistence of long range magnetic order with superconductivity and valence instability; etc. Physical background for these features of electron subsystem may be eliminated by detailed neutron scattering experiments, first of all by magnetic neutron scattering spectroscopy and diffraction.

The analysis of previous and new results of the experimental study for a number of rare earth intermetallics which deals with some features of these unusual ground states are presented in the report.

For the series of solid solutions $\text{Yb}_{1-x}\text{Tm}_x\text{B}_{12}$ based on the Kondo-insulator YbB_{12} it is observed [1] that substitution of Tm to Yb resulted in essential increase of thermopower (Seebeck) coefficient. The effects of Tm-substitution on the dynamical magnetic response of $\text{Yb}_{1-x}\text{Tm}_x\text{B}_{12}$ ($x = 0, 0.08, \text{ and } 0.15$) and $\text{Lu}_{0.92}\text{Tm}_{0.08}\text{B}_{12}$ compounds have been studied using time-of-flight inelastic neutron scattering [2]. Major changes were observed in the spectral structure and temperature evolution of the Yb contribution to the inelastic response for a rather low content of magnetic Tm ions. A sizable influence of the RB_{12} host (YbB_{12} , as compared to LuB_{12} or pure TmB_{12}) on the crystal-field splitting of the Tm^{3+} ion is also reported. The results point to a specific effect of impurities carrying a magnetic moment (Tm, as compared to Lu or Zr) in a Kondo insulator, which is thought to reflect the “undercompensation” of Yb magnetic moments, originally Kondo-screened in pure YbB_{12} . The correspondence is discussed between magnetic dynamics and strong effect of Tm substitution on the temperature dependence of the Seebeck coefficient in $\text{Yb}_{1-x}\text{Tm}_x\text{B}_{12}$, which was reported previously [1].

For $\text{EuCu}_2(\text{Si}_x\text{Ge}_{1-x})_2$ system it is found the coexistence of spin-fluctuation resulted from homogeneous mixed valence state of Eu, with long range magnetic order [3]. The experimental data are provided by several complementary technique: X-ray absorbing spectroscopy, Mossbauer spectroscopy of isomer shift, neutron spectroscopy and diffraction. The possible mechanisms of the discovered behavior are discussed.

The studies are supported partially by RFBR grants: 14-22-01002 and 14-02-00272.

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THEORETICAL CALCULATIONS OF RESOLUTION OF POWDER NEUTRON DIFFRACTOMETERS

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Besides the phase content contemporary methods of analysis of neutron diffraction patterns let us to determine such fine characteristics of the materials in study as inner stresses, size of coherent regions and dislocation density. The description of instrumental effects in powder neutron diffractometers is a problem whose solution is of great importance for analyzing experimental data and taking instrumental contributions into account, as well as for optimizing existing and future constructions of neutron diffractometers [1-3]. Solving of this task by means of analytical methods unlike numerical simulation of devices makes it possible to understand a qualitative aspect of observed effects, many of them being of pure geometrical nature. In addition, it is often possible to reveal certain restrictions imposed on assumptions used in numerical calculations.

We developed correct and rather universal approach to analytical description of resolution of different neutron devices taking into account so called spatial effects related to the change in the characteristics of the neutron beam with respect to its cross section during its propagation along the tool. It is remarkable that the rather complicated formulas obtained can be simplified radically if the Gaussian approximation is used. Within the framework of this approximation, it becomes possible to obtain thoroughly analyzed and geometrically interpreted analytic expressions for the parameters of the neutron beam, the line forms, and the intensities of Bragg peaks in the case of devices of various constructions.

Also the principal importance of the inclusion of spatial effect in the correct description of neutron diffractometers is shown. In particular, neglecting them leads to the violation of the established rules for the characteristics of neutron fluxes following from conservation laws formulated in [3]. It is shown that spatial effects can be sufficient even if a planar monochromator is used in a device.

The work was done at IVV-2M Neutron Material Science Complex within the State Task "Potok" № 01201463334 with partial support of RAS UB Program «Fundamental problems of Metallography and Electrophysics», grant № 15-17-2-3.

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EXAFS SPECTROSCOPY OF HIGH TEMPERATURE SUPERCONDUCTORS

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Temperature dependent X-ray absorption spectra of the hole-doped $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ (LSCO) and electron-doped $\text{Nd}_{2-x}\text{Ce}_x\text{CuO}_{4-\delta}$ (NCCO) high temperature superconductors were investigated above the Cu-K absorption edge in temperature range of 5-300K at E4 beamline of the DORIS III (HASYLAB, DESY, Hamburg, Germany) storage ring. Polarized fluorescence LSCO spectra were measured on a single crystal sample at Ellab polarization; transmission spectra for NCCO were measured on a pressed polycrystalline sample with the optimal thickness. The oxygen environment of copper ions in the CuO_2 plane for the first Cu-O shell was studied. We observed abnormal anharmonicity in the superconductive plane at low temperatures. It was shown, that for superconductive compositions ($x = 0.15$) the pair radial distribution function (PRDF) of the first Cu-O shell is strongly differs from the Gaussian one. So we simulated the experimental EXAFS-function with help of the vibration potential described by parametric function of interatomic distances and found that at low temperature a part of oxygen ions in the CuO_2 plane oscillate in a double-well potential, and their vibrations correlate with the transfer of the local hole (electron) pairs [1,2].

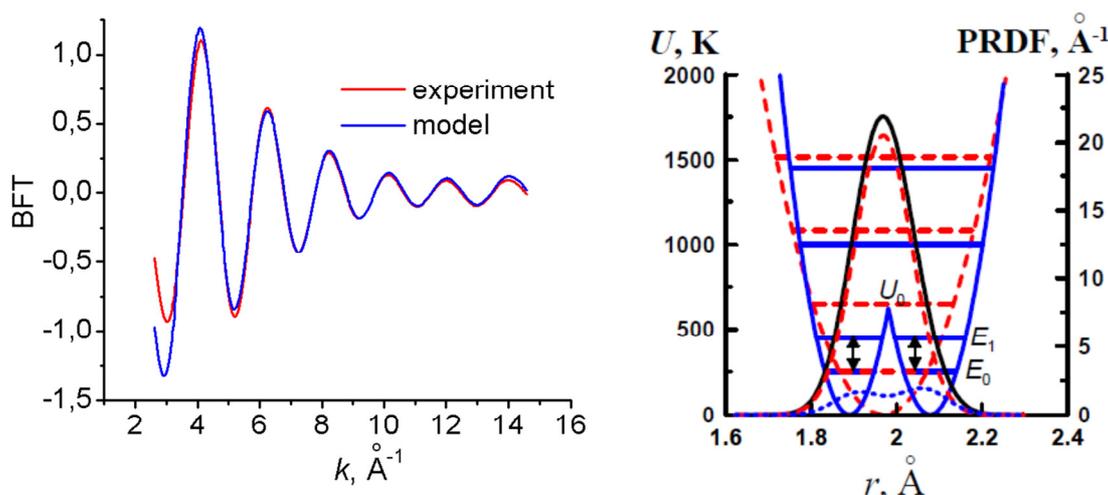


Figure. Experimental EXAFS-function $\chi(k) k^2$ of the first Cu-O(1) shell for $\text{Nd}_{1.85}\text{Ce}_{0.15}\text{CuO}_{4-\delta}$, measured at 10 K, with model (left panel). The model potential (harmonic and double-well) with energy levels E_0, E_1, \dots and total PRDF (black line) (right panel).

We suppose that doping of parent compounds La_2CuO_4 and Nd_2CuO_4 with Sr or Ce leads to formation of the local hole (electron) pairs in the upper antibonding molecular orbital $\text{Cu}3d_{x^2-y^2}-\text{O}2p_{\sigma^*}$ of part of CuO_n ($n = 4; 6$) complexes. These pairs can tunnel between neighbouring complexes in correspondence to dynamic exchange $\text{CuL}^2\text{O}_6 \leftrightarrow \text{CuL}^1\text{O}_6$ (for LSCO) and $\text{CuL}^2\text{O}_4 \leftrightarrow \text{CuL}^1\text{O}_4$ (for NCCO), giving rise to oxygen ion vibrations in a double-well potential. This work was partly supported by Russian Scientific Foundation (grant 14-22-00098).

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VOLUME COLLAPSE STRUCTURAL PHASE TRANSITIONS IN STRONGLY CORRELATED f-ELECTRON SYSTEMS

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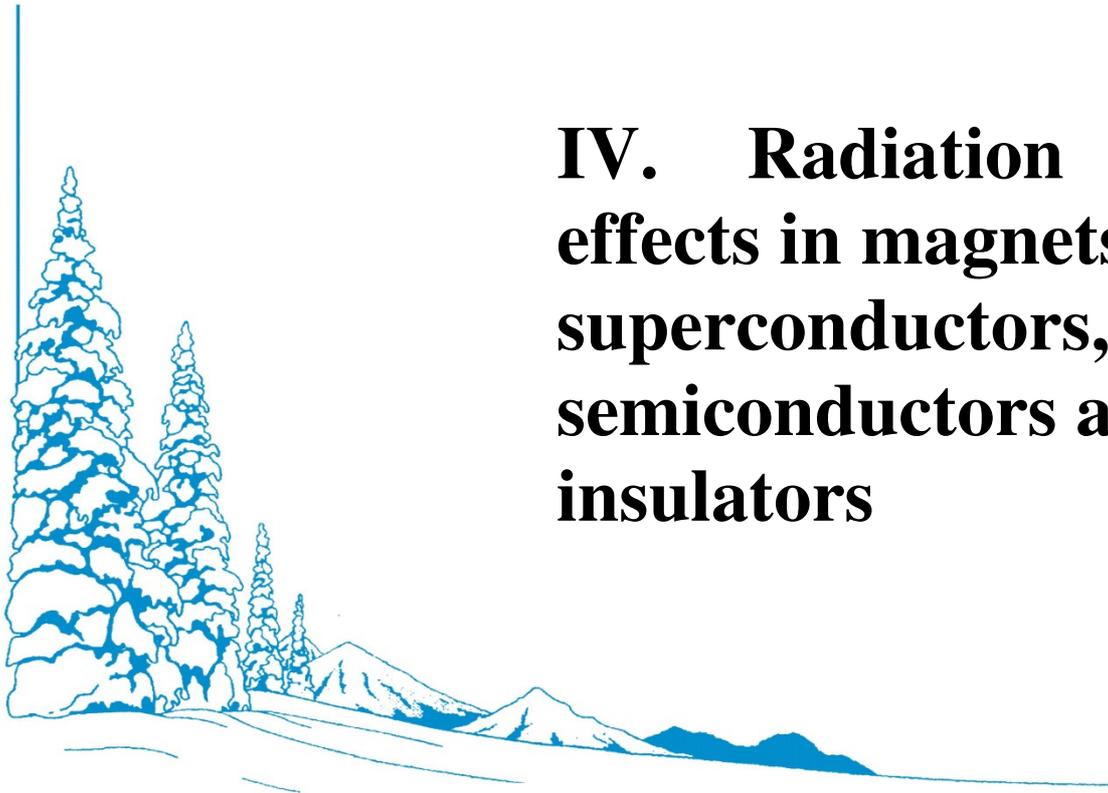
Transition between localized and delocalized (itinerant) behaviour embodies a forefront of physics of strongly correlated systems (SCES). The localized-itinerant transition of the 5f electrons is the central event within the actinide series, where the 5f electrons behave in a localized fashion for the heavy actinides but in a more delocalized manner for the light actinides, with a nexus in the vicinity of Pu and Am. There has yet to be a quantitative and definitive determination of this phenomenon [1]. In the lanthanides, where the 4f orbitals are spatially less extended than the 5f in the actinides, the localization-delocalization transition occurs at the beginning (around Ce), in the middle (Sm), and at the end (Yb) of the rare-earth series. Variation of temperature or pressure gives an increased overlap leading to a delocalization and, in some cases, to structural phase transformations accompanied by a volume discontinuity. The most famous example is cerium, which exhibits the isostructural $\gamma \rightarrow \alpha$ volume-collapse phase transition upon either cooling or application of external pressure. Similar transitions with volume discontinuity occur also in Yb-based compounds, e.g., in $\text{Yb}_{1-x}\text{In}_x\text{Cu}_2$ [2]. Mechanisms of these transitions are still widely discussed [3]. Even more intriguing is the $\delta \rightarrow \alpha$ transition in plutonium, especially $\delta \rightarrow \alpha'$ transformation in the Ga-stabilized fcc phase of plutonium [4].

Here we discuss recent progress in the investigation of isostructural $\gamma \rightarrow \alpha$ transition in cerium, structural transformation of americium under pressure [5] as well as recent results of inelastic neutron scattering study of plutonium δ -phase [6].

Extraordinaire cases can be also found in the 4f-CK Σ C. An example is the intermediate-valence compound CeNi which also experiences a pressure-induced first-order volume collapse phase transition [7]. Neutron scattering and synchrotron radiation experiments allowed us to determine the structure of high-pressure CeNi phase which remained unknown since 1985 [8].

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IV. 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 $\text{La}_2\text{SrMn}_2\text{O}_7$, LaMnO_3 , oxide CuO , Si , SmB_6 , GaN , 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.

SPIN GLASS STATE IN LAYERED COBALTITE $\text{GdBaCo}_{1.86}\text{O}_{5.32}$

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In layered perovskites $\text{LnBaCo}_2\text{O}_{5.5\pm\delta}$ the competition between antiferromagnetic (AF) and ferromagnetic (FM) interactions Co-O-Co leads to a series of magnetic transitions. In stoichiometric $\text{LnBaCo}_2\text{O}_{5.5}$ the cobalt ions have 3⁺ valency. Co^{3+} can have three spin states: HS ($S = 2$), IS ($S = 1$) and LS ($S = 0$) in depending on the crystallographic positions and temperatures. The influence of oxygen content on the physical properties $\text{GdBaCo}_2\text{O}_{5.5\pm\delta}$ has been studied in detail [1]. We present the results of investigation for the magnetic properties in cobalt-deficient sample $\text{GdBaCo}_{1.86}\text{O}_{5.32}$.

The single crystal $\text{GdBaCo}_{1.86}\text{O}_5$ was grown by zone melting. The sample contains 3% ions Co^{4+} . The ratio $\text{Co}^{3+}/\text{Co}^{4+}$ is close to one for composition $\text{GdBaCo}_2\text{O}_{5.53}$. Ferromagnetic state is realized in the temperature range $T = 240\text{-}300$ K that is similar to $\text{GdBaCo}_2\text{O}_{5.5}$. Magnetic dilution does not lead to the displacement of the $M(T)$ maximum to low temperatures.

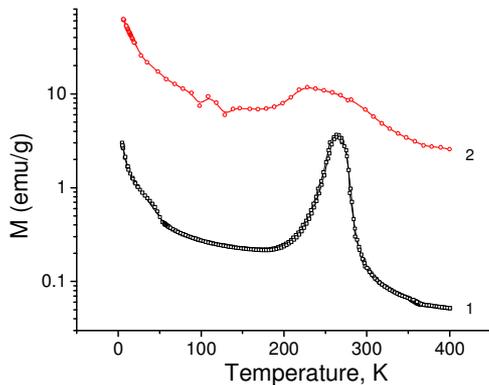


Fig. 1. Magnetization versus temperature for a $\text{GdBaCo}_{1.86}\text{O}_{5.32}$ at different magnetic fields:

1 – $H = 1000$ Oe, 2 – $H = 50000$ Oe

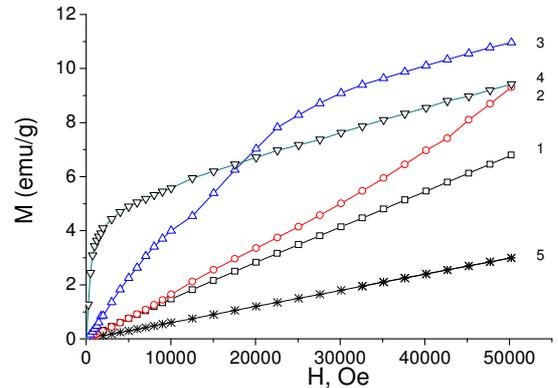


Fig. 2. Magnetization versus magnetic field for a $\text{GdBaCo}_{1.86}\text{O}_{5.32}$ at different temperature: 1–150

K, 2 – 210 K, 3 – 240 K, 4 – 265 K, 5–360 K

In the paramagnetic region ($T > 300$ K) the temperature dependence of the reciprocal susceptibility has a shape typical for ferrimagnetics with $T_N > T_C$. Below 200 K the magnetic state is determined by AF superexchange $\text{Co}^{3+}\text{-O}^{2-}\text{-Co}^{3+}$. However, the external magnetic field promotes the conservation of FM clusters in AF matrix at cooling. FM clusters can be formed near the defects by FM interactions $\text{Co}^{3+}(\text{IS})\text{-O-Co}^{4+}(\text{LS})$ at the expense of disproportionation $2\text{Co}^{3+} \rightarrow \text{Co}^{4+} + \text{Co}^{2+}$. The temperature and field dependences at cooling confirm the presence of FM clusters in region $T = 50 - 200$ K (Fig.1 and Fig.2). In the paramagnetic region $T = 450 - 650$ K all ions Co^{3+} have IS – state with the effective magnetic moment $\mu_{\text{eff}} = 8.78\mu_B$ which is close to calculated value μ_{eff} .

Work was supported by the State program № 01201463330 code "Spin" and UB RAS № 15-9-2-4 and RFBR 14-02-00432.

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EFFECT OF HIGH-ENERGY RADIATION ON THE QUANTUM-SIZED AlGaInP / GaAs LEDs PARAMETERS

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The influence of the high-energy electrons, gamma - rays and neutrons on the parameters of the quantum-sized light-emitting-diode (LED) heterostructures AlGaInP/GaAs has been researched. The object of the investigation were the experimental "red" (625-635 nm) and "yellow" (585-595 nm) LEDs have been grown on the GaAs substrate using MOSCVD technology with an active region on the basis of the multiple quantum wells (Al_xGa_{1-x})_{0.5}In_{0.5}P with the optical reflector, based on the distributed Bragg mirror, and the commercial «Epistar» "red" (630 nm), and "yellow" (590 nm) LEDs.

A common feature of the binary semiconductors AlP, GaP, AlAs and GaAs when exposed to the high-energy radiation is their transition into the highresistance state due to the free charge carriers compensation by the radiation-induced defects. The main factor that reduces the light effectiveness of the semiconductor LEDs is the reduction of the free charge carrier's lifetime due to the capture of the non-equilibrium electrons in the quantum well by the non-radiative recombination defect centers. To obtain the information about the effects of radiation changes of the LEDs parameters the magnitude of the external quantum efficiency (EQE), the recombination currents value and the electrical resistance of AlGaInP/GaAs LEDs after the irradiation of the samples in passive mode power supply have been investigated. The limit state of LEDs upon the irradiation is determines as the 50% of the initial (before irradiation) EQE value.

The most sensitive parameters of semiconductor LEDs to the radiation exposure are the magnitude of the EQE, as well as the value of the recombination currents in the low range of the forward bias. It is shown that for the commercial AlGaInP/GaAs LEDs the 50% decrease of EQE is observed at the irradiation fluences is approximately half as large in the comparison with the experimental samples, which presumably are due to the smaller thickness (2 μm and 10 μm, respectively) and to the lower Mg doping level (concentration of free holes at about 2.1018 cm⁻³ in the commercial and 4.1019 cm⁻³ in the experimental samples) of the LEDs window layers. The "yellow" LEDs have less resistant to the radiation presumably due to the lower depths of the quantum well (at about 90 meV for "yellow" and 110 meV for the "red" LEDs) as supposed, which limits the level of the non-equilibrium electrons injection into the quantum wells of the "yellow" LEDs and thereby promotes a more rapid compensation of these electrons in the quantum wells upon the irradiation. No effects of the quantum wells number on the stability of the LRDs to the high-energy radiation did not revealed.

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SYNERGETICS OF CATASTROPHIC RADIATION FAILURES OF SEMICONDUCTOR DEVICES: INTERMITTENCY MODE

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In spite of the fact that modern radiation solid-state physics is very advanced base for the treatment of radiation effects, there are recent data concerning the effects, which can be difficult explained in frameworks of the conventional ideas. First of all we are talking about the radiation accident of Russian spacecraft "Phobos-Grunt" (2012), caused by the refusal of the integrated circuit WS512K32V20G24M [1]. Among the various features that characterize this radiation failure, 2 absolutely inexplicable ones should be underlined: 1) an abnormally fast first refusal, and 2) the sequence of failures and returns to the normal operation until the complete failure of the device. In our recent work [2] it has been shown in principle that it is possible to explain these features by the synergetic processes arising from the strong non-equilibrium of the impurity-defect system under the action of high-energy radiation.

In this paper, the kinetics of radiation processes is studied basing on the mathematical methods of synergy:

- the mapping theory describing the evolution of the number of defining defects of $n+1$ generation after the number of defects of n generation;
- the master equation allowing to describe adequately the kinetics of combinatorial quasi-chemical reactions between the defects and fluctuations of their concentration at the approaching to the bifurcation;
- ideas of dynamic chaos of transition of the "laminar" regime of radiation process to the "turbulent" one and vice versa,

and it is shown that, possibly, the intermittency mode of the 1st kind [3] is realized in the irradiated semiconductor, explaining easy the above-mentioned features of the devices' failure.

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SPACE CHARGE ACCUMULATION IN ELECTRON IRRADIATED OF POLYMERS DIELECTRICS

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Description of the physical processes of electric charges separation during the injection of fast electrons in the polymeric dielectrics is an important, but largely unsolved problem [1].

A current-transients method for the experimental electrical charging study [2]. Here a material sample is irradiated with a stationary mono-energetic electron flux with such energy (E_e) that their run was less than the thickness of the sample, then regularities of the accumulated charge and electric-field strength are used as a basis to estimate the kinetics of current change at the rear, relative to the flow of high-energy electrons, electrode [3-8].

Experimental and calculated results of the study electrification of various polymeric dielectrics under the action of electron fluxes with $E_e=0.04-2$ MeV, differing by the characteristics of radiation induced conductivity (RIC), were systematized in this paper. Polymeric dielectrics having RIC characterized by quick gaining of stationary value (PMMA, epoxy resins, rubber compounds, ceramic microlite), as well as polymers dielectrics with the kinetic dependence of RIC in the time, such as polystyrene (PC), polytetrafluoroethylene (polystyrol, PTFE) were examined. To describe kinetics of RIC changes a Rose-Fowler-Vaisberg model was used. A numeric simulation of electron transfer was conducted by the Monte Carlo methods. The reasons for the inversion of transient current observed in a number of polymer dielectrics. It was demonstrated that taking into account the specifics of radiation-induced conductivity (dependence on the dose rate, irradiation time and electric field) allows to describe in detail the observed regularities of the current-transients, providing detailed information about values of the generated electric field and a electric volume charge.

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SILICON-BASED HYBRID STRUCTURES WITH SCHOTTKY BARRIER: GIANT BIAS-DRIVEN MAGNETOTRANSPORT EFFECTS

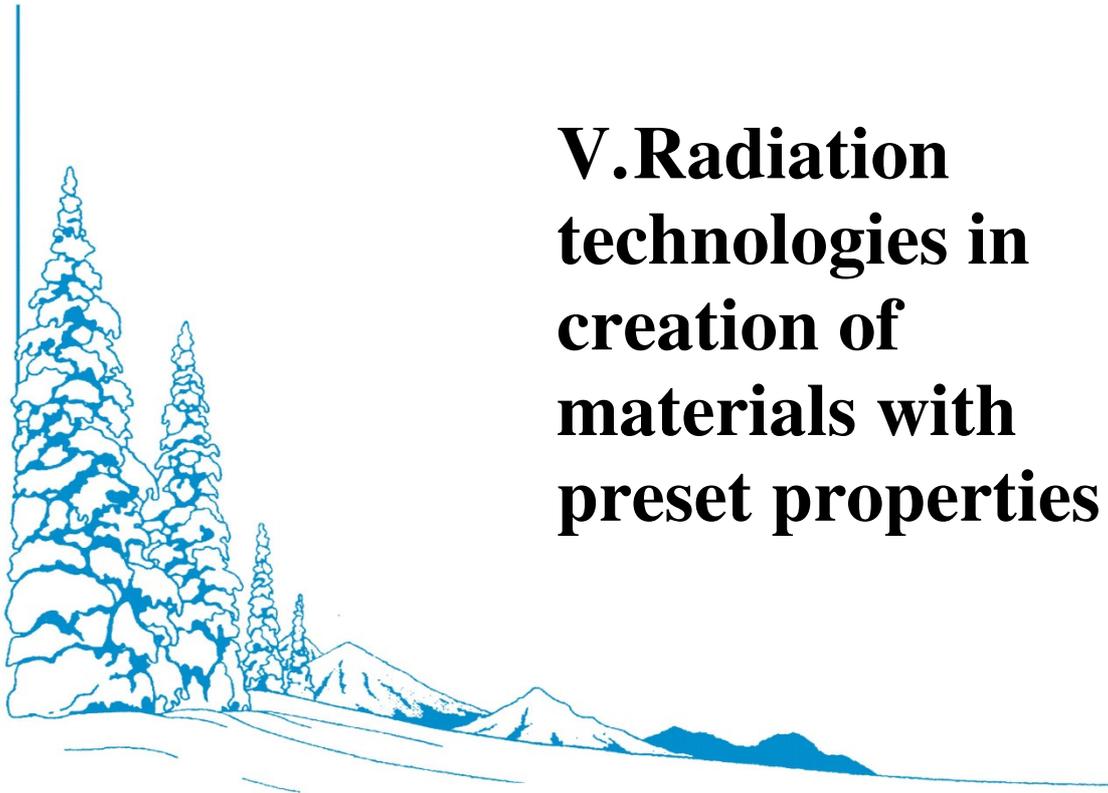
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The high application potential of the magnetic nanostructures is well known. They are already applied successfully in the storage devices. The devices fabricated on the base of the magnetic nanostructures are characterized by the high switching rate, fundamental non-volatility and inherently high stability. Also, these devices are more resistant to the influence of the ionizing radiation. The hybrid structure included the magnetic and semiconductor materials can be systems which allow to integrate the magnetoelectronics and semiconductor electronics.

The giant magnetoresistance (MR) effects in hybrid nanostructures attract much attention of researchers because they initiate nontrivial fundamental physical problems and offer the opportunity of integrating the MR functionalities in electronic devices. We demonstrate that the devices based on the *ferromagnetic metal/insulator/semiconductor* hybrid structures can exhibit specific magnetotransport properties [1–4]. For back-to-back Schottky diodes fabricated on base of the Fe/SiO₂/p-Si structures, dc MR was found. The MR ratio does not exceed 20 % at 9 T, but the value and the sign of the ratio can be driven by a change of a bias voltage on the device. More strong influence of magnetic field is observed in case of ac transport properties. A change of the impedance at application of a field was observed in Schottky diodes fabricated from Fe/SiO₂/p(n)-Si. Maximal effect is observed in frequency range of 10 Hz – 1 MHz where magnetoresistance and magnetoreactance ratios at 1 T exceed 300 % and 600 %, respectively. At that, the ratios can be by applying bias voltage. In addition, we observed giant dc MR effect induced by laser irradiation in the planar Fe/SiO₂/p(n)-Si-based devices. The conductance varies more than 500 times as magnetic field increase up to 1 T (MR ratio exceeds 10⁵ %). The MR value and sign can be effectively controlled by a bias and by changing the magnetic field polarity. We attribute observed magnetotransport properties to the interface states localized near the SiO₂/p(n)-Si boundary. The capture-emission processes for equilibrium and nonequilibrium charge carriers connect the interface levels, valence and conduction bands. The electron tunneling through SiO₂ barrier also occurs. Magnetic field causes the change of the energy structure of the interface states, thereby exerting influence on the processes of the centers recharging. The asymmetry relative to the magnetic field polarity originates from the Lorentz forces affecting carriers and the specific topology of the device.

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V. Radiation technologies in creation of materials with preset properties

The Seminar Program includes a section, which is intended to introduce the attendees to the latest developments in the sphere of radiation material science concerning the methods for production of new functional materials, including nanostructural materials. Presentations will be made on the formation of nanostructures by the method of radiation modification, specifically, the ion implantation and the shock-wave effect.

CHANGES OF COMPOSITION AND STRUCTURE OF COATED St3 STEEL'S SURFACE LAYERS DEPENDING ON ENERGY OF ARGON ION IRRADIATION

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Upon friction, the process of fracturing is localized in the surface layers of the material. Therefore, with the development of new technologies acting upon the surface there has arisen the problem of their efficient usage for the surface strengthening of machine parts. To such methods, different processes of depositing coatings can be referred. A combination of the process of applying coatings with ion implantation can increase the thickness of the strengthened layer and its physico-mechanical and service properties.

The aim of this work was to study the influence of irradiation with argon ions of different energies on the formation of composition and structure of surface layers, changes in the morphology and mechanical properties (microhardness and wear resistance) of carbon steel St3 with deposited ion-plasma coating of Cr and Ni₈₀Cr₂₀.

SIMULATION OF SHOCK WAVE IN THE AMORPHOUS ALLOY Fe₈₀P₂₀

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In the work, behavior of the amorphous alloy Fe₈₀P₂₀ under shock action was investigated. The amorphous alloy Fe₈₀P₂₀ was fabricated by means of heating and drastic cooling of a model crystallite with the help of computer simulation using the program package LAMMPS. To describe interatomic interaction, the potential of a immersed atom was used. It is discovered that upon shock loading a wave is formed that is spread over the model sample with a velocity that exceeds the sound velocity in this material at room temperature. The region of enhanced pressure is followed by an unloading wave.

It is established that after propagation of the shock wave, there takes place the formation of small clusters of phosphorous uniformly distributed over the bulk of the amorphous alloy. Linear dimensions of the system increase along the direction of the wave propagation. The velocity of wave propagation in the amorphous alloy was calculated. No crystallization of the material is found to occur upon such action.

INFLUENCE OF LASER IRRADIATION ON CHANGES IN THE COMPOSITION SURFACE LAYERS OF (Cu₅₀Ni₅₀)+C SYSTEM

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Effect of focused pulsed laser irradiation on changes in the content and microhardness of the surface layers of nonequilibrium copper-nickel foils with the deposited carbon layer was studied depending on the number of laser pulses. In all cases of the laser action it has been discovered that the carbon content changes and the oxides of the substrate's metals (copper-nickel foil) are reduced to pure metals. The decrease in microhardness of the irradiated surface compared to the initial state of the samples was fixed for all irradiation cases. Mechanisms that allow explanation of the changes observed in the samples under laser action are suggested.

FORMATION OF SURFACE LAYER OF THE AMORPHOUS IRON-BASED MATERIALS UNDER ION IRRADIATION

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This work is devoted to the study of structural transformations and thermodynamically related segregation processes in non-equilibrium systems such as amorphous alloys. The work concerns the investigation of structural and compositional evolution laws with respect to the surface layers of amorphous ion-beam treated iron-based alloys. One of the important fundamental and applied research trends is the question concerning the thermal stability of the amorphous alloys. In this connection it is useful to analyze the structure and segregation processes, especially at the early stages of crystallization, since the crystallization of amorphous alloys proceeds through a sequence of metastable states, and the properties of the alloy are changed drastically, so we can speak of two different materials with the same chemical composition. This raises the problem how to control the crystallization process in order to use it as a method of creating the new materials. Study of structural and phase transformations in going from the amorphous state to a stable crystal can help us to understand the nature of the amorphous state.

The subjects of research are the model Fe-B and Fe-P alloys irradiated by different types of ions - Ar, He, N, O, B. Ion irradiation was performed using plasma source with a cold cathode of a pulse-periodic action with the variation of ion beam parameters – dose, ion current density, and ion energy. The depth profiling and X-ray analysis were used to investigate the samples. Additionally, the atomic force microscopy and micro hardness measurements were utilized.

It has been shown that structural changes of the amorphous phase and the crystals at the surface of the amorphous alloy depend not only on the exposure parameters - the dose and the energy of ions, but also on the type of the implanted ions. In this case it is important that the heating-up temperature of the target practically does not depend on the type of ions, and is determined by the current density and the energy of the primary atoms. From this we can conclude that the decisive role in the processes of diffusion and phase formation play the microscopic mechanisms of the atomic-atom collisions cascades development. Comparing the results of X-ray analysis and compositional analysis data of the surficial region, it can be assumed that there is active formation of metastable phases at the surface layers as a result of

radiation, and the composition of the phases varies depending on irradiation parameters.

The work have been financially supported by RFBR №13-02-96002.

FORMATION OF COPPER-NICKEL ALLOY'S SURFACE LAYER WITH HIGH-PRESSURE TORSION AND ION IRRADIATION

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The aim of the work was to study the influence of ion-beam irradiation on the change of surface morphology, structure, composition and mechanical properties of copper-nickel alloy deformed in Bridgman anvils. Of greatest interest is the study of changes in chemical composition and microhardness, for comparison with the results of previous studies [1, 2].

The samples were rolled alloy foil thickness of initially 250- μm -thick $\text{Cu}_{50}\text{Ni}_{50}$ subjected high-pressure torsion to eight turns with a load of 3 GPa. The thickness of the samples was in the center - 150 microns, at the edges - 100 microns.

Implantation was performed with argon ions in a pulsed mode with a current density of 30 mA / cm^2 and an ion energy of 30 keV to a dose of 10^{17}cm^{-2} . The sample temperature during irradiation did not exceed 170°C . Changes in the atomic structure after irradiation is not revealed. All samples showed similar results, both before and after the irradiation (within error).

Microhardness of deformed samples depends on the distance from the center of the sample and, consequently, the degree of deformation of the sample is determined. Higher degree of deformation at the edges leads to an increase in the microhardness, while in the center of the sample, where the deformation under torsion is minimal, microhardness is not changed. It can be concluded that there is a dislocation mechanism for increasing the hardness of the material. After irradiation, the value of microhardness increases, and at higher degrees of deformation it is more pronounced. This confirms the assumption that changes of the material properties under irradiation are caused by relaxation of initial defect structure.

XPS studies revealed large amounts of adsorbed carbon in samples, both from the irradiated and a unexposed sides. No carbides are formed. The distribution of the main components – nickel and copper – in samples with any degree of contamination have the same behavior on the unexposed side and, within the error, is constant at all investigated depths. However, the situation is different at the irradiated side – in the sample where the carbon concentration on the surface is lower than a constant value found, the concentration ratio of nickel to copper decreases linearly with distance from the irradiated surface. Earlier, at the rolled foil alloys $\text{Cu}_{60}\text{Ni}_{40}$, $\text{Cu}_{80}\text{Ni}_{20}$ and $\text{Cu}_{50}\text{Ni}_{50}$, this was not observed. The physical mechanisms leading to the formation of such a linear relationship are yet unclear.

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PLASTICITY RESTORATION OF ALUMINUM ALLOYS WITH THE USE OF DYNAMIC LONG-RANGE EFFECTS UNDER ION BOMBARDMENT

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This work suggests the comparative studies of the microstructure, phase composition, and mechanical properties of aluminum alloys VD1 and D16 (2–3 mm thick) of the Al–Cu–Mg system, and the third generation alloy 1424 (7.3 mm thick), which is hard to anneal, of the Al–Mg–Li–Zn system in the initial (cold-worked) and one-side irradiated states using various regimes of irradiation with accelerated Ar⁺ ion beams. Irradiation was performed on an ILM-1 setup equipped with a PULSAR-1M ion source developed at the Institute of Electrophysics, UB RAS [1].

The possibility of the radiation annealing of the cold-worked sheets of the studied alloys under one-side irradiation with accelerated Ar⁺ ion beams was experimentally proved, the projected range of implanted ions being only a few tens of nanometers. For this purpose, static tests for uniaxial tension, optical and transmission electron microscopy were used. Accelerated radiation annealing offers an alternative to prolonged furnace annealing at elevated temperatures. The regimes of ion-beam processing were found to ensure the most possible softening. The obtained *level of properties* satisfies the *regulated requirements* and makes further cold rolling possible.

The radiation-dynamic nature of rapid annealing induced by accelerated Ar⁺ ion beams is evident from the absence of the noticeable changes in the structure and properties of the VD1 and 1424 alloys after their common furnace annealing in the absence of irradiation under the same thermal conditions which were used for ion beam exposure.

Furthermore, it was found that the impact of accelerated Ar⁺ ions on the *cold-worked* D16 alloy, depending on the irradiation regime, can provide *various properties*, namely: 1) at a relatively low fluence of $\sim 5 \cdot 10^{16} \text{ cm}^{-2}$ a relative elongation increases (~ 2 times without changing the strength characteristics), 2) a further increases in the fluence to $\sim 7.2 \cdot 10^{16} \text{ cm}^{-2}$ increases the relative elongation 4.5 times and decreases the yield strength by 110 MPa, which is similar to *annealed* state (but at a higher value of the tensile strength) and 3) at fluence of $1 \cdot 10^{17} \text{ cm}^{-2}$, the obtained properties of the D16 alloy are close to those of *quenched* state. The difference between structural changes, mainly in the final stages of the thermal and radiation annealing, gives addition opportunities for controlling the structure and properties of aluminum alloys.

In order to check the results of the work, we performed cold rolling of the bands of the aluminum 1424 alloy from 7.3 mm down to the thickness of 1.2 mm. We succeeded in the cold rolling owing to short-term intermediate radiation anneals used by us instead of usually used complex technological sheet-by-sheet processing, which includes soaking in a saltpeter bath for softening the alloy (similar to heating for quenching).

This work was supported by the Russian Foundation for Basic Research (project no. 14-08-01049).

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RESEARCH OF AN INFLUENCE OF ION IMPLANTATION PROCESSING ON THE SURFACE LAYERS STRUCTURE AND MICROHARDNESS OF ROLLED TITANIUM FOIL WITH ALUMINUM SPUTTERING LAYER

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The methods of ion-beaming and ion-plasmous processing have several fundamental advantages as compared with the traditional methods of chemical heat treatment, so they are being actively developed in the area of modification of metals and alloys surface layers in order to improve their strength properties.

In addition to the traditional advantages of ion processing, such as exceeding a solubility limit, controlling the depth of impurity distribution, selective detail parts processing etc., in recent ten years some completely new methods of influence on the surface material layers.

In particular, forming one or several layers of different materials of nanometer thickness range on a target surface and then ion-processing them with high-energy particles, some new compounds and phases were formed in the surface layers of nanometer thickness range. The ultimate strength, yield point, impact strength, fracture strength, corrosion resistance, wear resistance and other characteristics of the subsurface layers, obtained in the above mentioned way, can diverge from those of the "parent" ones.

In addition, combining the coating with ion processing allows to change the modified layer thickness [1], so its physicomechanical and performance attributes can also be changed over a certain range.

During the friction process, the disruption is localized in the surface layers of material. So a task of effective usage of modern surface-affecting technologies for surface strengthening of machine components is now actual [2].

The aim of this work is to research the effect of different rates of argon ions exposure on forming the surface layers structure, changing the morphology and mechanical properties (such as microhardness and wear resistance) of production titanium "BT-01" with aluminum surface being coated by ion-plasmous and electron beam processing.

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COMPOSITION OF SURFACE LAYERS OF CARBON STEEL DEPENDING ON ACCELERATING VOLTAGE PULSE Cr⁺ ION IRRADIATION

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The results of study show that the maximal concentration of ion-alloyed chromium decreases from 22 to 13 at.% with increasing the accelerating voltage. It is detected that in the subsurface layers there form the oxides Cr₂O₃, CrO₂, CrO₃, and FeO, Fe₂O₃ whose amount decreases with increasing the accelerating voltage, which is due to a growth of the sample temperature and more intense sputtering. The microhardness is established to increase by 20% after irradiation with the accelerating voltage 20 kV. With increasing the accelerating voltage, the microhardness decreases to the value corresponding to that of the initial sample, which is a consequence of thermal and radiation-induced annealings generated by the ion irradiation of defects.

MODIFICATION OF COMPOSITION, STRUCTURE, AND PHYSICO- MECHANICAL PROPERTIES OF ARMCO IRON BY THE METHOD OF ION-BEAM MIXING OF GRAFITE FILMS

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Methods of PFES, PSA, and EELFS were used to study chemical composition and structure of thin surface layers of Armco iron alloyed with carbon by the method of ion-beam mixing. An extended (~ 20 nm) transition layer of a variable composition is formed in which there is observed the formation of chemical bonds C-Fe typical of iron carbides. The degree of mixing and fraction of carbon in the C-Fe bond depend on the temperature conditions of the film production and parameters of action (energy, dose). X ray investigation showed the formation of structure inhomogeneties over the layer of mixing, as well as of cementite structure in the transition region "film-metal". The corrosion-electrochemical properties are found to increase by the order of magnitude, while the nanohardness of the modified surface, by 30%.

STUDY OF CHEMICAL COMPOSITION AND ATOMIC STRUCTURE OF FINE OXIDE FILMS ON THE ION-MODIFIED SURFACE OF Cu-Ni AND Cu-Mn ALLOYS

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The work deals with the study of chemical composition and atomic structure of thin oxide films formed on the surface of Cu-Ni and Cu-Mn alloys at temperatures from room to 350 °C in air after irradiation with argon ions 30 keV in energy and $5 \cdot 10^{16}$ and 10^{17} ion/cm² in doses,

current density being 100 mA/cm^2 in a periodic-pulse mode (1 ms, 200 Hz) . It is shown that after modification with argon ions, there takes place a redistribution of the alloy components within the first coordination shell. Chemical composition, distribution profiles, and chemical state of the component of oxide layers on the surface of the ion-modified alloys Cu-Ni and Cu-Mn are studied depending on the parameters of irradiation, temperature of oxidation, and alloy composition.



VI. 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.

DEVELOPMENT OF THE METHOD OF ATOMIC-PROBE TOMOGRAPHY IN ITEP

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In the work, a conception of the development of atomic-probe method in ITEP is given and prerequisites and the results achieved on the realization of an experimental facility are discussed. Advantages and limitations of this method and of software tools for collecting and processing data are demonstrated on the example of a linear geometry and laser evaporation. A number of problems related to the reconstruction of a 3D distribution of atoms in the volumes under study are paid attention.

EXPERIMENTAL DETERMINATION OF THE PARAMETERS OF ENERGY RELEASE IN DENSE CASCADES OF ATOMIC DISPLACEMENTS UNDER ION IRRADIATION

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The intensity of energy release was experimental determined in the region of dense cascades of atomic displacements in pure metals W, Zr, Fe, Al, and aluminum alloy 1424, depending on the regimes of Ar⁺ ion irradiation. The base of the method developed for this purpose is the measurement of the spectral densities of emittance of ion-irradiation-heated metal targets and their approximation by Planck functions of the thermal radiation. These functions are consistent with: (1) nanoscale regions of short-term ($\sim 10^{-12}$ s) thermal spikes and (2) cumulative heating of all the volume of the targets.

The targets made of pure metals and the 1424 alloy were irradiated with continuous Ar⁺ ion beams using an ILM-1 ion beam implanter equipped with a PULSAR-1M technological ion source based on a low-pressure glow discharge with a hollow cold cathode, developed at the Institute of Electrophysics, UB RAS [1]. The emission spectra of the targets under Ar⁺ ion irradiation with an energy of 5–50 keV were measured with a multichannel photodetector based on an OC-12 diffraction spectrograph and a CCD array in the range from 360 to 850 nm. We used a quartz optical fiber, the receiving end of which was set at a distance of 1 cm from the edge of the sample and was directed onto the sample surface at an angle of 60°.

The energy release density was experimentally determined in areas of dense cascades of atomic displacements during irradiation with argon ions with an energy of 5–20 keV reaching 0.3–0.5 eV/atom. The respective estimates of the temperature of thermalized cascade regions are in the range 3600–6200 K. Rapid heating of these regions during the period of time of ~ 10 –12 s to the experimentally determined temperatures explains the nature of the mechanism of post-cascade shock waves emission performed by those regions, which was predicted by the theory. These regions can rearrange metastable media. The pressure estimated in the area of dense

cascades is 4–10 GPa and more, which is an experimental confirmation of the probable emission of post-cascade solitary waves with the stresses at their fronts exceeding the yield strength of the material (including aluminum and its alloys). Such waves may be responsible for the rearrangement of condensed matters under ion bombardment.

The data obtained for pure iron are in a good agreement with those of a work devoted to ion sputtering [2], as well data obtained for iron and other targets are in a good agreement with the results of calculations made using molecular dynamics and Monte Carlo (TRIM) methods [3]. The experimental data also are in an agreement with the Planck theory of thermal radiation. The results of the study are the direct proof of the formation of nano-sized (about 10 nm in diameter) zones of explosive energy release near the target surface during ion bombardment, which was predicted by the theory of atomic collisions. The rate of energy release (about 10^{16} - 10^{17} W/cm³) is comparable to the rate of energy release during nuclear explosion.

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INVESTIGATION OF RADIATION POROSITY USING A SCANNING ELECTRON MICROSCOPE

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Radiation swelling of cladding tubes is one of the causes that limit a service life of fuel elements of fast neutron reactors. At present time radiation porosity is being studied mainly by two methods: hydrostatic weighing and transmission electron microscopy. A hydrostatic weighing it is integral method which does not show a structure of microinhomogeneity of porosity. TEM studies are local method permits to observe practically the whole spectrum of pore sizes and give a possibility of a quantitative analysis. However, TEM method has a problem related to preparation of samples, i.e. difficulties with distant cutting of a thin foil out of massive samples with a high radioactivity. Besides, a small volume of the region under study and an insufficient accuracy of positioning it along the thickness of the material make it difficult to obtain statistically representative results and estimate their macroscopic uniformity.

This work was aimed to develop a procedure (method) of a radiation porosity study with the usage of scanning electron microscopy (SEM), which permits to obtain both a more representative data amount and the information on a degree of a macroscopic nonuniformity of radiation porosity in structure elements under study.

Modern SEM with a Schottky field emission electron gun under optimal condition attains a resolution of 1.5 - 2 nm with a detector of back-scattered electrons (BSE), that permits to use them for a radiation porosity study. For the pore observation an annular detector of back-scattered electrons of scintillation type was used. In this case the area of investigation is limited only by a technique of a preparation of a metallographic section.

In this work were considered a formation of image contrast from changes of parameters of electron beam and work distance from a sample to the detector. Optimal parameters of electron

beam were determined experimentally. Also were comparisons results of SEM with TEM carry out.

THE CONTACTLESS METHOD OF CURRENT FLOW VISUALIZATION WITH A HIGH SPATIAL RESOLUTION

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Results of visualization of electric current paths in the various test planar structures like meander, ring, triangle, arrow, stripe with an artificial defect are presented. The experimental studies were performed for gold conductive layer with a thickness of 300 ± 10 nm and ~ 1 μm width. The two-dimensional (2D) images were obtained by use of magnetic force microscopy (MFM) as a sensor to measure a magnetic response of current flow. The 2D current maps were reconstructed from MFM phase images (see Fig.1) by numerical inversion of Biot–Savart law. By using such a method we obtained the parallel and perpendicular components of the current as well as module of total current with the spatial resolution better than 100 nm. We clearly observed the changes of X and Y components of current density related to changes in the cross section or form of the structures. Developed magneto-transport techniques can be used for detection and control of the current flow in real nano-electronic devices, for studying of defects of current paths in nano-strips due to, for example, electromigration processes and in other applications.

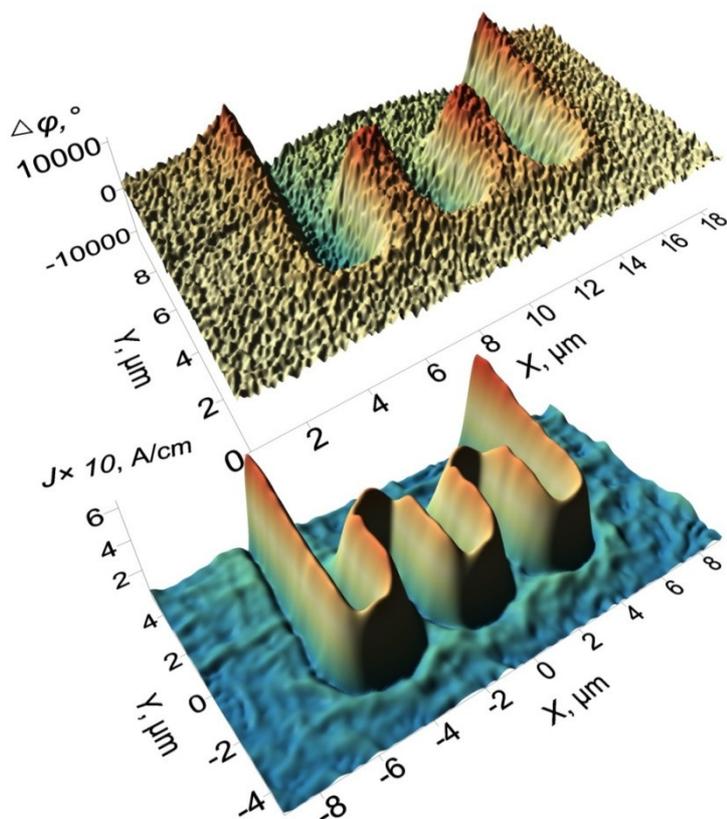


Figure 1. Phase shift (top) and 2D image of electrical current density (bottom) in the meander. The value of current is 5 mA.

INFLUENCE OF ELECTROMAGNETIC PROCESSING OF THE MELTS METAL ON THE STRUCTURE AND PROPERTIES OF CAST METAL

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Cast metals has many drawbacks - heterogeneous composition, coarse dendritic structure, porosity, etc. At the same time, the technology of the manufacture of many details involves producing high quality metal already in the casting stage. Currently, there are several ways to external influence on the molten metal before casting, provides the required structure and properties. These methods include: modification, alloying, electromagnetic stirring, ultrasonic treatment, vibration treatment. The first two methods are related to changes in the composition of the melt, the last involves only external physical impact. One of the new areas of the external action is the treating metal melts by powerful electromagnetic pulses [1].

Alloys of the ferrous and non-ferrous metals: alloy of Al-4 wt. % Cu, and zinc of commercial purity, steel 35L were subjected treatment by powerfull electromagnetic pulses. The compositions of the alloys is given in Table. 1.

Table 1

Alloy	Elements, wt. %
Al-Cu weight 3 kg	0,16 % Si, 3,7 % Cu, 0,04 % Mn, 0,05 % Mg, <0,01 % Ti, 0,35 % Zn, 0,53 % Fe, else Al.
Zn weight 300 kg	0,005 % Pb; <0,001 % Cd; 0,0041 % Fe; 0,001 % Cu; 0,001 % Sn; <0,001 % Sb; <0,0005 % As, else Zn
Steel 35L weight 1,9 tons	0,32-0,4 %C, 0,2-0,52% Si, 0,5% Mn, до 0,3% Ni, до 0,045% S, до 0,04% P, до 0,3% Cr, else Fe.

Two melting in identical temperature-time conditions was carried out for all alloys. In the first case (getting test samples) metal was overheated above melting temperature by 100 ° C the, and was holding for homogenization. Then the furnace was switched off and the treatment of melt powerful electromagnetic impulses for 10-15 minutes was made. Generator with pulses parameters: the amplitude of 10 kV, the duration of 1ns, the leading edge of 0.1 ns, repetition rate of 1 kHz was used for pulse electric treatment of metal melts. The generator consumes cardinality from the network maximum 100 watts. One electrode of the generator was dipped into the melt the second was connected to a graphite crucible.

The procedure of second melting process (receiving the initial samples) was the same, but the melt was holding in the furnace for 10-15 minutes and did not electromagnetic treatment. In both cases, prior to casting was fixed a temperature, melt casting was conducted in sand form.

Initial and test samples have a differences in the macro, the microstructure and properties. For all test samples there is an increase of density, the almost complete absence of shrinkage porosity in castings, grinding eutectic precipitates and secondary phases. In addition, the solubility of the main component in the α -phase increased after electro pulse treatment. In the table 2 are shown the characteristics test and initial samples of the steel.

Table 2

The results of mechanical tests of steel 35L

Sample	σ_T , MPa	σ_B , MPa	δ , %	ψ , %	KCU ^{+25°C} , J/sm ²	KCU ^{-60°C} , J/sm ²
Initial	386	520	6	12	12	9,8
Test	454	772	15	39	45	13

As regards the mechanical characteristics of the test samples recorded simultaneously improving the properties of strength, ductility and toughness.

Comparing our results with the data of different types of external influence on the melt, it was possible to find some similarities. The closest results are obtained by ultrasonic treatment of the melt. Apparently, we can say that the electromagnetic pulses was generated the pressure acoustic field in the melt.

There are studies [3-5], in which it is shown that ultrasonic vibrations are occur in the metal melts when exposed to electromagnetic oscillations. The calculation performed for induction melting aluminum [6] shows that vibrational melt pressure in the crucible diameter of about 300 mm and with the constant field $5 \cdot 10^4$ A/m is reach 2 bar. It is assumed that this pressure is sufficient to produce useful metallurgical effects.

The excitation of mechanical vibrations in the used plant for treatment of metal melts by electromagnetic pulses occurs by contact method. The current flows from the emitter to the surface of the melt.

For a theoretical justification given hypothesis carry out a comparative calculation of the fluctuations intensity. In some studies of the effects of ultrasound on the melts there are some dates that allow you to calculate the resulting mechanical pressure.

The sound pressure and the particle displacement in metals, as for planar or spherical waves are related as follows [3]:

$$p = \rho c \omega \xi = z \omega \xi, \quad (1)$$

where the product of the density of the metal and the speed of sound $\rho c = z$ – acoustic impedance (resistance); ω – the angular frequency ($\omega = 2\pi f$); ξ – particle displacement from the equilibrium position.

In [7] for the radiator in the aluminum alloy is given: $f = 21$ kHz, $\xi = 25$ mm, power of generator 1 kW, intensity of the fluctuations 100 W/cm², the mass of the treated metal is not specified. The speed of longitudinal waves in solid aluminum 6260 m/s [8]. The speed of fluctuations in the melt can reach 70% of the speed in the solid metal [8]. The density of the molten aluminum in 2390 kg/m³ [9]. Calculation by formula (1) gives the value of the pressure at the radiator 29 MPa or 290 bar.

In [10] for the radiator in the aluminum alloy is given: $f = 20$ kHz, $\xi = 4$ m, the generator power of 150 W, the weight of the treated metal is 200g. The calculation gives a value of the sound pressure of 4.6 MPa (46 bar).

In [2] for the radiator in the aluminum alloy is given $f = 19,5$ kHz, $\xi = 30$ m, the generator power of 600 W, the intensity of fluctuations of 109 W / cm², the mass of the treated metal is 210 gr. The calculation gives a value of sound pressure 34 MPa (340 bar).

It is noted In [6] that for the creation of positive metallurgical effects electrodynamic pressure in the melt should be 1-4 bar or $1-4 \cdot 10^5$ Pa.

To determine the vibrational pressure from the effects of electromagnetic pulses can use the formula to calculate the wave pressure at the surface:

$$p = E(1+R)/c, \quad (2)$$

where p – wave pressure N/m²; E – the power of the incident wave, per unit area and per unit time, W/cm²; R - coefficient of reflection ($R = 0$ for total absorption, $R = 1$ for total reflection); c – the speed of propagation of the wave in m/s. Wave propagation velocity in molten metals is about $4 \cdot 10^3$ m/s [8].

In the case pulsed excitation of oscillations incident, the pulses power is can be calculate as:

$$P = \frac{U^2}{r}, \quad (3)$$

where r - the cable impedance (50 ohms); U - voltage of generator (10 kV). The value of the incident pulse power calculated by formula (3) is $P = 2 \cdot 10^6$ watts. Free surface area of the metal used in the crucible of 80 mm diameter is $5 \cdot 10^{-3} \text{ m}^2$. Consequently, the pulse power per unit area is equal to $4 \cdot 10^8 \text{ W/m}^2$.

Substituting the value of the pulse power in equation (1), we obtain $P_{\text{imp}} = 1,3 \cdot 10^5 \text{ Pa}$ (or 1.3 bar). This value is close in magnitude to those given in [6] and [10]. Thus, we can say that electromagnetic pulse treatment generate in the melt ultrasonic vibrations similar to those that occur during ultrasonic treatment. And this means that there are such phenomena as cavitations and acoustic streaming.

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USE OF MONOCRYSTAL DIFFRACTOMETER TO RECORD DIFFUSE SCATTERING

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The close relationship between phase transitions and diffuse scattering was demonstrated in a number of works with perovskites [1, 2]. Later, the methodology was carried over to metals and mathematical expressions were obtained to calculate the intensity of diffuse scattering and its dependence on temperature [3]. Nowadays, works concerning measurements of diffuse scattering on metals are few. For these measurements, it is possible to use the STOE monocrystal diffractometer. Figure 1 gives typical patterns of intensity distribution on the detector: points are

projections of piercings made with rods in the sphere of reflections in the reciprocal space while arcs are projections of the Ewald sphere intersection with the planes in the reciprocal space. The parameters measured numerically are coordinates and intensities of both reflections and arcs. Different sections are obtained through sample rotation against the primary beam. The results given in figure 2 show the smooth change in intensity of reflections (each reflection being the cross-section of a rod by the Ewald sphere at different distances between “braggs”).

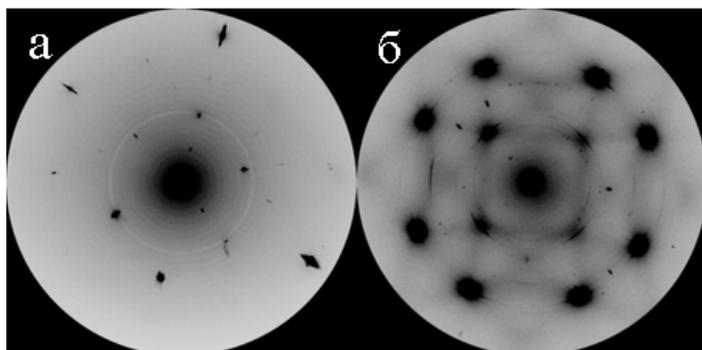


Figure 1. Experimental results. Projections on the detector from (a) piercings of the Ewald sphere with rods and (b) intersections of the planes with the Ewald sphere in the reciprocal space.

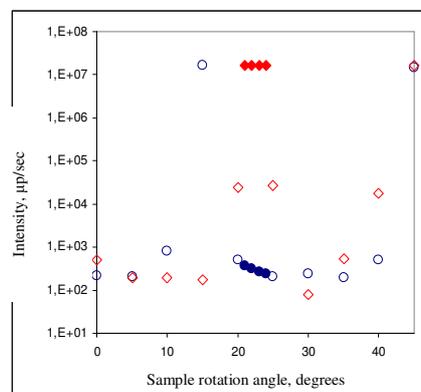


Figure 2. Intensity of reflections in X-ray images versus sample rotation angle.

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NEUTRON DIFFRACTION STUDY OF DISLOCATION STRUCTURE OF Cr-Ni-Mo-Ti AUSTENITE STEEL AFTER COLD PLASTIC DEFORMATION AND IRRADIATION WITH FAST NEUTRONS

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In the work, the dislocation density and relative volumes of edge and screw dislocations in the samples of reactor steel 16Cr-15Ni-3Mo-1Ti after preliminary cold deformation and subsequent irradiation with fast neutrons have been estimated using neutron diffraction data. When calculating, a modified method of Williamson- Williamson, G.K., and Hall Hall was used [1]. The irradiation with fast neutrons is found to result in a decrease in the dislocation density arisen after deformation. The applicability of the method of neutron diffraction to studying the dislocation structure of deformed and irradiated samples is shown.

A series of four samples of austenite steel 16Cr-15Ni-3Mo-1Ti was investigated. Aiming at the production of austenite solid solution with a FCC crystal lattice and at the stress relaxation, samples were heated to 1100C, held for 2 h, and cooled into water. Then, the samples were subjected to cold plastic deformation to a reduction of 10, 20, 30, 40 %. For neutron diffraction experiments, samples were prepared in the form of cylinders 6.45 mm in diameter and 55 mm long. Irradiation with fast neutrons ($E > 0.1$ MeV) was carried out in a vertical channel of the research reactor IVV-2M at a temperature of ~ 800 C. The neutron diffraction spectra of the X16H15M3T1 steel samples under study were taken at room temperature on a neutron diffractometer D7a (angular resolution $\Delta d/d = 0.2\%$) located at the output of the neutron beam from the horizontal channel of the research reactor IVV-2M. The main structure parameters were obtained from the profile analysis of the spectra measured using Rietveld method and FullProf software.

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