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RADIATION DAMAGE PHYSICS OF METALS AND ALLOYS

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Abstracts

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Compiled by Denis Perminov

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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 transmutated gas mixtures.

RADIATION-INDUCED TRANSFORMATIONS IN Fe-Ni AND Fe-Ni-P ALLOYS WITH DIFFERENT MICROSTRUCTURES

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The methods of residual resistivity and the thermal expansion coefficient (TEC) were used to study processes of radiation-induced separation of the solid solution and evolution of radiation defects upon irradiation in the range of 240-570 K and annealings of Fe-34.7 at % Ni and Fe-34.7 at % Ni-0.1% P alloys with different microstructures produced under cold deformation. The alloys were compared in the following states: quenching from 1373 K; aging at 780 K after quenching; deformation to 40%; and deformation and annealing at 573 K for removal of vacancy clusters. Dose and temperature dependences of the separation of solid solutions upon electron irradiation and subsequent isochronal annealings were established.

It was found that the process of radiation-induced separation of the solid solution, which causes a considerable growth of residual resistivity (up to 20%) and the TEC (up to 10^{-5} K^{-1}), takes place under 5 MeV electron irradiation to doses of about $5 \cdot 10^{18} \text{ cm}^{-2}$ and upon subsequent annealings. Vacancy clusters of both deformation and radiation origins act as sinks of point defects and decrease the radiation-induced separation of the solid solution. They dissociate during annealings in the range of 350-500 K and lead to the formation of migrating vacancies, which, in turn, ensure that the process of separation of the solid solution continues. However, the principal and strongest effect on separation under electron irradiation is due to the dislocation microstructure, which reduces the solid solution separation by more than three times at 573 K. An interaction between vacancies and phosphor atoms facilitates the formation of vacancy clusters in both the deformed and the irradiated invar, reduces the separation of the solid solution, and leads to a growth of the TEC. It was shown that concentration inhomogeneities in the matrix of the aged at 780 K alloy are not considerable sinks of point defects. The effect of different microstructures at low irradiation temperatures is insignificant, and at high temperatures the effect of aging and deformation vacancy clusters is neutralized.

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POSITRON ANNIHILATION STUDY OF EFFECTS OF SOLUTE ATOMS ON EVOLUTION OF VACANCY DEFECTS IN ELECTRON-IRRADIATED Fe-Cr-BASED ALLOYS

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The evolution of vacancy-type defects in Fe-Cr alloys (13-16 at. % Cr) undoped and doped with C, N, Au, or Sb and in conventional ferritic-martensitic steel (~13 % Cr) has been investigated using positron annihilation spectroscopy under electron irradiation at room temperature and subsequent stepwise annealing. Small vacancy clusters are formed in the undoped Fe-16Cr alloy, which anneal out between 320 and 550 K. It has been shown that oversized substitutional solute atoms (Sb, Au) in the Fe-Cr alloy interact with vacancies and

GENERAL PROBLEMS OF RADIATION DAMAGE PHYSICS

form complexes, which are stable up to 600 and 420 K, respectively. It has been found that the accumulation of vacancy defects considerably increases in the alloys and the steel with a enhanced content of interstitial impurities. It has been shown that this effect is related to the formation of vacancy-carbon complexes. It is known that chromium in iron decreases the diffusion mobility of carbon. Therefore, the structure of vacancy-carbon complexes and the kinetics of their annealing in Fe-Cr alloys differ from those in the Fe-C system.

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THE EFFECTIVENESS OF MATHEMATICAL MODELS FOR FUSION MATERIALS

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The development of new materials for cost-effective fusion power generation has recently become one of the priority issues for the international magnetic fusion programme. While the criteria related to the stability of materials under 14 MeV fusion neutron irradiation, where candidate alloys and/or composite materials for the tritium breeding blanket are expected to retain mechanical strength, fracture toughness, and creep resistance over the period of 5 to 10 years, are broadly similar to those for the next generation fission reactor materials, there are significant differences, primarily associated with the different energy spectra of fusion and fission neutrons. Recent advances in mathematical modelling show that a synergetic approach, based on concepts drawn from theoretical condensed matter physics, novel computer simulation algorithms, and model validation experiments, offers a cost-effective knowledge-based way forward in the development of fusion materials.

The recent renaissance in mathematical modelling, and the rapid accumulation of new knowledge about microstructural evolution of materials under irradiation are driven by the realization of the fact that it is unlikely that a prototype fusion reactor, or a large-scale fusion neutron source for testing materials are going to be constructed in the immediate future. At the same time, the operation of the present generation of fusion devices, including Tore-Supra in France, Asdex Upgrade in Germany, and JET at Culham Centre for Fusion Energy in the UK, has highlighted a range of generic materials-related problems not foreseen in the past, like the problem of materials compatibility in the divertor, and the fact that a substantial amount of tritium is retained in the plasma-facing materials. The ITER programme is expected to present an even broader range of materials–related challenges.

Despite the fact the engineering design criteria and constraints imposed by radiation swelling, hardening, embrittlement, and thermal and irradiation creep, and the loss of mechanical strength of materials, are macroscopic, analysis shows that all of them are fundamentally related to microscopic reactions associated with the formation and migration of radiation defects and dislocations. These processes are controlled by energy scales of the order of an electron-volt characterizing the dynamics of atoms in the material. This fact highlights the dominant role of the atomic scale in the hierarchy of multiscale mathematical modelling methods, involving electronic, atomistic, mesoscopic (Langevin and Monte Carlo) and dislocation-based approaches. This presentation will also highlight a range of outstanding problems in the field of radiation

stability of iron-based alloys and steels that have recently been addressed and partially resolved using new mathematical concepts and algorithms, validated by dedicated experiments.

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CURRENT STATE OF THE RADIATION DAMAGE THEORY

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Efforts of many scientists for more than a half of a century have resulted in substantial understanding of the response of various materials to irradiation. The contribution of theory to this process is significant. Consequently, some phenomena have been predicted before their observation: void swelling, radiation-induced segregation and existence of one-dimensional mass transport under high-energy cascade-producing particle bombardment. Development of the NRT standard for a common measure of the irradiation dose, the Standard Rate Theory and its further development, the BEK model, and finally the Production Bias Model have established a framework for analyzing microstructure evolution in different materials. However, the theory has not acquired a status allowing it to play a decisive role in creating radiation-resistant materials. Moreover, some theoretical predictions are in contradiction with observations, which indicates that something important has escaped attention. In the present paper, the current theoretical framework and experimental data are analyzed and the reasons for the situation described are discussed. A way of developing a self-consistent predictive theory is outlined.

DEPENDENCE OF VACANCY-SOLUTE INTERACTIONS ON MAGNETIC STATE IN DILUTE IRON-BASED ALLOYS

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A vacancy-solute interaction is determined the diffusion of foreign atoms and whereby all diffusion-controlled processes in solids. The knowledge of these interactions is especially important for understanding the structural changes developing in reactor materials under irradiation where the concentration of vacancies is high. The traditional approaches consider the size misfit between foreign and matrix atoms as main reason of vacancy-solute interactions (deformation interaction). At the same time according modern concepts, the important role plays the mechanism of chemical vacancy-solute interaction which depends on the electronic structure of impurity.

Using the density functional theory methods implemented in the locally self-consistent Green's functions (LSGF) scheme, we performed systematic *ab initio* calculations of the interaction energies of vacancies with dissolved atoms 3p (Al, Si, P, S), 3d (Sc - Cu) and 4d (Y -

Ag) elements in the bcc Fe based alloys. The calculations were done for ferromagnetic as well for paramagnetic state modeled in the disordered local moments (DLM) approximation.

It was shown that of the vacancy-solute interactions for the first two coordination spheres are determined by the chemical contribution and interaction energy is regular change along the element series when the number of valence electrons increase. Magnetism is found to have a very strong effect on the vacancy-solute interactions for 3p and 3d elements. The transition of bcc iron from ferromagnetic to paramagnetic state is accompanied by a decrease in interaction energy which is the most significant for the impurities Mn, Cr and Cu. The obtained results are important for understanding of the diffusion-controlled kinetics of structural changes in the iron based alloys and above all for temperature nearby Curie point.

SEGREGATION OF SUBSTITUTIONAL ELEMENTS ON GRAIN BOUNDARIES. AB INITIO CALCULATION

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Segregations of dissolved atoms on grain boundaries (GB) have significant influence both on phase stability and on physical and chemical properties of alloys. Though thermodynamic principles of segregations formation are known for a long time, the microscopic mechanism of this phenomenon continues to remain a subject of discussions. Traditional approaches consider dimensional discrepancy of ionic radii as a principal cause of interaction of the dissolved element with GB (deformation interaction). According to modern conceptions the determining role in segregations formation on GB is played with the chemical interaction dependent on electronic structure of an impurity.

For finding-out of electronic effects role in segregations formation a calculations of electronic structure and full crystallite energy, containing special GB and an atom of an alloying element in various positions relative to GB, was carried out by methods of electronic density functional theory (PAW-VASP) taking into account a nuclear relaxation. As an example, tilt boundaries $\Sigma5\{210\}[001], \Sigma5\{310\}[001]$ in Al, alloyed by Mg or Si, and $\Sigma3\{112\}[001]$ in bcc Fe, alloyed by Ni or Cu are considered. It is shown, that in the case of GB with high density of coincident lattice points in boundary plane ($\Sigma5\{310\}[001]$ and $\Sigma3\{112\}[001]$) energy of interaction is determined by reorganization of electronic structure in GB nodes with the nuclear coordination distinguished from volumetric. Thus its value depends on charge transfer and on a magnetic state of an impurity (for GB in bcc Fe). At the same time, for GB with low density of coincident lattice points in boundary plane, such as $\Sigma5\{210\}[001]$, having significant nuclear relaxation, the essential contribution to interaction energy is given with deformation interaction. The conclusion is drawn that nonequilibrium GB, for which crystallographic nuclear coordination is broken in wide adjoining area, have the increased tendency to formation of alloying elements segregations.

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INFLUENCE IONIZING RADIATION ON DURABILITY CHARACTERISTIC OF METALS AND ALLOYS

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Metals and flloys are in the states, which are highly far from the state of thermodynamic equilibrium. Energetic parameters of defect and impurity reconstruction prevent to acheve this state. While irradiating the metals and alloys the states, which are improbable from the standpoing of equilibrium thermodynamics, appear.

Investigation influence ionizing radiation from stop industry uranium–graphite reactor at power doze 0.15 - 0.18 *R/s* and ionizing radiation from sourse of radiation ${}^{60}Co$ at power doze 240–250 *R/s* on durability characteristic of austenitic steel, titanium, wolframium, molibdenium, solid alloys BK8 and T15K6.

There has been investigated of basic samples (without treatment) and samples, irradiating by doze $10^4 R$ of ionizing radiation. The investigation results of solid alloys BK8 are shown in table.

Regime	Crippling load, kgs	Cross- breaking strength, kgs/cm	Deformation, n, <i>mm</i>	Stiffnes, kgs/mm	Micro handnes, kgs/ mm ²
Basic samples	443	9843	0,058	7637	1290
Uranium– graphite reactor	392	8756	0,042	9350	1634
Sourse of radiation ${}^{60}Co$	390	8754	0,041	9348	1632

As visible, results of measure depend only from doses of irradiation and not depend from power doses irradiation. After irradiation decrease the cripplingload, cross-breaking strength, deformation and increase stiffnes and microhandnes solid alloys.

Decreasing the quantity of defects and ordering the structure metals and alloys while irradiating by low power doses of ionizing radiation, when less than one hundredth part of atoms is ionized, is possible only owing to the energy release, which was stored in the crystal lattice in the result of chain processes of defect annihilation. Annihilation processes of vacancies and genetically associated interstitial atoms are initiated by ionization.

Investigation carried out with the help of P \ni M-200 raster-type electron microscope showed that samples of BK8 alloy contained the particles of fungsten carbide in the form of polygonal prisms or cylinders of cross dimensional of 1 – 10 *mkm*. In this defect ranges are accumulated on the WC – Co interface. Decrease of the number of defects in the crystal under the ionizing radiation in the result of annihilation or defect reconstruction should be accompanied by the heat-evolution, which in due to liberation of the energy stored in the crystal. In this case change of the material structure takes place.

For identification of connection the basic and irradiated samples were analysed by the massspectrometer method of secondary ions. Peaks corresponding to CoWC ions were observed in mass spectrometers of irradiated samples. Appearing the new phase and the change of material defectness also changed the character of sample break. This is confirmed while studying destruction surface by the replica method on the $\Im BM-100$ translucent electron microscope.

Irradiation of solid alloys essentially changed the rupture character:

- the pores along cleavage planes ribs disappear and their numbers on the WC – Co interface decreases;

- the elements of viscous failure – combs separation and grooved patter on the tungsten carbide surface;

- elongation of the cobalt bond around tungsten carbide particles decreased.

Thus, ordering of solid alloy structure in ionizing radiation is accompanied by considerable transformations. The greatest changes take place in the ranges of high defects density, for example, on the WC-Co interface. The structure transformations being observed are likely to be the result of a local heating owing to chain reaction between defects.

The method of acoustic emission, whish generated in process of irradiation, is the most perspective method to control the quality and diagnostics of metals and alloys. The measurement of acoustic emission have been carried out in process irradiation of tungsten, alloys BK8 and T15K6. These alloys are used in manufacturing crowns and boring bits for drilling rocks. There is direct correlation of the number of acoustic emission pulses with strength properties of a solid alloys. Besides, direct correlation between the number of acoustic emission pulses of heading per bit and a mechanical cutting speed of boreholes while boring by a carbide tool. In the defect annihilation region a heat flash appears owing to the released energy stored in the crystal lattice. This energy is about 19 eV. The stored energy release results in the sharp temperature increase in the limited range. Then the temperature immediately decreases up to the temperature of the annihilation region surroundings. In the results of there processes the pressure pulse arises causing the acoustic wave, which is scattered on defects in the materials.

ON THE INTERACTION BETWEEN RADIATION-INDUCED DEFECTS AND FOREIGN INTERSTITIAL ATOMS IN α-IRON

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An interaction has been investigated between Frenkel pair (FP) defects and nitrogen atoms in α -iron by means of a resistivity recovery (RR) method. Both FP partners are captured by nitrogen atoms. A release of self-interstitial atoms (SIA) takes place at 165 K and that of vacancies occurs at 250 K. Binding energies of the both FP defects with nitrogen atoms are about 0.1-0.15 eV. A weak carbon-induced stage is observed at 340 K. Analysis of a position of the stage allows concluding on a decay of the vacancy-carbon atom pairs characterised by a dissociation (binding) energy about 0.9 (0.35) eV.



RR curves of Fe1 (70 appm N) and Fe2 (130 appm N) samples at temperatures above stage I after electron irradiation. Line graphs represent RR data of Takaki et al [1] above stage II depicted for comparison. The dash-dotted vertical line marks 280 K. In the inset: high temperature portions of the Fe1 and Fe2 curves; the dashed horizontal lines mark a stage at 340 K.

Initial Frenkel pair concentrations in samples: Fe1, Fe2 – 130 appm;

Fe – 84 appm; Fe-15C and Fe-67C – 90 appm; Fe-15C(II) – 490 appm.

RR spectra of Fe1 and Fe2 samples. Interpretation of the peaks: 150 K – the onset of free migration of Fe-Ni mixed dumbbells (our iron contains ~100 appm Ni):

<u>165 K</u> – dissociation of SIA from atoms of N;

220 K – trapping of vacancies at atoms of N;

250 K – dissociation of vacancies from atoms of N;

340 K – dissociation of vacancies from atoms of C (estimated concentration does not exceed 1 appm).

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ELECTRICAL RESISTIVITY STUDY OF THE α' PRECIPITATION IN Fe-Cr SYSTEM AT 773 K

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Four Fe-Cr alloys (Cr8.7, Cr10.8, Cr16 and Cr22) were tempered at 773 K during 240 h. Changes in microstructure were monitored by measurements of electrical resistivity at 4.2 K

(residual resistivity, ρ^0) and 374 K, $\rho(374K)$. The temperature-dependent part of the whole resistivity, so called "ideal resistivity", i.e. $\rho^{374K} = \rho(374K) - \rho^0$, was separated. The following correlations between changes in microstructure and ρ^0 and ρ^{374K} were found based on dependencies of ρ^0 [1] and ρ^{374K} on a Cr concentration and data of the small angle neutron scattering and atomic probe tomography [2].

Residual resistivity of Cr16 and Cr22 is insensitive to depletion of the solid solution with Cr, while that in Cr10.8 drops slightly. α' precipitation leads to a rise of residual resistivity when a Cr concentration in precipitates exceeds ~60 at % (C_{crit}). α' precipitates with a Cr concentration below C_{crit} do not affect ρ^0 .

Changes in ρ^{374K} reflect two processes. First, depletion of the solid solution with Cr reduces ρ^{374K} . Second, inhomogeneity induced by formation of α' precipitates (with any Cr concentration) increases ρ^{374K} . Changes caused by the first process dominate in Cr22, while the second process is responsible for increase of ρ^{374K} in Cr10.8 and Cr16 where depletion with Cr is low.



Any reliable changes of ρ^0 and ρ^{374K} were not detected in Cr8.7.

1. The α - α' miscibility gap at 773 K is positioned between 8.7 and 10.8 at % Cr, most probably, closer to 8.7 at % because the Cr10.8 alloy demonstrates clear features of α' precipitation.

2. α' precipitates may nucleate with a Cr concentration below C_{crit} . Incubation time to achieve C_{crit} is very short (<3h) in Cr22 and sufficiently long (~100 h) in Cr10.8 and Cr16.

3. Volume fraction of α' precipitates after 240 h of aging is estimated as 0.15 and 0.4 vol % in Cr10.8 and Cr16, respectively.

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FEATURES OF RADIATION DEFECT FORMATION IN MEDIA WITH NEGATIVE HUBBARD ENERGY

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The negative Hubbard energy taking place in media with strong electron-ion interaction (Anderson, 1975), changes considerably the defect processes, even leading to the transformation of defect into the defecton-inverson [1,2]. Not less essential changes will be shown in the elementary radiation defect formation (RDF) act too. The modern RDF theory for ionic and electronic irradiation based on the interpretations about an output of knock-out atom outside of an instability zone (IZ) during random wandering (Oksengendler, 1978; Vinetsky, 1979), results to the following expression for the cross-section [3] of RDF:

$$\sigma_{d} = \frac{C_{m}}{\left(T_{\max}E\right)^{m}} \left\{ \frac{1}{m} \left(\frac{T_{\max}^{m}}{\widetilde{U}_{0}^{m}} - 1 \right) - \frac{T_{\max}^{m}}{\widetilde{\varepsilon}^{m}} \left(\exp\left(\frac{\widetilde{U}_{0}}{\widetilde{\varepsilon}}\right) \right) \left[\Gamma\left(-m, \frac{T_{\max}}{\widetilde{\varepsilon}}\right) - \Gamma\left(-m, \frac{\widetilde{U}_{0}}{\widetilde{\varepsilon}}\right) \right] \right\}$$
(1)

Here E is the energy of attacking particle, \tilde{U}_0 is the potential energy of displaced atom on border of IZ, $\tilde{\varepsilon}$ is the energy loss of displaced hot atom at his movement in IZ, T_{max} is the largest possible transfer of energy (in a head-on collision) from the fast particle to regular atom, C_m and m are the parameters of colliding particles, $\Gamma(a,x)$ is incomplete gamma function.

In the media with the positive Hubbard energy the radius of IZ by Vineyard-Koshkin is defined by the expression $R_0^+ = a\sqrt{e^2/(\xi a Q_m)}$, where a is the interatomic distance, Q_m is the heat energy of migration of interstitial atom, ξ is the dielectric constant of medium. In the media with the negative Hubbard energy at every recharge of interstitial atom the inversed barrierless migration takes place ($Q^*_m \rightarrow 0$), and the problem of displacement of regular atom at irradiation becomes similar to the Onsager problem (1938) of division of charges. Adapting this result to the RDF problem, it is possible to introduce a new expression for the IZ radius: $R_0^-=e^2/(\xi kT)$. In this case the formula (1) is true. In case of defecton (inverson) character of interstitial atom [1] the theory of RDF based on a formalism of Chapman-Kolmogorov [4] is required.

The proposed theory is able to explain a number of abnormal low-temperature radiating effects in semiconductors.

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MODELING OF RADIATION-INDUCED SEGREGATION IN Fe-Cr ALLOYS AND FERRITIC - MARTENSITIC STEELS

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Under irradiation alloys turn into a steady state with nonhomogeneous composition. Radiation-induced segregation (RIS) leads to significant changes in alloy composition near main microstructural features (point defect sinks): grain boundaries and sample surfaces, dislocations, precipitates, voids and strongly effects the precipitate phase content, swelling, corrosion, embrittlement and other radiation phenomena. When developing new radiation-resistant structural materials one should study the RIS and formation of the radiation-induced phases in these materials.

Experimental data and mechanisms of RIS in Fe-Cr alloys and ferritic - martensitic steels are considered. Physical models and numerical methods are developed for modeling of RIS near plane (grain boundaries and sample surfaces), cylindrical (dislocations) and spherical (precipitates and voids) point defect sinks in ternary substitutional alloys with composition dependent component diffusivities. Molecular dynamics is used for investigation of component diffusivities and mixed dumbbells binding energies in Fe-Cr alloys. Calculations of RIS are performed in Fe-Cr alloys and ferritic - martensitic steels with a special attention to ion irradiation.

The models developed allow to calculate RIS in alloys near various microstructural features under neutron irradiation after modeling of RIS near sample surface under express heavy ion irradiation.

EFFECT OF HETEROGENIUS PRECIPITATION ON VACANCY-DISLOCATION INTERACTION

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The cold working of austenitic steels is most effective method that used today for reduce of a vacancy swelling. However, experimental data show [1], that the effect of dislocations at large degrees of deformation (> 30 %) essentially decreases. It is caused by that processes of origin and growth of pores influence not only a dislocation density, but also their distribution. At large deformation the non-uniform microstructure is generated. Besides, such structure is formed at an irradiation as a result of dislocations climb and also intensive formation of dislocation closed loops. Thus the swelling of steels not only does not decrease, and is incremented in some cases. This effect is intensified by preference of dislocations and dislocation closed loops to interstitials. It is possible to reduce dislocation preference to interstitials and to stabilize dislocation structure of steels by alloying impurities which interact with dislocations.

Positron is a sensitive probe for vacancy-type defects and dislocations. Besides, it has been shown in [2,3], that segregation of impurity atoms and nucleation of intermetallic precipitate particles $Ni_3Ti(Al)$ -type on dislocations results in to change of annihilation parameters. It allows to study heterogeneous nucleation of precipitates on dislocations and effect of this process on

interacting of dislocations with point defects at early stages of an irradiation (<1 dream) when small defect clusters in the form of closed loops or tridimensional clusters are only formed.

In this work Fe-36.5wt. % Ni and Fe-36.5wt.%Ni-2mas%Ti alloys cold worked on 40 % and irradiated with electrons at 300K and 573K were investigated. It has been defined, that vacancy clusters are formed in cold worked Fe-Ni alloy during irradiation though high density of dislocations. It is caused by presence of a lot of regions which are free from dislocations. The accumulation of defects in cold worked Fe-Ni-Ti alloy during irradiation is essentially lowered in comparison with cold worked Fe-Ni alloy. This effect is caused by that Ni₃Ti precipitates particles which are present on dislocations reduce preference of dislocations to interstitials and, thereby, reinforce the mutual recombination of point defects.

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IRRADIATION INFLUENCE BY HEAVY PARTICLES (⁸⁴Kr, E=1.56 MeV/NUCLEON) ON MARTENSIT TRANSFORMATION INTO STEEL 12X18H10T

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The radiating damages and structural-phase transformations in materials of covers fuel element fast reactors are caused not only neutrons, but also heavy fission fragments. At present work we studied martensit transformation into steel 12X18H10T under the influence of an irradiation heavy ions. We used the samples with the sizes of a working part 10x3.5x0.3 mm, which we annealing at 1050°C during 30 minutes. Then a part of samples had been stretching at 20°C, and other part had been irradiation by high-energy ions ⁸⁴Kr (E = 1.56 MeV/nucleon, F₁ = $1x10^{15}$ sm⁻²; F₂ = $4x10^{15}$ sm⁻²) on accelerator DC-60 (Astana, Kazakhstan).

The scanning electron microscope JSM-7500F equipped with the back scattered electrons (HKL) detector was used for study of the structural-phase transformations in steels. We used analysis method of diffraction pattern of back scattered electrons (EBSD) for reception of textural characteristics of samples.

It was founded that α - and ϵ – phases are formed in near-surface layer of the irradiated sample. The increasing of the fluence is reducing to increasing of the α - phase. The morphology features of the α -phase, generated during irradiation were investigated.





Austenite

 α - martensite

Рис. 1 The phases card, which have been removed in mode EBSD, x7500 for the sample of a steel 12X18H10T, irradiated ⁸⁴Kr, F=1•10¹⁵ион/см² (a step of scanning is 0,05 microns)

The results of martensite deformation features in the material are cited in this paper. The similarities and differences of the α – phase, which was formed as a consequence of irradiation and deformation, were analyzed.

KINETIC MONTE-CARLO SIMULATION OF SELF-POINT DEFECT DIFFUSION IN DISLOCATION ELASTIC FIELDS IN BCC (Fe, V) AND FCC (Cu) CRYSTALS

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Simulation of self-point defects (SPDs) diffusion in elastic fields of dislocations of different types in the temperature range (293-1000) K was performed by object kinetic Monte Carlo method for bcc (Fe and V) and fcc (Cu) crystals with dislocation density $\sim (0.1 - 3) \cdot 10^{14} \text{ m}^{-2}$. Interaction energies of SPDs (elastic dipoles) with dislocations were calculated by means of the anisotropic theory of elasticity.

Long-range elastic fields of dislocations determines the efficiency of dislocations as sinks of SPDs to a large degree in the considered temperature range. Dislocation sink efficiencies decrease with the temperature increase and tend to the limit value for linear sink without interaction field. An analytical expression was selected which accurately described the simulation data.

The dependence of dislocation sink efficiency on the dislocation density is described by linear dependence on the square root of the dislocation density with good accuracy in the considered dislocation density range. The specified linear dependence tends to the value approximately equal to $\pi/2$ when dislocation density tends to zero.

Considered features of SPDs behavior in elastic fields of dislocations can exert considerable influence on evolution of material microstructure under damage irradiation.

RADIATION-ENHANCED DIFFUSION PROCESSES IN POLYCRYSTAL AND ULTRAFINE MATERIALS.

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For the first time in experimental practice the method of calculating radiation-enhanced grainboundary diffusion (REGBD) has been developed and the coefficients and parameters of REGBD Ti^{44} in α -Ti and α -Zr have been calculated. It was shown that values of coefficients of enhancement of REGBD are equal to 3,5-7,0 orders and are in good agreement with similar values for radiation-enhanced self diffusion in volume in α -Ti and α -Zr for investigated interval of temperature [1,2]. The estimated value for vacancy migration energy for grain boundaries is equal to 0,9 eV for α -Ti and α -Zr and is in agreement with data for volume diffusion.

The methodological scheme of calculating of REGBD coefficients for different types of boundaries in nanocrystall materials with hierarchic structure has been developed. For calculating radiation enhanced diffusion coefficients for boundaries of nanoclusters the model, which was earlier developed by authors and takes into account influence of impurities and vacancy-impurity complexes, has been used [1,2]. Using the earlier obtained REGBD data for α -Ti and α -Zr and the methodological scheme the calculating of coefficients and parameters of REGBD for nanoclusters boundaries has been done.

It has been shown that at certain values of binding energy of complexes and high content of impurities on flows the significant decreasing (several orders) of vacancy migration energy and increasing of mobility level at low temperatures is observed at nanoclusters boundaries in comparison with nanograin boundaries.

For the first time in experimental practice the calculating of radiation-enhanced grain boundary interdiffusion coefficients (REIGBD) in Zr-Nb system has been done using the estimation method for calculating radiation-enhanced interdiffusion coefficients in the same system. Using this system the estimation model for temperature dependences of radiationenhanced interdiffusion coefficients in nanocrystall materials has been developed for different share of boundaries. The effective coefficients of REGB and REIGBD in Zr and in Zr-Nb alloys have been received.

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KINETICS OF DEFORMATION- AND RADIATION-INDUCED SEGREGATION DEVELOPMENT IN Fe-Cr-Ni ALLOY

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Kinetics of nickel segregation on grains boundaries (GB) in Fe-Cr-Ni alloy at intensive plastic deformation and in conditions of an irradiation is investigated in the present work. In both cases formation of segregations is connected to the directed flows of dot defects from volume to GB which result in an alloy component division in near-boundary area (reverse Kirkendall effect). Kinetics of segregations formation was studied by the numerical solution of the diffusion equations which are taking into account a birth and absorption of dot defects and also their mutual recombination [1, 2]. It is shown, that nickel concentration dependence of time for radiation-induced segregation (RIS), which is usually observed at elevated temperatures, can be nonmonotone both for motionless, and for moving boundary. Thus nickel segregation to GB at an irradiation at a stationary state can be less than during previous time. It is shown, that occurrence of such nonmonotonicity is caused by development of "fast" RIS stage [2]. The width of a near-boundary zone of enrichment by nickel grows monotonously in time reaching stationary value. Similar dependence of time of a near-boundary zone width of enrichment by nickel has qualitatively similar character in case of deformation-induced segregation (DIS). Unlike a case of RIS, dependence of nickel concentration of time at DIS was monotonous owing to the fact that DIS is usually observed at moderate temperatures (in the present work the temperature was close to room temperature) at which the stage of "fast" DIS does not develop.

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RADIATION-INDUCED PLASTIC DEFORMATION

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Under radiation influence plastic deformation can be developed within nanoscale distances. At the ionic or the laser influence this is connected with the effect of volume increasing of the thin implanted layer or with thermal extension of the scin-layer. In the cascade radiation influence the plastic deformation appears due to the unelastic relacsation of the cascade regions with the sizes up to 10 nm of very high energy up to several eV per atom.

In the case when the scale of the plastic deformation development is less than minimum of the possible distance between dislocations the nondislocation hardening of the metals appear. This is characterized by the greater (on order) modul of strengthening. Plastic deformation is connected

with formation and evolution two-measured defects (shear planes, twins, stacking faults and so on). The density of such defects can be more than 10^7sm^{-1} .

The possibility of the process of nondislocation plastic deformation has been showed. In this case the structurization within nanoscale. The consideration made in the frames of Ornstain-Tcernice theory used to plastic deformation. The growth of the fluctuations of the plastic current leads to specific nanoclusteric structure of a metal which can be registered by the microscopic methods. Appearance of such structures is accompanied by the great changes of material properties (mechanical, electrophysical). These can be also connected with the known radiation phenomena in materials (swelling, brittlement, electrical conductivity degradation and so on).

The experimental results showed illustrate the peculiarities of the plastic deformation at the ionic implantation of metallic materials (pure metals Zr, Ti and alloys Fe-Ni-Cr, Ni-Cr, Cu-Ni, Fe-Cr, V-Ti-Cr, intermetallics – Heusler phases Cu₂MnAl and Ni₂MnGa). After irradiation by ions of various nature within the narrow range of radiation parameters (doses, target temperatures and ionic flux intensities) the so-called R-states appear. These states have the characteristic clusteric structures (cluster sizes of 3 - 100 nm)which extend for great depths (long range effect). In the materials there are increasing of microhardness several times and also increasing of magnetization (in the Heusler phases).



The most topical problem of today is development of new metal materials for fusion and fission-type reactors. Ion 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 hightemperature 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).

NANOSTRUCTURE CHARACTERIZATION OF OXIDE DISPERSION STRENGTHENED STEEL - ODS EUROFER NEUTRON IRRADIATED UP TO 32 DPA

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Oxide dispersion strengthened steels possess better high-temperaturecreep and radiation resistance than conventionally produced ferritic/martensitic steels. This behavior is mainly caused by the presence of highly dispersed and extremely stable oxide particles with diameters of a few nanometers.

In this work the nanostructure of European steel ODS Eurofer (9%-CrWVTa) in unirradiated as well as in irradiated at 330°C to 32 dpa were investigated. The irradiation was performed in the research reactor BOR-60 at SSC RF RIAR (Dimitrovgrad, Russia). Nanoscaled clusters of typically 2 nm diameter containing not only yttrium and oxygen but also vanadium and nitrogen were found in unirradiated state. Moreover, concentration of vanadium in particles was found to be higher than that of yttrium, which indicates the importance of these elements in cluster formation. The estimated average cluster number density is about 2×10^{24} m⁻³.

For irradiated state TAP needles were prepared from the rests of Charpy samples after impact testing. For all samples except one, which was tested at 500° C, the tests were performed at temperatures not exceeding the irradiation temperature. A high number density $2 \div 4 \times 10^{24} \text{ m}^{-3}$ of ultra fine 1-3 nm diameter nanoclusters enriched in yttrium, oxygen, manganese and chromium was observed in the as-irradiated state. It was observed that after neutron irradiation vanadium atoms had left the clusters, moving from the core into solid solution. The concentrations of yttrium and oxygen in the matrix, as it was detected, increase several times under irradiation. This effect can be tentatively explained by dissolution of the larger yttrium oxide particles (more than 10 nm in diameter) which can be hardly detected by tomographic atom probe, but commonly observed with TEM.

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INVESTIGATION OF GAS POROSITY FORMATION AND DEVELOPMENT IN U-Mo FUEL UNDER IRRADIATION CONDITIONS

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U-Mo dispersion fuel is a promising candidate for research reactors with U^{235} low-enriched fuel. During the irradiation process the porosity is formed due to GFP release in the fuel. The post-irradiation examination of irradiated U-Mo/Al dispersion fuel has revealed that its stable behavior under irradiation conditions depends on the ability of the U-Mo alloy to keep hold of gas fission products. Therefore it is important to research the dependence of morphology of forming voids and changes of its quantitative characteristics on the irradiation parameters.

Methods of scanning microscopy and quantitative stereometric analysis [1] investigate the

parameters of the porosity in the U-Mo/Al dispersion fuel after IVV-2M irradiation burn-up up to 93% (Zarechny).

During the raster electronic-microscopic analyzing of the secondary and back-scattered electrons it was defined that gas bubbles have a crystallographic octahedron cutting and layer thickness, that forms the contrast from the gas bubbles, was determined. The porosity parameters were estimated regarding the thickness of the surface layer where the contrast from pores is formed.

The minimal porosity is observed in the fuel particles with minimal burn-up. The gas bubbles are formed mainly at the boundaries of the fuel grains. With increasing burn-up gas bubbles distribution become homogeneous over the fuel particles volume. The bubble size increases. Parameters of the porosity depend on the burn-up as well as the fuel fission rate and irradiation temperature. It was established that the specific volume of the gas bubbles increases with the burn-up up to the maximum and the further decreasing coincides with the beginning of the critical form change connected with the fuel-cladding swelling formation. Thus the specific volume of the gas bubbles in the fuel particles can be used as a generalized parameter for the determining of boundary conditions of fuel elements safety operation.

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ANISOTROPIC SWELLING OBSERVED DURING STRESS-FREE REIRRADIATION OF AISI 304 TUBES PREVIOUSLY IRRADIATED UNDER STRESS

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A "history effects" experiment was conducted in the EBR-II fast reactor that involved the reirradiation of AISI 304 cladding and capsule tubes. It is shown that when irradiated tubes had not previously experienced stress, subsequent irradiation led to additional swelling strains that were isotropically distributed. However, when tubes previously irradiated under a 2:1 biaxial stress were reirradiated without stress the additional swelling strains were not isotropically distributed. The tubes retained a memory of the previous stress state that appears to be attempting to distribute strains in the directions dictated by the previous stress state. It is clear, however, that the memory of that stress state is fading as the anisotropic dislocation microstructure developed during irradiation under stress is replaced by an isotropic dislocation microstructure during subsequent exposure in the absence of stress.

It is also shown that once the transient regime of swelling nears completion, further changes in stress state or irradiation temperature have no influence on the swelling rate thereafter. A comparison is made with another published experiment on AISI 304 where specimens previously irradiated in the stress-free state were subsequently reirradiated under stress. In both cases the terminal swelling rate was $\sim 1\%$ /dpa, regardless of the distribution of strains.

ENHANCEMENT OF IRRADIATION CREEP OF NICKEL-BEARING ALLOYS IN THERMALIZED NEUTRON SPECTRA CHARACTERISTIC OF LWR AND CANDU[©] REACTORS

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Studies of neutron-induced stress relaxation conducted in water-moderated reactors prior to the early 1980s derived irradiation creep rates that were later found to be significantly higher than were derived from later experiments conducted in fast reactors. More recent experiments continue this trend. The question arises whether water-moderated neutron spectra indeed cause more irradiation creep per dpa or whether some other factor is acting to cause an apparent increase. The answer now appears to be a combination of these two possibilities, but with both arising from an identical but previously unanticipated source.

It was in the early 1980s that the ⁵⁸Ni (n, γ) ⁵⁹Ni reaction with thermal neutrons was recognized as a precursor to enhanced helium production via the ⁵⁹Ni (n, α) reaction and in the late 1980s to enhanced hydrogen production via the ⁵⁹Ni(n, p) reaction. Both of these reactions are highly exothermic and produce unexpectedly high amounts of additional atomic displacements. The unrecognized increases in dpa rate lead to calculations of artificially high rates of creep in thermalized spectra. Additionally, the generation of helium and hydrogen-filled bubbles causes a new creep mechanism called bubble-enhanced creep to occur, with the creep rate increasing by both processes.

In some extreme cases of very highly thermalized spectra operating on high nickel alloys (X750 in CANDU far-from-core positions) the ⁵⁹Ni contributions totally dominate the production of dpa, producing total relaxation of preloaded springs in less than 3 years whereas previous predictions based on in-core tests predicted relaxation would occur only after 600-700 years.

PECULIARITIES OF PLASTIC DEFORMATION PHENOMENA IN HIGH-IRRADIATED AISI 304 AND AISI 316 STAINLESS STEELS

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Recently the "true stress – true strain" curves have been widely published and discussed for irradiated materials. The "true curves" have many advantages compared to the traditional engineering curves. For example, the true curves give the ability to investigate the real deformation behavior, but few papers are devoted to irradiated material "true curves" analytical presentation. In metastable chromium-nickel steels the deformation hardening is under the influence of other phenomena. These phenomena include formation and accumulation of α -martensite. It influences stress-corrosion resistance and other material performance measures. Despite practical importance and scientific interests of martensitic transformation, it has not been thoroughly investigated for irradiated materials. Other key factor influencing material property and plastic flow process is grain size. In some cases due to fabrication methods the material grain

size may be as large as 200-1000 micrometers. It is interesting from a scientific and practical point of view to investigate large grain irradiated material.

In the present work, the results of plastic flow, deformation hardening and martensitic $\gamma \rightarrow \alpha$ transformation are described and analyzed for AISI 304 and AISI 316 stainless steels of industrial heats and for the same steels with molybdenum and hafnium additions. Also samples of cast irradiated AISI 316 steel were tested. Flat samples with gauge dimensions 7.6×1.5×0.75 mm and round samples with gauge diameter 2.1 mm and length 12 mm were tested (tensile, compression and indentation tests) at temperature intervals of RT-290°C. Post-deformation structure investigation of samples was performed with using of scanning electron microscopy and EBSD-analyze. For martensite content measurement the ferroprobe unit and magnetic saturation methods were used.

The standard engineering mechanical properties, values of critical stress σ_{cr} and strain ε_{cr} corresponding to the start of martensitic transformation were defined. By using optical extensometry, the diagrams "true stress σ – true strain ε " were obtained. For some cases during archive and literature data analysis, calculated true curves were used. Kinetic curves of martensite accumulation were obtained as result of tensile, compression and ball indentation tests. The influence of irradiation on austenite stability of investigated steels are estimated and discussed. Peculiarities of irradiated steels plastic flow are investigated and analyzed with using of different plasticity physics equations (Ludwig, Swift, Holloman). Impact of irradiation on parameters of these equations is discussed.

EFFECTS OF OVERSIZED SOLUTES ON RADIATION-INDUCED SEGREGATION IN AUSTENITIC STAINLESS STEELS

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Radiation-induced segregation (RIS) in austenitic stainless steels is characterized, in part, by a detrimental loss of Cr at grain boundaries caused by irradiation-produced defect diffusion to sinks. Since RIS is considered to be part of a complex process that increases the susceptibility for irradiation-assisted stress corrosion cracking (IASCC) in light water reactors [1, 2], reducing RIS may benefit IASCC susceptibility.

One method that has been examined for suppressing RIS is the addition of oversized solute atoms to enhance recombination and reduce defect diffusion to sinks. The primary objective of this work is to establish the mechanism by which oversized solute atoms reduce RIS in stainless steel. A solute-vacancy trapping mechanism and its effect on RIS were incorporated into the composition-dependent modified inverse Kirkendall (MIK) model [3]. The trapping model (MIK-T) revealed that the binding energy was the most important parameter to the trapping model and that large binding energies were required for significant reductions in RIS [4].

Austenitic stainless steels with Zr or Hf additions (compositions shown in Table 1) were irradiated with 3.2 MeV protons at 400°C and 500°C [5]. Grain boundary Cr depletion was measured with transmission electron microscopy and showed a dramatic reduction in the amount of Cr depletion up to 3 dpa at 400°C and 1 dpa at 500°C. However, the experiments showed that the effectiveness of the solute additions disappeared above 3 dpa at 400°C and above 1 dpa at

500°C. The loss of solute effectiveness with increasing dose is attributed to a loss of oversized solute from the matrix due to growth of carbide precipitates. Solute concentrations were based on an imaging analysis of carbide sizes and densities to determine the amount of solute remaining in solution. Atom probe measurements observed the loss of oversized solute in solution as a function of irradiation dose. These observations were supported by diffusion analysis suggesting that significant solute diffusion by the vacancy flux to precipitate surfaces occurs on the time scales of proton irradiations.

POSTIRRADIATION EXAMINATION OF AFC-1 METALLIC TRANSMUTATION FUELS AT 8 AT.%

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Introduction

The U. S. Advanced Fuel Cycle Initiative (AFCI) seeks to develop and demonstrate the technologies needed to transmute the long-lived transuranic actinide isotopes contained in spent nuclear fuel into shorter-lived fission products, thereby dramatically decreasing the volume of material requiring disposition and the long-term radiotoxicity and heat load of high-level waste sent to a geologic repository. The AFC-1 irradiation tests are designed to evaluate the feasibility of the use of actinide bearing fuel forms in advanced sodium-cooled fast reactors for the transmutation of transuranic elements from nuclear waste [1]. AFC-1B, AFC-1F and AFC-1Æ are the first set of irradiation tests to be studied. AFC-1B and AFC-1F contain non-fertile and low-fertile actinide bearing metallic alloy fuel compositions and is discussed in a separate paper [2]. The tests were irradiated in the Idaho National Laboratory's (INL) Advanced Test Reactor (ATR) to an intermediate burnup of 4 to 8 at.% ($3.5 - 8.9 \times 10^{20} \text{ fiss/cm}^3$). Sibling tests are being irradiated to reach a final burnup up to 40 at.%.

Experiment Description

The AFC-1B. AFC-1F and AFC-1Æ irradiation tests were designed to provide irradiation performance data at intermediate burnups of 4 to 8 at.% on non-fertile and fertile actinide transmutation fuel forms containing plutonium, neptunium and americium isotopes [Refs. 3-6]. AFC-1B contains four non-fertile metallic fuel compositions (Pu-12Am-40Zr, Pu-10Am-10Np-40Zr, Pu-60Zr and Pu-40Zr). There are two Pu-12Am-40Zr rodlets irradiated to different burnup. AFC-1F contains four fertile metallic fuel compositions (U-28Pu-4Am-2Np-30Zr, U-27Pu-3Am-2Np-40Zr, U-34Pu-4Am-2Np-20Zr, and U-29Pu-7Am). There are two rodlets each of the U-28Pu-4Am-2Np-30Zr and U-27Pu-3Am-2Np-40Zr compositions.

The rodlet assembly is designed as a miniature fast reactor fuel rod with a standard diameter and reduced length. The rodlet assembly consists of the metallic or nitride fuel column, sodium bond, stainless steel Type 421 (HT-9) cladding and an inert gas plenum. A stainless steel capsule assembly contains a vertical stack of six rodlet assemblies. Details of the capsule and rodlet radial dimensions of the metallic and nitride fuel specimens were provided previously [3].

Postirradiation Examinations (PIE)

Preliminary results of postirradiation hot cell examinations of AFC-1B and AFC-1F

irradiation tests are reported for five non-fertile metallic alloy transmutation fuel rodlets and six low-fertile metallic alloy transmutation fuel rodlets. Non-destructive examinations on the eleven metallic fuel rodlets included visual examination, dimensional inspection, gamma scan analysis, and neutron radiography. Detailed examinations, including fission gas puncture and analysis, ceramography and isotopics and burnup analyses, were performed on three non-fertile and two low-fertile metallic alloy transmutation fuel rodlets.

Figure 1 displays the fission gas release burnup dependence of the 11 metallic alloy rodlets and includes data of U-xPu-10Zr (i.e. x=0 - 26) for comparison. The fission gas release behavior of the transmutation metallic fuels follows the same trend as the U-xPu-10Zr fuel when correlated with fission density. There is negligible amount of fission gas release until a fission density of ~6.0 x 10^{20} f/cm³. At this threshold burnup, there is a rapid increase in fission gas release that is displayed by the AFC-1 metallic fuel samples similar to U-xPu-10Zr behavior. It is significant that there is no difference in fission gas release behavior between the non-fertile (non-uranium bearing) and low-fertile (uranium bearing) compositions. Based on gas release at this intermediate burnup, it is predicted that fission gas release of the transmutation fuels at higher burnup will show a slightly increasing plateau. This will be evaluated during PIE of the sibling experiments AFC-1D and AFC-1H.



Fig. 1. Fission gas release from metallic alloy transmutation fuel rodlets irradiated in AFC-1B and AFC-1F Capsules and U-xPu-10Zr (x=0-26)

Comparisons of metallography indicate the transmutation metallic alloy fuels show similar microscopic and swelling characteristics as U-xPu-10Zr fuels at equivalent burnup. The U-xPu-10Zr metallic alloy fuels historically exhibit three stages of porosity development and swelling: 1. incubation characterized by very low swelling rate up to a threshold burnup of ~0.35 x 10^{21} f/cm³ (1.0 at.% U-xPu-10Zr), 2. transition characterized by rapid fission gas driven swelling until a network of open porosity fully develops, the fuel contacts the cladding and majority of fission gas is released occurring at burnups between ~0.35 – 1.0 x 10^{21} f/cm³ (1.0-3.0 at.% U-xPu-10Zr) and 3. stable irradiation change characterized by a slowly increasing plateau due to accumulation of solid fission products and retained fission gas occurring at burnups > 1.0 x 10^{21} f/cm³ (3.0 at.% U-xPu-10Zr). Fig. 2 displays metallograph of the low-fertile metallic alloy composition at two burnups, 0.42 and 0.60 x 10^{21} f/cm³, indicating that the transmutation metallic fuels transition stage occurs at fission density around 0.6 - 1.2 x 10^{21} f/cm³. The observation of the transition behavior being similar to U-xPu-10Zr fuels provides another indication that the fuel performance of metallic fuel forms is similar for a wide range of actinide compositions and zirconium content.

Results

Preliminary postirradiation hot cell examinations of AFC-1 irradiation tests were completed
for eleven metallic alloy transmutation fuel rodlets. The preliminary conclusions are summarized below:

- 1. No rodlet failures were observed in the actinide-bearing metallic fuel forms irradiated to 4-8 at.%.
- 2. The irradiation swelling and fission gas release of metallic alloy transmutation fuel compositions Pu-TRU-40Zr and U-xPu-TRU-yZr (x=25-34: y = 20-40) is similar to that of U-xPu-10Zr (x=0-26).
- 3. The threshold burnup of metallic alloy transmutation fuels to exhibit significant swelling and fission gas release is $\sim 0.6 \times 10^{21}$ f/cm³.



Fig. 2. Transverse cross-sectional metallography montages of U-29Pu-4Am-2Np-30Zr samples a) Mount LAR-3A irradiated to 0.42×10^{21} f/cm³ (6.4 at.%) burnup showing developing porosity and swelling and b) Mount LAR-3B irradiated to 6.0 x 10^{20} f/cm³ (8.9 at.%) burnup showing fuel fully swollen leading to fuel-cladding contact and initial formation of fuel restructuring with central dense zone and porous annular zone

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STUDIES OF THE CORROSION OF MATERIALS BY LEAD BISMUTH EUTECTIC (LBE) AT UNLV, USA

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This paper will review the work investigating the corrosion of materials by oxygen controlled LBE conducted at UNLV and elsewhere in the US, focusing on the efforts the Johnson/Farley group. The effects of composition and surface modification on LBE corrosion were examined. Austenitic stainless steels (316 and D9) (exposed at IPPE, Russia) will be highlighted. Studies of the effects of silicon in iron-silicon alloys (exposed to LBE at Idaho National Laboratory, USA) and aluminum in steels (exposed in the DELTA loop, Los Alamos National Laboratory, USA) in LBE corrosion will be discussed. Theoretical descriptions of the corrosion mechanism (based on the available space model) and new characterizations using Raman spectroscopy will be mentioned. Work in progress with radiation enhanced LBE exposures will be outlined.

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STRUCTURAL AND CONCENTRATION CHANGES IN THE ZONE OF THERMAL FACING OF HIGH-PRESSURE-VESSEL STEEL

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The methods of light and electron microscopy were used to study changes in the structure of both the facing of the 12Kh18N10T austenitic stainless steel and the base material of the reactor vessel made of the 12KhGMFA steel. The structural investigations of the buildup layer of the stainless steel showed that the austenitic structure contained a small quantity of the delta ferrite in the form of thin interlayers at grain and subgrain boundaries. An examination in a JEOL scanning electron microscope with a stepwise microanalysis revealed intragranular segregation of chromium and nickel in the cast facing. The local concentration of chromium in the grains changed from 20 to 17.8 wt %; nickel, from 10.5 to 9%; and manganese, from 1.2 to 2.1%. The persistence of the dendritic segregation is explained by the fact that the facing operation was performed without high-temperature homogenization.

The depth of the mutual penetration of the alloying elements in the transition layer between

the austenitic facing (12Kh18N10T steel) and the martensite-like structure of the 12KhGMFA structural steel during the build-up operation was determined. The concentrations of Fe, Cr, Ni, and Mn were measured in a step-by-step microanalysis performed every 3 μ m of the transition zone. This transition zone of size 33 μ m spreads approximately an equal distance from the buildup boundary towards the stainless steel and the structural steel. This factor should be taken into account since martensite, which impairs the plastic characteristics of steel, is formed in the chromium- and nickel-depleted transition zone of the stainless steel. This zone has approximately the same composition as the Kh12N7G1 steel, in which the martensite point is near 100-200°C, and a martensitic transformation is implemented as the facing is cooled.

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THE COMPARATIVE STUDY OF BN-600 REACTOR RADIATION EFFECTS ON CHANGING ELASTICITY AND ELECTRICAL RESISTANCE IN "CHS68" AND "EK164" AUSTENITIC STEELS.

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There are test results on physical and mechanical properties of "ChS68" steel, the main material for BN-600 fuel claddings, and of "EK164" steel, a more radiation-resistant material. In the initial condition the specific resistance of the "EK164" steel is 5% higher than that of the "CHS68" steel and Young's modulus is as low as that. In physical properties the greatest differences is observed for a Poisson's ratio: its value is ~0.17 for the "EK164" steel and about 0.30 for the "ChS68" steel.

The electrical resistance of both steels increases considerably after irradiation in BN-600 up to damage doses of ~78 d.p.a. and their elastic moduli decrease. The initial condition characteristics were obtained for the regions of the fuel claddings irradiated up to a dose of ~0,5 d.p.a. at 370 °C from the analysis of the changing physical and mechanical properties (PMP). The value of PMP change in the "EK164" steel specimens is lower than in the"ChS68" ones. The changes of the electric resistance and elastic moduli are mainly contributed by swelling that is significantly lower in the "EK164" steel than in the "ChS68". The relative PMP change can be easily described by the formula obtained from the two-component model developed earlier [1]:

$$\frac{\Delta R}{R_o} = \frac{5 \cdot S}{4 \cdot S + 6},\tag{1}$$

$$\frac{\Delta E}{E_0} = \frac{1}{\left(1+S\right)^2} - 1,$$
(2)

where $\Delta R, \Delta E$ - the changes in electric resistance and Young's modulus;

 R_o, E_o - the electric resistance and Young modulus in the initial condition;

S - the swelling.

The Poisson's ratio is different for different fuel elements claddings and practically does not change with the damage dose. A correlation between the Poisson's ratio and the fuel cladding swelling values of both steels has been noted.

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MECHANICAL ACTIVATION OF SURFACE-OXIDIZED METAL MATRICES AS A METHOD FOR CREATION OF HIGH-PRESSURE-VESSEL ODS ALLOYS OF A NEW CLASS

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In recent years mechanical alloying attracts the increasing attention of specialists dealing with the development of materials for the atomic power engineering, including high-temperature oxide-dispersion-strengthening (ODS) steels and alloys, which resist high-temperature swelling on exposure to fast neutrons. The traditional use of yttrium oxides with a high atomic binding energy as oxygen carriers is not always technologically expedient. They require much more time for their processing in ball mills and dissolution in steel than unstable iron oxides (e.g., hematite Fe₂O₃). It was found that deformation of iron oxides mixed with metals having a high affinity for oxygen (Ti, Y, Zr) and steels alloyed with chemically active metals is accompanied by an active dissolution of the initial iron oxides of the matrix elements. This method can be simplified still more if a fine powder of iron or steel is oxidized first on the surface to form particles of an oxide film, and then this film is dissolved in the steel matrix under deformation.

Oxidized powders of iron and alloyed steels were severely deformed in a mill for 4...16 h in argon so that the surface oxides dissolved in the iron and steel matrices. The disks were compacted by pressure shear and annealed at temperatures of 750, 900, and 1100°C (30 min) so as to synthesize nanoscale strengthening oxides of iron. As the annealing temperature was elevated, iron oxides were formed; the density of dislocations decreased; the ferrite matrix recrystallized; and the grain size increased. Particles of the iron oxides became more visible against the recrystallized matrix. The particles preserved their fineness even upon heating to 1100°C. The average size of the particles was 3.1 nm, and their concentration was $2.8 \cdot 10^{22}$ m⁻³. This was sufficient for a considerable improvement of the strength and the high-temperature strength of the initial metal matrices.

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EFFECT OF COMPRESSIVE AND TENSILE STRESSES ON SWELLING AND CREEP STRAIN OF Fe-18Cr-10Ni-Ti AUSTENITIC STEEL

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At present, work in justification of the lifetime prolongation of the operated VVER-440 and VVER-1000 internals as well as of the operation of new VVER reactor internals up to 60 years is the most urgent. Fe-0.08C-18Cr-10Ni-Ti austenitic steel, being the material of operated and new VVER internals, was selected for the experiment. As the design of internals is very complicated and there are many holes for cooling, areas with compressive and tensile stresses may appear, so it is important to investigate the effect of stresses on the properties and structure of the material. Experiments to investigate the effect of tensile stress on the properties and structure of the material have been carried out both at "SSC RIAR", Russia [1-3] and abroad, but the effect of compressive stress have not been practically studied. Besides, we had to check if the known mechanisms and dependence of creep strain on stress type would remain. This paper presents the effect of compressive and tensile stresses on swelling, microstructure and creep strain of Fe-0.08C-18Cr-10Ni-Ti steel.

Pressurized specimens of complicated geometry fabricated from austenitic steel Fe-0.08C-18Cr-10Ni-Ti were irradiated in the BOR-60 reactor at ~400°C up to a damage dose of over 30 dpa.

The specimens were located on one of the levels of the irradiation rig MP-146. Irradiation was performed in the BOR-60 blanket. During the reactor shutdowns for maintenance and reloading of fuel assemblies, the diameter and length of specimens were measured. There were four series of measurements with an interval of about 4 dpa.

Examination of the microstructure of both irradiated and unirradiated pressurized specimens were performed by means of transmission electron microscope JEM-2000FXII.

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THE EFFECT OF LOW DOSE NEUTRON IRRADIATION ON THE TENSILE AND IMPACT PROPERTIES OF A SERIES OF TITANIUM ALLOYS.

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Structural materials used in the first wall of fusion reactors or in any type of nuclear power device will become activated and will represent an environmental hazard during their storage. Titanium is the natural metallic element having the fastest radioactive decay. Binary or ternary titanium alloys can be designed to keep this basic property and at the same time improve the mechanical properties of pure titanium. The most widely used titanium alloy is Ti6Al4V. This metastable alpha-beta alloy suffers from structural instabilities when exposed to radiations. Vanadium precipitates are generated deteriorating the ductility and inducing brittleness. [1, 2].The situation is better with alpha alloys. A few available results indicate that the resistance to irradiation is improved compared to alpha- beta alloys [3]. Nevertheless the unique industrial alpha alloy available Ti5Al2.5Sn suffers also from phase instabilities and is not an excellent low activation material due to the presence of aluminium [2].

In this study, a series of simple ternary and binary alloys have been developed in the laboratory, based on the substitution of Al by Zr. Since the mechanical strength of titanium is strongly dependent on the forging process, a beta anneal heat treatment has been given to all alloys in order to have a better comparison perspective. The tensile and impact properties before and after irradiation to low dose, have been followed together with the properties of pure titanium.

Alpha and alpha-beta industrial alloys have the best strength but show poor low temperature impact properties after irradiation. Pure titanium has good impact properties after irradiation but has a relatively low strength. As expected, the impact strength correlates well with the strength. The impact properties deteriorate rapidly with irradiation hardening.

The results indicate that the most promising composition is a binary alloy with about 5% Zr.

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CHANGES IN THE MECHANICAL PROPERTIES OF "EK164" AND "CHS68" STEELS FUEL CLADDINGS AFTER A LONG-TERM OPERATION IN THE BN-600 REACTOR.

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At the present time an achievement of high fuel burn-up in fast reactors is an urgent problem in nuclear power. For this purpose the fuel claddings made of "ChS68" austenitic steel and manufactured by the advanced technology at the Moscow Staleliteiny Plant and those made of the new "EK164" austenitic steel developed by the Bochvar Institute, Moscow, were used.

In this work the "ChS68" and "EK164" fuel claddings after their operation in the BN-600 reactor for 560-572 effective days up to the highest burn-up of 9.1 -9.7% h.a. and a damage dose per cladding up to 82.4 d.p.a. were studied.

Mechanical properties were determined at temperatures of 20, 400, 600 °C and the operation temperature of investigated parts of the fuel claddings. The changes in diameters and swelling along the fuel elements are presented. The results of metallographic investigations and damaged surface fractography after mechanical tests of specimens of fuel claddings taken from different positions in core regions were also used.

It has been shown that in the core center a maximum swelling and diameter change of "ChS68" claddings are 1,5-2 times higher than those of "EK164" under similar operation conditions.

The determination of the mechanical properties was performed using the traditional method of a uniaxial tension of annular specimens as well as the new method of testing of tubular specimens by an internal pressure of plastic filler. Zero plasticity and low strength values were detected for the annular specimens of the claddings taken from the core center while determining the "ChS68" mechanical properties by the traditional method. By the internal pressure method the higher strength values and a non - zero plasticity were obtained.

The fractographic studies showed that the surface failure was mainly an transgranular one and, at some magnification, it could be referred to a quasi-cleavage. At high magnifications the cleavage facets had a small-cup pattern.

The results in total showed that the mechanical properties of the "EK164" steel after high dose irradiation are higher than those of the "ChS68" steel.

ATOM PROBE INVESTIGATION OF NANOSCALE STATE OF ODS EUROFER STEEL AFTER HEAVY ION IRRADIATION

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Understanding of radiation stability of promising nuclear structural materials actively implicates modeling experiments utilizing (exploiting) different types of influence. It is known that the fastest and most reliable way of creating radiation damage in investigated material is low-energy heavy ion beam irradiation. Exposure to low energy heavy ions leads to inhomogeneous defect distribution thus in such experiments specially prepared specimens for microscopic experiments are used. The present work is devoted to the workout of modeling experiments on specimen irradiation for further tomographic atom probe microscopy investigation.

The first experiments on irradiation of the promising steel for fusion and fission power plants were carried out in ITEP. Material – ODS EUROFER was subjected to the iron ions beam with energy of 150 keV up do damaging doses of 13 dpa. Irradiation had been performed on leniar accelerator TIPr (ITEP). ODS EUROFEr steel is dispersed strengthened with yttrium oxide and has high number density (~ 10²⁴ M^{-3}) of nanometer-sized clusters. Analysis of different chemical elements position in irradiated volumes showed alterations in nanocluster chemical composition. Increase of the dose results in concentration reduction of O, N and V, whereas concentration of Mn goes up. As for the clusters, their sizes and number density remain the same. Comparison of these data with the one obtained after neutron irradiation in BOR-60 up to 32 dpa shows correlation in nanoscale alterations in the ODS EUROFER.

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CHARACTERISTICS OF RADIATION POROSITY FORMED IN THE MATERIAL OF "EK164" AND "CHS68" STEELS FUEL CLADDINGS UNDER IRRADIATION IN THE BN-600 REACTOR.

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At the present time the austenitic steel "EK164-ID"c.w. is considered as a candidate material to reach a maximum fuel burn-up. For the estimation of its efficient use in BN-600 a combined fuel element assembly had been operated for 560 effective days; the assembly had fuel claddings made of the new "EK164-ID" and main "ChS68-ID" steels.

This work studies the characteristics of porosity formed in the "EK164-ID" c.w. in the fuel claddings material exposed to a maximum damage dose of 77 d.p.a. in the BN-600 reactor. For a comparison the specimens of unirradiated steel "EK164-ID" c.w. of the same melt that of the investigated fuel elements and the specimens taken from the lower region of a gas cavity of different fuel elements were studied. The differences in a dislocation structure and phase composition of the unirradiated specimen and those taken from the lower region of a fuel element gas cavity have been found.

The specimens of investigated fuel elements corresponded to the three irradiation temperature ranges, i.e. low, average and high. Histograms of size distribution of voids were plotted and described as a sum of unimodal logarithmic-normal distributions which corresponded to the voids of different types. At the same time the specimens of the similar temperature range for the fuel element with a "ChS68-ID" c.w. cladding were studied; the fuel element was operated as incorporated into the same assembly as the fuel elements with "EK164-ID" c.w. claddings. The porosity characteristics were determined on the "EK164-ID" c.w. specimens for all the temperature ranges.

The obtained porosity characteristics of the two steels in one and the same temperature ranges have been compared. The mean void size increases with irradiation temperature in both steels but

this size is higher in "ChS68-ID" c.w. than in "EK164-ID"c.w. The swelling was determined by using the hydrostatic weighing method and electronic microscopy. It has been found that the value of porosity and swelling of the "ChS68-ID" c.w. steel specimens is higher by a factor of 1.5 and more than that of the "EK164-ID" c.w. specimens irradiated under the similar conditions.

TRAPPING OF HYDROGEN ON NANOSCALE PARTICLES OF YTTRIUM OXIDE IN ODS STEELS

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Oxide-dispersion-strengthened (ODS) ferritic chromium steels have necessary properties for use as materials of fuel elements in fast neutron reactors. A large number of point defect sinks is formed in these steels by their production technology.

The ODS1 and ODS5 steels under study were produced by the method of pressure sintering of powdered iron, ferrochrome, and (in ODS5 steel) Y_2O_3 oxide. The FeCr alloy was prepared of carbonyl iron and ferrochrome.

The radiation-induced segregation of deuterium in the steel and alloy samples was measured using a D(d,p)T nuclear reaction. The depth of analysis was determined from the length of the projective path of a 700-keV deuteron in the analyzed materials. Taking into account the depth resolution of the nuclear reaction (0.5 μ m), it was ~4 μ m. The irradiation intensity was at a level of 1×10¹³ ion·s⁻¹·cm⁻².

The dose dependences of the average concentration of implanted deuterium and the dose dependences of deuterium trapping α (percentage of all the implanted deuterium trapped in the irradiated volumes) were determined during implantation.

The scheme used for preparation of the samples, measurements, and analysis of the data allowed us to distinguish trapping of deuterium on radiation-induced (RTs) and initial (oxide particles ~5 nm in diameter and sintering defects) traps and determine the capacity of the latter.

It was shown that yttrium oxide particles were highly efficient sinks of point defects at all the doses of deuterium implantation studied. It was found that the initial traps played a decreasing role in the total trapping of the implanted deuterium during irradiation. This was due to both filling of these traps and achievement of a high density of RTs, and to suppression of deuterium trapping on the initial traps. At the irradiation-implantation doses higher than 1.5×10^{17} cm⁻², all the initial traps (dispersed oxides and sintering defects) were saturated with hydrogen, and their role in hydrogen trapping was neutralized.

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TOMOGRAFIC ATOM PROBE STUDY OF NANOSACLED FEATURES IN STRUCTURAL MATERIALS OF NUCLEAR POWER PLANTS

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In modern approaches of nuclear material investigation microstructural peculiarities are of a great concern. The critical information can be derived from nano- and even atomic scales and should reflect not only structural alterations but also redistribution of chemical elements. These phenomena determine both stages of radiation damage and macroscopic changes such as swelling, embrittlement; which in some cases were driven by formation of structural nanoscale peculiarities. On the other hand, development of promising structural materials especially for reactor core requires formation of variety of nanoscale structural peculiarities that increase mechanical properties as well as radiation resistance. The most appropriate technique to investigate phase-structure peculiarities at atomic scale is tomographic atom-probe microscopy.

ITEP has a considerable experience in atomic-scale investigation of structural material properties including after irradiation. At present in ITEP atom-probe investigations are carried out in the following directions: pressure vessel steels of VVER power plants; precipitation hardening ferritic/martensitic steels (EK-181, Eurofer 97, ...); oxide dispersion strengthened steels (ODS Eurofer, Russian ODS steels); radiation stability of nuclear structural materials in model experiments using heavy ion beams. The purpose of the present work is to give a survey on the nowdays investigations in ITEP.

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ANOMALOUS PHASE TRANSFORMATIONS ON EXPOSURE TO DEFORMATION AND RADIATION EFFECTS AND NEW WAYS OF DEVELOPING OXIDE-STRENGTHENED REACTOR STEELS

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It is known that fast-neutron irradiation of Fe-Cr-Ni alloys causes migration of point defects to sinks (grain and subgrain boundaries), leading to enrichment of boundary regions with nickel (the reverse Kirkendall effect). Similar boundary segregations of nickel (up to 2-10% of the alloy volume) appear under severe cold deformation. We were the first [1] to detect a diffusion redistribution of nickel in the Fe-12Cr-30Ni alloy; therewith boundaries of nanograins and fragments formed under deformation were enriched in nickel (up to 50%). The boundary enrichment in nickel is explained by migration of deformation-induced point defects to sinks. From the appearance of deformation-induced segregations it is possible to predict formation of atomic segregations in alloys without reactor irradiation.

It was shown that fine particles of Ni₃Ti, Ni₃Al, and Ni₃Si dissolve in displacement cascades in the austenitic matrix under neutron irradiation at 340 K in an IVV-2M reactor. It was found [2] that Ni₃Ti (Al, Si, Zr) particles also dissolve in the austenite upon severe cold (300 K) deformation ($\epsilon \sim 6$). According to calculations, dissolution of Ni₃Ti intermetallics in the austenitic Fe-Ni-Ti matrix under cold deformation can take place on account of the deformationinduced transfer of Ni and Ti atoms to interstitial sites and their drift in the stress field of moving dislocations with an activation energy of 0.2-0.3 eV.

Investigations into anomalous low-temperature deformation-induced dissolution of Fe_2O_3 and Y_2O_3 oxides in the steel matrix during mechanical alloying allowed finding new ways to creation of oxide-dispersion-strengthened (ODS) high-temperature reactor steels. It was shown in particular that oxide strengthening of pure iron is possible through its interaction with air without addition of traditional expensive alloying elements.

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THE EVOLUTION OF MICROSTRUCTURE AND DEFORMATION STABILITY IN Zr-Nb-(Sn,Fe) ALLOYS UNDER NEUTRON IRRADIATION

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Abstract

A review is given on the effect of neutron irradiation on the microstructure and properties of Zr-Nb and Zr-Nb-Fe (Sn, O) alloys. The in-reactor performance of Zr alloys is dependent on their composition and microstructure and even small changes in the composition and processing lead to substantial changes in properties as a result of evolution of precipitates and matrix composition. Development of a new generation of alloys of Zr-Nb-Fe (Sn, O) system (that show higher resistance to the irradiation-induced growth, creep and corrosion) required examination of their microstructure during the manufacturing process and evolution after neutron irradiation.

The basic irradiation phenomena involve irradiation induced damages (formation of the **a**and **c**-component dislocations) and redistribution of alloying elements. The influence produced by precipitates containing Zr, Nb and particularly Fe on the properties under irradiation is demonstrated. Relationships between composition, microstructure and irradiation-induced growth of the Zr-Nb-Fe-Sn are described. An increased content of iron (over limit of the solubility) in Zr-Nb alloys leads to lower irradiation growth and creep and to strengthening of the matrix as a result of Fe leaving Laves phase (hcp) particles with their transformation into β -Nb (bcc). β -Nb precipitates become depleted in niobium (or enriched in zirconium) and finely dispersed irradiation induced secondary particles enriched in niobium are formed. The basic microstructures that illustrate neutron damage structures, precipitates instability, evolution and irradiation growth are shown and discussed.

KEYWORDS: irradiation, Zr-Nb-Fe-Sn alloys, microstructure, phase, precipitates, dislocations, electron microscopy, growth, examination.

MATERIAL'S CHALLENGES FOR TRAVELING WAVE REACTORS

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TerraPower, a private company, is developing and engineering a novel reactor called a Traveling Wave Reactor (TWR). The TWR exploits the "breed and burn" principle first proposed in 1958 by Russian scientist S. Feinberg as a way to extract more of the energy in uranium than can be reached in thermal reactors. Like all fast reactors, TWRs could significantly reduce enrichment requirements, but unlike many other fast reactor concepts, TWRs do not require reprocessing in order to achieve high energy utilization. TWRs use depleted or natural uranium as their primary fuel and require only a small amount of enriched uranium at start-up. A TWR core with a 60-year operating life, for example, would require as little as 7% of the separative work units (SWUs) that an LWR with equivalent electrical generation would consume. The TWR therefore would never need refueling. The longevity of a TWR core depends on the size of the initial charge of natural or depleted uranium and on the fuel burn-up achieved during reactor operation.

Last year a conceptual design was completed of a 3,000 MWt metal-fuel, sodium-cooled TWR plant. The conceptual design of a smaller (1,200 MWt) demonstration TWR labeled TP-1 will be completed this year. TP-1 is slated for beginning of operation in 2020. In addition, an aggressive fuels and materials development program has been initiated to answer issues associated with this class of reactor.

As a consequence of the TWR strategy the cladding and duct materials will be exposed to very high neutron exposures, exceeding 400 dpa. TP-1 envisions the use of ferritic martensitic alloys as the major structural material. A review will be presented of materials issues for TWRs and the actions being taken to address these issues.

PATTERNS OF CORROSION IN AUSTENITIC AND FERRITOMARTENSITIC STEELS USED AS – GRADE MATERIALS OF SPENT FUEL ASSEMBLY OF THE BN-350 REACTOR

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Results of comprehensive examination of the surfaces of hexagonal ducts of spent fuel assemblies (FA) are presented along with the scales of corrosion damage in stainless steels referring to austenitic (18Cr10NiTi, 16Cr11Ni3Mo) and ferritomartensitic (13Cr2MoNbVB) grades. The steels were irradiated up to high (~80 dpa) damage doses in course of reactor operation at comparatively low temperatures and dose rates and then were left in wet storage for more than 20 years.

Specimens are the $5 \times 5 \times 2$ mm plates cut out from the face segments located at different levels relatively to the core center reactor («0», "500», «900» and «1200» mm) and irradiated up to different irradiation doses. Specimens were studied by optical metallography techniques

(Neophot-2), by means of transmission (JEM100CX) and scanning electron microscopy (Amrey-1200); also their micro-hardness was measured (PMT-3).

It has been found that inner surfaces of the steel duct walls suffered from corrosion to the highest extent. Brown color and numerous cracks up to 50 μ deep imply, that a protective layer is loose; it consist, mainly, of three-valence iron oxide (Fe₂O₃). On the duct outer side the surface layer is denser; it isn't peels off and it is black. Difference in a degree of the corrosion damage of stainless steels has been established also on different height of the FA duct. It has been found that the material segments irradiated in the core center suffered from corrosion to the most extent. Inter-crystallite nature of corrosion damage of the reflector region FA duct wall H214 (1) (steel 18Cr10NiTi) has been established at a height by 75 mm higher than the core center, where damage regions are the most prominent. Elemental composition of the regions near the pits has been determined both in irradiated and unirradiated steel samples.

STRUCTURAL INHERITANCE IN U-6Nb ALLOY AND CONDITIONS OF ITS ELIMINATION FOR REFINEMENT OF AUSTENITE GRAINS

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Investigations of the U-6NB alloy, which were undertaken jointly by the Institute of Metal Physics and the Institute of Technical Physics, revealed structural inheritance, which consists in that the size, the shape, and the orientation of grains of the high-temperature γ -phase are restored during the α - γ transformation upon heating for quenching or normalization. Restoration of coarse austenite grains leads to appearance of coarse crystalline fracture regions and impairment of the mechanical properties at 20°C. Coarse initial grains of the high-temperature phase could not be refined earlier by a single thermal treatment. Recrystallization due to «phase hardening» did not take place in the γ -phase of U-6Nb alloy similarly to titanium alloys. Four basic regimes of preliminary treatment of initially coarse-grain samples—cold deformation of the α -phase, its recrystallization, eutectoid decomposition, and aging of the martensite-were proposed for elimination of the structural inheritance. After heating to 1000°C, some samples underwent eutectoid decomposition at 500-600°C; other samples were subjected to martensitic transformation and were aged at 600°C for 10 h; some quenched samples were deformed to 20-50% and recrystallized in the α -state at 600°C. The samples treated by this method were heated to 700-1000°C, and conditions for refinement of the initial austenite grains were determined. Marked refinement of the grains of the high-temperature γ -phase (by a factor of 5-10) was observed in the case of cold deformation, recrystallization in the α -state, and eutectoid decomposition. One might expect that elimination of the structural inheritance and refinement of grains will change the mechanism of plastic deformation of U-6Nb and improve resistance to nucleation and development of cracks under shock wave loading.



The subject of this Section was prompted by the need for a systematic and comprehensive research into actinides and their alloys, including fissionable alloys whose properties are determined primarily by the features of their electronic structure and selfirradiation-induced defects. These materials are related to systems with strong electronic correlations and are too complex to leave researchers confined to fragmentary data obtained from one or the other physical, physicomechanical, metallographic or dynamic experiment run on a few more or less randomly picked samples. It is for this reason that the papers included in the Program of this Section are dedicated to the study of the properties of fissionable actinides and their model analogs in different thermodynamic states, the mechanisms of phase transformations in them, to revealing the features of their electronic states and the interrelation of their crystalline structure, the electronic and magnetic properties of actinides and their compounds, the problems of their aging, radiation stability, and response to external dynamic and shock actions.

NEUTRON STUDY OF LATTICE DDYNAMICS IN f- AND d- METAL BORIDES WITH CLUSTER-FRAMEWORK STRUCTURE

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 MB_x systems (x \geq 6) belong to the so-called higher borides. Their structure is formed by the 3dimentional lattice of B_6 or B_{12} clusters, with metal atoms (M) embedded inside the free space between the clusters. In the present work we review the experimental studies of lattice dynamics in the cluster-framework MB_{12} systems (M=Yb, Lu, Zr) performed by means of inelastic neutron scattering in combination with phenomenological calculation based on the Born-von Karman force-constant model as well as *ab initio* calculation [1-3].

From the character of dispersion curves and the general shape of the vibrational spectrum the fundamental similarity have been found between MB_{12} and MB_6 systems [1-3]. In particular, dispersion curves demonstrate extended flat (q-independent) parts, the high-energy region of the spectrum (PhDOS) is related only to the vibrations of boron clusters, the maximum energy in the spectrum is of order 130-160 meV. Using the method identical to the isotope-contrast, we have determined the partial PhDOS for the rare earth dodecaborides. The observed peculiarities of the phonon spectra have been analyzed basing on the simple models and were found to originate from the considerable hierarchy of the interactions in the crystal lattice, where M-M interaction is the weakest, and B-B one is the strongest. The difference in their energy scale is ~ 10^2 [1,2].

Ab intio calculations of phonon dispersion curves and PhDOS have been carried out within the local density approximation of the density-functional theory [3]. They revealed a good agreement with experiment in the energy region, corresponding to boron vibrations. However there is a considerable discrepancy for some low-energy modes, where the contribution from the f-(d-)metal ions is essential.

Interconnection between the lattice dynamics and magnetic excitations of strongly correlated electrons in rare-earth dodecaborides, as well as electron-phonon interaction effects in superconducting ZrB_{12} have been studied. The correlation have been found between the parameters of the low-energy phonon branches in YbB₁₂ and its magnetic excitation spectrum transforming upon the transition to the non-magnetic ground state with spin and charge gaps.

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HIGH-FIELD MAGNETIC AND MAGNETOACOUSTIC ANOMALIES IN URANIUM INTERMETALLLIC ANTIFERROMAGNETS

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In the present work, we report on recent collaborative studies of the magnetism and magnetoacoustics of several uranium intermetallic antiferromagnets (AF) in pulsed magnetic fields up to 60 T. All studies were performed on the single crystals grown by the Czochralski method in a tri-arc furnace.

UCu_{0.95}Ge has $T_N = 48$ K and the magnetic moments are in the basal plane of the hexagonal lattice. The metamagnetic transitions (MT) to the forced ferromagnetic (FF) state occurs in the field of 38 T applied along the *c*-axis. The sound velocity and the sound attenuation exhibit well-pronounced anomalies at the transitions, both the spontaneous at T_N and the field-induced to the FF state. In the paramagnetic state, both acoustic characteristics show large frequency-dependent changes revealing the presence of unusual relaxation processes of a non-magnetic origin.

UIrGe has $T_{\rm N} = 16$ K. It exhibits a large magnetic anisotropy with the hard direction along the *a* axis of the orthorhombic structure. Along the *b* and *c* axes, the MT towards the FF phase is observed at 21 and 14 T, respectively. The sound velocity displays an anomaly of 1×10^{-4} at $T_{\rm N}$. The MT along the *b* and *c* axis are accompanied by a lattice softening with a sound-velocity change of about 1.5×10^{-3} . Thus, the MTs affect the sound velocity much stronger than the spontaneous AF ordering.

 UCo_2Si_2 has $T_N = 82.5$ K. The U magnetic moments are aligned along the *c* axis of the tetragonal lattice. In magnetic fields applied along this axis, we observed the MT in 45 T (at 1.4 K). The MT is extremely sharp but exhibits a very small hysteresis. Magnetization gain upon the MT corresponds roughly to 1/3 of the U magnetic moment. We can suppose that the state above the MT is ferrimagnetic with the ++- arrangement of the magnetic moments. The ultrasound measurements confirm the transition and show its rather complicated temperature evolution.

 U_2Ni_2Sn has $T_N = 26$ K. In fields applied along the *c* axis of the tetragonal lattice, three metamagnetic transitions are observed at 30, 40 and 52 T. The U magnetic moment reaches 0.7 μ_B in 60 T without any trend to saturation. High-field measurements along the *a* axis are planned to determine type of the magnetic anisotropy.

MAGNETORESISTANCE AND ELECTRONIC SPECIFIC HEAT IN MAGNETIC FIELDS IN SUPERCONDUCTING BaPb_{0.75}Bi_{0.25}O₃

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Non-monotonic behavior of the magnetoresistance R(H) at various bias currents and data of

the specific heat measurements in magnetic fields up to 90 kOe have been observed on superconducting polycrystalline $BaPb_{0.75}Bi_{0.25}O_3$. From the specific heat data the non-linear behavior of the value Cp/T vs. H in the low temperature region (2-7 K) have been obtained. These results correlate with the magnetoresistance data showing a maximum on the R(H) curves. This maximum depends on the value of bias current. Such behavior both for the specific heat and for the magnetoresistance can be successfully explained in the terms of the spatially nonuniform state superconductor-insulator model [1] for the system investigated [2].

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MAGNETISM AND VALENCE INSTABILITIES IN PSEUDOBINARY SYSTEMS

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The ground state and cerium valence in pseudobinary compounds $Ce_x(Gd,Pr)_{1-x}Ni$ has been studied by means of magnetic measurements, neutron diffraction and X-Ray absorption spectroscopy. The ground state formation in Ce-based intermetallics is a result of a complex interplay of the RKKY exchange interaction, the 4f electrons hybridization and the crystal field interaction. A clear evidence of a strong influence of the partially delocalized cerium 4f electrons on the magnetic ordering in the Gd and Pr sublattices in Ce(Pr,Gd)Ni-based intermetallic systems has been established. Pure CeNi is an intermediate valence system with peculiar magnetic properties [1]. Physical properties of Ce strongly depend on the chemical substitution in pseudobinary CeNi-based compounds [2]. The ferromagnetic systems GdNi and PrNi fundamentally differ in terms of the magnetic ordering mechanisms – conventional in the former and soft mode-driven in the later [3]. While Ce demonstrates valence instabilities, both Gd and Pr ions have a well-localized magnetic moments in RNi-intermetallics. All these compounds crystallize in the CrB-type structure (space group Cmcm) and show complete solubility with respect to the Ce/Gd/Pr substitution.

The magnetic properties of RNi-based pseudobinary systems (DC magnetization and AC susceptibility) were measured in the temperature range 1.8K to 300K using a PPMS instrument (Quantum Design). The magnetic neutron diffraction measurements have been performed at 2K < T < 50K. The cerium effective valence was investigated by means of L_3 -XANES spectroscopy in the temperature range 10K to 300K, the L_3 -absorption spectra of Ce were collected at HASYLAB, DESY (Germany) and at Kurchatov Centre for Synchrotron Radiation and Nanotechnology (Russia). The temperature and chemical pressure dependence of the Ce valence is discussed. The data obtained provide a strong evidence of Ce substitution-induced enhancement of the localized magnetism in (Pr/Ce)Ni and (Gd/Ce)Ni compounds. Most probably, this effect results from the enhancement of the RKKI interaction in the rare earth

sublattice mediated by the partially delocalized 4*f* electrons of Ce.

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NEUTRON SCATTERING STUDIES OF MAGNETIC DYNAMICS IN PNICTIDE SUPERCONDUCTORS

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Spin excitation spectra are studied by inelastic neutron scattering in the 122-ferropnictide superconductors and compared with the first-principles calculations. The studied samples of BaFe_{1.85}Co_{0.15}As₂ and BaFe_{1.9}1Ni_{0.09}As₂ exhibit neither static magnetic phases nor structural phase transitions. It is shown that in both the normal and superconducting (SC) states, the magnetic spectrum lacks the three-dimensional screw symmetry around the $[1/2 \ 1/2 \ L]$ axis that is implied by the crystal space group. This is revealed both in the 'in-plane' anisotropy of the normal- and SC-state spin dynamics and in the 'out-of-plane' dispersion of the spin-resonance mode. It is concluded that this effect originates from the higher symmetry of the magnetic Fesublattice with respect to the crystal itself so that the inelastic neutron scattering signal inherits the symmetry of the unfolded Brillouin zone (BZ) of the Fe sublattice. The in-plane anisotropy is temperature independent and can be qualitatively reproduced in normal-state densityfunctional-theory calculations without invoking a symmetry-broken "nematic" ground state that was previously proposed as an explanation for this effect. Below the SC transition, the energy of the magnetic resonant mode E_{res}, as well as its intensity and the SC spin gap, inherit the normalstate intensity modulation along the out-of-plane direction L with a period twice larger than expected from the body-centered-tetragonal BZ symmetry. The amplitude of this modulation decreases at higher doping, providing an analogy to the splitting between even and odd resonant modes in bi-layer cuprates. It is demonstrated that at odd L a universal linear relationship $E_{res} =$ 4.3 $k_{\rm B}T_{\rm c}$ holds for all the studied Fe-based superconductors, independent of their carrier type. Its validity down to the lowest doping levels is consistent with weaker electron correlations in ferropnictide compounds as compared to the underdoped cuprates.

STUDIES OF NEUTRON ELASTIC AND INELASTIC DIFFUSE SCATTERING IN SINGLE CRYSTALS

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The presence of defects in the regular periodic order of a crystal lattice is reflected by a modification of the intensity, position and shape of the δ -function like Bragg peaks of the original structure and sometimes accompanied by an additional signal appearing at other positions in the reciprocal space. While the effects concerning directly the Bragg peaks are

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frequently investigated, the supplementary signal, apart of superstructure reflections revealing the appearance of additional order, is subject of less attention. The main reason probably being interpretation difficulties arising from ambiguities in relation between experimental data and predictions of existing theoretical models [1]. It is also important to realize that in the X-ray case, generally more accessible and more favorable in terms of Q-resolution and intensity, at any temperature the measured diffuse scattering intensity contains in addition to defect scattering also the integral over the whole spectrum of lattice vibrations, themselves eventually affected by the presence of lattice defects.

In this sense the case of neutron scattering is more favourable due to the readily available energy analysis, which permits to separate the static and dynamic components of the response with a reasonable efficiency. Recent developments of the neutron three-axis spectrometers (TAS), boosting their performance both by massive beam focusing and by implementation of arrays of analyzer/detector channels have provided access to very efficient data collection schemes. Maps of diffuse scattering over several Brillouin zones can be collected in less than an hour from crystals of a modest size ($\approx 10^{-1}$ cm³). Moreover, the flat-cone geometry [2] can be employed to map response in nonequatorial planes, permitting to carry out systematic surveys over three-dimensional intervals in reciprocal space.

In this lecture, we will mainly concentrate on fundamentals of diffuse scattering phenomenology and on experimental possibilities offered by the ILL *FlatCone* [3] multianalyzer, illustrated by examples in diverse fields ranging from relaxor ferroelectrics to quantum magnetism. We shall also give an outlook into energy-resolved polarized neutron diffuse scattering.

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PLUTONIUM PROPERTIES IN MULTIPLE INTERMEDIATE VALENCE MODEL

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The ground state issue in plutonium metal is one of the central problems in modern solid state physics. In order to explain high value and weak temperature dependence of magnetic susceptibility of α - and δ -Pu, we assumed Pu to be in the multiple intermediate valence (MIV) state, in which fluctuations occur not between two (as in ordinary intermediate valence (IV) systems), but between three electronic configurations with valence states 4+, 3+, and 2+ [1,2]. Using modified Fermi-liquid relations valid for the ordinary IV systems, in terms of the MIV model it is possible to describe the experimentally observed behavior of magnetic susceptibility and electronic (more precisely, magnetic) specific heat of α - and δ -Pu, as well as to explain significant difference of their atomic volumes [1]. Besides, the MIV model gives reasonable estimate of the entropy change associated with the $\alpha \rightarrow \delta$ structural phase transition [1]. Furthermore, the temperature dependence of linear thermal expansion coefficient (TEC) α (T) can be calculated using the classical relation:

$$\alpha(T) = \left[\gamma C_V^{ph}(T) + \Gamma C_{mag}(T)\right]/3B_T(T), \qquad (1)$$

where BT(T) is the isothermal bulk modulus, CVph(T) is the Debye phonon specific heat, Cmag(T) is the magnetic specific heat calculated in terms of the MIV model, γ and Γ are the lattice and magnetic Grüneisen parameters, respectively. We show that in general Eq. (1) reproduces main features of thermal expansion of Pu, including anomalously high TEC of α -Pu and "invar" effect observed in the fcc Pu-Ga alloys.

We also discuss a microscopic approach to describe thermal expansion of Pu which allows to estimate temperature variation of the effective Pu valence.

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STUDIES OF SPIN DYNAMICS IN GEOMETRICALLY FRUSTRATED MAGNETS AT CNCS, ORNL

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The Cold Neutron Chopper Spectrometer (CNCS) at the Spallation Neutron Source in Oak Ridge is a flexible and versatile direct-geometry multi-chopper time-of-flight spectrometer that provides both good energy and Q resolution at low incident neutron energies (1-50 meV). Two high-speed choppers, one to shape the neutron pulse from the moderator and a second to cut down the pulse length at the sample position, provide an adjustable energy resolution, ranging from ~ 1:2% to ~ 10% of the incident energy. The secondary flight path, with a length of 3.5 m, has a highly pixilated detector covering scattering angles between -50⁰ and +135⁰ in the scattering plane and ±16⁰ perpendicular to the scattering plane. The detector array with a total solid angle of 1.7 sr consists of 400 two meter long tubes filled with ³He gas.

The instrument has been operating since May 2009 with external users. Looking back over a little more than one year of user operation, it can be concluded that CNCS offers excellent intensity and resolution for inelastic and quasielastic neutron scattering experiments in the thermal and cold energy ranges. A wide range of scientific fields has already been covered, most notably in studies of collective excitations in single crystal samples (spin waves and phonons), magnetic nanoparticles, and diffusive processes in soft matter. The instrument promises to become a workhorse for the neutron scattering community for quasielastic and inelastic scattering experiments. This presentation highlights a few examples including spin dynamics in

spatially frustrated multiferroic $CuCrO_2$ and magnetic excitations in lightly hole-doped $La_{1-x}Sr_xCoO_3$.

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GENERALIZED DYNAMICAL MEAN – FIELD THEORY IN STRONGLY CORRELATED SYSTEMS

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Basics of dynamical mean – field theory (DMFT). Shortcomings of traditional DMFT. General scheme of DMFT+ Σ approximation. Applications of DMFT+ Σ approach: anomalies of electron dispersion in strongly correlated systems, "kinks" of electronic nature and the role of electron – phonon interaction. Mott – Anderson transition in disordered systems, evolution of the density of states, optical conductivity and phase diagram. Nonlocal effects – introducing the length scale into DMFT. Pseudogap fluctuations: spectral density and density of states, dynamic conductivity. Electronic structure of "real" HTSC copper oxides within LDA+DMFT+ Σ scheme.

NONLINEAR CURRENT-VOLTAGE CHARACTERISTICS OF (La_{0.5}Eu_{0.5})_{0.7}Pb_{0.3}MnO₃ SINGLE CRYSTALS: POSSIBLE MANIFESTATION OF THE INTERNAL HEATING OF CHARGE CARRIERS

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Temperature evolution of the current-voltage characteristics (CVC's) of a single-crystal lanthanum manganite $(La_{0.5}Eu_{0.5})_{0.7}Pb_{0.3}MnO_3$ is investigated in a wide (up to 1 A) range of instrumental currents. The effects of a transport current and an applied electric field on the resistance of the material are studied in view of possible implementation of charge ordering break in dielectric regions occurring due to phase separation in manganites. A negative differential resistance portion observed in the CVCs suggests the presence of a current switching effect. Below the temperature of the metal-dielectric transition in $(La_{0.5}Eu_{0.5})_{0.7}Pb_{0.3}MnO_3$, the hysteresis is observed in the CVC. The detailed analysis of the internal sample heating on the basis of experimental data on thermal conductivity showed, however, that these CVC features can be explained within the concept of nonequilibrium heating of electron gas [1].

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POLYMORPHISM AND PHASE TRANSITIONS IN CRYSTALS WITH BCC LATTICE

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On the basis of pseudospin Ising Hamiltonian with allowance for the interactions between nearest and next-nearest neighbors, a theory of single structural transformations from BCC structure to HCP, FCC, DHCP and cascades of structural phase transitions is developed [1]. The high-temperature diffuse scattering diffraction pattern and also combined rearrangement of initial Bragg reflections and the evolution of diffuse scattering to Bragg reflections at transitions to low-temperature phases are calculated [2]. The approach developed explains the existing transitions and predicts new transitions to other close-packed structures (see Figure).

Possibilities of the theory generalization onto the multiple polymorphic transformations in crystals with other high-temperature structures, in particular, the blende structure, are discussed.

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MANGANESE OXIDE Pb₃Mn₇O₁₂ WITH MIXED-VALENCE MANGANESE IONS: STRUCTURAL, MAGNETIC, AND DIELECTRIC PROPERTIES

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The recent intense interest of researchers has been attracted by the manganese oxides where the ratio between the manganese ions of different valence can be varied by substitution, which allows drastic variations in the magnetic and electrical properties of the materials. Previously, we reported the magnetic, dielectric, and calorimetric properties of single crystals of one of these materials, $Pb_3Mn_7O_{15}$ (Mn^{3+}/Mn^{4+}). We found anomalies in the temperature dependence of magnetization at $T_1 = 160$ K, $T_2 = 70$ K, and $T_3 = 20$ K [1], which are in good agreement with those revealed in the temperature dependence of specific heat [2]. The first temperature (T_1) corresponds to the occurrence of the short-range magnetic order; the second temperature (T_2) is the temperature of the transition to the state typical of a weak ferromagnet; at the third temperature (T_3) , the antiferromagnetic structure is rearranged. Considering a large number of nonequivalent positions of Mn ions, the magnetic structure should be fairly complex, which is confirmed by data of the neutron study. The temperature dependences of the real and imaginary parts of permittivity show the relaxation behavior in the temperature range 110 - 180 K, which is related to the processes of ordering the Mn³⁺ and Mn⁴⁺ ions in the crystal [3]. We interpreted the data obtained assuming the hexagonal P63/mcm space group. The results of recent structural studies with a high-resolution synchrotron at the temperatures 15-295 K [4] are unambiguously described by the orthorhombic structure with the Pnma space group over the entire temperature range. Additional X-ray studies showed that with increasing temperature Pb₃Mn₇O₁₅ transforms from orthorhombic (*Pnma* space group) to hexagonal ($P6_3/mcm$ space group). The grouptheoretical analysis made it possible to establish general regularities of the structural variations and reveal the features of the atom behavior upon the phase transition.

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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 La₂SrMn₂O₇, LaMnO₃, oxide CuO, Si, SmB₆, 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.

STUDY OF THE MECHANICAL PROPERTIES OF LIF AFTER VARIOUS TYPE OF IRRADIATION

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The paper presented microhardness measurements for pure lithium fluoride (LiF) implanted with Ar, Kr and Xe at doses ranged from 109 up to 10^{12} ion/cm². Measurements were also performed for the microhardness after irradiation by electron and gamma rays. The data exhibited a large increase of microhardness of LiF using heavy ions in comparison with the unimplanted and irradiated samples with electrons and gamma rays. The influence of annealing the samples on the microhardness is also studied. The obtained results were interpreted according to the formation of F-centers in LiF.

EFFECT OF NONSTOICHIOMETRY AND ELECTRON IRRADIATION ON THE PARAMAGNETIC STATE OF LANTHANUM MANGANITES

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Structural, charge, and electron inhomogeneities in LaMnO₃-based manganites play a key role in the effect of colossal magnetoresistance (CMR). Stoichiometric LaMnO₃ is an orbitally ordered antiferromagnet of the A-type. When the parent compound deviates from the stoichiometric composition or is doped with bivalent Ca, Sr, and Ba ions, the magnetization, appears due to $Mn^{3+}-Mn^{4+}$ ferromagnetic interactions. The CMR effect in the paramagnetic region cannot be explained by the mechanism of double exchange alone, and therefore, all the models assume the presence of polarons. According to [1], correlated and uncorrelated nanoscale polarons can remain above T_C. In correlated polarons with an orbital order of the CE-type, the local ferromagnetic order is due to a double exchange due to delocalization of e_g electrons of Mn³⁺ ions. Such polarons can exist only in the orthorhombic phase. Uncorrelated polarons are formed near a defect in all the crystallographic phases.

Nonstoichiometric LaMnO_{3+ δ} samples are nonconducting at all temperatures. However, along with an AFM phase of the A-type, FM regions of a new E-type can exist, which are analogous to the CE-phase, and, therefore, the CMR effect is possible in these regions [2]. Magnetic measurements in the paramagnetic region of nonstoichiometric and doped manganites can give information about the "composition – structure – properties" correlation. Electron irradiation can lead to local structural changes, without varying the composition of samples.

This study deals with the magnetic properties of initial and electron-irradiated nonstoichiometric LaMnO_{3+ δ} compositions and doped manganites at 80 K < T < 650 K. The samples with different concentrations of Mn⁴⁺ ions and different symmetries of the crystal lattice were prepared by additional annealing at different temperatures (T = 1100°C to T = 650°C) and subsequent quenching.

As the annealing temperature T decreased, the lattice symmetry changed from orthorhombic to rhombohedral. The concentration of Mn^{4+} ions was evaluated from the unit cell volume. As

the Mn⁴⁺ concentration increased, the ferromagnetic Curie temperature was elevated from $T_C = 122 \text{ K}$ to $T_C = 186 \text{ K}$ because of the increase of number of Mn³⁺–Mn⁴⁺ pairs. In the samples with a low concentration of Mn⁴⁺, an anomalous behavior of the temperature dependence of the reverse susceptibility in the region T = 500-600 K results from the structural transition O'(c/ $\sqrt{2}$ <a<b) \rightarrow O (a<c/ $\sqrt{2}$ <b). In the rhombohedral samples, this transition shifts towards lower temperatures T < 200 K. The temperature dependences $1/\chi$ (T), the field dependences of magnetization, and high values of the effective magnetic moment indicate that uncorrelated magnetic polarons are present up to 620 K according to Varma's model. Electron irradiation of nonstoichiometric LaMnO_{3+ δ} to a dose $\Phi = 5 \times 10^{18} \text{ cm}^{-2}$ decreases T_C by 2-6 K. In conducting La_{0.67}Ba_{0.33}MnO₃, the transition near T_C = 338 K is smeared, pointing to a nonuniform distribution of defects. A possible reason for the change in the magnetic properties of the irradiated manganites is a displacement of oxygen ions from their sites and a change in the distances and the bond angles in Mn-O-Mn, which influence the superexchange and the double exchange.

This work was performed according to the program of scientific cooperation between the Ural and Siberian Divisions of the Russian Academy of Sciences and the program "Physics of New Materials and Structures" of the Department of Physical Sciences at the Russian Academy of Sciences.

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CHARACTERISTICS CHANGE OF LIGHT-EMITTING DIODES ON BASIS GaAs AND GaN AT GAMMA-NEUTRON IRRADIATION

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Gamma-neutron radiation influence on kinetics, intensity and spectrum of light-emitting diodes electroluminescence, voltage-current and voltage-capacity characteristics were investigated. The irradiated light-emitting diodes annealing at different temperatures and currents flowing through the structure was investigated.

The first batch of samples for light-emitting diodes research was taken on the GaN/InGaN basis structure with peak wave length ~480 nanometers. They were made by RFNC-VNIITF. The second batch of samples was formed from light-emitting diodes A Π 108AM on the AlGaAs/GaAs basis structure with wave peak length ~845 nanometers. They were made by Russian industrial manufacturing.

The irradiation influences most essentially on samples kinetics and luminescence intensity. It is shown, that GaN-light-emitting diodes luminescence intensity is two orders less sensitive to neutron fluence in comparison with sensitivity of GaAs-diodes.

The experimental results analysis was made on base of the kinetics equation of charge nonequilibrium carriers in device active area. This analysis describes experimental results well.

Injection annealing prevalence for GaN-light-emitting diodes is shown experimentally. The effect has threshold character and proves at current densities more than 2 A/cm². Temperature

annealing did not observe. Irradiated samples heating up to 130 °C resulted in luminescence intensity reduction.

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FORMATION OF NANOSTRUCTURE AND NANOPARTICLES IN LITHIUM FLUORITE CRYSTALS AT GAMMA-IRRADIATION

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It is well known [1] that under radiolysis of alkali halide crystals at first separated Frenkel pairs are generated, then they are accumulated and interact to form aggregates of halogen vacancies and colloid metal nanoparticles giving rise to wide band absorption at 2.3-3.3 eV. We showed earlier, when {100} oriented LiF crystal is subjected to γ -irradiation to 108 R, the twin pattern of the lattice changes and LiOH nanocrystals are formed with an average size of ~28 nm [2].

Using X-ray diffraction and optical absorption techniques the process of nanostructure and nanoparticles formation was studied in LiF crystals cleaved in $\{100\}$ upon dry 60Co- γ irradiation in the dose range of 105–108 R at the dose rate of 530 R/c and 300 K. We found for the first time that at the dose of 106 R the optical density of F-center absorption band 250 nm grows up D>3, which is characteristic for charge transfer transitions. Then it splits into symmetric doublet of narrow resonances, which are similar to the surface plasmons of s1-metal nanoparticles [3]. According to Mie-Gans theory, cylindrical nanorods support two distinct plasmon modes: a longitudinal one can move toward red side, and a transverse one shifts toward UV-side of spectra, with their frequencies dependent on a/b aspect ratios of the nanoparticles [3]. The maximal concentration of F-centers was 3.3.1017 cm-3 (corresponding to the average distance between them ~ 14 nm), when an exchange interaction becomes effective. With the dose growth to 107 R, the longitudinal 280 nm and transverse 220 nm resonances attain D~4, being separated by the photon gap with D=0 that evidences F-centers ordering. Absorption band of Mcenters (divacancies) 450 nm splits into triplet at a dose >107 R, when Lin nanoparticles (λ =380 nm) are formed and laser generation occurs. In the case of LiF the accumulation of the defects at twins provides their ordering and assembling of Lin nanorods. The observed coherent optical response of superlattice of M-centers and lithium nanoparticles in LiF crystals is interesting for non-linear optics and nanophotonics.

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ATOMIC-FORCE MICROSCOPY STUDY OF (100) SILICON SURFACE AFTER IRRADIATION BY 21- MeV PROTONS

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The work is dedicated to investigation of changes of morphology of the surface of singlecrystal silicon irradiated with 21-MeV protons. With the use of the method of atomic force microscopy, isles of nanometer size were detected on the irradiated samples surface, forming local clusters in a number of cases (with nanoisles density in them differing from the mean values by 1-2 orders of magnitude). It is shown that formation of the detected radiation-induced changes on the surface of silicon presents a post-irradiation temperature activated phenomenon. The said changes are the result of annealing of the defects formed in the course of irradiation of silicon.

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EFFECT OF SEVERE PLASTIC DEFORMATION AND THERMOBARIC TREATMENT ON THE STRUCTURE AND PHYSICAL PROPERTIES OF HEUSLER ALLOYS BASED ON X-Y-Z (X = Co, Ni; Y = Cr, Mn; Z = Ga, Al, Sn)

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Heusler and Heusler-like alloys are of great interest due to their magneto- and temperature operated shape memory, half-metallic state, giant magnetocaloric effect and other functional properties [1, 2].

We investigated structural, optical, electrical, magnetic and galvanomagnetic properties of some Heusler and Heusler-like alloys based on X-Y-Z (X = Co, Ni; Y = Cr, Mn; Z = Ga, Al, Sn) in ordered (cast samples) and disordered (deformed samples) states. Ordered state was obtained by annealing after casting. Severe plastic deformation by torsion under pressure in Bridgman anvil pressure and thermobaric treatment of alloys were carried out for disordered state.

According to our results severe plastic deformation and thermobaric treatment lead to decreasing of the grain size and significant change the optical, electrical, magnetic and galvanomagnetic properties of the alloys. Meanwhile, the crystallographic structure of alloys is preserved. The obtained results are discussed in the frameworks of modern theoretical concepts.

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EFFECT OF THERMOBARIC TREATMENT ON THE ELECTRO- AND MAGNETORESISTIVE PROPERTIES OF NANOCRYSTALLINE Nd_{0.7}Sr_{0.3}MnO₃

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Orthomanganites $R_{1-x}D_xMnO_3$ (R is rare-earth, D is alkaline earth metal) attract a significant interest due to their potential application in resistance random access memory elements. Both the magnetoresistive and electroresistive properties can be used for the information reading and storage. As the electrotransport properties of ceramic manganites strongly depend on the nanosized grains and intergranular layers [1], a new way to regulate the electroresistance by tuning microstructure under thermobaric treatment was explored.

Polycrystalline sample of $Nd_{0.7}Sr_{0.3}MnO_3$ was subjected to thermobaric treatment at a quasihydrostatic pressure of 9 GPa and the temperature of 1000 ^{0}C for 10 min with subsequent quenching to room and liquid nitrogen temperatures. X-ray and SEM-analysis show that the treated samples have preserved the orthorhombic pseudoperovskite structure, however, the length and bond angles Mn-O-Mn and, accordingly, the lattice parameters were slightly changed. The microstructure details such as grain size and intergranular borders were significantly modified. The average region of the coherent scattering estimated from X-ray data decreases from 90 nm to 60 nm.

The quenching under high pressure resulted in a remarkable change of temperature dependence of the resistivity. The value of resistivity increased by two orders of magnitude and the Metal-Isolator transition temperature was decreased by 50-100 K in the $Nd_{0.7}Sr_{0.3}MnO_3$ samples treated under high pressure and high temperature conditions. For these samples also a significant electroresistance (700%) and magnetoresistance (30%) effects were observed.

Acknowledgements

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EFFECT OF THE CHEMICAL COMPOSITION ON AMORPHIZATION OF TITANIUM-NICKELIDE-BASED ALLOYS WITH FAST NEUTRONS

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The structural state of a $Ti_{50}Ni_{47}Fe_3$ single crystal irradiated with fast neutrons (F= 2.5×10^{20} cm⁻², T_{irr} =340K) was studied using thermal neutron diffraction at a temperatures 78K and 295K. The alloy of this chemical composition was chosen in searching for a radiation-resistant shape memory material. It is established that this alloy retains its crystalline state after irradiation, whereas the $Ti_{49}Ni_{51}$ crystal studied previously is completely amorphized after similar irradiation. The martensite transformation not occur nevertheless retain essential structural distinctive.

INFLUENCE OF THERMO-RADIATION TREATMENT ON THE FORMATION PROCESS OF DEFECTS IN DOPED SILICON

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Processes of the impurity-defect complex formation were studied in silicon doped by the rapidly diffusing copper and palladium.

Copper and palladium dopants in the samples were shown to lead to changing of the crystal elastic energy and accumulation of these impurities around microdefects. Increasing in the palladium concentration up to $3 \cdot 10^{16}$ cm⁻³ or irradiation with neutron fluencies above 10^{17} cm⁻² result in decay of the contaminant aggregates and decrease in the microdefect core sizes and also to rearrangement of palladium atoms into needles.

Doping silicon with copper causes generation of defective complexes of $5 - 10 \ \mu m$ sizes which grow like trees to 70-100 $\ \mu m$ sizes depending on the crystal direction [100], [110] or [111].

The possible mechanism of the impurity-radiation defect complex formation in doped silicon is discussed and the quasi-chemical reactions taking place at the heat and radiation treatments are suggested.



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.
STRUCTURE CHANGES IN HAUSLER ALLOYS Cu₂MnAl AND Ni₂MnGa INDUCED BY ION BOMBARDMENT.

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The Hausler alloys are intermetallids, which possesses unusual magnetic properties. But elements, the Hausler alloys consist of, don't exhibit ferromagnetic behavior (what we can see in classical Hausler alloy Cu_2MnAl). Ferromagnetic properties in these alloys are result of transformations of a crystal structure. Of course the range between non compensed spin atoms in lattice is very important.

In this investigation the structure changes induced by ion bombardment are studied. After ion beam treatment of Cu_2MnAl and Ni_2MnGa alloys, high growth of magnetization had obtained. Furthermore, changes of X-ray diffraction pattern had obtained. Such diffraction changes were mentioned in our earlier works with solid solutions [1, 2]. The ion bombardment leads to nanoclusteric structure appearing and mechanical and electro-physical properties are strongly changing. Thus when we obtain the X-ray diffraction changes similar to solid solutions, we had expected that these changes had resulted from formation nanoclusteric structure.



Fig.1. X-ray diffraction line (422), Cu₂MnAl alloy.

Initial alloy (top) and irradiated by the 30 keV Ar+ ions with different target temperatures (bottom).

Fig.2. The magnetization changes of 30 keV Ar+ ions irradiated Cu₂MnAl alloy (arbitrary units) depending on target temperature.



(422) X-ray diffraction line of Cu₂MnAl alloy, irradiated by the 30 keV Ar+ ions with target temperature 450° C, is splitted with significant angle range. Some changes of X-ray diffraction line (422) we can see with target temperatures 400° C and 500° C too. The crystal structure of materials obtained is metastable one and it can be destructed by annealing with temperature same the target temperature when irradiation had proceeded. The arrows on figures 2 and 4 shows magnetization changes after irradiated specimens were annealed with temperatures similar to irradiation one. Same results were obtained both Cu₂MnAl and Ni₂MnGa alloys, but for Ni₂MnGa annealing were not results in full return of magnetization to initial. Similarity of diffraction and of physical properties changes of Hausler alloys in one hand and solid solutions in the other hand shows similarity of structure changes in both cases. Such magnetization changes in Hausler alloys formation of clusters with different symmetry can be explained. It can lead to range between atoms with non compensed spin changing and thus exchange interaction increasing.

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ION-BEAM TREATMENT AND MAGNETIC PROPERTIES OF SOFT MAGNETIC MATERIALS

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It is known that magnetic properties of soft magnetic materials can be changed by using thermomagnetic or ion-beam treatments (TMO or IBT) at a final stage of their manufacture [1, 2]. Combined application TMO and IBT is of interest.

It is found that the electromagnetic losses in the bcc-alloy Fe-3 mass. % Si is reduced by 10% after IBT+TMT, and the magnetostriction remains unchanged. But the magnetostriction increases from $1.5 \cdot 10^6$ to $16.0 \cdot 10^6$ and the electromagnetic losses P_{0.7/1000} grows from 12.8 to 31 W/kg (but it is a permissible level) after TMT+IBT.

In the first instance a reason of electromagnetic losses reduction is the fact that IBT leads to a considerable increase the low-temperature mobility of atoms; and it facilitates a refinement of the alloy volumes in vibration zones of magnetic domain boundaries during TMT. In volumes, which are cleaner of defects, the magnetic domain structure becomes more perfect and destabilized.

In the second instance IBT increases the defects content of an alloy. As a result, compression stresses appear in the surface layer and tensile stresses appear in the rest of the volume. Compression stresses cause a change in the magnetic domain structure; namely, a system of small domains with transverse magnetization appears (90-degree domains). It provides the maximum magnetostriction deformations of the sample at its magnetic reversal during operation, but tensile stresses give magnetic softness. As IBT operates only on the surface layer, so it is possible to receive a necessary combination of electromagnetic loss and magnetostriction by changing IBT parameters and a thickness of a processed material.

Thus, application of the combined processing (TMT+IBT) allows to operate properties of materials over a wide range.

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NANOSTRUCTURED STATES IN METALLS, INDUCED BY THE HIGH-DOSE ION BOMBARDMENT

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The specific nanostructured states (R-states) appear at high dose ionic irradiation of the metallic materials in the accelerators or in the plazma devices at the various nature, energies and densities of the ion fluxes. We observed this phenomenon after irradiation by the Ar^+ , Ni^+ , V^+ ions with 30-50 keV or 1 MeV energies (current 30-50 μA) and at irradiation in the plasma device by the 10 mA Ar^+ ions current and 1 κV voltage.

The formation of the R-states have been registered within some narrow range of the radiation parameters (doses, the target temperatures and ionic flux intensities). This phenomenon was accompanied by the large change of the structure and material properties which was different from as initial these states and from other states after irradiation.

The materials used in our experiments were the metallic materials of the various structure and morphology (the model solid solutions and technical alloys of Fe-Ni, Fe-Cr-Ni, Ni-Cr, Cu-Ni (FCC structure), Fe-Cr, V-Ti-Cr (BCC structure), pure metals Ti and Zr (HCP structure) and also some intermetallics (Hausler alloys).

Characteristic features of these states:

- nanoclusteric morphology;

- anomalous large changes of the properties of the both ionic and electronic subsystems of a metal;

- changes in the X-ray diffraction patterns.

The appearance of nanoclusteric structure (R-state) is possible due the ionic irradiation. In this case the lattice is reinforced by the little clusters of the same atoms as in the matrix but they have different from the matrix symmetry. So we obtain the specific composite.

It can be supposed that the formation of such composites can be perspective for the creation of the new properties of the materials.

COMPARISON OF CHARACTER OF RADIATING DAMAGES TO PURE PLATINUM AT AN IRRADIATION FAST NEUTRONS AND IONS OF AVERAGES ENERGY

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The present paper gives the results of field ion microscopy (FIM) study of subsurface volumes of pure (99.99 %) platinum irradiated with accelerated Ar^+ ions and fast neutrons. The FIM method allows to provide direct precision study of crystal lattice radiating damages of

material in atom scale, to identify a pores including the smallest one, up to single vacancies and their accumulations, to establish their concentration, sizes, shape, their distribution in volume of the irradiated material, change of the specified parameters depending on fluence irradiations etc. As a result, the quantitative analysis of spatial distribution of radiation damages (vacancies and their complexes and also radiation-induced nanopores) in volume of irradiated Pt has been carried out.

Irradiation of Pt tip-samples was provided using Ar^+ ions accelerated up to 30 keV with fluencies of F=10¹⁸ cm⁻² and ion current density of j=200 mkA/cm² at T=573 K. For study of defects generated under neutron bombardment, platinum samples were irradiated in the RWW-2M reactor at ~310 K to fast neutron (E > 0.1 MeV) fluencies 6.7×1017 cm-2.

Platinum irradiation with neutrons to fluency 6.7×10^{21} m⁻² resulted in formation of defect structure characterized by increased concentration of single point defects and their complexes, which sizes are comparable with interatomic distances. The quantitative information on spatial distribution of vacancy complexes depending on their frequency rate on grain boundary and in volume of the irradiated material is received.

In the course of investigation of the structure of Pt irradiated to $F=10^{18}$ ion/cm², accumulations of the nanopores filled with atoms of argon was observed in subsurface volume. It was observed that nearly 40 % of nanopores are concentrated in subsurface layer with thickness of 10 nm, nanopores concentration decreases in linearly dependence y = -0.084 x + 5.154 with the distance from irradiated surface. The given numerical values of coefficients of linear dependence have been calculated only for conditions of the executed experiment.

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INFLUENCE OF Ar⁺ ION IRRADIATION ON D16 ALLOY MICROSTRUCTURE AND PHASE COMPOSITION IN DIFFERENT INITIAL STATES

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Specimens (3 mm thick) of D16 alloy of the Al-Cu-Mg-Mn system, in cold deformed and quenched state, were irradiated with accelerated Ar^+ ions (E = 40 keV, j = 400 μ A/cm², D = $2.5 \cdot 10^{15} - 10^{17}$ cm⁻²). The irradiation time was 1–40 s. The maximum temperature of continuous heating of specimens with ion beams did not exceed 450 °C (in the absence of exposures at constant temperatures).

Metallographic and electron-microscopy investigations of cold deformed specimens of D16 alloy in initial (cold deformed) and irradiated state have shown that irradiation with accelerated Ar^+ ions to a dose of 10^{17} cm⁻² (the time of irradiation is 40 s, T_{max} ~450 °C) promotes the processes of recrystallization: formation of new recrystallized grains in the entire volume of a specimen is observed. The structure formed under irradiation is similar to that formed in the course of *heating* and *exposure* at 495 °C during 20 min (before the standard quenching operation).

After irradiation of D16 alloy to a dose of $2.5 \cdot 10^{15}$ cm⁻², in the specimen section parallel to the irradiated surface, at a distance of ~150 µm from it, there is observed formation of a cellular dislocation structure. Increase of the irradiation dose promotes increase of the alloy degree of homogeneity, narrowing of cells boundaries and their growth in diameter. Besides, there occurs practically complete dissolution of lath-shaped Al₆(CuFeMn) intermetallides of crystallization origin. At a depth substantially larger than 150 µm, the action of irradiation on the specimen structure and phase composition is not so strong: irradiation does not lead to formation of a cellular dissociation structure or Al₆(CuFeMn) phase particles. At the same time, irradiation influences the composition in the whole volume of the specimen: in the course of supersaturated solid solution decomposition in the whole volume of the specimen: in the course of the process of *natural ageing*, metastable θ'' phase (GP2 zones) based on CuAl₂ phase precipitates in nonirradiated alloy, but *under irradiation*, Mg₂Al₅Cu₅ phase-based zones are eliminated.

The work is supported by the Ural Branch of RAS (Grant for young scientists and postgraduate students "Influence of irradiation with medium-energy Ar^+ ions on the structure and properties Al-Cu-Mg system alloys in cold deformed and quenched state", 2010).

SPECIALTIES OF FORMATION OF ROLLED COPPER-NICKEL FOILS' SURFACE LAYERS' CHEMICAL COMPOSITION DUE TO LONG-RANGE EFFECT OF ION IMPLANTATION.

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<u>Goal</u>: solution of fundamental problem of nanometer compositional layers formation with given physical and chemical properties in metallic systems under ion and electron irradiation.

<u>Methods</u>: ion beam and pulse electron irradiation, study and control with X-ray photoelectron and Auger-electron spectroscopy (XPS and AES), Atomic force microscopy (AFM), Secondary-ion mass-scpectroscopy (SIMS).

Results:

Studies of surface layers' of rolled $Cu_{80}Ni_{20}$ foils elemental composition after irradiation with Ar⁺ and B⁺ under various ion energies and summary doses have been made.

It showed that changes of microhardness of beamed side of foils depending of irradiation parameters corellate with changes of B concentration. Dose variation leads to much greater change of microhardness and chemical composition than variation of ion energy.

Studies of composition on un-irradiated side showed that ion implantation causes redistiribution of material's components inspite of thickness of foils being several periods greater that calculated depth of compositional and structural changes of material due to ion implantation.

We suggest that such behaviour is caused by initially non-equilibrium state of samples due to rolling. Ion implantation causes self-expanding structural changes: during irradiation of sample ions generate local thermal peaks. The excessive energy dissipates from these peaks in form of elastic waves and is enough for transformation of initial defect structure, such as de-blocking of dislocations and annihilation of different types of defects, which leads to generation of more excessive energy. This allows elastic waves to support themselves. As a result, we observe redistribution of material's components on un-irradiated side due to connection of atoms of specific type with flows of defects.

In conclusion, ion beam treatment of metallic systems with corresponding set of parameters

could be used for intended modification of thin-measured products for creation of ultrathin surface layers of given composition, structure and topography.

The study is done with phinancial support RFBR (project 10-02-96039_ural)

EMISSION SPECTRA OF THE SURFACES OF IRON, ZIRCONIUM AND TUNGSTEN BY MODERATE ENERGY Ar⁺ ION IRRADIATION

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Samples of pure iron (99.99 Fe), tungsten (99.96 W) and zirconium (99.98 Zr) were irradiated with continuous Ar^+ ion beams as the energy of ions varied from 5 to 20 keV and ion current density from 50 to 150 μ A/cm², under vacuum at a residual pressure of <10⁻⁵ mm Hg. Emission spectra of targets were measured by a multichannel photodetector based on an OS-12, diffraction-grating spectrograph and a CCD-line in the range from 360 to 850 nm. The luminous flux from the irradiated sample to the photodetector was transmitted through a multistrand quartz optical fiber, the receiving end of which was installed at a distance of 1 cm from the edge of the sample and directed to the sample surface at an angle of 60°.

In the spectra of all the targets there are two broad bands of Planck thermal radiation. The first band with an intensity maximum at wavelength λ_{ml} varying in the range of approximately 500 to 570 nm corresponds to the equilibrium Planck radiation of the surface plasma. A maximum of the second band with wavelength λ_{m2} is located in the IR (infrared region). This band appears only as a short-wavelength wing and is due to thermal radiation from the target integrally heated by beam exposure.

Against the background of these wide bands in all spectra there are much more narrow ones of non-equilibrium radiation. Some of these bands match well the ones of the irradiated metal atoms emission and the argon atoms one. These bands show up mostly clearly at an accelerating voltage of less than 10 kV. Extra investigations are needed to identify the other bands.

On the assumption of achievement of a quasi-equilibrium state in thermal spikes region resulting from the thermalization of atomic displacement dense cascades and being as a source of the surface plasma, and using the Wien displacement law, estimates of temperature in the thermal spikes have been obtained for different metals. This temperature varies from 5000 to 6000 °C, depending on the target material and the parameters of the irradiation.

The obtained results agreed satisfactorily with the molecular dynamic calculations by the program SRIM [1] for the corresponding values of ion energy and target mass. The intensity growth of IR radiation wing for all three metals correlates with an increase in ion energy, ion current density, the radiation dose and corresponds to integral heating of the targets.

The work was carried out within the scope of the Cooperation Agreement between UB RAS and RFNC- VNIITF, project No 10-2-48-YAZ.

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IMITATION EXPERIMENTS ON RADIATION-INDUCED PORES-FORMATION IN ALLOYS UNDER EXPOSURE TO ACCELERATED MEDIUM-ENERGY IONS

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Many of recent researches of swelling processes in model and reactor materials aim at reducing the degree of swelling in fcc austenitic steels to that of bcc ferritic and ferritic-martensitic steels, while preserving the advantages of mechanical properties of austenitic steels.

Modeling of such a process, applying ion irradiation, is of interest in connection with the vacancy swelling problem of reactor materials.

In this study an effort was made to get an effect of radiation-induced swelling in initially deformed nickel-bearing fcc alloys, such as $Fe_{69}Ni_{31}$ and ChS-68, using 20 keV accelerated Ar⁺ ions. The temperature of irradiation was held at 650 °C, which corresponded to the swelling peak of nickel-bearing fcc alloys under irradiation with Kr⁺ (1.5 MeV) ions. The required irradiation temperature was obtained at the expense of the radiant energy of a heater consisting of a coil and a reflector, with simultaneous exposure to an ion beam. In the absence of a beam the temperature of the target (a 8×8 mm² 20 µm thick sample) was ~200 °C. The sample was suspended in vacuum on thin threads with low thermal conduction in such a way that heat exchange of the sample with the environment occurred via radiation only. The temperature field inhomogeneity across the sample surface and over its thickness did not exceed 1 K. The irradiation dose of $5 \cdot 10^{17}$ ion/cm² matched the damaging dose of > 200 dpa in the ion range region.

Electron microscopic investigations showed the presence in the demaged layer of pores with linear dimensions from 10 to 100 nm in Fe₆₉Ni₃₁ μ ChS-68 alloys after irradiation. Their volume fraction was at least 2-3 %. Voids had clearly defined faceting, some of them being elongated in specific crystallographic directions, and were up to 100 nm long.

So, it was found that irradiation with Ar^+ ion with 20 keV energy to a dose of $5 \cdot 10^{17}$ ion/cm² at 650 °C results in vacancy swelling of Fe₆₉Ni₃₁ and ChS-68 alloys.

It was shown that, by applying the well-known procedure of one-sided samples etching to a specified thickness, irradiation with low energy ions may be quite effective in evaluative research of resistance of model and reactor materials to irradiation-induced swelling.

The work was carried out within the scope of the Cooperation Agreement between UB RAS and BNPS (Beloyarskaya nuclear power station), project No. 10-2-47-BYA.

ION BEAM EFFECTS ON ELECTRICAL PROPERTIES OF CARBONYL IRON R-20 AND VK POWDERS

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Carbonyl iron presents disperser powder of pure metallic iron particles of sizes from 0.5 to

 $25 \,\mu\text{m}$. and an average bulk weight from 3 to 4.5 g/cm^3 . Powders of carbonyl iron are used for products manufacturing using the technology of metal injection molding (MIM-technology), as well as in traditional powder metallurgy. Owing to their fine structure, they serve as an excellent raw materials for production of high-precision complex-shaped components possessing high density, high strength and good magnetic properties. In the production of carbonyl iron much attention is paid to the stability of the structure and composition of produced powders, and to improvement of electrical properties.

In the investigation, the ion-beam treatment of carbonyl iron powders of two grades, R-20 and VK was carried out. The powders were irradiated with continuous 30 keV Ar⁺ ion beams in the PULSAR unit outfitted with a cold hollow-cathode glow discharge ion source. From an Ar⁺ ion beam of a circular section, using a collimator, a strip beam of $20 \times 100 \text{ mm}^2$ section was cut, under which a tray with powder passed at a speed of 4 cm/s. The thickness of the irradiated powder laid on the surface of the stainless steel tray, was comparable with the average size of its particles. In the course of irradiation, varying ion current density j = $100 - 300 \,\mu\text{A/cm}^2$ and dose D = $10^{15} - 10^{17} \,\text{cm}^{-2}$ were used.

Irradiated carbonyl iron of mentioned grades was used to obtain ring cores by cold pressing. On the prepared cores, windings of PEV grade copper wire 0.4 mm in diameter were placed. The values of inductance and induance coil Q-factor were measured with RCL-meter Hioki 3532-50, and, using the values of inductance, permeability was calculated in a wide frequency range. The values of energy loss in the core were determined from the B – H dependencies in the frequency range from 10 to 500 kHz and induction of 8 to 50 mT. The measurements were conducted using the UVT 82-A-93 high precision setup of the Ural Institute of Metrology.

As a result of the researches it was established that ion-beam treatment of carbonyl iron powders at certain parameters of irradiation increases their saturation magnetization by 1.8% and ensures a 5-10-times increase of the Q-factor of inductors for electronics with a core of 'insulator-carbonyl iron' composite in the frequency range of 10 - 500 kHz.

The work was carried out under the Federal Program, "Scientific and Scientific-Pedagogical Personnel of Innovative Russia" for 2009-2013 years (State contract No P650 dated 05.19.2010).

STRUCTURE AND PROPERTIES OF TITANIUM-BASED COATINGS PREPARED BY METAL PLASMA IMMERSION ION IMPLANTATION AND DEPOSITION

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In this work titanium-based coatings with different microstructure, phase composition and properties were prepared by Metal Plasma Immersion Ion Implantation and Deposition (PIIID). The metal plasma is produced by a cathodic arc discharge operated in direct current mode and filtered by means of a curved magnetic filter. Ions from the plasma are accelerated to the sample surface by applying a negative bias voltage to the substrate. In this way, deposition and ion implantation are combined. The method of PIIID provides a promising technique to control structure and phase composition of Ti-based coatings with superior adhesion properties, and fast treatments with complex, three-dimensional shapes. Ti oxides and/or nitrides are formed by

additional supply of oxygen and/or nitrogen into the vacuum chamber near the substrate, respectively. The method of PIIID allows to obtain dense Ti-based coatings with different stoichiometry and phase composition in dependence on the deposition parameters.

The phases observed are strongly dependent on the relation of the gases partial pressure forming the ambient in the vacuum chamber. For pure nitrogen flow the XRD patterns are typical for titanium nitride TiN with a face centered cubic lattice. For an additional low oxygen flow rate with a relation of the gases partial pressures $p(O_2)/p(N_2) = 1/3$ the TiN peaks shift to higher angles indicating lattice shrinking due to the oxygen incorporation. For equal partial pressure of N₂ and O₂ broad diffraction peaks are observed, which can be interpreted as phase mixture of fcc TiN and fcc TiO. The average atomic composition in the Ti-based layers measured by AES shows, that the main component of the layers produced by the relation of the partial pressures $p(O_2)/p(N_2) = 1/3$ and $p(O_2) = p(N_2)$, is an amorphous TiO₂ phase. The crystallization of this amorphous TiO₂ phase to rutile and anatase modifications of TiO₂ begins at higher O₂ partial pressure $p(O_2)/p(N_2) = 3/1$. At pure O₂ flow, according to the XRD results a crystalline mixture dominated by rutile with a contamination of anatase is formed.

The polar component of the surface energy and the hydrophobicity of the surface show a dependence on the chemical composition and on the amorphization rate of the coating. The highest hardness and the best compatibility have been found by the oxynitride coating $TiN_{0.4}O_{1.6}$.



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.

RESEARCH OF PERMEABILITY OF HYDROGEN HEAVY ISOTOPES THROUGH NICKEL ALLOYS.

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During work of a fusion reactor with molten salt active zone (MSR) formation of significant amounts of tritium is predicted. For the control of tritium, having high mobility, chemical and radiating activity, are necessary to understand its behaviour in constructional materials and fluorine molten salt.

Now within the framework of project ISCT #3749 the first series of experiments in this direction is done. Measurements of deuterium permeability from a gas phase through flat samples of nickel alloys HN80MTY (Russia) and EM721 (CNRS, France) are carried out.

Factors of deuterium permeability, diffusion and solubility in the given alloys in a range of temperatures 473 ... 1123°K and deuterim pressure 0,14 ... 89,8 kPa are determined. It is shown, that temperature dependence of factors of deuterium permeability and diffusion in alloy HN80MTY has arrenius-like curve with a deviation in the field of temperatures 723 ... 823°K.

For understanding of this phenomenon researches of a structural condition of alloys before and after permeations experiments was done.

DIFFUSION OF HYDROGEN IN METALS UNDER THE INFLUENCE OF IONIZING RADIATION AND ACOUSTIC WAVES

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Usually considered the diffusion of hydrogen in metals separately under the influence of ionizing radiation and a separate acoustic. Occurrence of acoustic emission during irradiation is used to analyze the structure of hydrogenated metals, ie used as a tool change method of defects. This paper shows that the acoustic emission arising from the action of ionizing radiation is enhanced hydrogen diffusion, both on the ascending branch of the acoustic signal and its descendants (decaying) branch of / 1 /. Thus, there is an anomalous non-linear diffusion of hydrogen in titanium and steel. Equations describing the process under study, obtained on the basis of the theory Goltsov-Smirnov / 2 /. Shows the experimental setup for in situ study of diffusion processes in these conditions and discuss the mechanism of hydrogen transfer on the basis of the interaction of radiation with a hydrogen subsystem of metal. To establish the correlation between the energy of ionizing radiation in hydrogenated metals and their acoustic properties analyzed the dependence of the dielectric constant of hydrides of the metals on their composition.

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MODIFICATION OF NANOSCALE STRUCTURE OF RUSFER EK-181 UNDER THERMAL TREATMENTS

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Heat resistant reduced activation steels are most promising structure materials for new generation fusion and fission nuclear reactors. The main interest in Russia is applied to 12%-Cr ferritic martensitic steel Rusfer EK-181 development. This steel has an increased heat resistance, which overcomes foreign analogues at temperatures up to 700°C [1-2]. This improvement of mechanical properties is connected with nanometer sized structure features formation (different types of clusters and segregations) during thermal treatment [1-2]. Tomography atom probe study of Rusfer EK-181 steel showed that some thermal treatments enable to create a nanometer features in the material [3]. In order to reduce particles size and to increase a homogeneous distribution these particles it is needed to modify standard thermal treatments. Information about nanoscale structure is required for this modification. At present nanoscale structure features are studied by tomographical atom probe [3].

In this work nanoscale characterization of Rusfer EK-181 steel by tomography atom probe are performed after various thermal treatments:

- 1) annealing at 800-850 °C
- 2) quenching at 1100 °C, 40 min; air
- 3) quenching at 1100 °C, 40 min; air + tempering at 720 °C, 3 h; air
- 4) quenching at 1100 °C, 40 min + thermal cycling 810 °C, 30 min + tempering at 720 °C, 1 h

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FEATURES OF THE PROPERTIES OF NANOPHOSPHORS AND THEIR USE FOR RADIATION DETECTION

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Interest to the properties of nanoscale materials has spurred active investigations into specific

features of the luminescence of nanophosphors. Currently there are two main lines of research. The first is the search of effective nanophosphors for light emitting devices. The second is development of high-dose luminescent detectors of ionizing radiation.

The report deals with the fundamental features of nanophosphors, which are due to their structural state. It has been noted that the luminescence characteristics of nanophosphors are affected by a high concentration of surface trapping centers and quantum confinement effect. A higher probability of recombination of charge carriers in the nanoparticles and a decrease in the number of nonradiative transitions lead to a growth of the luminescence intensity. Therefore, nanophosphors are promising materials for use in illuminating devices based on ultraviolet light emitting diodes. In this case, nanophosphors act as converters of UV radiation to visible light.

Specific features of the luminescence of nanostructural materials also determine some new properties, which are important for radiation detection. They include an increase in the luminescence output under high-dose irradiation and improvement of the radiation resistance, which is explained by an intensive drain of the radiation defects formed in the nanoparticles to their numerous boundaries. Therefore, the process of accumulation of radiation defects is retarded, and, hence, the properties of luminescent detectors are impaired less under high-dose irradiation.

The report presents the results of the investigation of nanophosphors luminescence and dosimetric properties having different compositions used for monitoring of gamma radiation and high-energy electron and ion beams. These properties of commercial and nanoscale samples of thermoluminescent detectors have been compared. It has been stated that nanophosphors show promise for use in high-dose dosimeters.

STRUCTURE FORMATION IN ION-PLASMA-NITRIDED Fe-Cr ALLOYS UNDER SEVERE PLASTIC DEFORMATION

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At present an active area of research is the development of nitrogen-containing high-strength steels and alloys as competitors of stainless steels with deficient nickel and manganese. The Fe-Cr-N system is among such systems. The phase equilibrium diagrams of the Fe-Cr-N system and the effect of nitrogen on the processes of structure formation and the physical-chemical properties of these alloys have been studied comprehensively in [1]. Nitriding of the surface of alloys in this system provides conditions for modifying the structure during the subsequent deformation and thermal treatments.

The present work deals with the effect of the chromium concentration on the phase composition and the structure of Fe and Fe-Cr alloys (Fe-4Cr, Fe-20Cr, and Fe-22Cr) in surface regions nitrided using an ion-plasma method. It has been shown that as the chromium concentration increases, nitriding leads to the growth of the nitrogen concentration in the alloys and the replacement of γ '-Fe₄N nitrides by ϵ -Fe_xN and CrN. The formed nitrides are submicrocrystalline, facilitating their later dissolution during the deformation-induced transformations of nitrides and the formation of solid solutions of nitrogen and chromium in the α Fe matrix.

The effect of the structure of the matrix and the nitrides, which were formed by the ionplasma method, on the processes of the mechanically activated phase transformations, the formation kinetics of the solid solutions, and the nanostructurization has been analyzed.

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MATERIALS CHOICES FOR THE FLEXIBLE MYRRHA IRRADIATION FACILITY.

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MYRRHA [1] is designed as a multi purpose irradiation facility for radio-isotopes production, reactor materials irradiation studies and nuclear fuel developments. It will also be the first of the art Accelerator Driven System (ADS) to be constructed in the world. It will serve as a demonstrator for the transmutation of radioactive waste in a high power (100MW) subcritical core cooled with lead-bismuth eutectic (LBE). For this purpose, it will be connected to a proton accelerator delivering a beam of 2.5 mA at 600 MeV.

The physical damage processes taking place during operation into the liquid metal cooled MYRRHA system have been identified to be swelling, irradiation creep, corrosion in LBE, radiation embrittlement, creep, fatigue, LBE embrittlement and stress corrosion cracking. Degradation of mechanical properties is due to the combined action of LBE and radiation damage. Based on the open literature and from previous sodium cooled fast reactors expertise the following list of materials has been chosen as candidate materials, pointing out that the final chemichal composition will be determined on a latter stage: the austenitic steel 15-15Ti, also denominated D9 in the US, the austenitic steel 316 L and finally the ferritic martensitic steel T91.

The materials selected [2] in the different reactor components have been chosen taking into account the following factors, as received mechanical and physical properties, fabricability and materials availability, materials database qualification in nuclear environment and degradation of properties by irradiation and LBE.

Safe operation of the components is possible if the materials limits are not transgressed. Nevertheless the materials limits need to be validated by urgent R&D, especially in the domain of corrosion where long term results are missing and in the domain of fatigue where results are scarce.

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INVESTIGATIONS OF MARTENSITIC γ-α' TRANSFORMATION DURING COMPRESSION TESTS OF 12CR18NI10TI METASTABLE STEEL SAMLES IRRADIATED BY NEUTRONS

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Results of experiments, directed on researching kinetics of phase diffusionless γ - α ' transformation in 12Cr18Ni10Ti stainless steel during uniaxial compression and, for comparison, tensile tests are adduced and discussed.

Cylindrical stainless steel samples were austenizated (1050°C, 30 minutes), then were irradiated in WWR-K nuclear research reactor core at <80°C to fast fluencies up to $1.4 \cdot 10^{19}$ n/cm². Mechanical compression and tensile tests were conducted with strain rate 0.5 mm/min at room temperature. During one-step plastic deformation, amount of martensitic α '-phase, induced in the sample and sample's geometry changes were registered.

As a result of experiments, graphs of compression and tensile tests with axes "«true» stress – local «true» deformations" and martensitic α '-phase deformation dependence were constructed.

Processing of the received data has allowed to define the quantities, that defined parameters of $\gamma - \alpha'$ transition in both neutron-irradiated and unirradiated steel:

- Amount of "true" critical stress and deformations corresponding to the beginning of martensitic α '-phase initiation during the deformation;
- Parameters in the equation describing kinetics of martensitic γ - α ' transformation;
- Values of critical density of the mechanical work necessary for inducing $\gamma \alpha'$ transition.

The role of internal stress fields in initiation and accumulation processes martensitic α '-phase in the metastable stainless steels irradiated with neutrons is analyzed.

ABOUT CALORIMETRIC TECHNIQUE OF ION CURRENT DENSITY ESTIMATION BY THE CROSS-SECTION OF POWERFUL ACCELERATED ION BEAMS

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It is a complicated task to evaluate ion *current density* (μ A/cm²) or (with an accuracy of a multiplier equal to ion charge) ion *flow/flux* (ion/cm²·s), which is equal to the rate of *dose/fluence* (ion/cm²) build-up. Using the Faraday cup for this purpose is effective only for small current densities. At sufficient high ion current densities there is a possibility of conducting plasma formation inside the Faraday cup, which leads to distortion of measurements results.

To evaluate ion current density, complicated experimental schemes may be used promoted in [1], which, for each concrete type of ion source, can be suggested and realized only by experts in electrophysics measurements.

It should be pointed out that, in some cases, an error in flux measurement may be caused also by charge exchange of some part of ions. In this case heating of the target may be observed in the absence of an appropriate increase in ion current density due to the presence of neutral atoms in the particles flow, due to the charge exchange effect.

We propose the calorimetric technique of determining the accelerated particles flux and some other parameters, based on the analysis of processes accompanying the implantation of accelerated ions in a substance and the features of solution of a one-dimensional heat conduction problem in certain boundary conditions. Based on the energy conservation and the Stefan-Boltzmann laws in conditions of *temperature monitoring* of probes, there may be obtained a system of asymptotic equations ($t \rightarrow 0$ and $t \rightarrow \infty$) enabling us (with account for the experimentally found small corrections for sputtering and the increase in mass of the target at the expense of ion implantation) to determine a number of process parameters. Namely, it is possible to determine the *absorption coefficient* of ion beam energy by the target material, *sputtering coefficient* and the *radiating capacity (emissivity)*, as well as to measure the ion *current density* (accelerated particles *flux*), by the ion beam section.

The work was carried out within the scope of the Cooperation Agreement between UB RAS and BNPS, project No. 10-2-47-BYA.

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DEFINITION OF KINETIC PARAMETERS OF MARTENSITE $\gamma \rightarrow \alpha'$ TRANSFORMATION INTO STEEL 12CR18NI10TI, IRRADIATED THERMAL AND FAST NEUTRONS, DEFORMED AT LOW TEMPERATURES

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It is known that characteristics of many physicomechanical properties reactor austenitic stainless steels and their behavior at stressing are in many respects defined by kinetics of $\gamma \rightarrow \alpha'$ transition in the course of plastic deformation. This circumstance is especially important for considering at work on transportation fulfilled fuel assemblages of reactors at their stage decommission.

In the present work studied the phase-structural changes induced by cold deformation in a steel 12Cr18Ni10Ti – a material of a six-sided cover fulfilled fuel assemblages of fast reactor BN-350 (CC-19, «-160 mm», T_{irr} =370°C, 55 dpa), and also in a similar steel in austenite condition (1050°C, 30 minutes), subjected to a neutron irradiation in the research reactor VVR-K (4·10¹⁸; 1,9·10¹⁹; 9·10¹⁹ n/sm², T_{irr} < 80°C).

Flat steel samples not irradiated and irradiated, deformed at temperatures 20,-20,-60,-100°C step-by-step registering change of magnetic properties and the geometrical sizes of samples. Curve dependences of "true" pressure and quantity formed α' -phases (Mf) from "true" deformations (ϵ) are as a result constructed. It has given the chance, in particular to define "true" critical parameters of the beginning of formation of a α' -phase.

The received experimental data analyzed, using the equation of Ljudvigson-Berger. Values of factors in this equation for various conditions of an irradiation and test are defined. It is shown that parameters autocatalytic of process and propensities of a steel to martensite transformation into strong degree depend on stretching temperature. The present results are compared with received earlier [1,2].

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INVESTIGATIONS OF MARTENSITIC γ-α' TRANSFORMATION DURING NEGATIVE TEMPERATURE DEFORMATION TESTS OF 12CR18NI10TI STAINLESS STEEL IRRADIATED BY NEUTRONS

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Recently the great attention in reactor materials technology is given to investigations of martensitic (direct and inverse) transformations coursing in austenitic stainless steels under the influence of a neutron flux, temperatures and gradients of stresses. It is also known that low temperatures of tests and irradiation of high-energy particles intensify phase transformations.

Thereupon revealing of regularities and features of $\gamma \rightarrow \alpha'$ -transition during low temperatures deformation tests of metastable chromium-nickel steels represents certain interest.

In the present work results of studying martensitic transformations during negative (up to -100°C) temperatures (T) deformation tests with using a method of electro resistance (R) measurements of 12Cr18Ni10Ti reactor steel irradiated by neutrons (max $Ft=2\cdot10^{20}n/cm^2$, E>0.1MeV) are resulted.

Diminutive steel samples after austenization (1050°C, 30 minutes) were irradiated in the reactor WWR-K at T<80°C. Then not irradiated samples (initial) and irradiated with neutrons were deformed at various negative temperatures by universal test installation «Instron-1195» in specially created low temperature test cell. The current experimental data received during experiment, automatically were registered on personal computer.

As a result of experiments dependences R=f(T) and R=f(Ft) which analysis has allowed to establish parameters of martensitic $\gamma \rightarrow \alpha'$ -transformations under various conditions of tensile tests of the steel irradiated by neutrons are received. Mechanisms of structural phase transformations of irradiated metastable stainless steels in low temperatures deformation tests are discussed.

IRRADIATION OF MELT METAL POWERFUL ELECTROMAGNETIC IMPULSES

V.F. Balakirev*, V.V. Krumsky**, <u>N.A. Shaburova</u>*** *Institute of Metallurgy UB RAS, Ekaterinburg, Russia ** South Ural State University, Chelyabinsk, Russia (kvv@susu.ac.ru) *** South Ural State University, Chelyabinsk, Russia (sarnata34@rambler.ru) Properties of metals can be adjusted at the early stages of preparation - with the help of external influence on the melt. It is known that varying the modes and methods of processing the melt can widely change the structure and properties of the metal.

The technological processes of production of metallic melts are associated with the transfer of charges materials in the molten state and subsequent crystallization of the system. Traditionally, researchers pay great attention to the second stage, i.e. search for optimal crystallization conditions. Attempts to influence on the metal on the melt stages are limited the only an additional alloying and refining. That allowed to optimize the composition and removal of harmful impurities. The paper proposes a method of external influence on the melt that led to get the metal with a high set of properties.

As an external physical impact on the melt in the paper the processing of powerful nanosecond electromagnetic impulses (NEMI).

Exposure to melt metals with powerful electromagnetic impulses - a new, little explored area. For the first time in the world in 1996 action of high-power electromagnetic impulses to the various substances studied in the South Ural State University. Generators are used with impulse power exceeding 1 MW, with impulse duration of 1 ns. The generators have a small size and weight. Power consumption less than 100 watts.

The processing of melts were carried out with the plant shown in Fig. 1.

In experiments conducted using the generator of electromagnetic radiation GNP type, with the following characteristics: pulse duration 0.5 ns, the amplitude more than 8 kV, power in one impulse more than 1 MW, frequency of impulse repetition up to 1000 Hz. Generator (1) create a unipolar impulse of current. One electrode (2) of generator was immersed into the crucible with the melt, and the second was mounted on the body of the crucible.

The impact of NEMI on the melt carried immersion transducer (4) in the melt metal (6).

Black metals (steel 20L, 20GL, grey iron, cast iron with 3.7 wt.% C) and non-ferrous metals (copper, tin bronze, aluminum, industrial silumins, zinc alloys, lead-zinc and etc.) are processed by with powerful electromagnetic impulses [1, 2, 3]. Some changes in the chemical composition of the metal and changes in the structure and properties of the metal have been observed in the all independently experiments [2, 4].

The results of the majority of independent studies of metals and alloys are in good agreement with each other and clearly show the positive influence of electromagnetic impulse process on the structure and properties of the metal. However, it should be noted that subsequent heat treatment of cast metal negate the results of impulse process.



The work of authors is a continuation of this direction and is devoted to study the impact of electromagnetic impulses on the lead melts and simple fusible alloys.

Electromagnetic effects on melt lead held in the above installation. The duration of impulse exposure to melt lead was different - 0, 15, 30 and 60 minutes.

Fig. 1. Apparatus for processing of metallic melts: EMI: 1 - EMP generator, 2 - wire, 3 - asbestos cover; 4 - emitter, 5-crucible with the melt; 6 - heating element resistance

Investigation of the microstructure, conducted by scanning electron microscope JEOL JSM-6460LV showed:

1) The phase composition of samples is the same and has the following phases: a matrix of lead, antimony crystals which dispose on the boundary of the lead grains, sulphide (Cu, As, Sb, S) - and PbS- phases.

2) The microstructure of the samples, there are some characteristic differences. The sample not processed by electromagnetic impulses, have in microstructure dominated the allocation of non-equilibrium eutectic crystals of antimony. They are in some areas form a grid of grain boundaries. Sulfide inclusions are relatively large, compact and unevenly distributed over the surface microsection.

In the other samples the proportion of eutectic precipitates crystals of antimony is decrease and they are becoming more dispersed. It was marked increase of the proportion of sulphide inclusions in the structure.

Thus, the results of the study can conclude that the effect of powerful electromagnetic impulses on the molten lead contributes the development of equilibrium crystallization in the whole volume of the metal.

The samples untreated by impulses are characterized by the presence of phase separation on





the main impurity elements (Sb, Cu, As). Consequently, in some areas of the metal concentrations of these elements reaches the eutectic composition and is formed of nonequilibrium eutectics.

Investigation of the influence of electromagnetic processing of molten lowmelting alloys showed. Investigated alloys: 38% Pb - 62% Bi and 32% Pb - 50% Bi - 18% Sn were prepared in the laboratory from technically pure components.

First, in the crucible melted batch of more refractory component (lead), then, as the molten lead bismuth and tin added. After melting of all components of the melt was stirred, and cast control sample for the analysis of chemical composition.

The microstructure of the samples was studied on polished microsections. Typical microstructure of alloys are shown in fig. 2, 3.

Fig. 2 - Microstructure of the unirradiated sample (a) and irradiated (b) two-component 38% Pb - 62% Bi alloy

According to the state diagram of Pb-Bi alloy belongs to hypereutectic. The phase composition of hypereutectic alloys in the system Pb-Bi is presented in two phases - the primary crystals of bismuth, crystallizing from the liquid phase and a double lead-bismuth eutectic crystallizes at 125 $^{\circ}$ C. The number of lead-bismuth eutectic in the structure of this alloy at equilibrium crystallization should be around 70%, and primary crystals of bismuth - 30%.

Studies of the microstructure of untreated sample pulses showed that the amount of eutectic in

the structure of the sample does not exceed 45-50%, respectively, the primary crystals of bismuth - 50-55%.

The observed phase relation indicates the occurrence of non-equilibrium crystallization, due, apparently, accelerated cooling.

A characteristic difference between the microstructure of the alloy, processed by impulses in the molten state is significantly smaller number of primary crystals of bismuth. The number of primary crystals of bismuth does not exceed 5%.

Consequently, in the case of the alloy, processed by electromagnetic impulses can talk about the course of equilibrium crystallization.

On fig. 3 shows the typical microstructure of alloys of Pb-Bi-Sn.

In the structure of the original, and processed by powerful electromagnetic impulses samples containing 3 phases - primary crystals of bismuth, fringed double tin-bismuth eutectic, ternary lead-tin-bismuth eutectic is located on the periphery.

In the microstructure of the alloys noted significant difference in the number of phases. The structure of the treated alloy is practically no primary crystals of bismuth, markedly reduced the number of binary eutectic.

For all alloys density are measured by triple-weighting method. Results presented in table 1.



Table	1

Density of the alloys

Sample	Density, g/sm ³
The alloy Pb-Bi, the initial	10,421
Alloy Pb-Bi, processed NEMI	10,537
The alloy Pb-Bi-Sn, the initial	9,779
The alloy Pb-Bi-Sn, processed NEMI	9,905
Pure lead, the initial	11,132
Pure lead, processed with NEMI for 15 minutes	10,934
Pure lead, processed with NEMI for 30 minutes	10,482
Pure lead, processed with NEMI for 60 minutes	11,080

Fig. 3 - Microstructure of the unprocessed sample (a) and processed (b) Pb-Bi-Sn alloy

Comparison of the hardness of lead samples showed in the table 2.

Thus, the results of the study can conclude that exposure to melt lead, and fusible alloys nanosecond powerful electromagnetic impulses contributes to the development of equilibrium crystallization in the whole volume of the metal, structure refinement and improvement of properties of the metal. That confirms the results of earlier experiments.

Table 2

Hardness of the lead samples

Sample	average hardness,
	HB
Pure lead, the initial	11,6
Pure lead, processed with NEMI for 15 minutes	12,6
Pure lead, processed with NEMI for 30 minutes	10,9
Pure lead, processed with NEMI for 60 minutes	13,4

Scientific research is being done in the framework of Federal Target Program "Research and scientific-pedagogical cadres of Innovative Russia" for 2009-2013.

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SEU EFFECT AT 14 MEV ENERGY NEUTRONS INFLUENCE ON THE STATIC RAM SOI IC.

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Energy allocation local areas are formed at 14 MeV energy neutrons interaction with a semiconductor. Modern microelectronics develops on the way of separate active elements geometrical sizes reduction. So the local area of energy allocation becomes comparable in the sizes with active area of the integrated transistor. Therefore 14 MeV energy neutrons can cause single upset (SEU) in digital IC.

The most probable mechanism of 14 MeV energy neutrons and silicon interaction is primary - knocked-on atoms (PKA) formation. Threshold nuclear reactions are present at this process as well, but their interaction cross-section is insignificant.



Figure 1 - Failures SRAM quantity dependences (received in several experiments) on size fluence at an irradiation 14 MeV energy neutrons.

Primary knocked-on atoms have average energy about 960 keV and linear energy loss (LEL) about 6 MeV·sm²/mg.

This work purpose was research of SEU effect in SOI IC of static RAM (SRAM) at14 MeV energy neutrons influence on them. The received results confirm, that 14 MeV energy neutrons cause SEU.

SEU quantity dependences (received in different experiments) on 14 MeV energy neutron fluence are presented on figure 1. The values disorder on dependences initial site has stochastic character. Characteristics start to converge at neutron fluence big values.

Possible SEU quantity from 14 MeV neutrons fluence estimation is submitted in this work also.

DYNAMIC STRAIN AGING OF 12CR18NI10TI STAINLESS STEEL AND ARMCO-IRON IRRADIATED BY NEUTRONS

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There are all bases to believe that change of strength and plasticity of constructional reactor materials during operation in a temperature interval 200-600°C are in many respects defined by processes of dynamic strain ageing (DSA) – interaction impurity and alloying atoms with mobile dislocations. As a rule, DSA it is accompanied by occurrence of serrated plastic flow associated with macrolocalization of deformation.

Earlier some regularities and features of phenomenon DSA in iron, 12Cr18Ni10Ti stainless steel and 03Cr20Ni45Mo4NbBZr high-nickel alloy, irradiated in the research WWR-K reactor have been established [1]. The present work is executed in development of these researches.

Flat samples (gauge dimensions of 10x3.5x0.3 mm) were irradiated in the WWR-K reactor to maximum fluence of neutrons $1.4 \cdot 10^{19}$ n/cm² (E> 0.1M3B). A mechanical tensile test was made

with strain rate $8.3 \cdot 10^{-4}$ c⁻¹ in a range of temperatures 20-700°C. Some of the experiments were video-recorded for the purpose of deformation localisation features at DSA studying.

In neutron-irradiated 12Cr18Ni10Ti stainless steel two temperature regimes of DSA were revealed — 200-400°C and 425-625°C which differ by intensity and type of serrated plastic flow. In low-temperature region reduction of DSA intensity as a result of irradiation is observed.

It was found that neutron-irradiation of Armco-iron leads to reduction of embrittlement effect in DSA temperature range and suppression of serrated plastic flow observed in unirradiated specimens. This effect increases with increasing damage dose.

The microstructure evolution of irradiated Armco-iron during deformation hardening and DSA mechanisms in the irradiated materials are discussed and analyzed.

[1] Sakbaev M.Zh. Dynamic strain ageing of iron and some iron-chromium-nickel alloys, subjected radiation exposure, Abstract of a PhD thesis, Almaty, 1994, 22p.



The Seminar is aimed at organizing a broad discusson of the presented projects, clarifying the programmes of further research, establishing contacts and defining the range of problems with a potential to serve a basis for new joint investigations, including those falling within the scope of the ISTC projects. One of the tasks of this Seminar is discussion of project proposals from the ISTC database (approved without financing), which are potentially realizable in the interests of nuclear power engineering and nanomaterials science.

EFFECT OF HYDROGEN AND LOW-TEMPERATURE NEUTRON AND ELECTRON IRRADIATION ON RADIATION DAMAGE OF AUSTENITIC STEEL

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The methods of resistivity, electron microscopy, and measurements of the mechanical properties under low-temperature (77 K) electron and neutron irradiation were used to study accumulation and annealing of radiation defects in 16Cr15Ni3Mo1Ti austenitic steel, which shows promise as a radiation-resistant high-pressure-vessel material when it is unsaturated and saturated with hydrogen to 300 at. ppm.

The effect of hydrogen and low-temperature (77 K) neutron and electron irradiation on the mechanical properties and residual resistivity of the austenitic steel was analyzed. It was found that saturation with hydrogen led to an increase in the yield stress. Irradiation with fast neutrons also increased the yield stress. Hydrogen practically had no effect on radiation hardening, but it influenced the residual resistivity increment.

Overlapping of displacement cascades under irradiation began at a fluence larger than 1.5×10^{18} cm⁻². An almost linear relationship between the yield stress increment $\Delta \sigma_{0.2}$ and the square root of the residual resistivity increment (the concentration of accumulated radiation defects) $\Delta \rho^{1/2}$ was observed at all the neutron and electron fluences. This was an indication that the contribution of radiation defects to residual resistivity was independent of whether they were distributed either homogeneously or resided in the region of displacement cascades, even when the cascades overlapped.

The effect of displacement cascades and homogeneously distributed Frenkel pairs on the physicomechanical properties of 16Cr15Ni3Mo1Ti austenitic steel was analyzed.

This work was supported by the International Scientific and Technical Center (project no. 3074.2), the Russian Foundation for Basic Research (project no. 11-02-00224), and the Presidium of the Ural Division of the Russian Academy of Sciences (projects nos. 09-M-23-2004 and 10-2-05 YaTs).

NEUTRON-DIFFRACTION STUDY OF MICRO- AND MACROSTRESSES IN STRUCTURAL AGEING ALLOYS FOR NUCLEAR POWER ENGINEERING AFTER THERMAL AND RADIATION EXPOSURE.

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The Project objective is investigation of micro- and macrostresses in samples of radiationresistant steels used in nuclear reactors. The Project work envisages a comprehensive study of samples of the test materials. The main method will be high-resolution neutron diffraction analysis providing measurements of both microstresses, which arise under irradiation or during decomposition of solid solutions (followed by the formation of intermetallics, carbides, radiation clusters and other precipitates) in a preset volume of samples, and stresses in welded joints. The central task of this Project will be measuring internal microstresses in the bulk of samples from radiation-resistant ageing alloys in the process of formation, growth and coagulation of second-phase disperse particles noticeably influencing pores formation.

For the needs of the investigations necessary set of samples was prepared. The samples were subjected to thermal treatment, and their composition and structure underwent certification. Microstresses in samples of aged steel 0.40C-4Cr-18Mn-2V, 16Cr-15Ni-3Mo-1Ti and 26Ni-5Cr-3Ti were studied by the methods of electron microscopy and neutron diffraction. Besides that using the method of high-resolution neutron diffraction analysis, macro- and microstresses in the initial state (before neutron irradiation) were studied in samples of PWWR reactor vessel steel (grade 0.12C-Cr-Mn-Mo-V ferrite). There was found a great difference in the structural mechanism of intermetallic and carbide ageing of steels 26Ni-5Cr-3Ti and 0.40C-4Cr-18Mn-2V. The effects of electron and neutron irradiation on radiation-induced structural-phase transformations in Fe-Ni-Ti alloy at irradiation temperatures of 320-340 K were studied. Dose dependences of microstresses variation at formation of vacancy clusters in Fe-Ni austenitic alloy in the process of irradiation with fast neutrons and electrons at 340 K were derived.

The Project will pull together the efforts of participants from Institute of Metal Physics UB RAS (Ekaterinburg), the Laboratory of Neutron Physics at Joint Institute of Nuclear Research (Dubna), and Russian Federal Nuclear Center – All-Russia Scientific-Research Institute of Technical Physics (Snezhinsk), who have already accumulated a wealth of experience in studies of the radiation damageability of constructional materials and determination of internal stresses by the neutron diffraction method.

CHANGE OF LATTICE STRUCTURAL PARAMETERS AND OF ELECTRON SPECTRA OF *n*-GaN FILMS ON THE SAPPHIRE UPON REACTOR IRRADIATION

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Real conditions of the semiconductor nitrides films growth on the substrates result in the occurrence of the tensile or the compressing single - axis and biaxial elastic stresses. To the additional stress also promotes the high concentration of the radiation-induced defects in nitrides. All this causes high interest, which is shown now to studying the influence of an external hydrostatic pressure and the anisotropy elastic stresses of compression / tensile nature on the structural and the electronic parameters of the semiconductor nitrides A³N.

In the present work the executed calculations of the external hydrostatic pressure up to 10 GPa, the single-axis elastic stress of the tensile and compression along the hexagonal axis c with at the account of a relaxation of a crystal lattice along an axis a and the biaxial elastic stresses of the tensile and the compression up to ± 10 GPa in the basal plane of an elementary cell (0001) at the account of the relaxation of a crystal lattice along an c – axis on the lattice structural parameters a, c and u, the length of the valence chemical bonds, the electronic bands structure and the energy position of the local charge neutrality level (LCNL) in the wurzite n-GaN have been investigated.

The measurements of n-GaN films structural parameters a and c in the heterostructure n-

GaN/Al₂O₃ upon reactor neutrons irradiation up to $7.25*10^{19}$ cm⁻² reveal an expansion of the *c* - lattice parameter up to 0.38% while the *a* - parameter values in *n*-GaN film and in Al₂O₃ substrate almost unchanged. As follows from the theoretical estimations, the tensile stress along the *c*-axis comes up to 1.5 GPa-value in the irradiated *n*-GaN film, while the compressive stress in the basal plane of the unit cell of GaN is about 0.5 GPa. The expansion of the irradiated *n*-GaN film along *c*-axis results in the decrease of the band gap and the lowering of the local charge neutrality level (LCNL) of GaN by 37 meV and 22 meV respectively in the comparison with theirs initial values in *n*-GaN film on sapphire before irradiation. On the base of these data the pressure coefficients of the forbidden gap and LCNL are estimated.

The restoring of the structural parameters of the reactor neutrons irradiated *n*-GaN films upon isochronal annealing at $100-1000^{\circ}$ C of the irradiated heterostructure *n*-GaN/Al₂O₃ has been investigated.

EXAMINATION OF THE REGULARITIES AND MECHANISMS OF THE NEUTRON FLUX INFLUENCE ON THE RPV MATERIALS EMBRITTLEMENT

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Accelerated irradiation does not allow receiving conservative estimation of the PWR RPV embrittlement, which may be caused by Cu-enriched precipitates formation in low neutron flux irradiation regime at the region before to plateau.

Difference in opinions on embrittlement dependence on flux level may be explained for the presence in the embrittlement kinetics of the oscillation component. In the upshot result is depends from both flux and fluence of neutrons. Proposed interpretation of the flux effect manifestation based on assumption that competition between precipitates formation and their cascade dissolution exists.

Summing the previously mentioned on the subject discussed one might conclude, that depending of the fluence level reached manifestation of the «flux effect» in reference to Guide pattern may be quite different, namely: negative, positive and «two zeros».

RADIATION-DYNAMIC EFFECTS AT IRRADIATION WITH NEUTRONS, IONS, FISSION FRAGMENTS, NON-TRADITIONAL METHODS OF MATERIAL PROPERTIES MODIFICATION AND THE PROBLEM OF NUCLEAR REACTORS SAFETY

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In the recent investigations carried out at the Institute of Electrophysics, Ural Branch of Russian Academy of Sciences (IEF UB RAS), a phenomenon was displayed of initiating by irradiation of explosive phase transformations in metastable media, in particular, in the metastable metals and alloys due to exceedingly high rate of energy release in the region of dense cascades of atomic collisions, exceeding by one or two orders of magnitude an analogous

rate (J/atom/s) at nuclear explosion. The effects are observed at irradiation of model and reactor materials with neutrons and accelerated ions. It was established that the named phenomena are not related to radiation defects formation and mass transfer. They are caused by collective radiation-dynamic effects not studied before. Based on the available data, abrupt increase in the rate of fissionable materials ageing under external irradiation may be predicted. Investigation of the revealed processes is vital both from the point of view of developing non-traditional methods of material properties modification, and from the point of view of nuclear power plants safety.

Main Project object: The main object of the ISTS project No 2228 is the experimental and theoretical investigation of radiation-dynamic (RD) effects: irradiation-induced fast processes and phase transformations externally similar to combustion and detonation phenomena, observed at irradiation with neutrons, fission fragments and heavy ions in metastable metals and alloys, including radiation-resistant steels and alloys from the standpoint of nuclear reactors safety and development of basically new radiation methods of material modification. The unique research nuclear reactors, ion accelerators and analytical techniques available to the participating institutions will be used.

DEVELOPMENT OF THEORY OF CREATION OF RADIATION-RESISTANT ZIRCONIUM STRUCTURAL ALLOYS FOR REACTOR CORE REGION

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VANADIUM ALLOY PLATED BY FERRITIC STAINLESS STEEL – MATERIAL FOR FAST REACTORS FUEL CLADDING

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INVESTIGATION OF MOLTEN-SALT FLUORIDE SYSTEMS FOR INNOVATIVE NUCLEAR POWER ENGINEERINGS

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RFNC-VNIITF INVESTIGATIONS OF INTERACTION OF HYDROGEN ISOTOPES WITH STRUCTURAL MATERIALS FOR THERMONUCLEAR FACILITIES AND TRITIUM SYSTEMS

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