

Седьмой Международный Уральский Семинар

# РАДИАЦИОННАЯ ФИЗИКА МЕТАЛЛОВ И СПЛАВОВ

Тезисы докладов

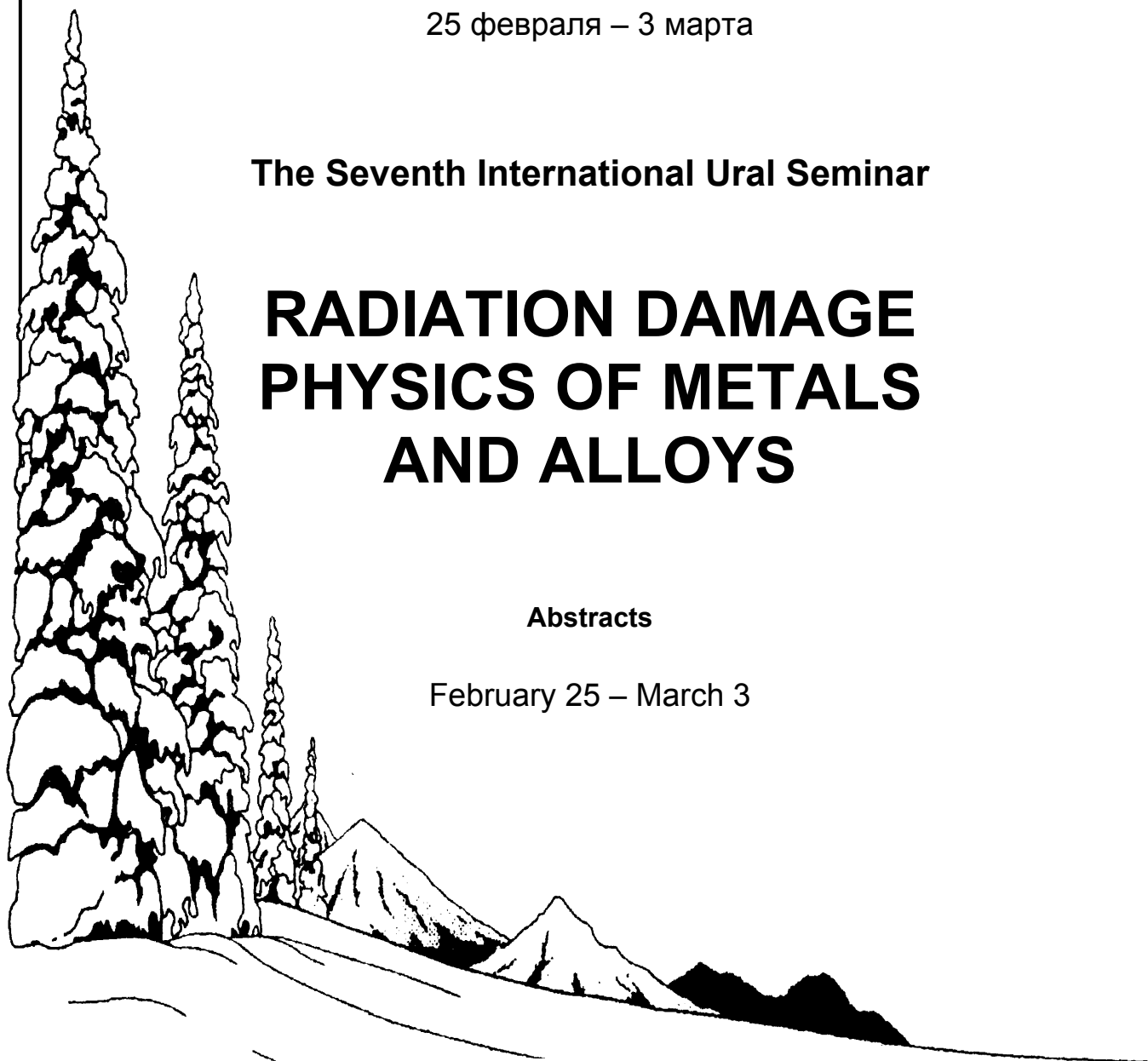
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The Seventh International Ural Seminar

# RADIATION DAMAGE PHYSICS OF METALS AND ALLOYS

Abstracts

February 25 – March 3



Снежинск  
Россия

Snezhinsk  
Russia

2007



**The Seventh International Ural Seminar**

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PHYSICS OF METALS  
AND ALLOYS**

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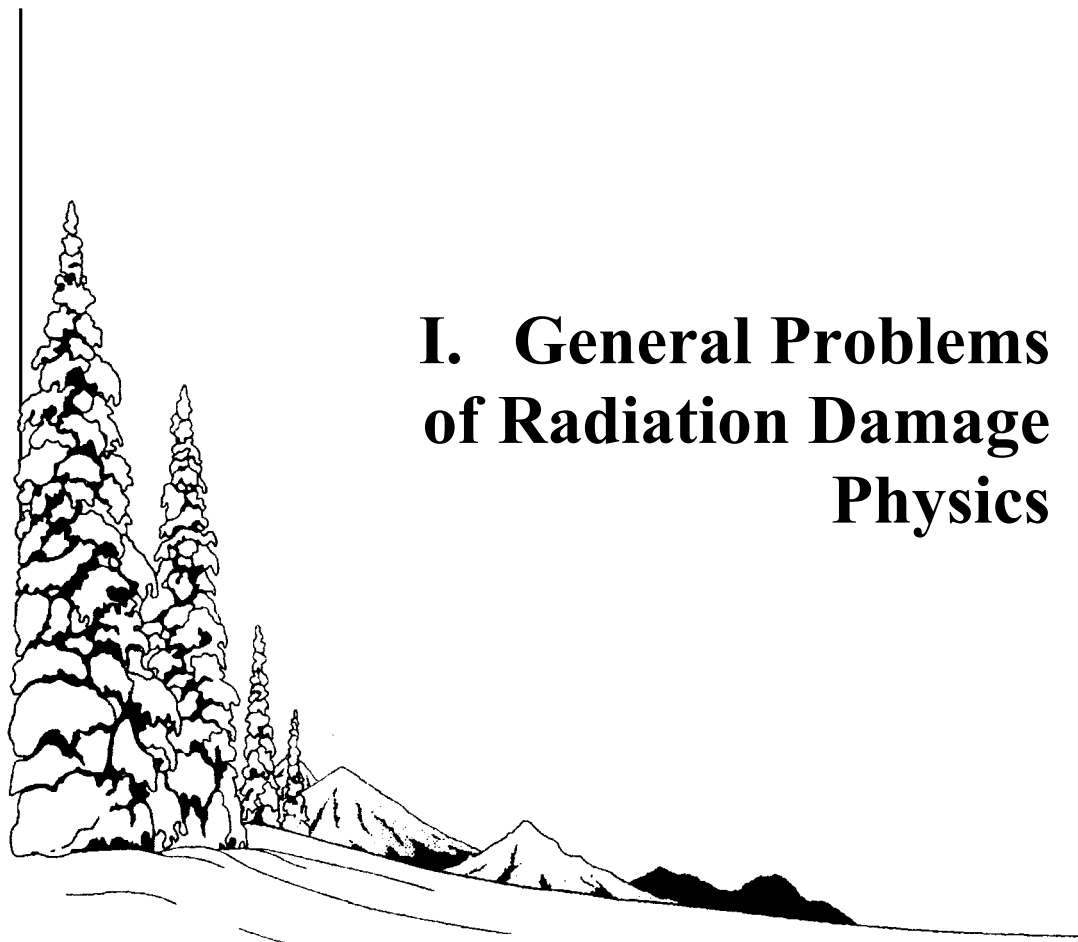
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# I. General Problems of Radiation Damage Physics

The topical problems of radiation damage physics of metals and alloys today are: the properties of point defects in metals and concentrated alloys and their interaction with alloy additions, radiation-stimulated segregation and phase transformations in alloys under irradiation, void swelling, and transmuted gaseous impurities behaviour. The properties of point defects have been investigated in the majority of metals, however, from the point of view of fundamental science, the interest lies in the laws governing the interaction of defects with impurities, particularly in bcc and hcp lattices, where they remain underinvestigated as compared with fcc metals. This Section includes papers dedicated to specific point defects behaviour in various alloys and compounds, including Fe-Cr(Ni) systems on which many radiation-resistant reactor materials are based. Serious attention is given to the formation of atomic segregations, mechanisms of vacancies migration, voids formation, and acceleration of mutual diffusion of elements. The results of modeling of radiation processes in irradiated materials are presented. The mechanisms of radiation-induced nanostructural state formation in metals are discussed.





## Features of Free Migration Stage of Vacancies in Electron-Irradiated Fe-Cr Alloys

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The resistivity recovery (RR) data in electron irradiated Cr4 and Cr9 alloys are obtained over 130-400 K at several initial defect concentrations. It is found that, over the temperature range of 130-250 K, the RR spectra have a complicated structure consisting of 5 stages at 155, 170, 190, 205-210 and 220 K, some of which demonstrate unusual and anomalous features.

It is shown that combined manifestation of several specific features, both in the electronic structure and in the mobility of defects, makes vacancy free migration onset at 205-210 K (stage III) almost invisible by conventional experimental means, such as RR and positron lifetime (PL).

Specific features in electronic structure lead to anomalously high specific resistivity of small vacancy agglomerates formed in stage III. Resistivity contribution of these agglomerates masks resistivity reduction due to defect recombination and makes stage III invisible in conventional RR spectra because of negative (inverted) or nearly zero amplitude of the stage.

A modification to conventional RR method is proposed and applied. It makes use of the difference between RRs in two samples with different defect concentrations and allows separation, detection and identification of free migration stages to be performed, irrespective of the sign of resistivity changes in them and the particular structure of RR spectrum.

Di- and tri-vacancies are immobile in stage III. Therefore vacancy agglomeration in stage III is characterised by dominating di-vacancy formation, which does not lead to detectable increase in positron long-lifetime component due to close PLs in di- and mono-vacancies. At the same time, it is the high specific resistivity of di-vacancies that causes the anomalous RR behaviour. Thus specific combination of resistivity and migration properties of di-vacancies makes stage III almost invisible for conventional experimental means.

Interstitial atom (IA) release from configuration traps and onset of IA long-range migration take place at 220 K that is only slightly above stage III.

Recombination of mobile IAs with immobile di-vacancies initiates the processes similar to those usually occurring in stage III. On the one hand, it leads to reduction in the defect-induced resistivity and emergence of a well-expressed RR peak. On the other hand, the released mobile single vacancies associate with the immobile di-vacancies resulting in vacancy agglomerate enlargement. This enlargement is very similar to a classical signature of stage III as seen by increase in positron long-lifetime component. Thus proximity and sequence in temperature positions of long-range migration onsets for vacancies and IAs, and

common origination of RR stage at 220 K and detectable onset of vacancy clustering, make IA migration stage easily confusable with stage III.

Other RR stages are identified. The stage at 155 K does not correspond to any defect annealing process and is caused, most probably, by a small change in FP specific resistivity. The stages at 170 and 190 K are attributed to close-pair and correlated recombination, respectively, caused by short-range vacancy migration, in part suppressed in stage I due to configuration trapping of IAs.

### **Representation and Analysis of Characteristics of Point Defects and Diffusion Processes on Databases on Radiation Damage Physics of Reactor Materials**

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The paper makes an introduction to complete databases on characteristics of point defects in metals, diffusion coefficients in principal reactor non-metallic systems, self-diffusion coefficients, impurity diffusion, diffusion of components and interdiffusion in zirconium and its alloys, actinides and actinide-based alloys, iron and its low alloys, and steels. Presentation is made of databases analysis by point defect averaged characteristics for FCC and BCC metals, and of analysis of the basic regularities of such characteristics. Computer processing of the most reliable data has been performed..

### **Metal Surface Erosion under Powerful Submicrosecond Ion Beams**

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The capabilities of powerful submicrosecond ion beams with particles initial energy from 50 to 1000 keV, power density  $10^7 \dots 10^9$  W/cm<sup>2</sup> ensuring metal surfaces erosion were investigated.

A mathematical model of surface erosion was developed for conditions of exposure to powerful pulsed charged particle beams.

The calculated dependences of erosion coefficients allow beam parameters to be found, at which the efficiency of erosion treatment on metal surface would be maximum. The efficiency of ion beams with current pulse duration not exceeding 200 ns is the highest.

## The Strong Influence of Atomic Displacement Rate on Radiation-induced Void Swelling of Structural Alloys

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Void swelling was recognized as the major-limiting process for structural materials of various national fast reactor programs. Extensive experimental programs were conducted to study parametric sensitivities of this phenomenon and to mitigate its consequences. Many of the most definitive experiments were interrupted however, as most nations chose to terminate their fast reactor programs. Recently, experiments in the USA, Russia and Kazakhstan have shown that the influence of atomic displacement rate on void swelling was previously strongly underestimated. The implications are significant for design of Gen 4 reactors.

The EBR-II fast reactor was constructed using annealed AISI 304 before the void swelling phenomenon was discovered in the United Kingdom. When EBR-II structural components were examined it was found that swelling as high as 10% had accumulated in some components. AISI 304 was quickly abandoned in favor of other more swelling-resistant steels such as cold-worked 316. It was later shown that all austenitic steels would eventually swell at  $\sim 1\%/dpa$  and that swelling resistance resided in the duration of the transient regime of swelling. It was demonstrated that many material and environmental variables influenced the transient duration, including the atomic displacement rate. However the latter was strongly underestimated, and in development of predictive swelling equations the dpa rate or flux dependence was usually ignored and thereby combined with that of the temperature dependence.

The power of displacement rate to strongly influence the transient regime of swelling was clearly observed in simple solute-free Fe-Cr-Ni alloys irradiated at  $\sim 400$  °C in the FFTF fast reactor. An experiment conducted on annealed 304 hexagonal ducts from EBR-II showed a similarly strong effect of dpa rate.

A series of previously unexamined experiments conducted in EBR-II, FFTF and various fast reactors in states of the former Soviet Union show that increases in displacement rate cause increases in the transient duration of swelling and thereby reduce swelling. Reanalysis of both published and unpublished swelling data for AISI 304 in particular show the competitive effects of both temperature and dpa rate to produce the swelling profiles.

The most significant consequence for Gen 4 application arises in use of published swelling correlations. Identical materials irradiated at high flux in FFTF and BN-350 have been demonstrated to swell much less when irradiated at identical temperatures and doses in EBR-II and BOR-60 at dpa rates approximately one-third that of the higher flux reactors. Since most high fluence data were produced in high flux reactors such as FFTF, derived swelling correlations will strongly underestimate swelling produced in lower-flux reactors and in lower-flux zones of all reactors.

There are other consequences of this flux-sensitivity. First, studies of variables such as helium/dpa ratio or compositional variations will yield misleading results when conducted in two or more reactors with large differences in dpa rate.

Second, in components spanning significant flux gradients, available predictive equations will over-predict the resultant swelling gradients and associated distortion.

Third, flux and temperature profiles are strongly coupled in most reactors such that the extent of the swelling temperature regime has been consistently underestimated. In particular, the lower end of the austenitic swelling regime extends as low as ~280 °C.

Fourth, in thick components there is interaction between gradients in flux and temperature to produce gradients in swelling and irradiation creep rates. These interactions are coupled through the stress-local state and thereby produce the macroscopic distortion. Failure to recognize the effect of flux gradients will lead to incorrect predictions of distortion.

Fifth and finally, since most charged particle simulation experiments are conducted at higher-than-reactor-relevant rates, the swelling is usually much less than would occur during reactor irradiation.

Numerous examples are presented from published and as-yet unpublished studies to demonstrate these consequences. It is important that this new insight be applied to design of new fast reactors, but also be applied to other reactor concepts where swelling is anticipated. While the majority of the cited studies were conducted on austenitic steels, there are studies that suggest similar behavior occurring in ferritic steels.

### **Mechanisms to Maintain Radiation Resistance of Structural Materials**

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Steels and alloys for shells of nuclear reactor fuel elements are exposed to the action of neutrons which damage the structure and generate vacancies, interstitials and their complexes in the lattice. There is an opportunity to achieve radical improvement of radiation stability proceeding from the non-equilibrium characteristic of the structural-phase state of transition metal alloys. The paper is dedicated to comparative evaluation of efficiency of the mechanisms of bringing down radiation damage to structural materials.

## **Crystal Lattice Defects and Magnetoresistance of W and Mo Single Crystals in Strong Magnetic Fields**

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The paper presents investigation of magnetoresistance of pure and perfect single crystals of tungsten and molybdenum and crystals with lattice defects in the temperature range from 2 to 75 K and in magnetic fields up to 15 T.

It was established that conduction electrons scattering on radiation defects and dislocations strongly influences transverse magnetoresistance of metal crystals. An effect of deformation (dislocation) breakdown resulting in change of electron orbits shape in magnetic field was revealed. The electron-dislocation scattering in magnetic field may lead to dc electric current concentration in the vicinity of dislocation walls, i.e., to an internal static skin effect.

## **Modeling of Radiation-Induced Segregation near Spherical Point Defect Sinks in Ternary Substitutional Alloys**

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The phenomenon of radiation-induced segregation (RIS) plays an important role in structure and phase changes in structural materials under irradiation and may influence significantly on phase composition, swelling, corrosion and embrittlement of the materials. In particular, RIS of nickel near voids, precipitates and grain boundaries in Fe-Cr-Ni alloys may result in the formation of austenite regions in ferritic alloys or ferrite regions in austenitic alloys near such point defect sinks. A mechanism of the strong dependence of swelling in these alloys on Ni content caused by RIS near voids has been put forward in the literature. Also, a correlation between the onset of fast swelling stage and the significant Ni enrichment near the void surfaces and the decrease of Ni content in matrix has been observed.

In the present work the methods of modeling of RIS near spherical sinks of point defects in ternary substitutional alloys were developed. An approach of the identical spherical sinks ordered in space was used for modeling. In such an approach any void captures point defects from a spherical Wigner-Zeitzi cell with the radius determined by the void number density.

In the framework of the approach, calculations of RIS near voids in Fe-Cr-Ni alloys for various irradiation temperatures and point defect generation rates were performed. It was shown, that at all irradiation temperatures considered the characteristic dose at which Ni concentration reaches a stationary level near a void surface increases with increasing the void radius, but the dependence of this level on the void radius is non-monotonic. It was

shown also that at the stage of well developed voidage the Ni segregation near void surfaces may result in a decrease of Ni content in the matrix.

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### **Influence of Thermal and Ion Treatment on Change in Atomic Structure and Mechanical Properties of Alloys with Long-Range Ordering**

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Long-range ordered alloys have strong dependence of the structure and properties on thermal and ion treatment. These types of alloys are a useful object in field emission microscopy, because their atom ordering simplifies the analysis of the microscopy images.

Author reviews his own results in connection with this problem. PdCu and PdCuAg ordered alloys were studied after thermal treatment and implantation by argon ions with 21-keV energy, dose range from  $10^{13}$  to  $10^{18}$  ion/cm<sup>2</sup>, current density up to 340  $\mu$ A/cm<sup>2</sup>, to investigate the structural and phase transitions stimulated by implantation. In these experiments, the methods of field emission microscopy were used in combination with mechanical properties test.

It was found that ion implantation leads to phase transformations in the investigated materials. Ordered alloy PdCu undergoes transition to a disordered state. The initially disordered alloy PdCuAg undergoes the cellular reaction during the irradiation with the formation of an ordered phase PdCu and a solid solution PdAg. As a result, the ion implantation leads to disordering of the irradiated sample surface and the undersurface volume.

The thermally stimulated order-disorder transition in PdCu was accompanied by recrystallization, which significantly reduced the grain size and led to yield limit increase.

In the tempered alloy PdCuAg, ion implantation was initiated by the cellular reaction with formation of particles of PdAg in the solid solution matrix PdCu. Such type of a structure gives the alloy a higher strength.

Vacancies pores in the phase PdCu formed as a result of the cellular reaction of alloy PdCuAg was investigated. This type of pores has typical contrast on the field emission images.

### **The Regularities of Irradiation Embrittlement Caused by Swelling**

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Investigation of the principal regularities of irradiation embrittlement caused by swelling is part of approach to another, greater problem of substantiation of the service term of in-vessel devices and reactors in general. The paper discusses the principal results of investigation into this type of embrittlement observed at SSC RF "Research Institute of Atomic Reactors" in the period of over twenty years.

## **Positron Annihilation Study of Nanostructural Features in Reactor Materials**

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At present positron annihilation spectroscopy (PAS) is a powerful tool for the study of the structure of vacancy defects in reactor materials. The affinity-induced confinement of positrons in coherent nanosized precipitation particles or nanoparticles, specifically copper nanoparticles in bcc Fe-Cu alloys and intermetallic nanoparticles in fcc Fe-Ni-Al alloys, was revealed recently. These discoveries launched a new successful application of PAS, namely the diagnosis of the local atomic structure and the chemistry of nanoparticles, and the analysis of the interaction of nanoparticles with irradiation-induced defects in constructional materials. This field of research is topical today since it is necessary to determine reasons for embrittlement of reactor vessel steels and methods for retardation of void swelling of austenitic stainless steels.

The report covers PAS data concerning the kinetics of formation and growth of incoherent nm-scale TiC particles on dislocations in Ti-modified stainless reactor steels. It also deals with positron studies of characteristics of Y-Ti-O nanoclusters in a new class of materials, that is, nanostructured ferritic alloys. The excellent mechanical behavior and a high irradiation resistance make this class of materials suitable for their use as structural materials in future fusion power reactors.

The PAS analysis of characteristics (the chemical composition, the defect structure and the density) of nanoparticles suggests that PAS can compete with such an expensive method as the atom-probe tomography. The report consists of four main parts describing (a) basic principles of the positron annihilation method, (b) positron confinement in precipitates, (c) the interaction processes of radiation-induced vacancies with precipitates in bcc and fcc alloys, and (d) recent positron studies of characteristics of nm-scale carbides in stainless steels and Y-Ti-O nanoclusters in nanostructured ferritic alloys.

## **Effect of Different-Type Sinks on Decomposition of Solid Solution in Electron-Irradiated Fe-Ni Alloys**

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Radiation-induced structural and phase transformations (SPT), which actually determine changes in properties of multicomponent materials during irradiation, are directly related to the free migration of point defects generated under irradiation. Separate Frenkel pairs are only generated upon exposure to electrons. The diffusion length of migrating radiation defects, which governs the radiation efficiency with respect to SPT, depends on the type and the concentration of point defect sinks presented by dislocations, vacancy clusters, interfaces, etc. The solid solution of most-used austenitic chromium-nickel steels can decompose under irradiation. This decomposition is accompanied by a considerable growth of the electrical resistance, which can serve as an indicator of the on-going decomposition. In this study such decomposition was taken as the measure of the radiation damage.

The object of study was the Fe-34.7 at.% Ni alloy quenched from 1323 K (N-Q). This alloy was subjected to plastic deformation ( $\varepsilon = 40\%$ ) so as to obtain an alloy with a high concentration of dislocation sinks and vacancy clusters (N-D). The part of deformed alloy was annealed at 573 K to prepare an alloy, with only dislocation sinks (N-DO). Irradiation with 5-MeV electrons and the isochronal annealing treatment were realized in pure helium.

It was shown that deformation led to depression of decomposition processes under irradiation nearly by a factor of 5 in the N-D alloy and 4 in the N-DO alloy as compared to the solid solution decomposition in the N-Q alloy, in which the concentration of sinks was a minimum. These coefficients changed insignificantly with growing irradiation dose.

When the irradiated and deformed alloys were isochronal annealed, vacancy clusters dissociated in two substages with the dissociation energies of 1.2 and 1.5 eV. These energies were independent of the VC generation method, be it deformation or electron irradiation. The efficiency of freely migrating vacancies, which were formed during dissociation of vacancy clusters, was improved with growing dose. The temperature dependence of the efficiency of different-type sinks was discussed.

This study was supported by the RFBR (Projects Nos. 07-02-00020 and 07-02-96052-Ural).

### **Radiation-Beam Exposure as a Method for Creation of a Gradient Structural-Phase State in Material for Nuclear Power Engineering**

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The paper presents generalization of experimental data on investigation of the opportunity of creation of corrosion-resistant structural-phase states or, if fact, creation of gradient materials from alloys used on fuel element shells of thermal and fast breeders. Study of corrosion processes of modified samples has shown that, in the modified samples, leveling-out of the oxide-metal boundary takes place, with the film becoming more uniform. A serious role belongs to implanted atoms redistribution in the process of corrosion, which changes the mechanism of oxide film growth.

### **Research of iron carbides structural stability with first principle methods**

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Though experimental data specify that  $\varepsilon$ -cementite (carbide  $\text{Fe}_3\text{C}$  representing an interstitial alloy on the basis of hcp lattice of iron) could play a role of an intermediate phase at formation of cementite, its theoretical studies are absent till now. Also the question on structural stability of cementite and the breadth of its homogeneity field on the phase diagram is debatable. Accounts of the present work are performed within the framework of the modern pseudo-potential variant of the electronic density functional theory on the plane waves basis, allowing the complete optimization of a structure to be carried out by the first principle molecular dynamics method.



Formation energies of different modifications of iron carbides  $\text{Fe}_3\text{C}$  in different magnetic states are calculated and first principle simulation of their structural stability is performed, including at a deviation from stoichiometry. For the first time  $\text{Fe}_3\text{C}$  lattice parameters have been defined from the first principles with the use of the complete structural relaxation procedure with variable cell geometry. Formation energies of iron carbides essentially depend on its magnetic state. It was found, that the formation of cementite at cooling cannot proceed through an intermediate phase as it was supposed in series of models: energy of paramagnetic  $\epsilon$ -carbide is too high, and low solubility of carbon in ferrite does not yield an opportunity to approach the necessary composition. The formation of cementite in supersaturated with carbon fields of  $\gamma$ -Fe happens to be energetically preferable. It is shown, that cementite is enough stable structure and can simultaneously be considered as a solid solution of carbon in Fe lattice. The framework of Fe atoms is conserved almost without changes at a deviation from stoichiometry of 5 at.% by decreasing of carbon concentration. At the same time, so small deviation from stoichiometry is accompanied by substantial increase of cementite formation energy, which will result in narrow field of cementite homogeneity on the phase diagram. The feature of defect cementite in a paramagnetic state is an occurrence of the considerable local moment (about 3 Bohr magnetons) in a vacancy environment in carbon sublattice.

The results of the present work allow the mechanism of cementite formation in steels to be understood better and have enabled formulation of the scheme of its formation.

### **Laws Governing Oxide Film Formation and Growth on Ion-Doped Surface of Zirconium Alloys**

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The purpose of the work was to study the effect of implantation conditions on the growth and state of oxide films growth as applied to the service conditions in type VVER nuclear reactors.

In tests lasting over 600-1000 hours, a multiphase oxide film with a complex laminate structure was formed. It was established that in long-lasting tests the processes of oxide film formation on the surface of modified alloys E110 and E635 are similar, i.e., the properties of oxide films are mainly determined by the ion doping conditions, with very weak dependence on the metal matrix elemental composition (its structural-phase composition).

**The Influence of Defects Formation in Be and Si in  
the Process of Ion Mixing under He<sup>+</sup> and Ar<sup>+</sup> Ion beams  
of Broad Energy Spectrum**

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In this paper, on an example of “multilayer film – Be (Si)-substratum” system, within the scope of the “isotropic mixing” model, calculation has been made of radiation paths of atoms initially knocked-out from film, the profiles of their in-depth distribution, and comparison made with the experimental data C(x) at implantation of Al, Ti, Fe, Mo atoms in Be and Si matrices under irradiation with He<sup>+</sup> и Ar<sup>+</sup> ion beams with medium energies  $\langle E \rangle = 5, 10$  and 20 keV.

**Deuterium Capture under Radiation Exposure**

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The regularities of accumulation, desorption and distribution of hydrogen (deuterium) implanted in steels 06X18H10, 08X18H10T and 12X18H10T were investigated over a broad range of irradiation doses to concentrations characteristic of reactor VVER-1000 thermal-neutron spectrum and expectedly occurring in thermonuclear reactors. Data were gathered on capture and thermally activated release of hydrogen (deuterium) in the irradiation temperatures range 20...600 °C, and post-implantation annealing at 20...1300 °C.

**Peculiarities of Deformation-Plastic Behavior of Metal Polycrystals  
Irradiated with Neutrons up to High Damaging Doses**

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Systematic study of regularities of irradiated metal and alloy deformation is topical in development of the theory of setting mechanical properties formation and creation of new radiation stable materials for the reactor. Also it is necessary to pay attention to finding and analyzing the “true” characteristics of durability and plasticity instead of the engineering ones, as we usually do.

In this work we investigated the pattern metals with technical purity – copper, nickel, iron, molybdenum and industrial constructional alloys 08Cr16Ni11Mo3 and 12Cr18Ni10Ti non-irradiated and irradiated with neutrons. Pure metals were irradiated in active zone of the reactor WWR-K (Almaty) at the temperature of ~80°C up to maximum fluence  $5 \cdot 10^{24}$  n/m<sup>2</sup>.

The plates of austenitic stainless steel of 2x0.3x10 mm were cut out of edges of the casings of spent fuel assemblies of the reactor BN-350 from different levels («-300 mm», «-150 mm», «0 mm», «+150 mm», «+300 mm» from the center of active zone). The irradiation was carried out at comparatively low temperatures typical for BN-350 and damaging dose rates, which maximum value comprised 55 dpa.

Mechanical test was made at a universal testing machine “Instron-1195” using a developed system of computer vision – optical digital extensometer with the marks specially covered on the surface of the sample. During the statistic stretching we registered change of marks dimension and their intervals. This allowed, at using of specially developed programs, to determine “true” values of stresses  $\sigma_{\text{true}}$  and deformations  $\varepsilon_{\text{true}}$  and their distribution along the sample as well as peculiarities of localization of plastic deformation, in particular, the formation and development of a neck in small irradiated samples.

For all investigated materials there have been obtained both engineering and “true” curves of the plastic yielding  $\sigma_{\text{true}}=f(\varepsilon_{\text{true}})$ , there have been examined peculiarities of deformation strengthening. It is shown that for metals and alloys irradiated with neutrons in the majority of cases the dependencies  $\sigma_{\text{true}}=f(\varepsilon_{\text{true}})$  can be described with the following equation:

$$\sigma_{\text{truth}} = \sigma_0 + K \sqrt{\varepsilon_{\text{truth}}} .$$

For the steel 12Cr18Ni10Ti irradiated up to the damaging dose 55 dpa, there was revealed the anomalous high value of total lengthening accompanied by formation and moving of deformation band (or “strain wave”). This result is analyzed from the position of evolution of dislocation structure in the crystal, which contains radiation defects.

### **Peculiarities of Dissipative Processes at Static Deformation of Iron Irradiated by Neutrons**

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According to up-to-date conceptions, the irradiation of metal polycrystals with high-energy particles generally leads to their strengthening and embrittlement. Literature provides extensive data on investigation of radiation-stimulated processes, in particular, on changes of mechanical properties as functions of fluence of particles, type and temperature of irradiation.

At the same time, analyzing the processes of radiation strengthening and embrittlement the researchers omit from their considerations peculiarities of plastic flow of the material, kinetics of deformation strengthening, processes of accumulation and dissipation of energy as well as peculiarities of deformation localization.

In this work utilizing the methods of deformation calorimetry, optical extensometry, metallography and transmission electron microscopy we investigated peculiarities of plastic flow, deformation strengthening and dissipative processes in the armco-iron non-irradiated and irradiated with neutrons up to fluences from  $2 \cdot 10^{18}$  to  $1.4 \cdot 10^{19}$  n/cm<sup>2</sup>.

During deformation experiments there were obtained and calculated the strengthening and plasticity values, work of deformation, dissipated heat and latent energy  $E_s$  for non-irradiated and irradiated samples. It is shown that irradiation of samples leads to the decrease in the value  $E_s$  that accumulates till destruction. For iron irradiated with fluence  $1.4 \cdot 10^{19}$  n/cm<sup>2</sup> we revealed that the heat released at deformation exceeded the external work. This result has been subjected to analysis involving data from electron microscopy.

Using the deformation extensometry method there were analyzed the curves “true strength – true deformations” enabled to determine parameters of deformation strengthening of irradiated material. There has been determined a threshold stress rate for initiation of deformation localization development.

We considered distribution of stress and deformations along sample. Obtained data were compared with the results of structure investigations and microhardness measurements, the correlation of microhardness of armco-iron and flow stress values have been examined.

### **Diffusion Decay of Supersaturated Impurities Solution in Grain Boundaries**

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The paper is dedicated to diffusion decay of supersaturated solution of impurities in intergranular boundaries, in its first two stages.

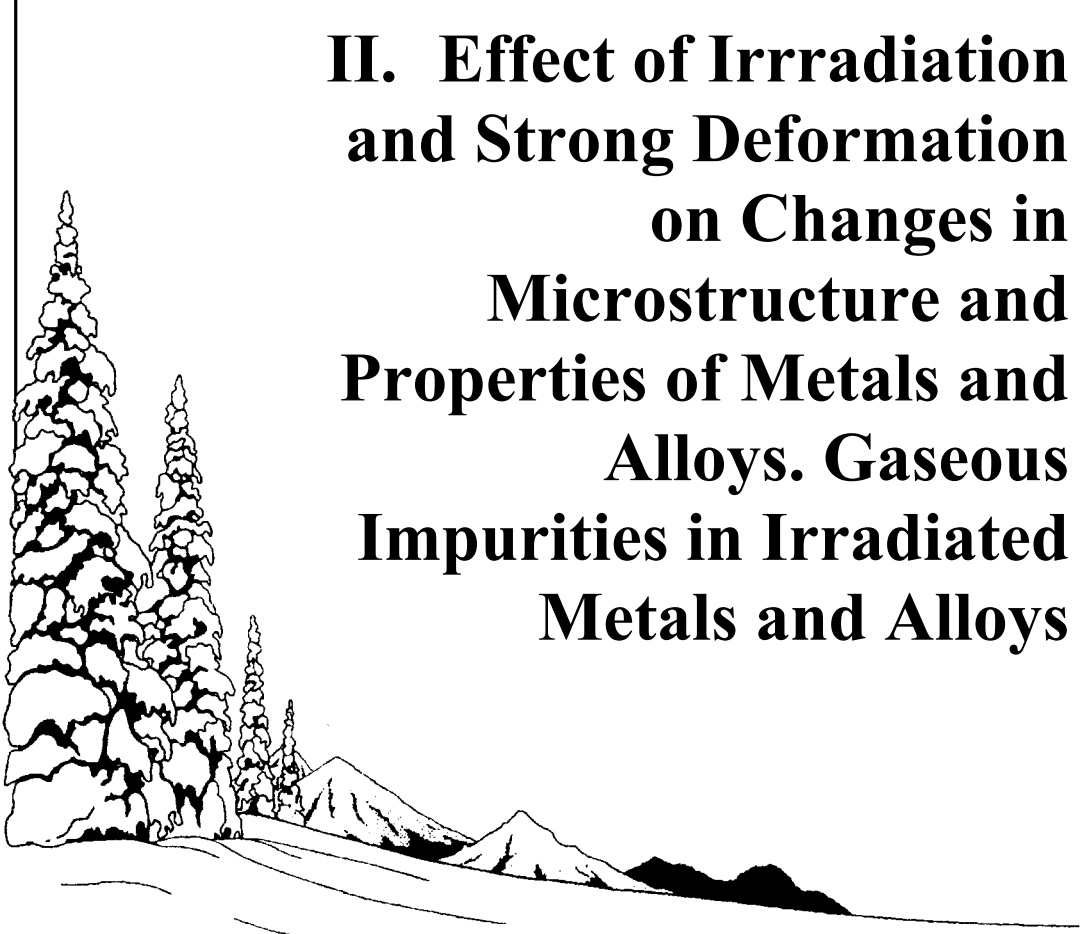
A set of basic equations describing the process has been derived. Estimate has been made of the time of system relaxation for achieving a quasi stationary state in the range of nuclear dimensions from a single atom to a value exceeding the critical size four-fold.

### **Hybrid Model for Simulation of Radiation Damage in Crystals**

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The paper proposes a hybrid model combining the molecular dynamics and the Monte Carlo methods in describing primary radiation defects in crystals caused by particles with energies not higher than several tens of keV. A comparison is made of the proposed model with the techniques based on the Monte Carlo method on an example of a problem of modeling primary radiation effects in plutonium caused by self-irradiation.



## **II. Effect of Irradiation and Strong Deformation on Changes in Microstructure and Properties of Metals and Alloys. Gaseous Impurities in Irradiated Metals and Alloys**

The study of the processes of interaction of point defects of radiation and deformation origin, as well as their complexes, with impurity atoms, dislocations, interphase and grain boundaries, with the application of modern investigation methods, at all stages of formation of complex defect structure in nano and submicrocrystal metal systems, and getting an insight into the effect of such interactions on deformation- and radiation-induced processes are necessary for revealing the true reasons of changes in the properties of irradiated materials and predicting their behaviour in radiation fields. The Program of the Section includes papers dedicated to phase and structural transformations in preliminarily deformed metals and alloys at their exposure to high-energy neutrons, ions and electrons, and to deformation processes in preliminarily irradiated materials. For the first time, attention at the Seminar will be drawn to analysis of structural-phase transformations caused by point defects generation under severe cold deformation. It has been found recently that, similar to neutron irradiation, intensive cold deformation may lead to alloys atomic layering and formation of segregations in the boundary regions, which is explained by migration of generated point defects to sinks in the form of grain boundaries and deformation fragments. There is made notice in the

**papers of the possibility of deformation-induced dissolution of carbides, intermetallics or nitrides in metal matrix at drift of formed interstitial atoms in the dislocations stress field. Phase and structural transformations are analyzed both under cold deformation and under alloys irradiation with high-energy nuclear particles; the processes of intermetallics dissolution in displacement cascades, atomic disordering, change of superconductivity conditions, and mechanical properties behaviour in irradiated metals are discussed. The effect of helium and hydrogen isotopes on physical and mechanical properties of irradiated alloys is analyzed.**

**Radiation-Induced Dissolution of Ni<sub>3</sub>Me (Ti, Al, Si, Zr) Intermetallic Particles in Displacement Cascades upon Exposure of Fe-Ni-Me Alloys to Neutrons at 340 K**

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Processes of the radiation-induced dissolution of fine particles of Ni<sub>3</sub>Me (Ti, Al, Si, Zr) intermetallics in matrices of quenched and aged FeNi<sub>35</sub>Me (Ti, Al, Si, Zr) alloys were studied using NGR spectroscopy. The alloys were exposed to fast neutrons in an IVV-2M reactor at a temperature of 340 K and a fluence of  $1.5 \cdot 10^{20}$  n/cm<sup>2</sup>. All the FCC alloys were aging compounds. Fine particles of Ni<sub>3</sub>Me intermetallics were formed in the FCC matrix of the alloys already during water quenching from 1373 K. Aging of the FeNi<sub>35</sub>Me (Ti, Al) alloys at 723-873 K caused a considerable reduction of the Ni concentration in the FCC matrix because of the increase in intermetallic phases [1].

Aging of the Zr- and Si-alloyed compounds was less intensive because they contained a considerable quantity of intermetallics already in the quenched state. Neutron irradiation of the binary FeNi<sub>36</sub> alloy was followed by short-range atomic ordering in displacement cascades and a redistribution of Fe and Ni similarly to cascade-free irradiation by high-energy electrons with  $E = 5.5$  MeV at a fluence of up to  $10^{19}$  e/cm<sup>2</sup> under the same temperature conditions [2]. When the FeNi<sub>36</sub> compound was alloyed additionally with Ti, Al and Si, the character of the atomic distribution in the particles and the matrix changed radically under exposure to both neutrons and electrons near 340 K. Processes of short-range ordering and redistribution (observed in binary FeNi<sub>36</sub>) were replaced by radiation-accelerated aging under cascade-free electron irradiation [2].

An exception was the quenched (aged or deformed) FeNi<sub>35</sub>Zr<sub>3</sub> alloy with zirconium, whose spectrum pointed neither to processes characteristic of binary FeNi<sub>36</sub> nor to those of radiation-accelerated aging. When all the additionally alloyed compounds, both quenched and aged ones, were exposed to cascade-forming fast neutrons in an IVV-2M reactor, the Ni concentration of their matrices increased due to the radiation-induced dissolution of intermetallic particles in displacement cascades. Changes in NGR spectra of the FeNi<sub>35</sub>Me (Ti, Al, Si, Zr) alloys after neutron irradiation were analogous to those observed after intensive cold plastic torsional deformation in Bridgman anvils and caused by the deformation-induced dissolution of intermetallics [1]. However, spectra of the Zr-alloyed compound exhibited, along with the growth of the partial contribution from high fields (the enrichment of the matrix with nickel from Ni<sub>3</sub>Zr particles), an additional component with nearly zero fields corresponding to paramagnetic regions of the structure.

This feature could not be related either to radiation-accelerated aging (similarly to aging of FeNi<sub>35</sub>Ti<sub>3</sub> alloys with growing neutron irradiation temperature [3]) or to the matrix decomposition like the radiation-accelerated decomposition in binary FeNi<sub>36</sub>. This conclusion was drawn considering the absence of any changes, which would point to the radiation-accelerated diffusion, in the spectrum of the quenched, aged and deformed

## **II. Effect of Irradiation and Strong Deformation on Changes in Microstructure and Properties of Metals and Alloys. Gaseous Impurities in Irradiated Metals and Alloys**

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FeNi<sub>35</sub>Zr<sub>4</sub> alloy subject to cascade-free electron irradiation. In this connection, it is interesting to consider specific features of the structure of the FeNi<sub>35</sub>Zr<sub>4</sub> alloy in displacement cascades and a low diffusion mobility of Zr having a large atomic radius. One may reasonably think that the radiation-induced dissolution of intermetallics, which leads to the increase in the concentration of mobile Ni in the FCC matrix of the alloy, is accompanied by disordering in Ni<sub>3</sub>Zr and a partial substitution of Fe atoms for Ni. Notice that the dissolution of intermetallics during cold deformation is also characterized by the predominant release of more mobile atoms to the alloy matrix as it was shown for the Al-Fe system [4]. Nickel atoms are more mobile in the Fe-Ni matrix during radiation dissolution of the Ni<sub>3</sub>Zr intermetallic.

This study was supported by RFBR-Ural (No. 04-02-96089), RFBR (No. 07-02-00020), the RAS Presidium for nanoscale materials (No. 20), and the fund of the Magnitogorsk Integrated Iron & Steel Works and Ausfer" JSC (No. 29-05-03).

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## **Effect of Helium Accumulation in Austenitic Steel on Microstructure Evolution and Radiation Damageability of VVER Internals Materials**

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The basing and prolongation of long-term designed operation life of VVER-type reactors demands to study the regularities of change of structure and properties of structural materials irradiated by neutrons under operation conditions typical to internals constructions (IC) of these reactors. One of the important parameters which characterize the irradiation conditions of IC materials is helium accumulation which may be used to verify the calculated damage dose in irradiated material and to characterize the spectral distribution of neutron flux [1]. Radiogenic helium effect on IC materials microstructure evolution under reactor irradiation is also significant. The evolution of dislocation loops, voids, radiation-induced formation of the second phases results in significant changes of the material mechanical properties [2-3]. This work aims at studying the helium effect on dose changes of microstructure and mechanical properties of Fe-18Cr-10Ni-Ti stainless steel after irradiation in the VVER reactors and their interrelationship.

TEM-examination of the Fe-18Cr-10Ni-Ti steel microstructure revealed the dependences of parameters (dislocation loops size and density) as function of damage dose. It was determined that helium accumulation measured by mass-spectrometric method with isotope dilution in (10-200) appm range may effect on strength and plastic property levels of steel at early stage (damage dose less than 5-15 dpa) of irradiation hardening at temperature ~300 °C.



## II. Effect of Irradiation and Strong Deformation on Changes in Microstructure and Properties of Metals and Alloys. Gaseous Impurities in Irradiated Metals and Alloys

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### Effect of Dislocation Structure on Accumulation of Implanted Deuterium in Austenitic Steels

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The ion bombardment of hydrogen-containing metals and alloys leads to the radiation-induced segregation (RIS) of hydrogen. The RIS intensity strongly depends on the initial structure of test samples, specifically the density of dislocations (DD). Data concerning the interaction of dislocations and hydrogen atoms are contradictory. Therefore, the approach we used to determine the effect of the dislocation structure on the RIS allowed separating the contributions of dislocations and deformation vacancies (DVs).

Samples of Cr<sub>16</sub>Ni<sub>15</sub>Mo<sub>3</sub>(Ti<sub>1</sub>) austenitic steels with the DD equal to  $1 \times 10^7 \text{ cm}^{-2}$  (quenched) and  $3 \times 10^{10} \text{ cm}^{-2}$  (both after cold plastic deformation and after deformation and annealing of DVs) were prepared. Their structures were certified by the TEM method. The samples were exposed to D<sup>+</sup> ions having the energy of 700 keV at the irradiation intensity of  $1 \times 10^{13} \text{ ion} \cdot \text{s}^{-1} \cdot \text{cm}^{-2}$ . As the samples were irradiated continuously up to doses of  $\sim 1 \times 10^{18} \text{ cm}^{-2}$ , the concentration of deuterium in the RIS was measured discretely using the nuclear reaction D(d,p)T.

It was found that if the concentration of dislocations in these steels was high, the dislocations served as

- effective sinks for vacancies, which represented a constructional material for deuterium traps (vacancy clusters), and routes of the fast diffusion of deuterium atoms from the RIS zone to the bulk;
- effective deuterium traps when dislocation tangles, deformation twins and deformation stacking faults were formed.

A scheme was proposed for determination of the contributions from initial traps (ITs) of the dislocation origin, radiation-induced traps (RTs) of different types, and sinks to the total trapping of deuterium in the RIS. The scheme allows determining the IT capacity, start doses of the deuterium capture in RTs, and the capacity of sinks in samples having different initial structures.

This study was supported by the Russian Foundation for Basic Research (project No. 04-02-16053).

### **Thermodesorption Investigation of Helium Behaviour in Nickel Alloys and Austenitic Steel**

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With the use of the thermal desorption spectrometry method, the features of capture and emission of ion-implanted helium ( $\text{He}^+$ -40 keV,  $\Phi = 5 \cdot 10^{20} \text{ m}^{-2}$ ,  $T_{\text{irr}} = 20 \text{ }^\circ\text{C}$ ) in Ni, nickel alloys with Mo, W, Ti, KhNM-1 and austenitic steel ChS-68 were investigated.

### **The Influence of Irradiation Conditions and Samples State on Deuterium Capture and Retention in Cr-Ni-Ti Steel**

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Using the methods of thermal desorption spectrometry and nuclear reactions, quantitative data were obtained on accumulation and retention of ion-implanted deuterium in austenitic-class steels.

The influence of the temperature of preliminary thermal treatment, state of the surface, conditions of mechanical and electrolytic samples preparation, irradiation temperatures, and particle flux densities per number and position of gas emission stages on the temperature scale, and their intensities redistribution were shown.

The conditions of appearance of high-temperature component in the thermal desorption spectrum were determined.

### **Irradiation Influence on Impurity Grain Boundary Segregation in Polycrystalline Materials**

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The phenomenon of an impurity precipitation on the grain boundary of polycrystalline materials is studied. The model problem is considered. In the problem requirement was suppose that atoms that diffuse from the grain body into grain boundary were generated from originally immobile admixture by irradiation. The irradiation influence is taken into account along with transmutation by the application of radiation-modified diffusion coefficient. By means of method that has been used in paper [1] for the first time the complete system of differential equations were obtained. That system describes the phenomenon of an impurity precipitation on the grain boundary of polycrystalline materials in terms of the

approximation of separate grain.

With a view to get an analytical solution one supposed that grain had parallel-sided, spherical or cylindrical shape. In such a way one can received a rough-and-ready algebraic equations system that described the grain boundary impurity concentration as time function. This rough-and-ready system is just both weak and strong grain boundary impurity solution. The concentration of grain boundary solution was found as time and radiation treatment power function.

The kinetics of an impurity resolution under the influence of sharp changing of the temperature or irradiation intensity is studied. It was found the case when the grain boundary absorbed the additive impurity and the case when the equilibrium concentration level was less than initial one.

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### **Phase Transformations in Austenitic Steels under Gamma-Irradiation**

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In this paper, with the use of Moessbauer spectroscopy on Fe<sup>57</sup> nuclei in back scattering geometry, at registration of internal conversion electrons, investigation of steels 12X18H10T and 08X16H11M3B after cold-work, thermal annealing and  $\gamma$ -irradiation was carried out.

### **Methods and Results of Investigation of Hydrogen Diffusion in Reactor Zirconium Materials**

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The literature data were analyzed and statistically processed on coefficients of hydrogen diffusion in  $\alpha$ -Zr (sponge and iodine forms), its commercial alloys and real components of water-cooled nuclear power plants, over the period from 1954 to 2004.

**Investigation of Changes in the Structure and Properties of Aluminum Alloy Al-Mg at Medium-Energy Ions Implantation**

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Judging by the results of investigations carried out in the recent decades, ion beams application is one of the most promising trends in modern technology of materials treatment. In this connection, study of the effect of ion-beam treatment on the structure, phase composition and properties of commercial aluminum alloys is of great scientific and practical interest. In this work, the object of investigation was an alloy of aluminum-magnesium system (Al-6MG) from the group of alloys not hardened by thermal treatment.

A 3-mm thick strain-hardened alloy sample was subjected to ion-beam treatment. In the course of irradiation, the energy of ions ( $E = 20\text{--}40$  keV), ion current density ( $j = 150\text{--}400$   $\mu\text{A}/\text{cm}^2$ ), irradiation dose ( $D = 2.25 \cdot 10^{15}\text{--}5 \cdot 10^{17}$   $\text{cm}^{-2}$ ) varied. In the course of irradiation, the maximum temperature of samples did not exceed the temperature of intermediate annealing (320 °C) for work-hardening removal in the course of this alloy rolling.

Uniaxial tension tests have shown that ion irradiation to doses  $1 \cdot 10^{17}$   $\text{cm}^{-2}$  and higher result in higher plasticity and loss of strength characteristics of the alloy. According to TEM data, the effect is due to cellular structure transformation, formation of subgrain structure with low-angle borders, and intermetallics  $\text{Al}_6(\text{Mn}, \text{Fe})$  refinement and dissolution.

Analysis of samples cross-section has shown that the above structural changes take place under irradiation not in the thin subsurface layer of Al-6MG alloy only, but throughout the whole 3-mm depth of the sample, while the  $\text{Ar}^+$  ions projected range at 20 keV energy in an aluminum alloy (according to calculation done by the TRIM method) is 40 nm only.

Internal strain relief under the beam takes place within  $\sim 100$  s, which is 20 times faster than in the course of annealing for strain relief.

**Features of Accumulation of Microdefects in Copper under Shock-Wave Loading**

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The optical microscopy and X-ray diffraction methods were used to investigate distribution of micro-, and macro-defects in copper samples recovered after shock-wave loading. The electric gun GNUV was used to generate the shock wave. Sample loading dynamics was recovered by the free surface velocity measured based on the Doppler shift in the wavelength of the probe laser radiation. In-depth distribution of defects was compared for two modes of sample loading. In the first mode, samples were loaded by the shock wave whose amplitude was insufficient for spallation, which was initiated by the shock wave in the second mode.

**Influence of  $\alpha$ - $\epsilon$  Phase Transformation in Iron and 30XГCA Steel on Features of Spallation in Wedge Samples under Explosive Loading**

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Set-ups and results of explosion experiments using optical lever techniques with wedge-shaped samples from non-alloyed high-purity iron and steel 30XГCA hardened to HR<sub>c</sub> 35...50 at samples loading, with tangential and square-directed detonation of charges of explosive of various power and thickness, are considered.

**The motion, rotation and annihilation of dislocation loops in the stress fields originating from line dislocations**

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In this study, we present the behavior of glissile loops generated either by agglomeration of self interstitial atoms or formed directly by collision cascades. These loops tend to glide by elastic interaction with the stress field, and to climb toward the line dislocation.

We have evaluated the elastic interaction between the infinitesimal interstitial loop and the line dislocation by incorporating the change in the normal vector of the dislocation loop in the stress field of the dislocation. The rotation of the dislocation loop strongly changes the interaction between the line dislocation and the loop. The results obtained by the modified elastic theory were compared with the MD simulation, and good agreement was obtained for the stable position of the dislocation loop near the line dislocation.

We have also evaluated the mechanisms by which a line dislocation absorbs dislocation loops existing in close vicinity by the conservative climb of the dislocation loop. The driving force of this process is assumed to be the pipe diffusion of vacancies along the loop line.

**Effect of Intermetallic Nanoparticles on Accumulation of Vacancy Defects in Iron-Nickel Alloys**

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The main factor limiting the use of stainless austenitic steels in fast reactors is their vacancy swelling. Swelling can be considerably suppressed through the use of aging alloys, which form dispersed intermetallic precipitates Ni<sub>3</sub>Ti(Si,Al). However, the mechanism of influence of precipitates on point defects behavior under irradiation is not completely known.

## **II. Effect of Irradiation and Strong Deformation on Changes in Microstructure and Properties of Metals and Alloys. Gaseous Impurities in Irradiated Metals and Alloys**

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This study deals with positron spectroscopy of the evolution of point defects in iron-nickel alloys under electron irradiation at early stages of the radiation damage ( $10^{-4}$ - $10^{-3}$  dpa), when just small defect clusters are formed, depending on the initial state of the alloys (quenching or aging for different periods of time).

The objects of study were Fe - 36.5 mass % Ni - 2.5 mass % Ti (Fe-Ni-Ti) and Fe - 34.5 mass % Ni - 5.4 mass % Al (Fe-Ni-Al) alloys. Some samples were aged at 823-1023 K for different periods of time. All samples were irradiated to 5-MeV electrons in a linear accelerator at temperatures of 300 K, 423 K and 573 K. The angular correlation of annihilation radiation (ACAR) was used to investigate defect structure. The S-parameter, which is defined by the concentration and the type of vacancy defects, characterized changes in ACAR spectra.

The study demonstrated that  $\text{Ni}_3\text{Ti}$  precipitates considerably reduced the accumulation of vacancy defects at the irradiation temperature of 573 K, whereas at lower temperatures the effect of these precipitates on the behavior of defects was not detected. At the same time, defects were accumulated less already at 423 K in the alloy containing  $\text{Ni}_3\text{Al}$  particles. It was found that the extent of this effect under irradiation at 423 K strongly depended on both the density and the size of  $\text{Ni}_3\text{Al}$  particles. However, when aged Fe-Ni-Ti and Fe-Ni-Al alloys were irradiated at 573 K, the accumulation of vacancy defects depended little on the particles dispersion. Possible mechanisms, by which the precipitates influenced the accumulation and annealing of radiation defects, were discussed.

This study was supported by the Russian Foundation for Basic Research (project No. 04-02-16053).

### **Hydrogen Penetration through Structural Materials at Ion Bombardment**

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Investigations of hydrogen penetration have a great importance due to material issues solving for driven thermonuclear synthesis.

In the present work the results of investigation of border conditions and number of structural factors influence on hydrogen penetration through structural materials during the hydrogen ions bombardment are provided. It was shown that hydrogen penetration factor strongly depends on border conditions (penetration from gaseous phase during hydrogen ions bombardment). Preliminary irradiation by He, O, N, c ions sample's surface layer significantly effects on hydrogen penetration through investigated materials.

The results of investigation of hydrogen penetration through structural materials with certain characteristics during accelerated hydrogen ions bombardment are presented. Influence of preliminary cold deformation (5, 15, 30, 50%) and grain size (3-5, 30-50, 400-600  $\mu\text{m}$ ) on hydrogen penetration factor is studied at ion irradiation. It is found that preliminary cold deformation as well as grain size decreasing increases hydrogen penetration through investigated materials during their bombardment by hydrogen ions ( $T = 200$  °C).

### Irradiation Influence on Gas Bubbles Movement under Temperature Gradient

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A theoretical investigation of a gas swelling of the uranium dioxide is made. An isotropic material has been used as a theoretical model. The atoms of a gas, the bubbles filled with gas, Frenkel pairs and dislocation loops been conceived in the modeling materials under irradiation was taken into account. It was shown that the presence of the temperature gradient can give rise to an arranged bubbles movement which is caused by the mechanism of the matrix atom surface diffusion. The full equation system that described a gas swelling is written. The system includes the kinetic equation for the bubbles distribution function, a balance equation for the gas atoms, and two kinetic equations for both vacancy and interstitials dislocation loops distribution functions. It was carried out that by stationary case that is realized because of the gas atoms are caught by moving voids and carried away of the material two balance equation for own defects can be combined to a unique one. The expressions for the bubbles growth velocity and their filling up with the gas in two limits cases are derived. In the first case that is so-called little bubbles diffusion processes are matched in such a way that the characteristic time of the bubble size changing is much more than characteristic time of the bubble filling up with the gas. In the second case that is so-called big bubbles the reverse ratio takes a place. It is cleared up that mechanical loading result in an anisotropic distribution of the dislocation loops. In some conditions of the loading that defined by the mechanical properties of the material's skin the loops can fully dissolve. An analytic expression for the relative swelling velocity of the material is found for both limit cases [1].

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### Effect of Ion Irradiation on Structure, Phase Composition and Mechanical Properties of Strain-Hardened Aluminum Alloy 1441

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The effect of accelerated argon ion beams on mechanical properties, structure and phase composition of commercial aluminum alloy 1441 of Al-Cu-Mg-Li system was investigated. The samples for investigation were cut from strain-hardened clad 3-mm thick sheet metal manufactured at the Kamensk-Uralsky Metallurgical Plant. Samples were irradiated with continuous Ar<sup>+</sup> ion beams on the ion-beam implanter PULSAR with a glow-discharge hollow-cathode ion source. Ion beam density was 150  $\mu\text{A}/\text{cm}^2$ , ions energy  $E = 20$  keV, irradiation dose varied within  $D = 1 \cdot 10^{15} - 1 \cdot 10^{17} \text{ cm}^{-2}$ . In the course irradiation, the target temperature was continuously controlled with the help of a chromel-alumel

## II. Effect of Irradiation and Strong Deformation on Changes in Microstructure and Properties of Metals and Alloys. Gaseous Impurities in Irradiated Metals and Alloys

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thermocouple. The maximum temperature of samples heating under low irradiation doses did not exceed 40 °C, and under high irradiation doses it was not over 220 °C.

It was established by mechanical properties measurement and transmission electron microscopy analysis that irradiation with Ar<sup>+</sup> ions at E=20 keV, j=150 μA/cm<sup>2</sup>, D=1·10<sup>15</sup>-1·10<sup>16</sup>cm<sup>-2</sup>, irradiation time 1–10 s, leads to strong change in the structure of strain-hardened alloy 1441: there takes place a transfer from cellular to a similar polygonal structure. At that, relative elongation increases, while strength characteristics are preserved. At D = 1·10<sup>17</sup> cm<sup>-2</sup> (irradiation time ~ 100 s), there are observed an abrupt increase in plasticity and yield point drop, which is due to formation of a homogeneous coarse-crystal grain structure with grain diameter over 10 μm. A similar recrystallized structure is observed after 2-hour annealing.

It was also established that, under ion irradiation, there takes place dissolution particles β'(Al<sub>3</sub>Zr), Al<sub>8</sub>Fe<sub>2</sub>Si in the structure of both the deformed and the annealed (T ~ 400 °C, 2 h) alloy, and new phase Al<sub>2</sub>LiMg formation occurs.

Changes in alloy 1441 dislocation structure and phase composition in are noted both in the subsurface layer adjacent to the ions penetration zone and throughout the whole bulk of the sample (sample thickness is tens of thousands times larger than the ions projected range).

### Deformation-induced segregations in reactor Fe-Cr-Ni alloys

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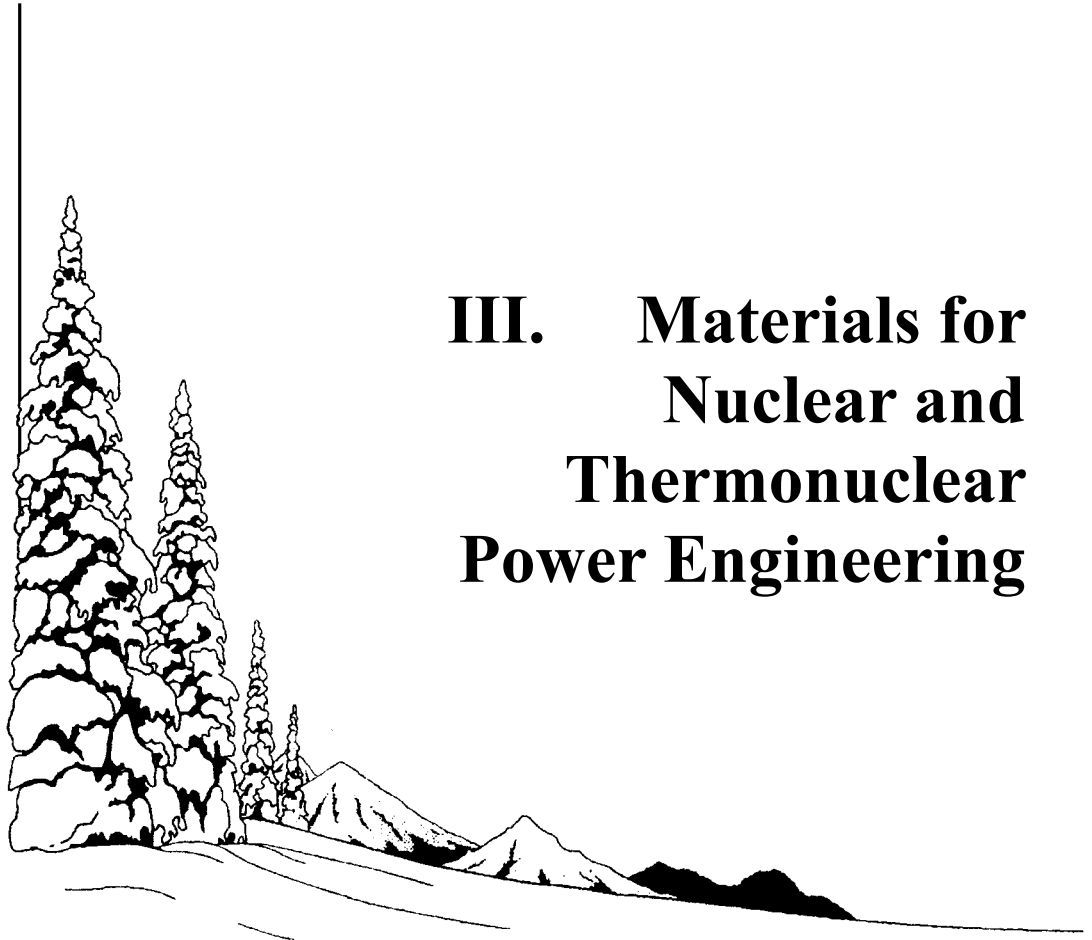
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The model of deformation-induced segregation on a moving grain boundary in three-component alloy subjected to intensive plastic deformation that take into account generation and absorption of point defects, as well as their mutual recombination, is proposed. Redistribution of alloy components atoms and formation of boundary segregations at intensive plastic deformation in stable austenitic alloys Fe-12Cr-30Ni are investigated. Based on a numerical solution of diffusion equations it was shown, that motion of grain boundary leads to formation of deformation-induced segregation with asymmetric alloy components profiles across grain boundary. The grain boundary have been shown to be enriched with nickel and depleted with chrome and iron. It was shown too, that deformation- and radiation-induced segregations to grains boundaries in stable austenitic alloys of this type are described by analogous laws, which let make an estimate of radiation-induced segregations in different reactor alloys with the help of their plastic deformation data.

Concentration profiles for the components of a Fe-Cr-Ni alloy in the vicinity of a moving grain boundary for different temperatures, boundary speeds and nickel content are presented in our report. Studying results of effect of generation speed of point defects, deformation time and other parameters are also shown. Segregation features at relatively low temperatures (close to room temperature), particularly important for experiment interpretation of deformation by shear under pressure in Fe-Cr-Ni alloy and allowing change of magnetic properties of the alloy subjected to intensive plastic deformation to be explained, were revealed. An increase of nickel segregation at grain boundaries with increasing temperature to 600-700 K is awaited.

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### **III. Materials for Nuclear and Thermonuclear Power Engineering**

One of the topical problems of today is the problem of creation of new metal-based materials for fusion and fission-type reactors. For example, the reactors currently under construction (BN -800) and future fast-neutron reactor projects (BN-1800) still expect the construction materials showing high radiation resistance to withstand the damaging dose of 100-130 dpa, which would ensure the required depletion of fissile material. The Section includes a great number of material-science presentations on the subject of radiation-induced changes of physical and mechanical properties of different reactor materials (those currently in use and showing promise). There will be discussed the material-science problems of prospective salt-liquid nuclear reactors, the corrosion behaviour of ferritic-martensitic steels in lead melt, the problems of high-temperature creep, fcc and bcc steels and beryllium swelling, the effect of irradiation on austenitic reactor steels, including the only “standard” austenitic grade ChS-68 steel for the BN-600 reactor fuel elements. The results obtained for real reactor materials are analyzed proceeding from the general principles of radiation physics of solids.



### **Manufacturing of Experimental Fuel Elements with Plutonium Dioxide for Tests in the MIR Research Reactor**

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At the present time it is necessary to confirm an opportunity of manufacture an efficient fuel element for burning plutonium with production of electric power in the VVER-1000, PWR, BWR reactors. The advantage of the fuel element type is absence of secondary accumulation of plutonium as it occurs, for example, in the MOX fuel. The first stage of work is manufacture of the first experimental number of fuel elements with plutonium dioxide ( $\text{PuO}_2$ ). These fuel elements are intended for tests in a water-water loop of the MIR reactor and for the subsequent material science investigations.

Manufacturing of fuel elements was carried out in view of experience and results of the previous research, developmental and experimental works, and also in view of positive results of fuel elements mass operation with uranium dioxide in which similar design and technological decisions of fuel element cores were used. In the framework of the work the equipment of covering by a fuel composition, assembly, hermetic sealing and the control of fuel elements is carried out. Then fuel elements have been transferred for reactor tests.

For comparative tests in the "Garland" irradiation facility of the MIR reactor two variants of the fuel elements design have been developed. These fuel elements are identical in external geometrical parameters. The distinction between them consists only in the structure of the fuel core. In both variants of the fuel element design the fuel cladding is made from the E-110 zirconium alloy, hermetically sealed on the ends closers from the same alloy. For the № 1 variant in the core on an axis of the fuel element it is placed displacer from the E-110 alloy as a hollow pipe of the crosswise structure hermetically sealed on the ends by welding. The internal volume between the fuel cladding and displacer is filled with aluminum granules between which the  $\text{PuO}_2$  particles are located. Other space is filled with aluminum silumin alloy. For the № 2 variant the separator from an aluminum alloy is inserted inside the fuel cladding, in which four vertical slots wire mini fuel elements are located of. These mini fuel elements were manufactured from tube with 2.4 mm in diameter by the welding hermetically sealed from two sides. The space formed between a separator, mini fuel element and an internal surface of fuel cladding is filled with an aluminum alloy.

During carrying-out of work, the development and manufacturing several kinds of the manufacturing equipment was carried out, in particular, vibration compaction device, furnaces for fuel elements infill with silumin, the technology of the fuel elements hermetic sealing by welding was perfected.

**Theoretical and Experimental Examination Dependencies of Incubation Swelling Doses of 16Cr-15Ni-2Mo-Ti-2Mn-Si (ChS-68) Steel on Neutron Irradiation Temperature**

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In order to illustrate radiation-induced point defect fluxes to different sinks the method of statistical thermodynamic of solid body describing both vacancy and interstitial migration as a probable process of their random walk in a crystal was used. A quantitative model of point defect migration was developed according to the assumption that a probability of point defect jumping from one position with  $U_1$  energy to the neighbor position with  $U_2$  energy is proportional to  $\exp((U_1 - U_2) / kT)$ , where  $T$  is temperature taken into account with normalizing conditions. Expressions for vacancies and interstitials average diffusion time to grain boundaries, dislocations, into vacancy voids, and their recombination were derived by this model. Using only point defect migration energies and their binding energies with different sinks without diffusion coefficients permitted to derive expressions for vacancy and interstitial concentration change under average-temperature irradiation of fcc-metals without additional presumptions. At that, average-temperature neutron irradiation means irradiation under temperatures when vacancies and interstitials have mobility enough to dissociate vacancy clusters for a short time as well as to ensure concentrations of these defects higher than thermal balance values.

Expressions for both point defect fluxes to different sinks and vacancy and interstitial concentration change in crystal matrix were derived for austenitic steels. Conditions for neutron radiation-induced origin growth of vacancy voids have been formulated; expressions for critical void radius depending on steel structure characteristics, temperature, radiation damage rate, and irradiation duration were shown. Calculation temperature irradiation dependencies on incubation dose derived for ChS-68 steel were verified with experimental data.

**Concentration Change of Packing Defects in ChS-68 CW Steel under Neutron Irradiation and Its Connection with Radiation Swelling**

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At present ChS-68 steel is a standard material for fuel element cladding of the BN-600 reactor. Its final treatment is cold working to ~20 % realized mainly by twinning and it is applied for radiation swelling depression. Long-lived examination experience of “INM” and “BNPP” has shown that fuel element claddings included into one and the same fuel assembly and operated under identical conditions in the BN-600 have swelling differed two or several times between themselves. One of these reasons is considered to be probable scattering of degree of cold working at claddings final treatment. Another probable reason is different Boron content dissolved in crystal matrix of this steel. Boron also is applied for swelling depression being itself the centre of point defects as well as the element blocking growth of interstitial dislocation loops, so it causes in loop formation containing packing defect. There is very little amount of structural examination results of such effects on a radiation swelling value.

The paper presents the results of X-ray and electron- microscopy examinations of unirradiated specimens of the ChS-68 steel subjected to 0, 5, 10, 15, 20, 25 % of cold working and its effect on packing defect concentration. Concentration of packing defect was determined by test results on claddings irradiated to high damage dose in the BN-600 and had different swelling whose change was analyzed. Correlation between packing defect concentration and swelling of the ChS-68 steel was found out.

#### **Correlation between Physical and Mechanical Properties Changes of Austenitic Steel ChS-68 under High Dose Irradiation**

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Complex definition of physical and mechanical properties was performed on specimens of austenitic stainless steel ChS-68 (16Cr-15Ni-2Mo-Ti-2Mn-Si) after its long-term operation as fuel element claddings in the fast neutron reactor BN-600. Areas irradiated under temperatures from 400 °C to 590 °C were examined. Peak damage dose of the claddings achieved 78 dpa at ~530 °C. Changes of physical and mechanical properties determined on one and the same cladding areas after irradiation are correlated between themselves as well as with material radiation swelling. One can see that neutron radiation-induced density reduction of steel on 12 % is accompanied with reduction of both elasticity characteristic on 15-20 % and ultimate strength on 50-70 % as well as increasing of material electrical resistivity on 6-7 % with respect to origin values. Peak changes of physical and mechanical characteristics as well as peak material swelling of tested fuel elements claddings are occurred slightly lower then the core area at ~480 °C and ~70 dpa.

At present, the literature about a mechanism of physical and mechanical properties changes of materials under irradiation is insufficient; it is poorly coordinated, and sometimes contradictory. That is why results derived in the paper can be used not only in design and

testing calculations on strength but also under simulation of solid body matrix evolution as well as structure and properties of radiation-induced structural steels.

### Amisotropic Swelling of Beryllium after Low-Temperature High Dose Irradiation

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The investigation results of influence of neutron irradiation in the SM reactor at the temperatures of 70 and 200 °C up to neutron fluence of  $(1.3-14.2) \cdot 10^{22} \text{ cm}^{-2}$  ( $E > 0.1 \text{ MeV}$ ) on swelling and microstructure of the TE-56 beryllium grade are presented.

It is known, that during hot extrusion of beryllium the basal planes are oriented mainly along the extrusion axis. It was revealed, that change of the geometrical sizes of beryllium samples - cylinders under neutron irradiation is anisotropic. In particular, change of diameter or height at the samples which have been cut out along and across of extrusion axis are various. In the work [1] it has been shown, that after an irradiation of beryllium monocrystals in the IVV-2M reactor at temperature of 80 K up to neutron fluence  $5 \cdot 10^{19} \text{ cm}^{-2}$  ( $E > 1.15 \text{ MeV}$ ) occurs anisotropic increase in the geometrical sizes of samples too. The ratio of relative length changes of samples oriented across and along the "c" axis of beryllium crystal lattice makes 1,5, which is practically equal in value the similar ratio  $(\alpha_{\perp c} / \alpha_{\parallel c})_{80K} \cong 1,5$  describing the degree of anisotropy of thermal expansion.

The results of TEM investigations shows, that the formation of dislocation loops in the (0001) basal and  $((10\bar{1}0)$  and  $(11\bar{2}0))$  prismatic planes under neutron irradiation in beryllium take place. The dislocation loops placed in the basal planes are to vacancy type, the loops placed in the prismatic planes are to interstitial type. After neutron irradiation at the temperature of 200 °C up to neutron fluence  $13 \cdot 10^{22} \text{ cm}^{-2}$  ( $E > 0.1 \text{ MeV}$ ) the gas bubbles were shown in the structure of beryllium. Average size of the grain boundaries and intragranular bubbles is 6.8 nm and 4.3 nm, correspondingly.

We made an assumption that anisotropic swelling of beryllium can be sequent of two processes superposition - swelling, caused by formation of gas products of nuclear reactions (helium and tritium) in a lattice of beryllium and the radiation growth due to directed condensation of defects results in settling of vacancies on basal planes, and interstitials on prismatic planes.

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### **Sensitivity to Delay Hydride Cracking of Material of E125 Alloy after Its Long-Term Operation as Tubs TC and CPSC**

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Delay hydride cracking (DHC) seems to be practically unique and significant mechanism of a growth of homothetic-cracking defects in Zr-alloy tube channels of water-coolant nuclear power reactors.

Now there are practically no experimental data on sensitivity to DHC of material of fuel channel tubes (FC) and channels of control and protective system (CPSC) of E125 alloy after there long-term operation in the RBMK-1000 reactor. Therefore it is important to estimate an influence of long-term operation of the alloy on change of its sensitivity to DHC.

DHC tests were performed on the special in-pile installation on compact specimens with a grown crack which were cut from tubes FC and CPSC after there long-term operation in the RBMK-1000 reactor. After these tests fracture toughness tests with determination of the critical coefficients of stress intensities ( $K_c$ ), fractographic and metallographic examinations were carried out.

The examinations have shown that DHC is realized at the temperature range of 140 – 200 °C. Incubation period up to crack movement onset continues from 15 minutes to 18 hours and it reduces with the following factors as increasing of test temperature, the coefficients of stress intensities, and hydrogen content. Movement crack rate is varied from 0.6 to 3.2  $\mu\text{m}/\text{hour}$ . It increases with the coefficients of stress intensities growth and achieves its maximum in the temperature range of 160 – 170 °C. Increasing of the hydrogen content leads to a shift of the upper temperature boundary of DHC realization. Additionally to a damage mechanism over hydrides a mechanism of toughness damage over bridges between hydrides is engaged under higher stresses ( $K/K_c > 0.3$ ).

Crack growth on the DHC mechanism was not observed in the material of tubes CPSC after long-term operation under temperature and stress ranges and long-term tests. Crack movement on shift mechanism causes by transverse strains is observed under higher stresses

### **Influence of Post-Irradiation Annealing on Deformation Localization, Structure and Properties of 12Cr18Ni10Ti Steel - a Aaterial of the BN-350 Spent Fuel Assembly Hexagonal Shroud**

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Within the framework of complex researches of structural materials of the BN-350 spent fuel assemblies the study of thermal annealing (450÷1050°C, 1 hour) influence on structure and physical and mechanical properties of the 12Cr18Ni10Ti austenitic stainless steel irradiated at 370 °C up to the damage of 55.4 dpa.

The experimental investigations of non-irradiated and neutron irradiated steel after thermal treatment were executed using mechanical tests with simultaneous control of microspecimen size (20×2×0.35 mm), optical metallography, measurement density, cartography of local magnetization and microhardness.

It was established that after annealing at 450°C the plasticity of 12Cr18Ni10Ti irradiated steel, was rather high (about 27 %). That is can be connected with the process of formation and movement of localized deformation bands Luderse-Chernov type accompanied with martensitic  $\gamma \rightarrow \alpha'$  transformation.

The comparative analysis of ferromagnetic  $\alpha$ -phase distribution, microhardness and change in geometry of a working area off specimens allowed reveal localized zones of plastic yielding in the deformed specimens.

In the result of isochronal annealing the density of non-irradiated steel did not change practically, however the density of irradiated steel increased with the growth of test temperature and became equal to that of non-irradiated specimen at 1050°C.

The influence of neutron irradiation and subsequent annealing on localization of plastic deformation accompanied with  $\gamma \rightarrow \alpha'$  transformation, changes in both structure and mechanical properties of the 12Cr18Ni10Ti steel is analyzed.

#### **Some peculiarities of martensitic transformations at deformation and annealing of steel 12Cr18Ni10Ti irradiated with neutrons and alpha-particles**

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Martensitic transformation in deformed and phase work-hardened non-irradiated steels has been extensively studied. At the same time there is a lack of investigations relevant to  $\gamma \leftrightarrow \alpha'$  transitions in austenitic steels subjected to irradiation with high-energy particles. Nevertheless, these works for reactor material science are undoubtedly topical, as stainless steel is the main structural material for fast reactors and thermonuclear facilities.

This work performs results of direct and back martensitic transformation in austenitic steel with similar composition with various content of nickel (9-10%) after reactor and cyclotron irradiation.

Plane samples with a work part of 10×3.5×0.3 mm were annealed at temperature of 1050°C within 30 min and dropped into water. A part of heat-treated samples was irradiated through at isochronous cyclotron U-150M INP NNC RK with alpha-particles of 50 MeV of a fluence 2·10<sup>17</sup> part/cm<sup>2</sup>. Another part of samples was irradiated in active zone of the reactor WWR-K up to fluence 2·10<sup>20</sup> n/cm<sup>2</sup> at temperature no more than 100 °C. There was tested expansion of non-irradiated and irradiated steel samples at room temperature at a universal testing machine “Instron 1195” as well as a micro-tearing machine combined with a calorimeter. In the latter case there were registered thermal effects connected with processes



of structural and phase transformations in steel at deformation. During expansion or after definite deformation by a flux-gate F-1053 there was determined the quantity of ferromagnetic  $\alpha'$ -phase in a neck of a deformed sample and in sites at some distance from it.

It was revealed that formation and accumulation of martensitic  $\alpha'$ -phase being induced with deformation pass more intensively in a neck than far from it both in irradiated and non-irradiated samples. It is determined that accumulation rate of  $\alpha'$ -phase is higher in irradiated steel than in non-irradiated one. It was revealed that release of extra heat is connected with formation of  $\alpha'$ -phase.

Isothermal annealing of samples of stainless nickel-chromium alloys have shown that irrespective of initial value of a magnetic  $\alpha'$ -phase its relative quantity decreases equally after annealing within 290-1073K. At temperature of 1073K the back martensitic  $\gamma \leftrightarrow \alpha'$  transformation is stopped.

In order to change the quantity of the martensitic  $\alpha'$ -phase depending on annealing temperature the presence of 4 temperature areas, in which  $\alpha'$ -phase decreases with different velocity, is typical.

#### **The effect of neutron irradiation of a high damage-dose on the electrochemical properties of ferrite–martensite steel 0.12C-13Cr-2Mo-Ni-V-B**

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The radiation resistant ferrite-martensite steel 0.12C-13Cr-2Mo-Ni-V-B is used as the main material of case tubes of fuel element assemblies in the BN-600 reactor. This steel is also a candidate for fuel cladding material for the BN-800 reactor. Though the steel is characterized by a high radiation resistance it reveals a higher speed of general and pitting corrosion observed in the spent fuel element assemblies stored in decay pool water. It is known that corrosion properties of metal materials are related to their electro-chemical characteristics.

This paper presents the results of the study on the affect of temperature and damage dose of fast neutrons on the electrochemical properties of 0.12C-13Cr-2Mo-Ni-V-B stored in the water with pH = 7.0. The steel was examined in the initial condition and after the exposure to neutron damage doses from ~ 4 to ~ 72 dpa. in the temperature range from 380 to 570 °C, which are the operation parameters of fuel claddings in the BN-600 reactor.

### **Influence of Neutron Irradiation Conditions on Characteristics of Defect Structure of E125 Alloy**

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An influence of radiation conditions of RBMK reactors such as temperature, flux density of fast neutrons on structural state of material of channel tubes (FC, CPSC) after their operation within 20 years was analyzed here. Dynamic balance state was shown to ensue approximately in the irradiated material after its operation within 10 years when microstructure parameters estimated by both X-ray analyses and analytical electronic microscopy are kept steady state or changed weakly.

Annealing (up to 580 °C) of irradiated specimens was performed to reveal how radiation-induced microstructure of the material of channel tubes is changed under temperature higher than operation temperatures.

Examinations of electrical-physical properties have shown that reduction of specific resistance occurs due to radiation defect annealing under heating of the irradiated alloy higher than operation temperatures. Isochronous heating experiment on specimens of FC tubs allows estimation of activation energy of diffusion processes at temperature around 400 °C that is close to vacancy migration energy in zirconium.

Estimation of parameters of hyperfine structure has shown that annealing led to significant growth of coherent scattering areas that is agreed with electron microscopy data on transformation of dislocation structure of the irradiated material after annealing. Lattice distortions were estimated by the method of harmonic analyses of forms of x-ray lines. Lattice distortions were estimated by the method of harmonic analyses of a form of x-ray lines. Microdistortions were determined to reduce in basical and prismatic faces and to increase distinctly in pyramidal faces. It causes in anisotropy behaviour of radiation-induced defects under annealing.

Anisotropy of the thermal coefficient of liner expansion (TCLE) in the grain-oriented material (channel tubes) was examined in the longitudinal and tangential directions to the rolling axis. Nonmonotonic was revealed on the temperature dependence of TCLE in the tangential direction. It disappears at duplicate measurements hence it causes in annealing of radiation-induced defects. Such defects are the vacancies and vacancy complexes since instead of specimen elongation under temperature increasing a specimen's shrinkage is observed in the measurement direction. Knowledge of Zr-tube texture allows presuming that vacancies in the tangential direction are to annihilate on sinks placed mainly in basical faces, in particular, on radiation-induced dislocations that are disappeared under annealing.

### **Thermal–Radiation Effect on Physical and Mechanical Properties of 13Cr-2Mo-Ni-V-B Steel**

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Claddings of 13Cr-2Mo-Ni-V-B steel (EP-450) are subjected to complex influence of neutron flux and higher temperature from 370 to 650 °C reactor conditions.

Structure-phase material change occurs during operation, it causes in strength and plasticity changes in different fuel element areas. The steel shows well-resistance to radiation swelling and exhibits plasticity loss in areas subjected to operation temperatures of 400-500 °C.

To study complex thermal-radiation effect on material physical and mechanical properties such as electrical resistance, modulus of elongation, and short-term mechanical properties the steel were determined after its irradiation in the BN-600 reactor. Correlation is found out to be between electrical resistance change and strength and elasticity properties of material. Increasing of electrical resistance is observed under reduction of strength and elasticity properties.

Changes of these properties were observed not only in the core area but also in the gas cavity of a fuel element where significant electrical resistance change was recorded. Cladding temperature is practically constant in the gas cavity, and it corresponds to the input coolant temperature of ~ 370 °C of a Fuel Assembly. Neutron flux here is by an order of magnitude lower than in the core. Significant property changes of EP-450 steel are assumed to be caused by neutron irradiation as well as long-term thermal effect on material (up to 10 000 hours) during which, due to material ageing processes, physical and mechanical properties changes occur.

### **Structural Materials for Fusion Reactors**

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A long term solution to problems of energy production, green house gas generation, and pollution control may rest with controlled nuclear fusion reactors. Candidate structural materials for such reactors include low activation ferritic steels. Understanding and eliminating deleterious irradiation effects in these materials is the goal of these experiments in this collaboration using the ANL facility.

In recent experiments on ferritic alloys we have recently found a significant difference with alloy composition in the microstructural response to irradiation, which corresponds to a bulk mechanical property change at a similar composition. In collaboration between the Department of Materials at the University of Oxford and the Materials Science Division at

Argonne National Laboratory, experiments which employ the unique transmission electron microscope and in situ ion irradiation user facility at ANL were performed on a series of Fe-Cr alloys. Enhanced nanometer-sized defect formation with Cr concentrations up to 11 % have been found and correlated with a decrease in mechanical hardening and embrittlement in similar alloys.

#### **Vanadium-Based Radiation-Resistant Alloys**

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A series of systematic investigations has shown that the most vanadium-based alloys are the most promising materials to be used as construction materials for nuclear reactors. Complex analysis of the requirements and conditions of operation of perspective fast reactors shows that such requirements may be met by vanadium-based ferritic stainless steel-clad alloys only, in which heat resistance is ensured by vanadium alloys, and compatibility with fuel and liquid-metal heat carrier, by ferritic stainless steel.

#### **Use and Improvement of E635 Alloy as applied to Fuel Cladding and FA structural Components of Water Cooled Reactors**

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The designed in Russia zirconium alloy E635 features high irradiation resistance (creep and growth), strength, and resistance to general and nodular corrosion. These properties of E635 alloy allowed it to be recommended as a material for fuel claddings and FA (fuel assemblies) structure components of VVER and RBMK. Currently E635 alloy is used to fabricate guide thimbles (GT), central tubes (CT) and rigid frame angles in FAA as well as GT and CT in FA-2 of VVER-1000. The alloy has been also adequately tried out as fuel claddings. The paper points out the most important stages of mastering and introducing E635 alloy into FA designs, reviews the favourable aspects of applying the alloy as a material for structure components of VVER-1000 core and presents the main results of PIE of fuel claddings, tubes of guide thimbles, CT and angles fabricated from this alloy. As applied to promising fuel cycles the directions of efforts are presented aimed at upgrading E635 alloy and items thereof to increase their operating reliability.

### **Experimental Study of Nickel-Based Alloys Corrosion Resistance in Fluoride Melts**

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The paper presents the methods and results of tests of nickel alloys in isothermal conditions with melt circulation realized in a thermo-convection loop. Melt temperature in the loop hot zone was ~700 °C, and ~600 °C in the cold one. The melt continuously circulated in the loop, inside which the investigated samples were placed, at a speed of ~5 cm/s during 1200 hours. In the process of tests, the circulating melt redox potential was measured at least once a day, and samples were regularly taken for chemical analysis of salt composition.

### **The Influence of Cold-work Level on the Irradiation Creep of AISI 316 Stainless Steel Irradiated as Pressurized Tubes in FFTF and EBR-II Fast Reactors**

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Development of dimensional change correlations for both fission and fusion service requires either a very large amount of relevant data covering all potential material and environmental conditions, or application of derived insight on broadly-applicable principles to a more limited amount of data. Previous studies have shown that irradiation creep is much less directly responsive to material and environmental variables, other than being directly dependent on the stress level, with most of its sensitivities at higher exposure levels arising directly and proportionately from the sensitivities of void swelling.

In a continuing attempt to better define the sensitivities of irradiation creep before swelling and its relationship to void swelling after it commences, previously unpublished data sets are being examined to test the generality of earlier proposed relationships. In the current effort the focus is on the dependence of irradiation creep on cold-work level.

In an earlier study on titanium-modified 316 stainless steel irradiated in FFTF it was shown that at relatively low irradiation temperatures (~400 °C) irradiation creep was essentially independent of cold-work level. At higher temperatures this trend was maintained until thermal creep began to assert itself, whereupon the stored energy at higher cold-work levels caused earlier breakdowns in creep resistance and produced non-linear stress dependence.

In order to test the generality of this conclusion the results of four earlier unpublished experiments conducted in the EBR-II fast reactor on pressurized tubes of AISI 316 stainless steel have been analyzed. While the onset of swelling is dependent on the cold-work level at

400, 425 and 480 °C, the post-transient irradiation creep is not dependent on cold-work. If the tube heats up and reaches pressures on reactor start-up that generate stresses above the yield stress of unirradiated steel, then plastic strains occur, but the post-transient strain rate is identical to that associated with material that did not exceed the yield stress on start-up.

At temperatures above ~540°C the influence of thermal creep and stored energy begin to assert themselves, with creep rates accelerating with increasing cold-work levels and becoming non-linear with stress level.

Based on these and more fragmentary data sources, it appears safe to assume that post-transient irradiation creep can be considered to be relatively independent of cold-work level, especially for lower temperature applications such as those experienced in light water reactors or anticipated for ITER.

#### **On Correlation of Radiation-Induced Sensitization in Austenitic Stainless Steel 12X18H9T with Chromium Content at Grain Boundaries**

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For the present the depletion of grain boundaries with chromium due to radiation-induced segregation is considered as a main cause of radiation-induced sensitization as well as of irradiation assisted stress corrosion cracking in austenitic stainless steels.

In the present work samples of austenitic stainless steel 12X18H9T non-irradiated and irradiated in the BR-10 fast reactor with doses 0,64, 10 and 33,1 dpa (NRT) at 350, 350 and 400°C respectively were tested for a susceptibility to intergranular corrosion (IGC) through the Strauss corrosion tests. Maximal depths of attack were 0, 320 and 640 µm respectively. After the annealing for 2 h at 600°C of the steel irradiated to highest dose maximal depth of attack was 180 µm. Modeling of the chromium segregation near grain boundaries during irradiation and the chromium desegregation during annealing were carried out. The microstructure of the steel 12X18H9T in initial condition, after neutron irradiation and after annealing was studied. It was found that grain boundaries in the steel irradiated with dose of 33.1 dpa at 400°C are decorated with almost continuous layer of precipitates, mainly of radiation-induced G-phase which don't fully dissolve during annealing performed. It is shown that peculiarities of IGC observed after irradiation and annealing are caused not only by Cr content at grain boundaries, but also by the presence of precipitates on these boundaries.

A comparison is made with the results obtained on radiation-induced sensitization in the 12X18H9T steel irradiated in the BN-350 fast reactor with doses up to 30 dpa at lower irradiation temperature range 280 to 330°C.

This work was supported by the Russian Foundation for Basic Research under Project № 04-02-17278.

### **Kinetics and Thermodynamics of Processes of Radiation-Enhanced Diffusion in System in Materials for Nuclear Power Engineering**

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Features and regularities of the processes of radiation-enhanced self-diffusion in alloys based on Zr, Ti and Pu were investigated. Evaluation of characteristics of radiation-enhanced self-diffusion was performed with the use of the originally proposed modified Darken equation. The results of calculations are in good agreement with the experimental and theoretical data on radiation-enhanced self-diffusion in Zr, Ti and Pu.

### **Technological Problems of Perfection of Structural Materials of a Fast High-Power Reactor**

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The achievement of deep burn-up of fuel is one of the most important conditions of increase of economic parameters of commercial reactors. Solution of this problem is hindered by a number of factors. One of the most limitative factors for a burn-up increase is a lack of sufficiently reliable structural materials of fuel subassemblies functional under high damage doses. The development and improvement of materials are mainly aimed at achieving the highest practical damage doses by cladding and wrapper materials of fuel subassemblies under the parameters as high as required by the operation of a commercial reactor.

In the report the review of results of post-irradiation examination of the service properties of structural materials of reactor БН-600 is done. On the basis of the analysis of the factors supporting the serviceability of fuel subassemblies, the basic problems of an increase of radiation resistance of cladding materials are considered.

### **Radiation Defects Annealing after Low-Temperature Electron Bombardment of Uranium**

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A specific structure of actinoids, including uranium, their polymorphous crystalline structures, a high sensitivity of phase modifications to alloying additions, a large anisotropy of their physical-mechanical properties and oxidation susceptibility – all these factors, on the one hand, make experimental studies very complicated and, on the other hand, are very significant for the physics of metals and alloys. A complex internal structure and properties of uranium can influence the behavior of point defects. The effect of neutron and ion

irradiation on specific radiation phenomena in uranium (radiation growth, gas swelling, embrittlement, etc.) has been long and comprehensively studied since 1950s. However, the behavior of point defects has received little study and is poorly understood.

The method of residual resistivity was used in this study dealing with annealing of radiation defects in uranium after it was exposed to 5-MeV electrons at 70 K. The isochronal annealing was realized at an average rate of 1 K/min in purified helium. The test samples represented uranium after slow cooling from the alpha-phase. Some of these samples were subject to plastic deformation at room temperature ( $\varepsilon = 12-40\%$ ). Other samples were quenched from the beta-phase at a rate of about 100 K/s.

The annealing spectra of radiation defects had a complex substructure. They contained narrow annealing peaks at about 77 K, 125 K, 170 K and 300 K, and complex peaks at about 220 K and 450 K.

This substructure had some regular features. As the degree of deformation increased, the amplitude of the peak at 77 K was enhanced and that of the peak at 170 K diminished. The other peaks exhibited little, if any, dependence on the degree of deformation. The amplitudes of the first three peaks in the spectrum of uranium quenched from the beta-phase were much smaller than they were in annealing spectra of the other samples. Oppositely, the amplitudes of the peaks at 220 K and 300 K were much larger.

Mechanisms of annealing of radiation defects were discussed and regular features of variations in the substructure of uranium annealing spectra were determined.

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### **Fatigue Behaviour of Ferritic-Martensitic Steels in a 590 MeV Proton Beam**

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Structural materials used in the first wall of fusion reactors will be stressed and deformed under simultaneous bombardment with high energy neutrons. In most investigations, material specimens are first statically irradiated and then only tested after irradiation, in a convenient laboratory. This usual method does not consider the mutual effects of irradiation and plastic deformation. In this work we present and discuss the results obtained in the past years in three different ferritic-martensitic steels tested *in situ* and compare them to those obtained in unirradiated and post irradiation tested material. The experiments were conducted into a 590 MeV proton beam line, using a specially dedicated *in situ* device [1]. In steels, the protons produce helium and hydrogen by spallation reactions at levels of 195 and 956 appm per dpa, respectively. The irradiation structure and the corresponding mechanical effects are similar to the ones observed after neutron irradiation [2].

The materials tested to doses up to 0.2 dpa were three candidate materials of the European fusion programme, the Manet II, the F82H and the Eurofer 97. The main



difference observed in terms of the mechanical behaviour was the clear reduction of the irradiation hardening. The observed irradiation hardening appeared to be controlled by the ratio of the dose rate and the strain rate. The fatigue lives of the in-beam specimens were shorter as compared to the post-irradiation tested and unirradiated specimens. Specimens tested with a tensile hold time showed a significant life reduction. The three investigated materials have a similar chemical analysis and microstructure. Nevertheless the behaviour of the Manet II steel was found to be different because the fatigue endurance was showing a temperature dependency. The steel was also very sensitive to previous cyclic plastic deformation imposed to the specimen before the irradiation. In this work, the possible mechanisms leading to the observed embrittlement will be reviewed and the various aspects of the microstructure observed in transmission electron microscopy, in the different material conditions will be described.

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### **Production of Oxide Dispersion Strengthened Reactor Austenitic Alloys Based on Fe-Ni Invar Doped with Ti and Zr**

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The study has been concerned with development of oxide dispersion strengthened (ODS) model austenitic alloys for fast reactors. The Mössbauer spectroscopy, the X-ray diffraction analysis and the TEM analysis were used to observe specific features of the mechanochemical dissolution of unstable metal oxides, which was followed by the formation of metal solid solutions and subsequent precipitation of secondary nanoscale oxides. A new approach, which was proposed [1] for synthesis of ODS alloys, helped realizing deformation-induced phase transitions in {Fe-Ni-Me (Me = Ti, Zr) + Me<sub>x</sub>O<sub>y</sub> (Me = Fe, Cu)} mixtures during compression shearing in Bridgman anvils and grinding in planetary ball mills.

It was shown that intensive plastic deformation (IPD) with the reduction  $\varepsilon = 7$  and subsequent annealing of the "alloy – iron oxide Fe<sub>2</sub>O<sub>3</sub>" systems led to the formation of an ODS alloy having a nanocrystalline structure saturated with fine inclusions of Fe, Ti and Zr oxides, which were products of mechanochemical reactions. If the copper oxide CuO was used, IPD ( $\varepsilon = 4..7$ ) caused a dynamic dissolution of the metal oxide and led to the formation of secondary iron oxides (Fe<sub>3-y</sub>O<sub>4</sub> and Fe<sub>1-x</sub>O), and titanium and zirconium nanooxides.

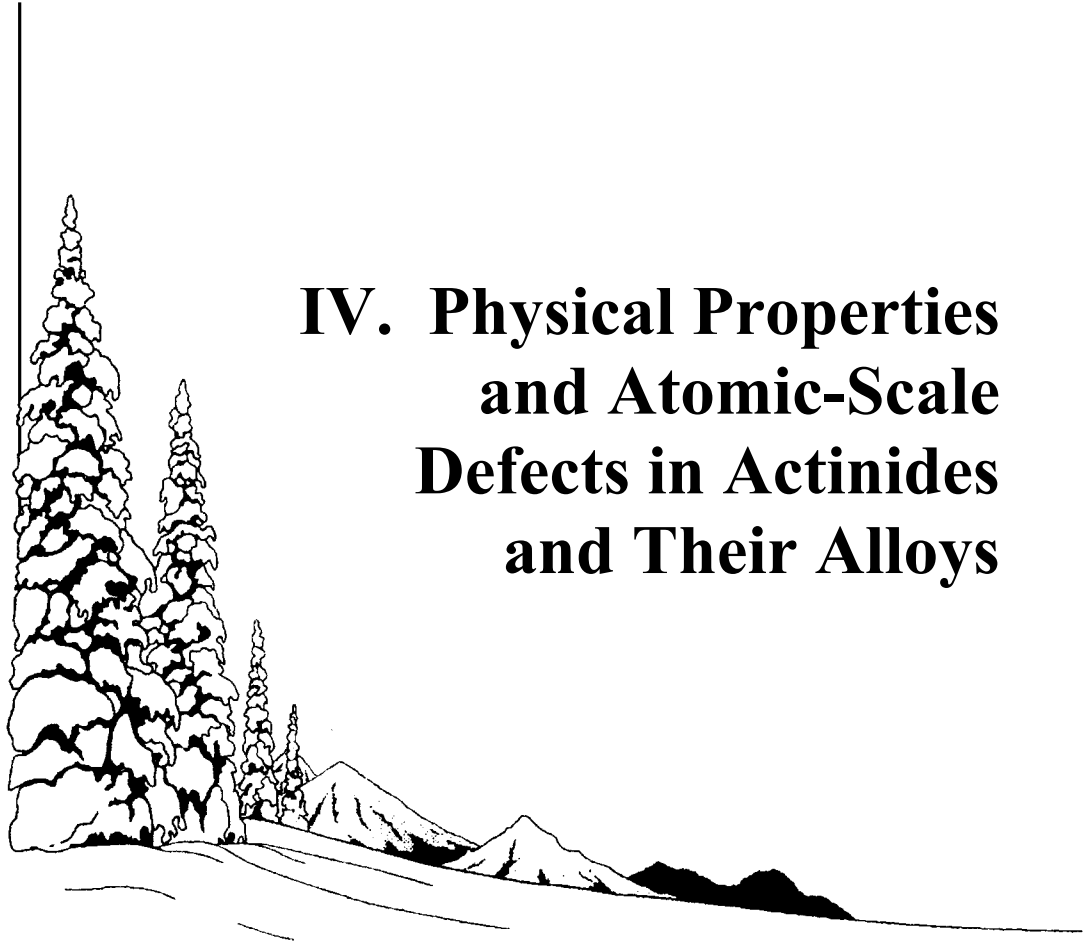
The process kinetics was governed by the degree of deformation. As the degree of deformation increased, the formation of oxides of the third element was promoted, while iron oxides were formed less intensively because of oxidation-reduction reactions, which caused migration of oxygen to more reactive elements, that is, Ti and Zr. The X-ray and electron

microscopic analyses confirmed the presence of particles of titanium and zirconium nanooxides in the structure of the synthesized alloys.

The study was supported by grants from the RAS Presidium for nanoscale materials (No. 20), the fund of the "Magnitogorsk Integrated Iron & Steel Works and Ausfer" JSC (No. 29-05-03), and RFBR (No. 04-02-16053).

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## **IV. Physical Properties and Atomic-Scale Defects in Actinides and Their Alloys**

The subject of this Section was prompted by the need for a systematic and comprehensive research into actinides and their alloys, fissionable alloys including, whose properties are determined primarily by the features of their electronic structure and self-irradiation-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 selected for this Section are dedicated to study of the properties of fissionable actinides and their model analogues 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 ageing, radiation stability, and response to external dynamic and shock actions.



## **AnFe<sub>x</sub>Al<sub>12-x</sub> (A=U, Np, Pu; 4<x<7): A Case Study of Magnetism in Actinide Intermetallic Compounds**

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The magnetic and other electronic properties of light-actinide intermetallic compounds are characterized by a partially occupation of the 5*f*-electron states. In contrast with the lanthanides, where the 4*f*-electrons are usually deeply implanted in the core of the atoms, these electrons are mainly delocalized. The magnetic properties of light-actinide intermetallic compounds are also determined by a strong spin-orbit interaction, which provides a powerful connection between the direction of the electron moments and the crystal structure. This leads to a large diversity of magnetic behaviours, as the nature of the light-actinide 5*f* electrons can be strongly affected by external variables like crystal structure type, nature of the nearest-neighbour atoms, magnetic field, pressure, etc.

U-Fe-Al phase diagram investigations have shown that there is a broad solubility range in the ThMn<sub>12</sub>-type structure, which extends from UFe<sub>3</sub>Al<sub>9</sub> to UFe<sub>7</sub>Al<sub>5</sub> at 850°C [1]. Moreover, phase relation studies indicated congruent melting between UFe<sub>3.8</sub>Al<sub>8.2</sub> and UFe<sub>5.8</sub>Al<sub>6.2</sub> compositions, which permits the growth of large single crystals suitable for a deeper characterization of their physical properties. The U atom can also be substituted by another actinide, like Np or Pu, and the coupling between the Fe and 5*f*-element magnetic sublattices is expected to change due to a higher localisation of the 5*f*-electrons in the heavier actinides. This can lead to a significant lower magnetic interaction and to different magnetic structures. The possibility of a considerable change of the composition in this family of compounds allows a deep study of the influence of the actinide and the neighbour elements on their magnetism, making this series a case study of magnetism in actinide intermetallic compounds.

This communication reports results on a systematic study of the UFe<sub>x</sub>Al<sub>12-x</sub> and (U,Np,Pu)Fe<sub>4</sub>Al<sub>8</sub> ternary compositions, with a detailed investigation of the preparation conditions and their influence on the phase homogeneity, crystal site occupancy and magnetic properties. Crystal structure refinements show that the variation of the UFe<sub>x</sub>Al<sub>12-x</sub> cell parameters with composition gives a clear hint on the different Fe occupations. Magnetization, electrical resistivity and neutron diffraction measurements, together with band structure calculations, point to complex magnetic structures of the UFe<sub>x</sub>Al<sub>12-x</sub> and (U,Np,Pu)Fe<sub>4</sub>Al<sub>8</sub> compounds, with a strong coupling between the actinide and Fe [2,3].

The dramatic influence of the final composition and crystal structure on the magnetic properties of these ternary compounds is emphasized, demonstrating the extra care needed in the preparation conditions for this type of materials.

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## Thermoelectric Energy Converters Based on Anomalous Rare Earth Materials

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Strongly correlated electron systems demonstrate dramatic anomalies of physical properties reflecting a very peculiar type of the ground state. In particular, the Seebeck coefficient of rare earth intermetallics is highest among metals, which makes them promising materials for the thermoelectric (TE) applications [1]. Cerium and Ytterbium-based compounds are capable of direct energy conversion from heat to electricity and vice versa. The current market niche for the TE materials includes both refrigerators and power generators of small and medium power. The main goal of the present work was to improve the power factor of the TE devices compared to the best semiconductor-based ones and keep rather high conversion efficiency. In order to increase the power factor we explored heterogeneous systems. One component of the composite TE devices is an anomalous rare earth system; another component is a simple metal. We report on the measurements of the Seebeck coefficient, thermal conductivity and electrical resistivity in promising TE material. The performance of the rare-earth based composites is discussed versus the best semiconductor devices.

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## Effect of Chemical and External Pressure on the Structure of Intermetallic Compound CeNi

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The study of mechanisms of the transitions between different structural modifications for the materials with almost empty or almost filled  $f_{5/2}$ -electronic configurations may provide a new insight into the origin of their ground-state properties, especially in the case if the structural transitions involve volume discontinuity driven by electron correlation effects. The intermediate-valence compound CeNi exhibits anomalous behavior of many physical properties including the first-order structural phase transition with volume discontinuity [1,2] and well-defined ground-state coherent effects in the magnetic response function observed by the inelastic neutron scattering technique [3]. Thus, CeNi is an attractive model system to investigate the influence of the coherent effects on the mechanism of the volume-collapse phase transitions. However, the structure of the high pressure phase remains unknown and

the  $P$ - $T$  phase diagram is established only at relatively low pressures ( $< 0.8$  GPa) and temperatures ( $< 150$  K).

The aim of the present work is to determine the structure of the high-pressure phase of CeNi. We report the results of neutron powder diffraction study of structural modifications in CeNi induced by either chemical (negative for  $Ce_{1-x}La_xNi$ ,  $0 < x < 1$ , and positive for  $Ce_{1-x}Lu_xNi$ ,  $0 < x < 0.4$ ) or external pressure up to 5 GPa. The results obtained are used for electronic structure calculations of CeNi in terms of the spin-polarized relativistic density functional theory in generalized gradient approximation.

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### Neutron Spectroscopy of Crystal Field Effects at Amorphization: Features of Local Structure and Thermodynamics

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On an example of stoichiometric intermetallic compound  $PrNi_5$  in amorphous and structurally-ordered states, the interrelation between microscopic properties,  $f$ -electrons excitation spectrum and microstructure was investigated. Thermodynamic, diffraction and neutron-spectroscopic experiments on a series of samples were carried out. The magnetic dynamic response spectra corresponding to different states of the intermetallic compound were analyzed. A phenomenological model for describing the structure of amorphous intermetallic compound and non-contradictory substantiation of the whole set of experimental data were proposed.

### Magnetic Properties of the Uranium Compounds

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In 1952, W.Trzebiatowski et al. [1] reported that the uranium trihydride and trideuteride are ferromagnetic below about 180 K. This discovery was an absolute surprise because the uranium metal is nonmagnetic (paramagnetic). Since this event, more than few

hundred uranium intermetallic and semimetallic compounds appeared to be magnetically ordered (for Ref. see e.g. [2]). Their magnetic and related properties have been examined by several complementary methods; however one cannot establish firmly if the localized or itinerant  $5f$  electrons are responsible for mysterious behavior of these materials. Additional complication arises from the interaction of the  $5f$  electrons with the band states resulting in strong hybridization, heavy fermion, Kondo lattice or mixed valence state.

Below we are going to review some most intriguing features of the uranium compounds investigated partly in our laboratories. We will describe complicated magnetic phase diagram of the UAs-USe system, then the coexistence of ferromagnetism and superconductivity observed under pressure in  $UGe_2$  and related compounds, assumed mixed valence state in some ternary intermetallics and possible heavy fermion state in the  $UCu_{8-x}Al_{8-x}$ . Finally we discuss some useful features of  $UFe_{10}Si_2$ .

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### Inelastic Neutron Scattering Study of Spin Polarons and Intrinsic Electronic Inhomogeneity in the Highly Correlated Perovskite Materials

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It now becomes more and more clear that an interplay between charge, orbital and spin degrees of freedom as well as effects of intrinsic electronic inhomogeneity are responsible for intriguing and rewarding properties of highly correlated systems, such as high- $T_C$  cuprate superconductors [1] and colossal magnetoresistance manganites [2]. A key aspect of cobalt oxides, which comes on the top of the conventional degrees of freedom, is the possible variety of the spin-states of the Co ions. Therefore, strongly correlated cobalt-based perovskite materials represent a unique system that allows studying the major open problems in this field.

Hole-doped lanthanum cobalt oxides  $La_{1-x}Sr_xCoO_3$  show a spin-glass ( $x < 0.18$ ), a cluster-glass ( $0.18 < x < 0.5$ ) and a ferromagnetic metallic state ( $x > 0.5$ ) at low temperatures [3]. So far, most of the investigations are concentrated on the middle- and high-level doped system  $La_{1-x}Sr_xCoO_3$ ,  $x > 0.1$ . However, already the lightly doped material  $x \sim 0.002$  (i.e.



with the estimated concentration of two holes per thousand  $\text{Co}^{3+}$  ions) exhibits paramagnetic properties at low temperatures, in strong contrast to the parent diamagnetic insulator  $\text{LaCoO}_3$ . The few embedded spins in a nonmagnetic background would give order of magnitude smaller magnetic susceptibility than observed [4, 5]. With this controversy in mind, we undertake an inelastic neutron scattering (INS) study of  $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$  to determine magnetic excited states of  $\text{Co}^{3+}$ . Firstly, using the INS technique, we identified the energy levels of the thermally excited states of  $\text{Co}^{3+}$  ions in both  $\text{LaCoO}_3$  [6] and  $\text{La}_{0.998}\text{Sr}_{0.002}\text{CoO}_3$  [5]. Further, we analyze a model of the localized magnetic polarons with unusually high spin number, which introduce an intrinsic electronic inhomogeneity and can be a precursor of the ferromagnetic metallic state upon further doping. We propose that holes introduced in the LS state of  $\text{LaCoO}_3$  by replacing the trivalent  $\text{La}^{3+}$  ions with divalent  $\text{Sr}^{2+}$  ions are extended over the neighboring Co sites, forming thus magnetic polaron and transforming all the involved Co ions (e.g. 8 per each Sr) to the high-spin state. The concept of such magnetic polaron in a diamagnetic matrix and intrinsic inhomogeneity in a spontaneously phase-separated system could be a key in understanding the peculiar properties of strongly correlated cobalt based perovskite materials.

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### Magnetoelasticity of Uranium Intermetallics $\text{UCoAl}$ and $\text{UFe}_2$

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Among a wide variety of the uranium intermetallics the most unusual properties are observed for the compounds where different magnetic interactions compensate each other. These compounds are usually very sensitive to the alloying, application of external pressure and magnetic field since they locate near the transition area between different magnetic states. A combination of their magnetic characteristics is frequently found to be unique. In present study, the peculiarities of the magnetic behavior of  $5f$ -electron systems, originated from a special (intermediate between localized and delocalized) character of  $f$  electrons, are illustrated on the example of two intermetallic compounds.

In  $\text{UCoAl}$  due to a fine balance between the ferro- and antiferromagnetic interactions,

the state of the exchange-enhanced Pauli paramagnetism is realized in the uranium subsystem, and a very seldom phenomenon of the itinerant 5f-electron metamagnetism is observed [1]. The experiments on the alloying within different sublattices and measurements of the magnetization under pressure, performed on the single crystals, allow us to establish the role of the change of interatomic distances and electron concentration in formation of the magnetic properties of this compound. The information on the influence of disturbance of crystallographic ordering on the itinerant metamagnetic behavior is obtained on the base of a comparative study of the single crystals of non-stoichiometric composition.

Cubic intermetallic  $\text{UFe}_2$  is a very unusual compound because of mutual cancellation of the uranium spin and orbital moments. Moreover, the magnetocrystalline anisotropy of this compound is compensated by the magnetoelastic contribution to the anisotropy [2]. We have studied the influence of non-magnetic lutetium substitution for uranium on the magnetic and magnetoelastic properties of the single-crystalline samples. Dilution of the magnetic uranium sublattice leads to the decompensation of the magnetic anisotropy, and the easy magnetization direction is changed. Additionally, the spontaneous magnetoelastic crystal structure distortions disappear, whereas a large field-induced anisotropic magnetostriction preserves.

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### **Electronic Structure of Actinides Seen by Photoelectron Spectroscopy and Magnetic Properties**

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It is generally accepted that light actinides (Th-Pu) exhibit an itinerant character of the 5f electronic states, whereas 5f localization takes place in the actinide series from Am onwards. The Mott transition with all the complexities of many body physics happens therefore between Pu and Am. This scheme is clearly reflected in magnetic behaviour of pure actinide elements, which are weak (Pauli) paramagnets up to Pu and localized atomic magnetism appears from Am onwards. Consistently, Am is a Van Vleck paramagnet due to its  $5f^6$  configuration, and magnetic order starts only at Cm, which is antiferromagnet. But one cannot simply associate occurrence of magnetic moments with the full 5f localization. Whole magnetism of intermetallic compounds of U is essentially a band magnetism based on fulfilment of the Stoner criterion in the narrow 5f band, tuned by the modifications of U-U spacing and 5f-ligand hybridisation. In this case, local magnetic moments are not based on localized 5f states. On the other hand, just at the localization threshold the proximity to the non-magnetic  $5f^6$  state means that even the dramatic lattice expansion between  $\alpha$ - and  $\delta$ -Pu,

which was expected to lead to a band narrowing and magnetic ordering, has practically no impact on magnetic susceptibility, which remains rather low and temperature independent. Although this regime is still being studied and discussed, we have to assume that only some types of defects or alloying, those which lead to the  $5f$  count reduced somewhere close to  $5f^5$ , give rise to magnetic moments, and to magnetic ordering in concentrated systems. Complementary information provided by photoelectron spectroscopy (valence-band) is often taken as a snapshot of the ground-state density of states. This is true only for real itinerant systems, in which the re-occupation of photoexcited hole is faster than the photoemission process. Localized  $f^n$  states (as in lanthanides) lead to  $5f^{n-1}$  final state multiplets dominating valence-band spectra. Again here, the proximity to the localization threshold brings extra difficulties, and spectra of various Pu phases and compounds clearly reveal traces of atomic multiplets even in cases when the full localization is far not reached yet. Thus the information on the ground state can be deduced only from the final state revealed in the spectra. Unlike valence-band spectra, in which the emission from localized and band states can overlap, the  $4f$  core-level photoemission exhibits two different types of response. The fact that the  $5f$  screening of the  $4f$  photoexcited hole, which takes place only in case of itinerant  $5f$  states, gives a lower energy of the final state than the non- $f$  screening process, provides an insight in the  $5f$  localization in actinide systems.

### **Intrinsic Inhomogeneities in $f$ -Electron Materials**

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The aim of this communication is to discuss the importance of existence of inhomogeneities (most of them of nanometric size) in the magnetic behavior of some magnetic rare earth alloys. We present some examples studied in our group of the University of Cantabria. We analyze the Rare- Earth intermetallic alloys in two ways: One is the effect of substitutions on the magnetic structures where inhomogeneities lead to very complex magnetic structures and the other is Ce compounds where new magnetic ground state phases as Non Fermi Liquids, NFL, Quantum Phase Transitions, QPT, no conventional superconductivity, NCS, were found.

Different examples of systems, where magnetic inhomogeneities (phase segregation, clusterization, structural disorder...) have been reported in literature, will be presented.

The example of new magnetic structures and its modifications with substitution is studied in the TbNi/PtxCu $_{1-x}$  series (1), a good example of the usefulness of neutron diffraction for the understanding of basic magnetic interactions. In the second part we will focus on the case of CeNixCu $_{1-x}$ , (2,3) which could be analyzed considering a wide spread of macroscopic and microscopic probes, ranging from muon spectroscopy to specific heat measurements under magnetic field. The very low temperature ground state is a nice

example of inhomogeneous magnetic behavior, observed by  $\mu$ SR, neutron diffraction and magnetic hysteresis cycles. The evolution of the magnetic behavior with the temperature supports the idea of a percolative magnetic system. We demonstrate that this is the origin of anomalous magnetic behaviors and then special care must be taken when we are dealing with substitutional compounds where magnetic disorder can not be avoided.

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**Appearance of Magnetism in Defect Plutonium**

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It is still a controversial issue, whether plutonium possesses magnetic properties. On the whole, the experimental investigations testify to nonmagnetic plutonium state. So-called the first-principle calculations of electronic structure in frames of electron density functional lead to opposite conclusion. The final solution of this question is absent, since both communities, experimental and theoretical, give strong arguments to their own advantage.

We hold the opinion that due to delicate balance between spin-orbital and exchange interactions in  $f$ -shell, the nonmagnetic ground state with  $f^6$  configuration and zero values of spin  $\mathbf{S}$ , orbital  $\mathbf{L}$ , and total  $\mathbf{J}$  moments is realized for pure plutonium. We confirmed this conclusion by band structure calculations in frames of  $LDA+U+SO$  approximation [1]. When the balance mentioned above is violated by, e.g. impurities and defects implementation into the crystal lattice of plutonium, the latter starts to demonstrate the weak magnetic properties revealed in various experiments.

It will be present a short review of recent advantages in theoretical description of the electronic structure of different allotropic states of pure plutonium in the talk. The main attention will be paid to our new results on the description of characteristics of magnetic interactions in defect plutonium  $f$ -shell in the presence of interstitial impurities, Ga impurities, and vacancies.

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### Temperature Dependence of PuGa(5at.%) Alloy Resistivity in –185...170 °C Temperature Range

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The paper presents the results of electric resistivity measurement of alloy PuGa(5at.%) in the temperature range –185...170 °C. The alloy sample used in the measurements had been manufactured 12 years ago. Resistivity measurements were made with the use of the MIES method. It was shown that the results of measurements are in agreement with the literature data. On the basis of the obtained temperature dependences a conclusion is made on phase stability in alloy PuGa(5at.%) in the temperature range –185...170 °C being preserved after 12 years in storage.

### TTT-Diagram of Undoped Plutonium $\alpha \rightarrow \beta$ -Transformation

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In complement to the data of known publications, and proceeding from the results of investigation of phase transformation kinetics in the process of intermittent volume heating of samples of plutonium in pulsed nuclear reactors of VNIITF, the authors of this paper propose a version of complete TTT (Temperature-Time-Transition) diagram of  $\alpha \rightarrow \beta$ -transformation of undoped plutonium, covering the change of the incubation period of phase transformation initiation within  $10^{-5} \dots 10^5$  s.

### Magnetism of Plutonium Monopnictides

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The physical interest in plutonium monopnictides is due to their not only nuclear properties, but also to the rich and unusual magnetic and electronic properties, which attract the attention of many researchers. The plutonium monopnictides with NaCl-type lattice structure have the general formula PuX, where X=N, P, As, Sb, Bi. These compounds have been extensively studied and now all of them are available as single crystals. Monopnictides PuP and PuAs are ferromagnets, PuBi is an antiferromagnet, and PuSb exhibits two magnetic phase transitions from paramagnetic phase to noncommensurate phase and then to ferromagnetic phase [1] (all of them possess very high magnetic anisotropy). No long-range

order is observed in PuN. In spite of much research, the magnetic properties of these compounds are still not completely understood.

We propose a model for describing the first-order magnetic phase transitions in these compounds, based on the combination of 6-state magnetic Potts model [2] and the model of structural phase transitions in cubic crystals [3], which had been earlier used for describing a cascade of magnetic phase transitions in UAs [4].

We derived a system of equations for multicomponent order parameter and after its numerical solution found the correlation function. With the help of this function we calculated the diffuse magnetic neutron scattering in the whole temperature range, positions and intensities of magnetic reflections (noncommensurate including), that allowed to describe magnetic phase transitions in plutonium monopnictides.

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### Monte Carlo + Molecular Dynamics Emulation of Radiation Damage Evolution in Pu

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The paper presents the results obtained in the simulation of damage cascades in self-irradiated unalloyed and gallium-alloyed delta-plutonium. The fast cascade stage was simulated by the Monte Carlo method. When the energies of cascade particles became close to the displacement energy, the resulting cascade configuration (coordinates + particle velocities) was transferred to a molecular dynamics (MD) code which tracked further evolution of the system to ~1–2 ns. The Modified Embedded Atom Model (MEAM) [1,2] was used to describe particle interactions.

Our simulations show that a cascade from the uranium recoil nucleus causes a large energy release into a lattice subsystem within a local region measuring about 20–25 nm; this causes material melting and subsequent recrystallization. Preliminary estimates showed that the energy transferred to the lattice is enough to cause melting in a region whose characteristic size reaches 15–17 nm; the region contains ~200 000 atoms. MD simulations show that heat conductivity reduces the characteristic size of the melting region to ~8–10 nm (~25 000 atoms) in a sample whose initial temperature was 300 K.

The evolution of temperature and density fields in the damaged region was tracked in simulations. The time of recrystallization was estimated to be ~1 ns. The distribution of point defects in the recrystallized region was obtained. Most of point defects created during a fast stage of the cascade vanish when melting and recrystallizing, residual defects evolve much slower than the nano-second time scale. The rate of the defects annealing as a function of temperature has been investigated.

To estimate the role of lattice-electron energy exchange, the electron-phonon coupling (EPC) was taken into account as an additional term in MD equations. It was shown that the account for EPC gives a 30 % reduction in the damaged region size [3]. These calculations were carried out in the assumption of infinite electron heat conductivity. Its finite value must slightly weaken the effect.

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### Structural Features of Behaviour of Uranium under Intensive Deformation

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Changes in the structure of uranium samples under plastic deformation by rolling and shock-wave loading of different intensities were studied. The main feature caused by the rolling deformation (reduction down to 40%) was the formation of coarse and fine twins and the increase in the density of dislocations up to  $10^{10}$ - $10^{11}$  cm<sup>-2</sup>. Split dislocations and stacking faults were observed too. The type of twins was determined in a transmission electron microscope by analyzing orientations of twins, the matrix and traces of twin plates. The presence of twins with the  $(130)_\alpha$  twinning plane was unambiguously established among possible twinning systems. A shock wave of a relatively small intensity (the amplitude of 21-50 GPa and the pulse length of 0.5-1.4  $\mu$ s) led to some elevation of the temperature and, hence, a redistribution of the available dislocations and the formation of a polygonal structure. Heating of the samples below the recrystallization temperature corresponded to results of analytical calculations for the given conditions of explosion loading.

If the intensity of the shock-wave loading was sharply increased (the volume loading of spherical shells of uranium), the following changes occurred in the structure. Separate fused spherical areas of uranium were observed on the internal surface of the preserved shell after the explosion loading. Thus, the dynamic loading regimes provided not only a strong deformation effect, but also the rise of the temperature from room temperature to 1130 °C and higher in fractions of a microsecond. The phase transformations  $\alpha \rightarrow \beta$ ,  $\beta \rightarrow \gamma$ , and  $\gamma \rightarrow$  melt take place over this temperature interval. The near-surface zone of the samples included deformed grains with a distorted shape. A large number of differently oriented twins and dislocations appeared in the grains. Carbides and carbonitrides of uranium were preserved in a large amount. A zone with cleavage cracks, which were due to tensile stresses, was observed at a distance of 7-8 mm from the external surface of the sample. Cracks and

discontinuities were also detected near the internal surface of the shell. A specific feature of the structure after this treatment was the formation of *localized deformation bands* (probably, adiabatic shear bands) as long straight and branching weakly etched plates, which crossed the grain. Wider regions of the localized deformation along grain boundaries were present. The boundary, where a coarse grain having the average size of 20  $\mu\text{m}$  transformed to a fine grain (5  $\mu\text{m}$ ), was observed at a distance of 14 mm from the external surface. If the coarse grain was severely distorted and contained many deformation twins, fine grains, which were adjacent to this region, were more equilibrium and were virtually free of deformation twins visible in a light microscope. The observed boundary corresponded, most probably, to the onset of the lowest-temperature phase transformation  $\alpha \rightarrow \beta$  during heating of the sample. The temperature of this transformation was 666  $^{\circ}\text{C}$  in the absence of the pressure and elevated to 720  $^{\circ}\text{C}$  when the pressure increased by 10 kbar. It may be assumed that the temperature increased to 700-750  $^{\circ}\text{C}$  in this region under the fast dynamic loading and first nuclei of the  $\beta$ -phase were formed by shear on grain boundaries. Then the nuclei began growing by the diffusion "recrystallization" mechanism and formed equiaxial small-size crystals of the  $\beta$ -phase. Crystals of the  $\alpha$ -phase appeared in regions with the equiaxial "fine-grain"  $\beta$ -phase upon cooling.

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### **Semi-Empirical Models Describing Thermodynamic Properties of *f*-Metals**

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The unusual thermodynamic properties (abnormal elastic and thermophysical features and complex phase diagrams) of a number of metals with unfilled *f*-shells (4*f* and 5*f*) are caused by close energies of different electron configurations. According to the modern concepts, electron states differ either in the degree of screening of the localized *f*-electron spins (Kondo volume-collapse model) or in localization and delocalization of *f*-electrons (Mott transition). At finite temperatures, thermodynamically stable state of a mixture of atoms with different electron configuration becomes feasible due to configuration entropy contribution, concentrations of atoms of different sort being determined from thermodynamic potential minimum. A successful semi-empirical model capable of describing behavior of material in this state is the Aptekar-Ponyatovsky (AP) model, which treats a system of atoms of different sort as a substitution solid solution with component-to-component ratio varying as a function of temperature and pressure. The extreme component concentrations correspond to the states of material in adjacent polymorphous modifications. This paper discusses capabilities of the AP model to describe thermodynamic properties of unalloyed cerium (4*f*-metal) as well as unalloyed  $\delta$ -plutonium and  $\delta$ -Pu-based alloys (5*f*-



metals). The paper presents the obtained within one model adequate description of abnormal behavior of these metals at varying external conditions that proves the common nature of these anomalies associated with the evolution of the f-electron subsystem.

### **Results of Dilatometric Study of Structural Materials**

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The present paper describes the design of a dilatometer developed by the authors for determining the thermal coefficient of linear expansion of fissionable and structural materials in controlled gaseous media in the temperature range from 0 to 1000 °C. The results of test experiments on investigation of expansion of some metals and a series of polymers in the temperature range from 40 to 400 °C are presented. The obtained data are in good agreement with the reference sources data, which confirms the serviceability capacity of the given measuring device and the opportunity for its application in study of the thermal coefficient of linear expansion of fissionable materials.

### **The Magnetic Properties of Defects in Pu, Pu(Ga) and Pu(Am)**

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The isochronal annealing of the low temperature accumulated damage from the radioactive decay of plutonium in  $\alpha$ -Pu and  $\delta$ -Pu<sub>1-x</sub>Ga<sub>x</sub> ( $x = 0.043$ ) [i] and  $\delta$ -Pu<sub>1-x</sub>Am<sub>x</sub> ( $x = 0.224$ ) was characterized using magnetic susceptibility. In each specimen, thermal annealing, as tracked by magnetic susceptibility, only commenced when  $T > 33\text{K}$  and the magnetic susceptibility changes due to defects were fully annealed at  $T \sim 300\text{K}$ . The  $\alpha$ -Pu magnetic susceptibility isochronal annealing data is similar to earlier measurements of resistivity characterized isochronal annealing [ii]. However, the  $\delta$ -Pu<sub>1-x</sub>Ga<sub>x</sub> ( $x = 0.043$ ) magnetic susceptibility isochronal annealing data, when compared with similar resistivity data, indicates that for this alloy magnetic susceptibility studies are more sensitive to vacancies than to the interstitials accumulated at low temperatures.

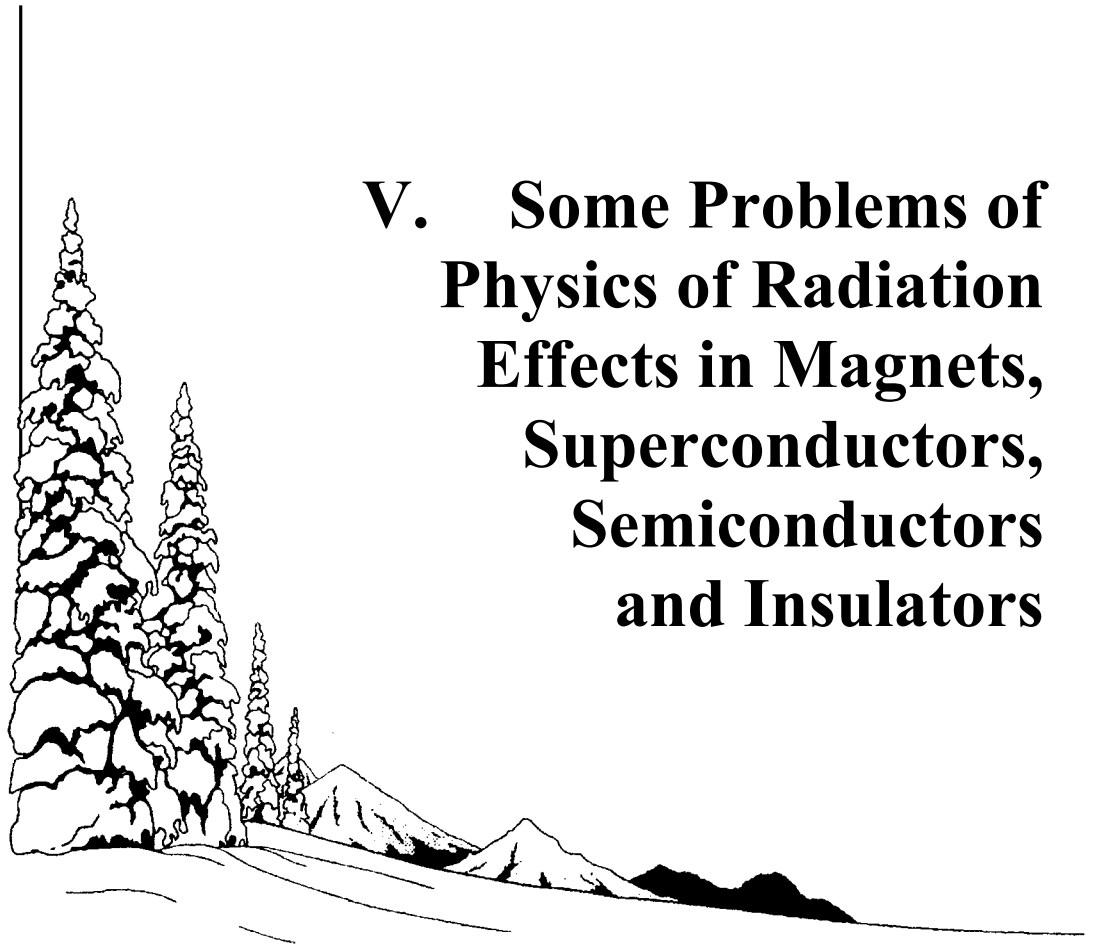
Pu(Am) is stable in the fcc  $\delta$ -phase from a few atomic percent to nearly 80 atomic percent Am, expanding the average interatomic separation as the alloy concentration of Am increases. Both Pu and Am spontaneously decay by  $\alpha$ -emission creating self-damage in the lattice in the form of vacancy-interstitial pairs and their aggregates. At sufficiently low temperatures, the damage is frozen in place, but can be removed by thermal annealing at

sufficiently high temperatures, effectively resetting the system to an undamaged condition. The magnetic susceptibility and magnetization are observed to increase systematically as a function of accumulated damage in the fcc  $\delta$ -Pu<sub>1-x</sub>Am<sub>x</sub> ( $x = 0.224$ ). Some results of these observations are reported here. The Pu<sub>1-x</sub>Am<sub>x</sub> ( $x = 0.224$ ) alloy shows a remarkable change in properties, over a limited temperature range beginning where interstitial defects are first mobile, and characterized by an induced effective moment of the order of 1.1  $\mu_B$ /Pu. This transient behavior may be evidence for a disorder driven low temperature phase transition, perhaps indicative of a compositional and structural proximity to a state possessing significant magnetic moments.

This work was performed under the auspices of the U.S. Department of Energy by the University of California Lawrence Livermore National Laboratory under Contract No W-7405-Eng-48.

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## **V. Some Problems of Physics of Radiation Effects in Magnets, Superconductors, Semiconductors and Insulators**

The subject of this Section is traditionally formulated with a view to introduce the Seminar attendees (mainly metal physicists) to the results of the latest research into radiation effects in superconductors, semiconductors and dielectrics (magnetic dielectrics including). In case of the first two materials, significant changes of their physical properties take place under irradiation already with quite low fluences of high-energy particles. Therefore, investigation of the causes of damage and the degradation 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 behaviour of radiation defects and changes in the physical and mechanical properties of such materials as manganites  $\text{La}_2\text{SrMn}_2\text{O}_7$ ,  $\text{LaMnO}_3$ , oxide  $\text{CuO}$ ,  $\text{Si}$ ,  $\text{SmB}_6$ ,  $\text{GaN}$ , etc. are analyzed. The amorphization of silicon under exposure to ion beams, the dielectric effect in HTSC ceramics, and the influence of radiation-induced disordering on semiconductor radiation detectors are discussed.



## Effect of Radiation Sensitivity of the Insulating Oxide on the Charge Resistance of SOI-Structures

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In SOI transistors subjected to ionizing radiation, the threshold voltage shear is experimentally shown to correlate with the current radiation sensitivity of SiO<sub>2</sub>. Decreasing sensitivity of particularly the insulating (embedded) oxide is the reason of the decrease in the radiation injury. Apparently, this effect is conditioned by “competition” of two processes, i.e. drawing of electrons from the bulk of oxide and their capture in oxide defects. Electrons capture causes decrease in the current radiation sensitivity of the oxide and correspondingly formation in it of a negative charge that partially compensates the positive charge of stabilized holes and, therefore, preconditions the threshold voltage shear of SOI – transistors.

Consideration is given to model approaches that describe experimental data on radiation-induced electroconductivity of SiO<sub>2</sub> within a wide range of changing absorbed ionizing radiation dose rate (10<sup>6</sup> – 10<sup>12</sup>rad/sec), as well as neutron fluence (up to 10<sup>17</sup>n/cm<sup>2</sup>) and allow quantitative explanation of the correlation effect.

The obtained results outline a possible way to improve the charge resistance of SOI-structures due to increased concentration of structural defects in the oxide. This task can be solved by means of the radiation modification: ion or neutron irradiation of the oxidized silicon plate using DeleCut-technology. Decrease in the sensitivity to the charge accumulation in the oxide can help to increase its thickness and thus create conditions for increasing the percent of suitable SOI-plates during their fabrication [1].

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## Relaxation of Radiation Defects in Nanocrystalline CuO

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Magnetic properties of nanocrystalline and coarse-grain samples may be different because of the large extent of interfaces in nanomaterials when the structural regularity is disturbed. The decrease in the size of particles in magnetic semiconductors may have a considerable effect since the magnetic order in oxides is determined by the superexchange via oxygen ions and depends on the distance between nearest magnetic ions. Distortions of the crystal lattice both in surface layers and the bulk of nanoparticles can frustrate exchange

couplings and disrupt the long-range magnetic order. This study deals with the effect of radiation defects on magnetic properties and their relaxation in coarse-grain ( $d > 2000$  nm) and nanocrystalline antiferromagnetic CuO with  $T_N = 230$  K. Relaxation processes after the electron bombardment and intensive deformation were compared. The high-density ceramic with the grain size  $d \sim 5-100$  nm on the average was prepared by the method of shock wave loading. The susceptibility  $\chi$  decreased in the CuO polycrystal with decreasing  $T < T_N$ . This is characteristic of 3D collinear antiferromagnetics. As the grain size  $d \leq 60$  nm decreased in the nanoceramic, the susceptibility  $\chi$  was enhanced and the paramagnetic contribution  $\chi \sim 1/T$  appeared at  $T < 140$  K. A correlation between the  $\chi$  value and the grain size was established. After the nanocrystalline samples were kept for three years in air at room temperature, the "paramagnetic" contribution to  $\chi$  vanished. This was due to the relaxation of long-range stress fields induced by microscopic deformations and defects.

The electron bombardment at a small dose  $\Phi \leq 2.5 \cdot 10^{18}$  electrons/cm<sup>2</sup> actually did not influence magnetic properties of the CuO samples studied, but at  $\Phi \geq 5 \cdot 10^{18}$  electrons/cm<sup>2</sup> the susceptibility  $\chi$  increased sharply in the magnetically ordered region. The most probable point defects under the electron bombardment were displaced oxygen ions. Therefore, exchange couplings could be broken and some Cu<sup>2+</sup> ions could change to the paramagnetic state. Measurements of  $\chi(T)$  in 3-year samples showed that the susceptibility decreased in both the nanoceramic and the polycrystal, but did not reach values typical of stoichiometric polycrystals. Probably, not all of the displaced oxygen ions returned to their spatial positions. Thus, the presence of a large number of bulk and surface defects led to disturbance of the long-range antiferromagnetic order and the growth of the susceptibility.

This study was supported under the program of the Department of Physical Sciences RAS and the Presidium of the Ural Branch RAS "New materials and structures» and the RFBR project № 04-02-16630

### **Magnetic Structure and Transportation Properties of Atomic-Disordered Crystal of Two-Dimensional Manganite $\text{La}_{2-2x}\text{Sr}_{1+2x}\text{Mn}_2\text{O}_7$**

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The magnetic structure and transportation properties of partially disordered crystals of two-dimensional manganites  $\text{La}_{2-2x}\text{Sr}_{1+2x}\text{Mn}_2\text{O}_7$  ( $x = 0.3, 0.4$ ) were studied in a broad temperature range. Through exposure to fast neutrons, there is suggested an opportunity to introduce, in a proportioned manner, the so-called antisite defects into a massive crystal of two-dimensional manganite  $\text{La}_{2-2x}\text{Sr}_{1+2x}\text{Mn}_2\text{O}_7$ , i.e., to bring about disordering of the initial distribution of cations in these oxides.

Single crystals of oxides  $\text{La}_{2-2x}\text{Sr}_{1+2x}\text{Mn}_2\text{O}_7$  ( $x = 0.3, 0.4$ ) were grown by the zone melting method. The scatter of mosaic blocks in crystals did not exceed 25 minutes of arc. The samples had a cylindrical shape, with the diameter and height 0.3 cm and 1 cm,

## V. Some Problems of Physics of Radiation Defects in Magnets, Superconductors, Semiconductors and Insulators

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respectively. Fast neutron irradiation of samples was carried out at 340 K in the IVV-2M reactor core. The fast neutrons fluence was  $2 \times 10^{19} \text{ cm}^{-2}$  ( $E_n \geq 1 \text{ MeV}$ ).

At fast neutrons fluence of  $2 \times 10^{19} \text{ cm}^{-2}$ , the substitutional defects average concentration over the crystal is  $\cong 4 \%$ . It was established that substitutional defects are the factor initiating transition of manganite of the given class from the state of a ferromagnetic metal to the state of an insulator with a spin glass structure.

The results testify to the fundamental role of parameters of the charge carriers kinetic energy and the localized spins magnetic energy in forming the basic physical properties of two-dimensional manganites. By varying the relation of these parameters through proportioned introduction of antisite defects in a massive crystal, its magnetic structure and transportation properties may be radically modified.

Work was carried out with partial financial support of the Federal Agency for Science and Innovations (FASI), State Contract No. 02.452.12 7004; the program of the Department of Physical Sciences (DPS) of RAS "Influence of atomic-crystal and electronic structure on properties of condensed matter"; RFBR, Project No. 04-02-16053a.

### Effect of Nonequilibrium States on Exchange Interaction in Nanostructured $\text{LaMnO}_{3+\delta}$

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Nanocrystalline materials are nonequilibrium metastable systems with largely extended interfaces between grains. If the size of crystallites  $d$  is smaller than the critical value and the defect content is high, their structural and magnetic properties can change as compared to those of polycrystals. The method used for preparation of nanoscale materials has a considerable effect on their properties. If processes taking place in nanomaterials are understood, it will be possible to realize capacities of nanotechnologies in creation of constructional and functional materials with assigned properties.

Two sets of nanostructured samples of  $\text{LaMnO}_{3+\delta}$ , which were prepared by the shock wave method and mechanochemical synthesis in ball mills, were studied. After the shock wave loading treatment, the manganite was shaped as a sphere with the radius  $r \approx 22 \text{ mm}$ . Samples were cut out at different depths in the sphere. They represented a dense ceramic with crystallites having the size  $d = 20\text{-}30 \text{ nm}$  on the average. Samples taken at the center had an orthorhombic structure. The structure of the samples from the surface layers of the sphere was rhombohedral. The volume of a unit cell and lattice strain increased in going to the sphere surface. The magnetic measurements demonstrated that these  $\text{LaMnO}_{3+\delta}$  samples had a relatively wide magnetic transition, which was indicative of the magnetically

inhomogeneous state. The Curie temperature  $T_C$  decreased in going from the center to the surface. The second set of nanostructured samples was synthesized by grinding for 1, 7 and 13 hours. The grain size decreased from  $d = 500$  nm to  $d = 15$  nm, while the lattice strain  $\epsilon$  increased. The nanopowders had an orthorhombic structure. The unit cell volume diminished with decreasing  $d$ . The drop of  $T_C$  could be explained by the decrease in the grain size and the rise of long-range elastic stresses.

The dependence between  $T_C$  and  $d$  was not unambiguous in nanostructured  $\text{LaMnO}_{3+\delta}$ . The temperature  $T_C$  could decrease or increase at the same  $d$  value. Magnetic properties of the nanocrystalline manganite depended on some factors, such as the grain size, lattice defects, elastic stresses, and the anisotropy energy, the exchange coupling between magnetic ions  $\text{Mn}^{3+}$  and  $\text{Mn}^{4+}$ .

This study was supported under the program of the Department of Physical Sciences RAS and the Presidium of the Ural Branch RAS "New materials and structures" and the RFBR project No. 04-02-16630.

### Modification of Structural and Magnetic States of Oxide Perovskite-Like Manganites through Introduction of Anti-site Defects

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The physical properties of oxide perovskite-like manganites of variable valence (e.g.,  $\text{La}^{3+}_{1-\nu}\text{Me}^{2+}_{\nu}\text{Mn}^{3+}_{1-\nu}\text{Mn}^{4+}_{\nu}\text{O}^{2-}_3$ , where  $\text{Me}^{2+} = \text{Sr}^{2+}, \text{Ca}^{2+}, \text{Ba}^{2+}$ ) being under close investigation in the recent ten years (see, e.g., the reviews [1-2]) are quite sensitive to structural imperfections, one of which is inter-lattice disordering of cations. In the thermodynamically equilibrium phases of perovskite-like compounds, such disordering is impossible for considerations of size (see, e.g., [1]). For this reason, such structural defects, formed, to all appearances, exclusively under irradiation with high-energy particles, are commonly referred to as "anti-site defects. The first investigations of manganites irradiated with fast neutrons have shown that anti-site defects exert an extremely strong effect on the parameters of crystalline and magnetic structures of manganites, resulting in various structural, magnetic and electronic transformations.

The present paper, on an example of La(Ba) and La(Sr) oxide manganites, deals with the laws of anti-site defects formation under neutron irradiation and analyses the possible mechanisms of their influence on structural, magnetic and electronic states. The scenario of electronic phase layering, when dielectric antiferromagnetic microregions are formed in the conducting ferromagnetic matrix, is discussed. Such a situation, as distinct from the case of thermodynamically equilibrium phase separation (characteristic of the system with strongly correlated electrons), may be force-realized through introduction of anti-site defects at manganites irradiation with fast neutrons [10].

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## Defect Production in Dense and Porous Glasses under Influence of the Separated Gamma-Component of the Reactor Radiation

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Radiation defect production in oxides is of great interest for atom and solar energy, and also for nuclear waste storage. The combined influence of neutron flux and gamma-radiation on materials was studied widely, and only neutrons were believed to be able to displace atoms. Although it turned out, that 1.25 MeV-  $^{60}\text{Co}$  gamma-quanta can shift light anions (O, F) by the inelastic mechanism. The aim of this work was to study defect production in pure fused quartz and barium-silica glasses, containing nano-crystalline inclusions, and also nano-porous glass under influence of 0.2 - 7 MeV- gamma-radiation in the quenched reactor. The periods were chosen when practically constant current of  $\sim 10\text{-}50$  nA is established in the ionization chamber, that corresponds to an average gamma-flux of 15-70 Gy/s. Single crystals of LiF were used for comparative dose monitoring of gamma-fluxes in the reactor and  $^{60}\text{Co}$  source by the well-known absorption band of F-center (a fluorine vacancy trapping an electron).

Spectra of optical absorption and photoluminescence, and also the glass structure were studied. The charged oxygen vacancy accumulation rate with the dose growth turned out to be higher in the barium silica glass than in pure  $\text{SiO}_2$  since at small  $Z$  the Compton scattering and photonuclear reactions prevail, and for Ba with high  $Z$  – just the opposite case of domination of the photoelectric effect. The radiation-induced growth of crystalline phase precipitates was found in the both glasses, which had been ascertained only to knocking out atoms (i.e. elastic displacements) by high fluencies of fast neutrons. The point defect production efficiency (for example,  $\text{E}'$ -centers and non-bridging oxygen atoms) in the glasses under the gamma-irradiation even in the quenched reactor turned out much higher than that under the exposure to the equivalent dose in  $\sim 1.25$  MeV  $^{60}\text{Co}$  gamma-source at the dose rate of  $\sim 7$  Gy/s (and even earlier when it was as high as 45 Gy/s).

Because of water vapor radiolysis on the glass pore surface under the gamma-irradiation, a 100-times increase in the surface proton conductivity occurred, that is important for hydrogen energy.

The work was accomplished by the grant F 2.1.2 from the Center of Science and Technology of Uzbekistan.

## Insulator-to-Metal Transition in $\text{SmB}_6$ Induced by Neutron Irradiation

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The semiconducting behaviour of temperature dependences of transport properties in some compounds, such as  $\text{SmB}_6$  [1], is probably caused by formation of an energy gap due to hybridization between the localized  $f$  states and conduction electron states; these materials are often referred to as “hybridization gap semiconductors” or “Kondo insulators”. To understand the response of the such systems to disordering on an atomic scale, without variation of the stoichiometric composition, we studied the behaviour of resistivity  $\rho(T)$  and Hall coefficient  $R_H(T)$  under neutron irradiation.

Samples of  $\text{SmB}_6$ , were irradiated at  $T_{\text{irr}} = (330 \pm 10)$  K in the reactor core (the fast and thermal neutron fluencies were approximately equal in value, each being  $\Phi \sim 1 \cdot 10^{19} \text{ cm}^{-2}$ ) and annealed in the temperature range  $T_{\text{ann}} = (100 - 900)^\circ\text{C}$ . Irradiation leads to a strong decrease in the low- $T$  ( $\sim 1.5$  K) values of both the resistivity (from  $\sim 700$  to  $\sim 1.5 \text{ m}\Omega\text{cm}$ ) and the Hall coefficient (from  $\sim 0.15$  to  $\sim 0.0002 \text{ cm}^3/\text{C}$ ), while the high- $T$  ( $> 20$  K) values remain almost unchanged. The formal value of Hall mobility  $\mu_H = R_H/\rho$  at  $T \sim 1.5$  K is very small (varying from  $\sim 0.15$  to  $\sim 1.0 \text{ cm}^2/(\text{V}\cdot\text{c})$  for all states of the sample), while estimate of the electron mean free path made using a simple expression  $l = (3\pi^2)^{1/3} \hbar / (\rho_0 e^2 n^{2/3})$ , where  $n = 1/(R_H e)$ , yields unreal values of  $l = (0.15 - 2) \text{ \AA}$ .

Although there is no doubt that fast drop of  $\rho(T)$  at decreasing  $T$  is due to a pseudogap effects, the low- $T$  behaviour of both  $\rho(T)$  and  $R_H(T)$  is rather logarithmic than exponential. The logarithmic-type dependences of transport properties are usually associated with Kondo scattering effects. This suggestion is corroborated by the observed negative magnetoresistance which varies (at  $H = 13.6$  T and  $T = 4.2$  K) from  $-5.0$  % for the initial sample to  $-0.5$  % for the irradiated sample due to field-induced ordering of Sm magnetic moments. The logarithmic-type dependences of  $R_H(T)$  in this case are due to the positive skew Hall effect, which is probably compensated by the negative contribution from the normal Hall effect.

Work was carried out with the financial support of the Federal Agency for Science and Innovations (State Contract No. 02.452.11.7004), the Presidium of RAS Programs of Basic Research: “Quantum Macrophysics” (Project No. 4 of UB RAS) and “Effect of atomic-crystalline and electronic structure on properties of condensed matter” (Project No. 9 of UB RAS), RFBR (Project No. 04-02-16053).

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### **Decomposition of Supersaturated Solid Solutions in Silicon Layers Damaged by Ion Bombardment**

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The paper draws attention to the fact that silicon layers exposed to ion bombardment with the purpose of solid-phase synthesis invariably get amorphized, and compounds formation in further annealing includes a stage of recrystallization. However the present knowledge of the processes of amorphization and recrystallization of ion bombardment-damaged layers does not allow the evolution of supersaturated defect and impurity subsystems to be predicted under conditions of their active interaction. The paper analyzes the opportunity for application of the representations of decomposition of model “pure-impurity” subsystems to the processes of formation and recrystallization of amorphous layers.

### **Silicon PIN- and APD- Detectors for Nuclear Particles Diagnostics in Gas-Dynamic Trap**

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The paper presents the basic principles of technology and design characteristics of PIN and avalanche APD-photodiodes developed at the Institute of Physics of Semiconductors. The distinctive feature of such photodiodes is the presence of a thin “dead” layer which allows the lower limit of energy of registered protons to be brought down to 10 keV.

### **Radiation Hard Devices Based on SiC**

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Modern military industry, nuclear power engineering, space station and aircraft engineering need electronic devices capable of operating under extreme conditions, i.e., at high temperatures, high radiation levels and in chemically active environments. Up to now such devices could not be fabricated with conventional semiconductor materials (Ge, Si, CdTe, and GaAs). SiC is the most promising material for registration of hard radiation due to the high value of threshold energy for defect production, chemical stability, electrical and mechanical toughness. The use of SiC devices in high level radiation and temperature conditions will serve to achieve better reliability of electronic equipment and reduce its weight for the reason that the SiC devices require no cooling.

## V. Some Problems of Physics of Radiation Defects in Magnets, Superconductors, Semiconductors and Insulators

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High-resistance pure  $4H$  and  $6H$ -SiC epitaxial layers were used for device formation. The initial material, Schottky barriers, high power ion implanted diodes and transistor structures were irradiated with protons (8 MeV), electrons (900 keV), neutrons (1 MeV), gamma-ray ( $5 \times 10^6$  rd), high power pulsed X-ray radiation [ $3 \times 10^{10}$  rd (SiC)/s], Al (150 keV), Kr (245 MeV) and Bi (710 MeV) ions as well. The spectrometry of short-range ions was produced using  $\alpha$ -particles (4.8–7.0 MeV) formed during the natural decay of the isotopes  $^{226}\text{Ra}$ ,  $^{238}\text{Pu}$ ,  $^{241}\text{Am}$ .

– It was found for the first time that diodes degraded after irradiation with fast neutrons and high-energy Kr and Bi ions, recovered their rectifying properties at a temperature up to 500 °C [1]. Consequently, the radiation resource and lifetime of devices based on SiC could be considerably increased under high working temperatures.

– The SiC structures with Schottky barrier allowed a thin structure of alpha decay spectrum up to 7 MeV to be obtained. The energy resolution of 0.34 %, commensurable with Si-detectors, has been achieved for the first time; this is true for all classes of short-range ions [2].

– For the first time the spectrometric characteristics improvement with temperature rise up to 180°C was revealed for the SiC detectors based on Al ion-implanted  $p^+-n$  junctions [3].

– For the first time the transistor structure SiC based detectors were realized with the signal amplification by a factor of tens under irradiation. It could be considered as equivalent to the signal from epitaxial layers with thickness of 300-400  $\mu\text{m}$ .

– The SiC power diode recovery time was determined to be equal to 25 ns under 22 ns pulsed X-ray radiation which points to the SiC devices strength under the high dose rate effects.

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## Gamma-Irradiation Effect on HTSC Structure

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In this paper, with the use of conversion electrons Moessbauer spectroscopy, investigations were carried out of phase composition of surface layers of high-temperature superconductors on the basis of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  with substitution of part of Cu atoms for  $^{57}\text{Fe}$  atoms.

### Proton Irradiation Effect on Gas Emission in Thin-Film System

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Thin-film devices change their characteristics in service in the proton irradiation zone. These changes may be connected with ion-stimulated gas emission on the surface of interface, which points to the necessity of study of the effect of proton irradiation on the properties of thin-film systems.

It is important to note the applied value of correlation of the gas emission process with the adhesive properties of the system. Based on the results of investigations performed, the authors propose a series of methods for measuring thin films adhesion to substrates by the stimulated gas emission method.

### Radiation Effects in Semiconductor Compounds of Group III-N (BN, AlN, GaN, InN)

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In the present paper, on the basis of heuristic models developed by the authors, calculation is performed of the energy position of the “neutral point” of a semiconductor crystal III-N saturated with intrinsic defects at the expense of high-energy exposure (ion, fast ion or electron bombardment). The calculated values of this energy position are compared with the limit position of the Fermi level in irradiated material.

### Radiation Effects in Filamentary Microcrystals of Indium Antimonide

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The results of investigation of change of properties of magnetic field sensors based on filamentary microcrystals  $n^+$ -InSb in the process of irradiation with fast neutrons in IBR-2 reactor are presented.

### Radiation-Dielectric Effect in Thick-Film Ceramics

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This paper presents investigation results for the radiation-dielectric effect earlier observed in multilayer composite plate [1]. This effect manifests itself in a reversible capacity change.

In contrast to previous works, the effect was investigated using the model plane capacitors whose dielectric layer is made with application of the thick-film technology. The pulsed X-ray accelerator IGUR-3 [2] and the pulsed reactor YAGUAR [2] were used for the experiments.

Possible mechanisms underlying the observed effect were considered proceeding from analysis of experimentally measured capacity and ionization current of model samples. The model based on the irradiation-induced charge exchange of micron and submicron-scale quartz inclusions was proposed following investigation of the heterogeneous-structure response by the Maxwell-Wagner mechanism [3] and considering the hypothesis on dipoles production as electron-ion pairs [4]. The charge-exchange mechanism is conditioned by the charge separation that depends on the significant difference in mobility of electrons and holes similar to the metal-oxide-semiconductor structures.

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### Superconductivity and Transport Properties in LaRu<sub>4</sub>Sb<sub>12</sub> Single Crystals Probed by Radiation-Induced Disorder

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A number of filled skutterudite compounds RET<sub>4</sub>Pn<sub>12</sub> (RE = rare earth; T = transition metal; Pn = pnictogen) show heavy-fermion behavior coexisting with an unconventional superconductivity (SC) state [1, 2]. To understand the SC features of this class of materials, we studied the response of electronic system in the SC and normal states in LaRu<sub>4</sub>Sb<sub>12</sub> to disordering induced by fast neutron irradiation.

Single crystals of LaRu<sub>4</sub>Sb<sub>12</sub> prepared by the molten-metal-flux growth method with Sb flux [2] were irradiated with  $5 \cdot 10^{18}$  cm<sup>-2</sup> neutron fluence at  $T_{irr} = (330 \pm 10)$  K and annealed up to  $T_{ann} = 500$  °C. Neutron irradiation leads to increase in residual resistivity  $\rho_0$  (from  $\sim 3.5$  to  $\sim 200$   $\mu\Omega$ cm) and some increase in the resistivity slope  $d\rho/dT$ . The SC state is suppressed by irradiation and reappears at  $T_{ann} = 300$  °C (SC temperature  $T_c > 1.4$  K at  $\rho_0 <$

120  $\mu\Omega\text{cm}$ ). The irradiation effects in both SC and normal properties are almost recovered after annealing at 500 °C. In the SC region  $\rho_0 < 120 \mu\Omega\text{cm}$ , the essential increase of upper critical field slope  $-dH_{c2}/dT$  is observed, which shows approximately linear behavior as a function of  $\rho_0$ . It is related to the constancy of “band” parameters such as electronic density of states  $N(E_F)$ .

The first possible explanation of the observed irradiation effects in  $\text{LaRu}_4\text{Sb}_{12}$  is that the  $T_c$  decrease results from pair-breaking effects of the  $d$ -wave order parameter, described by the AG formula  $\ln(T_{c0}/T_c) = \psi(\alpha + 1/2) - \psi(1/2)$ , where  $\psi$  is the digamma function,  $\alpha$  is the pair-breaking parameter,  $\alpha = \hbar/(2\pi k_B T_c \tau)$ . This equation predicts  $T_c = 0$  at  $\tau \leq \tau_c = \hbar/(0.88 k_B T_{c0})$  or  $\rho_0 \geq \rho_{0c} = (0.88 k_B T_{c0} m^*)/(\hbar n e^2)$ . Estimating effective electronic mass  $m^* \approx 10m_e$ , we have  $\rho_{0c} \approx 5 \mu\Omega\text{cm}$ . This value is significantly smaller than  $\rho_0 \approx 100 \mu\Omega\text{cm}$ , where the SC still exists. Thus AG theory overestimates  $T_c$  decrease in  $\text{LaRu}_4\text{Sb}_{12}$ .

There is another qualitative explanation to  $T_c$  suppression under disordering in exotic (non-phononic) mechanism of SC, associated with the disordering-induced damage of appropriate quasi-particles, which are responsible for exotic pairing.

Work was carried out with the financial support of the Ministry of Industry, Science and Technologies of the Russian Federation (State contracts Nos. 40.020.1.1.1166, 40.012.1.1.1356) and the Special Federal Program of Basic Research at Russian Academy of Sciences "Quantum macrophysics" (State contract No 1000-251/P-03/040-348-11054-269, Project UB RAS No 3).

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### **Changes in Crystal Structure and Magnetic Properties of $\text{Ce}_2\text{Fe}_{17}$ after Fast Neutron Irradiation and Subsequent Isochronal Annealing**

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The paper presents the results of structural and magnetic investigation of fast neutron-disordered intermetallic compound  $\text{Ce}_2\text{Fe}_{17}$ . The disordered structure was obtained by irradiation with fast neutrons with energy over 0.1 MeV in the IVV-2M reactor vertical channel at 80 °C. From the magnetic measurements, there was found complete suppression of the antiferromagnetic state, characteristic of the initial sample, with transfer to the ferromagnetic state at Curie temperature  $T_C = 320$  K. The crystal structure was studied by X-ray and neutron diffraction analysis at room temperature. As it follows from analysis, such changes are connected with increase in the volume at crystal lattice disordering. This is in agreement with the concept of Curie temperature dependence on interatomic distances. Isochronal annealing was carried out on the sample irradiated with fast neutrons to fluence  $1.3 \times 10^{19} \text{ cm}^{-2}$  under vacuum.

As magnetic experiments have shown, there are two stages observed in the course of isochronal annealing: in the first stage up to 400 °C, the Curie temperature rises to  $T_C = 355$  K, while in the second stage up to 700 °C, an abrupt drop down to  $T_C = 150$  K is observed. However, complete recovery of the initial magnetic properties had not been achieved in our

experiments, and the sample remained in the ferromagnetic state. Simultaneously, neutron diffraction study of the sample structure was carried out. According to preliminary conclusions, the elementary cell volume decreases with the annealing temperature rise, at that, the volume vs. temperature curve has a knee in the region of  $T = 400$  °C. Such behaviour agrees, in general, with the magnetic measurements data (two stages of annealing). The paper presents a detailed examination of the behaviour of interatomic distances and other structural parameters. There is given analysis of the obtained results aimed at clarifying the nature of the observed magnetic and structural properties.

Work was supported by the RFBR Project No. 04-02-16053 and the State Contract No. 02.452.11.7004

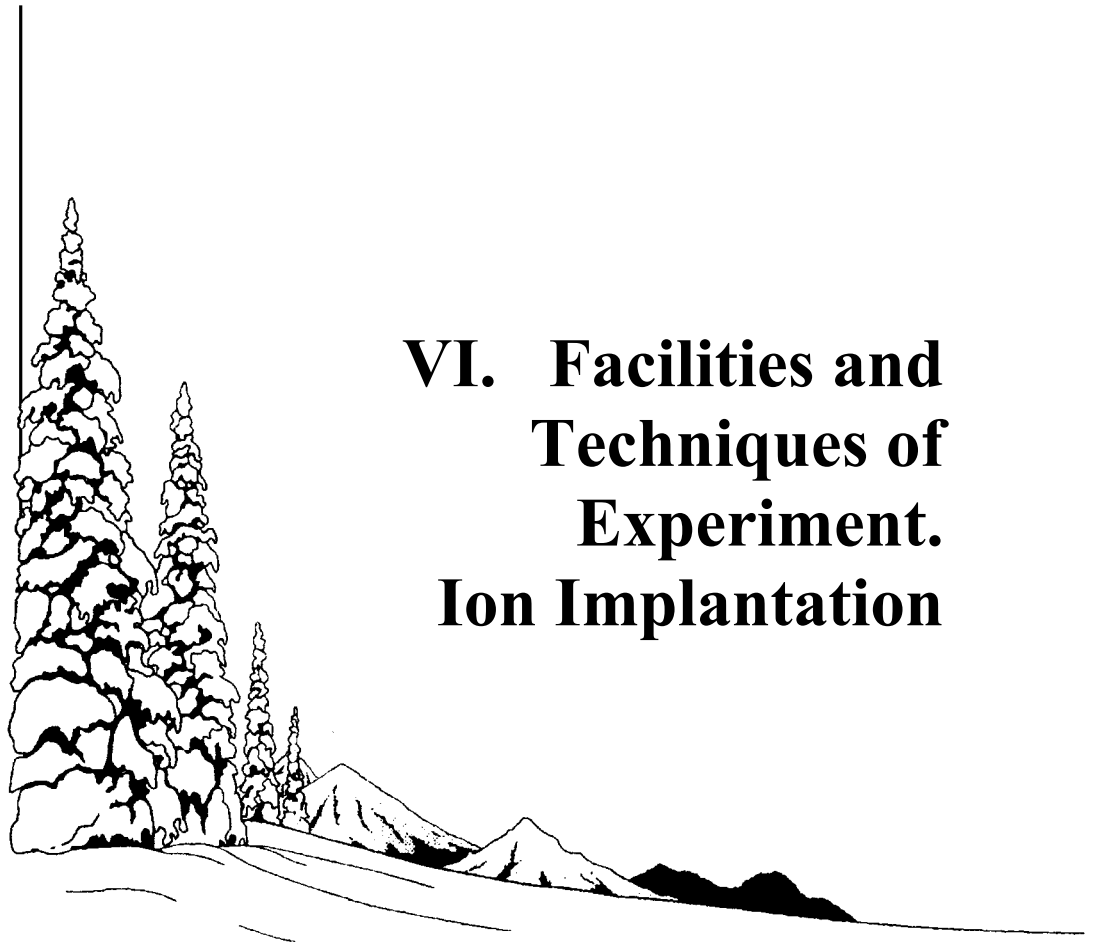
### Radiation Defects Localization in HTSC after Fast Neutron Irradiation

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As is known, the temperature  $T_C$  of superconducting transition in HTSC compounds is quite sensitive to defects of different type introduced in the lattice, and depends on their concentration and distribution in the bulk of the material. Fast neutron irradiation presents a unique and “clean” – from the physical point of view – method of generating defects on an atomic scale (vacancies, interstitial atoms, anti-site substitutions etc.) in the lattice of the investigated material. It is known that fast neutron irradiation leads to degradation of  $T_C$  in HTSC compounds, which, according to structural neutron diffraction analysis data, is accompanied with large non-correlated atomic displacements from lattice sites. It was suggested that such displacements may occur due to redistribution of the most mobile oxygen atoms over the crystal lattice positions. In the present work, an attempt was made to determine location of radiation-induced defects in typical HTSC compounds, YBCuO and LaSCuO, by plotting, using the results of neutron diffraction experiments, the Fourier maps of atomic density distribution within an elementary cell. The initial  $YBa_2Cu_3O_{7-\delta}$  and  $La_{0.83}S_{0.17}CuO_4$  samples were obtained using a standard ceramic technique. Structural investigations were carried out using the method of high-resolution powder neutron diffraction analysis ( $\delta d/d \sim 0.3\%$ ). The numerical values of structure parameters were obtained by processing the experimental neutron diffraction patterns by the Rietveld profile refinement method with the application of the FullProf program. The Fourier maps of atomic density distribution within an elementary cell were plotted using the GFourier subroutine. To exclude thermochemical processes, irradiation with fast neutrons with energy over 0.1 MeV was carried out in the IVV-2M reactor core at liquid nitrogen temperature ( $\sim 78$  K).

Work was supported by RFBR Project No. 04-02-16053 and State Contract No. 02.452.11.7004.





## **VI. Facilities and Techniques of Experiment. Ion Implantation**

**The Seminar Program traditionally includes a methodological section. Its purpose is to (a) introduce the attendees to the latest methodological developments in the sphere of radiation physics and radiation material science, and (b) inform about new radiation sources, application of the new condensed matter investigation methods, and the methods for obtaining new functional materials, nanostructural materials including. Details of stress waves recording and defects accumulation at high-speed loading of metals and alloys will be discussed, and conditions of spallation destruction in steels will be looked into. Presentations will be made on nanostructures formation by the method of radiation modification, and by the method of mechanical doping as applied to creation of new reactor construction alloys with hardening by nano oxides. The atom-probe methods of investigation, the experimental methods for determining nuclear reaction cross-sections on isotopes, on Zr and Ge in particular, the positron spectroscopy methods, and the features of microdefects accumulation in metals and alloys under shock-wave loading will be analyzed.**



## Features of Recording Short Stress Waves by Quartz Pressure Transducers

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At present, quartz pressure transducers made of the single-crystal x-cut quartz are widely used to measure the wave shape of mechanical stresses [1]. The relative ease of fabrication, high signal amplitude ( $\sim 10$  V/kbar) make their use preferable on installations with high level of electromagnetic interference, i.e. pulse electron accelerators, installations using metal foil explosion, etc. In standard experiments, the epoxy adhesive is used to fix the transducer to the tested sample. In this case, the mechanical strength of transducers comes to  $\sim 1$  kbar, what hampers their reuse. Measures favorable for decreasing mechanical stresses in the transducer, i.e. replacement of the epoxy adhesive with the diffuse oil and conversion of the stress wave from the backside of the transducer to acoustically matched load (aluminum), allow its fracture threshold to be increased up to 2-3 kbar.

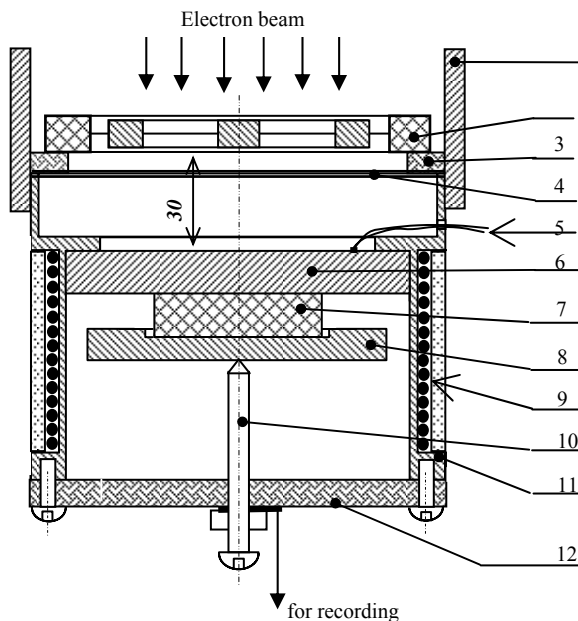


Fig.1. Experiment design

- 1 – mounting flange,
- 2 – ring and central calorimeters,
- 3 – textolite ring,
- 4 – fluoroplastic tape,
- 5 – thermocouple,
- 6 – impactor (sample),
- 7 – quartz pressure sensor,
- 8 – hold-down disk (SW absorber),
- 9 – heater winding,
- 10 – contact bolt,
- 11 – heater body,
- 12 – textolite disk.

Highly stable parameters of the single-crystal quartz in the wide range of temperatures make it possible to use the quartz transducer at elevated temperatures (up to  $400^{\circ}\text{C}$ ) [2]. Fig.1 gives the experiment design at elevated temperatures on the pulse electron accelerator IGUR-3 [3]. Significant attenuation of the signal when it passes through the material being an acoustic contact between the sample and the transducer was the greatest difficulty during the experiments. After graphite lubrication, diffuse oil, lithol, castor oil, silicone oil, and two-component epoxy VK-9T were tested, the two-component epoxy VK-9T without a hardener was chosen to be an acoustic contact between different materials. According to available information, it allows the most accurate transfer of the pressure pulse from the impactor to the transducer.

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## Calculation of Magnetic Field in the Region of the "Window-Frame" Type Dipole Magnet for Particle Accelerators

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Such dipole magnet (DM) represents an iron yoke with a hollow rectangular space inside and a winding at two opposite edges. The size and shape of the yoke are chosen in such a way that the resulting transverse field of the winding and the magnetized iron (which makes the main contribution) is sufficiently homogeneous inside the aperture. Since usually the DM length is many times larger than its transverse size, in this work all  $B(x,y)$  calculations were performed in the transverse plane intersecting the center of the magnet. In the central point, the field was always chosen to be equal to  $B(0,0) = 2$  T (which is the characteristic working value close to the maximum value for a DM of this type).

This work presents results of  $B(x,y)$  calculations for single- and double-layered windings with 8 and 16 turns from a circular tube-like conductor with magnetic permeability equal to unity.

## Experimental Determination of Cross Sections of (n,x) Nuclear Reactions on Zirconium and Germanium Isotopes

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Investigation of neutron reaction cross section at the energy range about 14 MeV is important for development of fusion reactor technology from the point of view of activation, radiation-damage and mechanical stability of construction materials, problem of radiation protection etc. Furthermore, measurements of nuclear reaction cross sections in this energy region are very useful for testing nuclear reaction models.

The cross sections of nuclear reactions  $^{92}\text{Zr}(n, p)^{92}\text{Y}$  and  $^{94}\text{Zr}(n, p)^{94}\text{Y}$  were measured in the neutron energy range of 13.56÷14.53 MeV. The discrepancies between existing results from different experimental groups in the selected energy range for the specified nuclear reactions achieve 20÷60 % [1]. Obtained results remove the uncertainty concerning values of cross sections of the investigated nuclear reactions and indicate the necessity of additional experiments to reduce the cross sections' determination errors.

In order to diminish the lack of EXFOR data base information regarding  $^{72}\text{Ge}(n,2n)^{71}\text{Ge}$  reaction cross sections, the excitation function of the specified nuclear reaction has been measured. For the first time such information can be introduced into the modern data bases.

All measurements have been carried out by neutron activation method. The samples in the form of foils of natural zirconium have been irradiated by DT-neutrons. Instrumental gamma-spectra of activation products have been measured by spectrometer with HPGe detector. The following issues are taken into account: instability of neutron flux in time, real geometry of experiment, the effect of true coincident summing of gamma-quantum during spectrum measurements of activation products and the effect of self-absorption gamma-quantum in a sample. The average neutron energy has been determined experimentally by Zr/Nb method [2].

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### Atom Probe Technique of Research of Subsurface Volumes of Irradiated Materials

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To the most powerful and modern methods of research of conductivity and semi conductivity materials by means of which it is possible to study directly a crystal lattice of solid states with the atomic spatial resolution, concern field ion microscopy (FIM) and various modifications of atom probes of a field ion microscope.

The purpose of the report is to show the efficiency of use of field emission methods for studying not only the radiation phenomena occurring in near-surface volumes of metals and alloys as a result of an irradiation, but also the atomic structure of various materials modified by intensive external influences. To show, how alongside with the defects of a crystal lattice typical for thermo mechanical, thermal, powerful deformation, etc. influences (mean point, linear, dislocation and other defects of a crystal lattice), in the investigated materials are observed such radiation-induced defects as: vacancy clusters, amorphous regions, segregation atoms of one of components in the ordered alloys, etc.

And, at last, one of the tasks of the report is to show the opportunities of new scanning atom probe tomography or an atom probe with a local electrode (LEAP 3000X Metrology System) manufactured by the IMAGO Scientific Instruments (6300 Enterprise Lane, Madison, WI 53719, 608.274.6880). Materials are examined by removing and analyzing individual atoms. Atoms are removed by either: (1) applying an ultra-fast voltage pulse or (2) simultaneously applying a high electrical field and an ultra-fast laser pulse. This

metrological system is a high-performance atom probe microscope providing 3D, atomic resolution, compositional imaging and analysis to research and industry. The optional Laser Pulsing Module expands the scope of applications to low electrical conductivity materials including semiconductors and ceramics. Recent experiments demonstrate that atom-probing with local electrode geometry provides a ~100 nm diameter field of view and collection rate of 20 million ions/hour. This allows for the analysis of a 100 nm diameter, 50 nm deep section of Si in 1 hour.

Work is executed with support of the Russian Foundation for Basic Research (Grant No. 07-02-00827-a).

### Energy Threshold of Low-Melting Material Spalling

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Absorption of pulse radiation energy (electron, X-ray, light, etc.) in materials can create mechanical stresses causing fracture of these materials (e.g. [1]). This loading method can be used to study dynamic strength of different materials. In this case, the dynamic load is understood as the maximum tensile stress that causes spalling of the material layer. Resistance of a material to spalling depends on its temperature. Strength of materials decreases when their temperature approaches the melting point [2].

For low-melting materials, amount of heat that shall be imparted to reach melting can be chosen a criterion characterizing their resistance to pulse radiation heating. The task of this work was to clarify a specific mechanism of low-melting material fracture: splashing (removal of the material melt) or spalling if absorbed energy is insufficient for melting. Difference between these mechanisms is in the heat of fusion.

Behavior of low-melting materials when irradiated by the electron beam was studied on accelerators IGUR-3 and EMIR available at RFNC-VNIITF [3]. Changes in the state of the samples surface were studied, thickness of the spalled layer was measured, and energy yield in the fracture section was determined.

For lead and solder POS-61, the average energy yield,  $Q$ , in the fracture section, which is calculated using measurement results, is given in Table 1 evidently showing that this value is independent of the radiation pulse duration in the range of electron pulse length (3...40)  $10^{-8}$  sec within the measurement error.

**Table 1. Mean energy yield in fracture section of lead and solder POS-61 with r.m.s. error**

Material	EMIR accelerator $Q, \text{J/g}$	IGUR-3 accelerator $Q, \text{J/g}$	Mean value $\text{J/g}$
Lead	$40 \pm 1.7$	$36 \pm 7$	$38 \pm 5.4$
POS-61	$36 \pm 5.8$	$41 \pm 6.3$	$38.5 \pm 6.7$

Table data show spalling to have the energy threshold at which the entire irradiated surface is removed. So, the least final thickness of the removed layer depends on the position

of the electron yield maximum that is in-depth of materials what is also confirmed by the analysis of irradiated samples [4].

The entire irradiated surface of the material is observed to separate in the location of the energy yield maximum with the electron energy transfer increase. The separated layer flows away being unmelted (Fig. 1b). The material layer is observed to remain intact along the irradiated-surface perimeter due to lateral unloading (Fig. 1a).

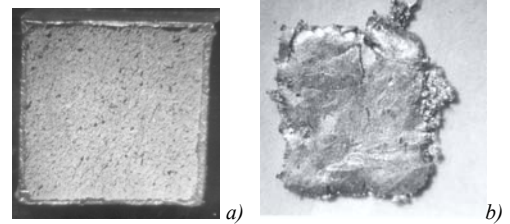


Fig. 1. Solder POS sample 4×4×1.5 mm characteristic damaged by electron beam: a – irradiated sample surface, ~16 J/cm<sup>2</sup>; b) separated part of same sample

The energy yield in the fracture section,  $Q$ , at the minimum thickness of the separated layer was compared with the amount of heat necessary to melt lead and solder POS-61 ( $Q_2 = c \cdot \Delta T + Q_{fus}$ , where  $Q_{fus}$  is heat of fusion,  $c$  is specific heat capacity, and  $\Delta T$  is heat-up amount), as well as with the amount of heat the material needs to reach its melting point  $Q_1 = c \Delta T$  (Table 2).

**Table 2. Comparison of threshold energy yield in fracture section at minimum thickness of removed layer with amount of heat lead and solder POS-61 need to melt, with r.m.s. error**

Material	$Q_1$ J/g	$Q_2$ J/g	$Q$ J/g
Lead	38.5	63.5	40 ± 2.5
POS-61	28.5	59.4	43.5 ± 5.4

From Table 2 it is obvious that the material is observed to remove over the entire irradiated surface when heated up to its melting point without accounting for its potential melting heat what is also confirmed by data in figure 1. So, the resulting conclusion confirms the conclusion from [5] which says that heating of low-melting materials up to the melting point is enough for them to loose their strength.

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### Field Ion Microscopy of Radiation Damages in FCC Metals Induced by Different Types of Radiation

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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 gaseous Ar<sup>+</sup> ions and fast neutrons. The FIM method gives an opportunity to perform direct precision investigation of material crystal lattice defects induced by intensive external influences.

Implantation of preliminarily FIM-certified needle-shaped samples was performed by 30-keV accelerated gaseous ( $\text{Ar}^+$ ) ions to doses (D)  $10^{14}$ ,  $10^{16}$  -  $10^{17}$  ion/cm<sup>2</sup> at ion current densities  $j = 70$  (T = 70 °C), 150 (T = 70 °C) and 200  $\mu\text{A}/\text{cm}^2$  (T = 200 °C), respectively. For study of defects generated under neutron bombardment, platinum samples were irradiated in the RWW-2M reactor at  $\sim 310$  K to fast neutron ( $E > 0.1$  MeV) fluences  $6.7 \times 10^{21}$  m<sup>-2</sup> and  $3.5 \times 10^{22}$  m<sup>-2</sup>.

Platinum irradiation with neutrons to fluence  $6.7 \times 10^{21}$  m<sup>-2</sup> resulted in formation in the material of a defect structure characterized by increased concentration of single point defects and their complexes comparable in size with interatomic distances. At fluence increase to  $3.5 \times 10^{22}$  m<sup>-2</sup>, radiation clusters (depleted zones with a “belt” of interstitial atoms) of average size 3.2 nm were formed in the irradiated platinum. The experimentally measured concentration of radiation clusters in the bulk of platinum was  $9 \times 10^{22}$  m<sup>-3</sup>.

It was found after ion implantation that, with fluence increase, the static and radiation-dynamic components of exposure cause transfer of the metal subsurface volume (not less than 20 nm into the depth from the sample irradiated surface) to a state which may be characterized as nanostructural by a number of essential features. Platinum nanostructurization in the subsurface volume was detected at  $D = 10^{17}$  ion/cm<sup>2</sup> only. At that, the state of metal varied from the nanostructural metal objects obtained after strong plastic deformations and mechanical fusion investigated earlier by the FIM and FIM tomographic atom probe methods [1, 2].

Work was carried out with the support of the Urals Division of RAS (the grant for young scientists and post-graduate students).

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### Tomographic Atom-Probe Microscopy of Reactor Materials

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Atom-probe investigations of VVER-440 reactor vessel steel show that the principal cause of embrittlement is formation of nanoclusters containing mainly copper and phosphorus. It was established that increase in nickel concentration and rise of irradiation temperature result in enhanced radiation-stimulated diffusion of phosphorus and may cause increase of its concentration on grain boundaries resulting in embrittlement by the intergranular mechanism. Investigations of the atomic structure of non-irradiated ferritic-martensitic steels hardened with yttrium oxides were carried out. Much lowered concentration of impurity elements inside grain was revealed. The investigations conducted have allowed the structure of clusters with increased concentration of Y and O atoms to be elucidated.



## **Ion-Beam Modification of Millimeter-Thick Subsurface Layers of Aluminum Alloys by Continuous Argon Ion Beams**

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The effect of accelerated medium-energy  $\text{Ar}^+$  ions on the mechanical properties and structural-phase state of aluminum alloys of different systems, AMg6 (Al-Mg), VD1 (Al-Cu-Mg-Mn) and 1441 (Al-Li-Cu-Mg) was investigated.

The samples for investigation were cut from strain-hardened clad 3-mm thick sheet alloy metal. In the course of irradiation, the following parameters varied: ions energy ( $E = 20$  and  $40$  keV), ion current density ( $j = 100 - 400 \mu\text{A}/\text{cm}^2$ ), irradiation dose ( $D = 1 \cdot 10^{15} - 2 \cdot 10^{17} \text{cm}^{-2}$ ); the target temperature was continuously controlled with a chromel-alumel thermocouple. The maximum temperature of samples heating did not exceed that of intermediate annealing performed for strain-hardening removal during rolling of the alloy of one or the other composition. As a rule, such kind of annealing takes 2 hours.

Strength characteristics of the original and irradiated samples were investigated. Multiparametric dependences of mechanical properties on ions energy, ion current density, and density of power applied to samples, irradiation temperature and dose were obtained.

It was established by the TEM method that, for the given alloys, the change of strength characteristics with increase of the irradiation dose, namely, a rise in plasticity accompanied with a considerable loss in strength characteristics, is governed by the processes of dynamic recovery and recrystallization during implantation.

The electron-microscopic examination of alloy structural state at sample sections parallel and square to the irradiated surface testify to the fact that radiation-induced structural changes take place not just in the thin subsurface layer comparable in thickness with the ions mean projected range (a few tens of nanometers), but throughout the whole depth of the 3-mm thick the sample, which is  $10^5$  times larger than the ions projected range.

The recorded structural changes occur at a high speed (within a few seconds under irradiation) and at a depth of  $\sim 3$  nm, which exceeds the ions projected range tens of thousand times and testifies to the fact of a radiation-dynamic contribution to the change in the structure of solids exposed to accelerated ion beams.

## Investigation of Electronic Structure of Pristine $^{13}\text{C}$ Isotope Powder and Composites based on $^{13}\text{C}$ Isotope for Neutron Target

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The creation of intensive source of high-energy neutrons based on proton accelerator is the important task of high-energy physics. The neutron target material creation is a principal part of the neutron source. Materials for making the neutron target should be with high tolerance to high temperature. While operating the neutron target should receive and dissipate 150–200 kW power from a beam at a spot 1 cm<sup>2</sup> size during continuous process. The carbon nanomaterial containing a lot quantity of the  $^{13}\text{C}$  isotope is the most perspective material for strict conditions. It was informed earlier [1] about research of structure and electronic properties of a carbon composite on the basis of an isotope  $^{13}\text{C}$  with density about 0.7 – 0.8 g/cm<sup>3</sup>. The aim of this work is to investigate the electronic properties of starting substance composed of  $^{13}\text{C}$  isotope and composites with higher density (up to ~ 1.55 g/cm<sup>3</sup>) on its basis, using the X-ray analysis, fluorescence spectroscopy, quantum chemistry calculation, microscopic investigation and conductivity measurements at low temperatures. The internal structure of the received material is represented extremely complex one and it is a combination several, sharply differing morphological forms of carbon. The analysis of the X-ray diffraction of the starting substance composed of  $^{13}\text{C}$  shows to presence of graphite particles with 20Å and 40Å thickness in equal parts. The X-ray fluorescence CK $\alpha$ -spectra of graphite and the starting substance composed of  $^{13}\text{C}$  were obtained. The density of C2 $\pi$ -state for the spectrum of substance composed of  $^{13}\text{C}$  in comparison with the spectrum of graphite is increased. The theoretical CK $\alpha$ -spectra of graphite composite based on  $^{13}\text{C}$  isotope agrees closely with experimental ones (Fig. 1).

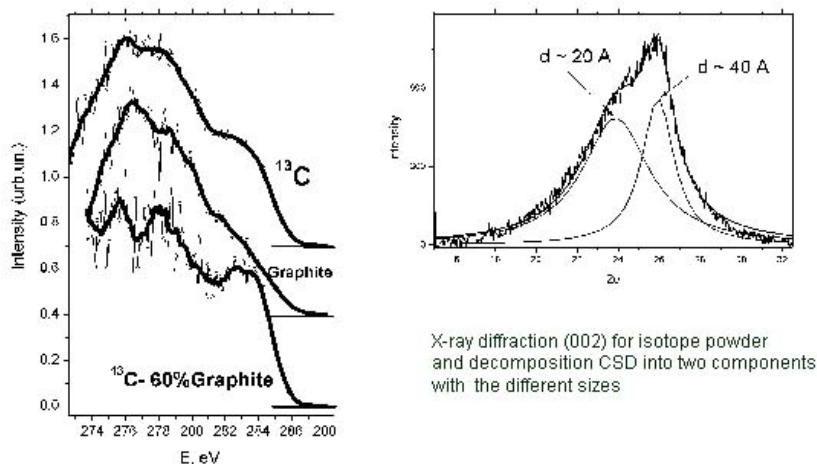


Fig. 1 Comparison of experimental CK $\alpha$ -spectra of  $^{13}\text{C}$  powder, graphite and their difference

It is represented, that the temperature dependence of conductivity for a pristine isotope powder  $^{13}\text{C}$  is given by  $\Delta\sigma(T) - \ln T$  and it is connected with a small fragments graphene layers. The decisive contribution to this temperature dependence by the quantum correction is brought. These quantum corrections are connected with the mechanism of the two-dimensional electron-electron interaction at least up to a temperature of 50 K.

The contribution of the quantum corrections connected with two-dimensional weak localization can be allocated also for an isotope composite with density  $1.55 \text{ g/cm}^3$  at temperature close to helium one. The possible reason of deviation of the temperature dependence of the conductivity from logarithmic law to the linear dependence can be not zero probability for carriers transition between graphene layers. That is connected with high faulted graphene layers and more complex microstructure of a composite based on  $^{13}\text{C}$  isotope. If the time of transitions between layers is less than the energy relaxation time then the three-dimensional Fermi-surface is formed [2]. The quantum corrections for such structure should be considered so, as well as for three-dimensional anisotropy conductors.

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### **Development and Investigation of Nanostructural Ferromagnetic $\text{Ni}_2\text{MnGa}$ -Based Alloys with Shape Memory Effects**

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Alloys  $\text{Ni}_{54}\text{Mn}_{21}\text{Ga}_{25}$  and  $\text{Ni}_{53,5}\text{Mn}_{20,25}\text{Fe}_{1,25}\text{Ga}_{25}$  were selected for investigation with a view to obtain nanostructural (nano- and submicrocrystalline) states in them. Investigations were carried out by the X-ray diffractometry and transmission electron microscopy methods in situ. Resistivity, thermo-emf and magnetic properties were also measured.

### **Technique of Preparation of Samples from Reactor Steels for Tomographic Atom-Probe Microscopy**

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The paper is dedicated to a technique developed for tomographic atom-probe microscopy of preparation of samples with shape meeting the following requirements: diameter 10 to 50 nm (depending on material), cone opening angle not over  $11^\circ$ .

The technique optimization was carried out on non-irradiated reactor steels of VVER-440 class. Preliminary data on elements distribution in them were gathered.

## Reprocessing of Nuclear Fuels in Chloride Melts: Some Thermodynamic and Electrochemical Aspects

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Pyrochemical reprocessing of wasted nuclear fuels includes among several steps the selective electroreduction of fuel components from dilute solutions of their chlorides in molten alkali or alkali-earth chlorides. The separation process of actinides and lanthanides can reduce the radioactivity of regenerated fuels and minimize the loss of fissionable species. Thus specific features of lanthanides electrochemical behavior in such electrolytes are now the subject of great interest. One of the main metal ion thermodynamic characteristics in such solution is standard conditional potential, the value of which measured versus chlorine electrode corresponds to the Gibbs energy of metal chloride formation in the solution.

The thermodynamic functions of lanthanide di- and trichlorides formation in two molten solutions: equimolecular mixture NaCl-KCl at 973K and eutectic LiCl-KCl at 723K were estimated on the bases of experimental data. The data for metals with ions having the same electronic structure as the corresponding lanthanide ions also were under consideration. For  $\text{Ln}^{2+}$  they are  $\text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Ba}^{2+}$  ions and for  $\text{Ln}^{3+}$  -  $\text{Sc}^{3+}$  and  $\text{Y}^{3+}$  ions. The thermodynamic solvation functions for  $\text{M}^{3+}$  (M – Sc, Y, Ln) and  $\text{M}^{2+}$  (M – Ca, Sr, Ba, Ln) in solutions referred to above were calculated by means of thermochemical cycle method. Empirical relationship among values of thermodynamic solvation functions and ionic  $\text{M}^{n+}$  radius were estimated.

Such calculations make it possible to estimate the stability of  $\text{Ln}^{3+}$  and  $\text{Ln}^{2+}$  in equilibrium with metal under given conditions and the values of the corresponding standard conditional  $E^* \text{Ln}^{3+}/\text{Ln}$ ,  $E^* \text{Ln}^{2+}/\text{Ln}$  and red/ox  $E^* \text{Ln}^{3+}/\text{Ln}^{2+}$  potentials.

## Recording Dispersion of Speed of Iron and Steel Samples Free Surface by the Optical Lever Method

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The results are presented of comparative analysis of records of experiments with loading of iron and steel samples with tangential and normal to surface detonation of explosive charges of different power and thickness, with recording of multiwave configurations at compression with the application of photochronographic technique of optical lever.

### Multi-pass Mode of Bremsstrahlung Generation

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The work is underway to create the intense bremsstrahlung sources with 20-100 keV quanta energy to study how bremsstrahlung impacts electronics materials and elements. A key problem in the process is the target assembly design, which shall both provide the maximum possible efficiency of electronic beam energy conversion into bremsstrahlung, and create large-area fields (up to  $1\text{m}^2$ ) with uniform radiation intensity. The multi-pass mode of bremsstrahlung generation is considered as a candidate solution of this problem. Its essence is that bremsstrahlung is generated on a thin tantalum target placed in the uniform magnetic field, which forces accelerated electrons to move the spiral paths and multiply interact with the target.

The ways of realizing the multi-pass bremsstrahlung generation mode differ mainly in mutual orientation of the external magnetic field, the injected beam direction and target position [1, 2]. This paper presents the version when the electron beam incidents at a slight angle ( $\sim 6^\circ$ ) as though it slides over the flat target, the beam axis and the direction of the external magnetic field being collinear [3]. The experiments in this geometry were performed on the pulse-periodic electron accelerator PPEA to estimate efficiency of the multi-pass generation mode, and on the pulse accelerator RAPID-M to study conditions of electron beam transportation to the target

The experiments on the PPEA showed that in the multi-pass generation mode, the bremsstrahlung dose increases no less than twofold, as compared to the one-pass mode [4]. In these experiments, the amplitude value of the beam current was not more than 1 kA and this eliminated problems with its transportation over the drift chamber under vacuum conditions.

Current amplitude in the accelerating tube of installation RAPID-M is 100...150 kA, i.e. it by far exceeds the limiting Alfvén current. Transportation of this electron beam without significant losses requires its charge and current to be neutralized [5]. In the performed experiments, these conditions were satisfied through filling the drift chamber with nitrogen. The obtained results show that in the gas-filled drift chamber under the pressure of  $\sim 100$  Pa and the external magnetic field with the 0.3 Tl induction, the electron beam is transported to the target practically without losses.

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### **Ion-Plasma Etching Method Application for Uranium Microstructure Investigation after Shock-Wave Loading**

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The paper discusses the features of preparation of uranium samples exposed to shock-wave loading, and different methods of revealing microstructure are compared. The paper gives the results of testing of optimal modes of ion-plasma etching, and the electron-microscopy images of the obtained microstructures.

### **ASU-TP for Corrosion Inspection Stand**

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Soecialists of RFNC-VNIITF have engineered a thermo-convection loop automatic control system (ASU-TP “Petlya”) with respective software for monitoring the thermo-convection loop temperature conditions and control of its parameters.

### **On the Opportunities of Application of the Methods of Pattern Identification in Material Structural Features Decoding**

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The paper presents the results of automatic identification (detection) of patterns with preset properties on static images obtained by digital methods.

### **The Formation of Nanodimensional Intermetallics in Nickel and Titanium during Implantation of Metal Ions**

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At the present time there is a tremendous growth in nanotechnology. Refining of polycrystalline grains and reduction in size of metal and ceramic materials increase their yield limit and deformation strength. The second possibility to improve the strength is going from ordinary metals and alloys to materials with tight interatomic bindings, typical representatives of which are intermetallic compounds. This is the only kind of materials with the metal binding whose the deformation strength can increase with increase in temperature. Intermetallic compounds of Ti-Al, Ni-Al, Ni-Ti systems, etc., among the plasticity, hold

their structure and strength at high temperatures, and possess good anticorrosion properties. Intermetallics whose grains have nanodimensional parameters are presently poorly known, however the prospects for using covers that consist of nanointermetallics are extremely encouraging. Considerable recent attention has been focused on the use of ion implantation for synthesis of intermetallic phases in surface layers of metal target. Micron thick surface layers formed in the condition of the high intensive ion implantation are several orders greater than the magnitude of the projective ion path. This is due to the high temperature in the process of implantation and the high rate of diffusion of a doping impurity from the target surface layer into the depth of a material. The above-listed advantages benefit the formation of nanodimensional intermetallic phases in modified layers.

The results obtained from the complex experimental investigation of the formation of nanodimensional intermetallic during ion implantation of metals (Al, Ti) into pure metals (Ni, Ti) are presented. It is found that during ion implantation nanodimensional intermetallics are formed in the surface layer of a metal target. Intermetallics are formed in accordance to the phase diagrams of Ti-Al, Ni-Al, Ni-Ti systems under study. The influence of parameters of ion implantation on the microstructure of surface ion-doped layers of targets and mechanical properties are considered.

The results of research performed in ISPSM of SB of RAS and TSABU on the program of integration projects of the Siberian Branch and the Ural Branch of RAS are presented in the paper.

### **The device on the alpha-activity detector from firm «Spectrum Science» Model 1950**

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The device Model 1950 from firm “Spectrum Science” is a simple pulse counter. Part of electronics is located in the system block of a computer. The power supply, a high-voltage source, a preliminary amplifier, a scaling amplifier, an analog comparator are located on the back wall of the detector chamber. The device is supplied by adjusting elements for change of discriminator threshold and working high-voltage feed.

The changes of electronics are required for decrease of account speed level and for expansion of measurement opportunities, especially for signal processing block. Some block diagrams of signal processing block of the containing various or identical basic units, collected on various block diagrams are presented, the information on influence of these changes on detector working is processed.

The device No. 2 has background level  $4.2 \pm 0.8$   $\alpha$ /hour and is supplied by the new amplifier, new high-voltage and low-voltage power supplies. The DC-DC converters are used in the power supply owing to galvanic outcome from a network 220B with small through passage capacity is provided.

The device No.3 has background level  $3 \pm 0.6$   $\alpha$ /hour and is supplied by the specialized preliminary amplifier with the frequency and pulse characteristics which calculated and have been picked up for the measured signal. Due to sharp selectivity of the

first cascade of strengthening sensitivity and noise immunity are improved. The scheme of anticorrelations for reducing handicaps level by a network 220V, high-voltage source and poor-quality grounding is used in the device. Reduction of false impulses is promoted by a full galvanic outcome of "ground" TEC and the strengthening block, the arrangement of the low level discriminator after the preliminary amplifier, instead of after cascades of strengthening. The device has satisfactory background fluctuations and measurement well generating for enough greater detector area.

### **Estimation of background constancy of device Model 1950 from firm «Spectrum Science» for low alpha-activity of materials measurements**

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The device Model 1950 of firm "Spectrum Science" presents a flowing proportional counter; it has the detector area of 1000 cm<sup>2</sup>. The estimation of device background constancy, reproducibility of low levels alpha-activity measurements is represented in this study. The analysis of device operation is investigated on information basis collected from June 1996 till May 2006. The background of the device was measured not less than 1 time a week for 48-96 hours.

The dependences of average speed account on years of device service and on dates of working are resulted in the study. Dependences of account speed fluctuations of background on duration of its measurement are shown.

The stream density measurements of samples with alpha particles from a surface 0.05 α/hour/cm<sup>2</sup>, 0.02 α/hour/cm<sup>2</sup>, 0.01 α/hour/cm<sup>2</sup>, 0.005 α/hour/cm<sup>2</sup>, 0.002 α/hour/cm<sup>2</sup>, 0.001 α/hour/cm<sup>2</sup> are presented. It is shown, that stream density measurement of samples with alpha particles from a surface down to 0.005 α/hour/cm<sup>2</sup> does not cause greater difficulties. The measurements of samples with density less than 0.005 α/hour/cm<sup>2</sup> require as long time measurement as alternation of measurement of device background and measurements of background with sample.

### **The device for measurement of ultra-low superficial alpha-activity of materials: development and measurement technique**

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In 1999-2006, the "Pure technologies" JSC, supported by the Foundation for Assistance to Development of Small Forms of Enterprises in the Scientific and Technical Sphere, carried out research into the radiating properties of microelectronics materials and created a device for measuring alpha-activity of the materials used in flip-chip



technology [1]. The purpose of research was to design and create an instrument for measuring ultra low alpha-activity of materials with sensitivity of 0.0005 alpha counts per 1cm<sup>2</sup>/hour. The separate components model tests and characteristics researches of a pre-production device model were conducted, a measurement technique was developed.

As a result of the research, an instrument for measuring alpha radiation level of materials and products with sensitivity 0.0005 alpha counts per 1cm<sup>2</sup>/hour with the working area of 1500 cm<sup>2</sup> was created. The low background and the automatic space background component account of the device allows measurements of ultralow alpha-activity of samples during comprehensible time (24-96 hours) with sufficient accuracy.

High sensitivity and accuracy of the device are ensured by the developed measurement technique including the way of selection and preparation of materials samples, the alternate account of background and sample activity, and design of result activity count procedure.

The application efficiency of the device is defined by its high sensitivity, the great area and low own background. The device can be applied to measurement of ultralow alpha-activity of microelectronics, geological and ecological materials.

The patent for the invention 2269839 "Detector of alpha radiation" is granted.

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## Neutron Physics Studies of Some Problems of Nuclear Material Science

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Foundations of the method of magnetic resonance and relaxation of polarized beta-active nuclei ( $\beta$ -NMR), method of multiple small angle neutron scattering (MSANS) and recently developed method of taking into account the correlations in the scatterers positions are considered. Next problems are discussed especially:

- 1)  $\beta$ -NMR: studies on atomic scale of radiation damage structure and their annealing kinetics, structure of substance and atomic diffusion, and influence of electronic structure on nuclear spin-lattice relaxation;
- 2) MSANS: investigation of structural inhomogeneities on the scale of 10-10000 nm;
- 3) theory and numerical simulations of the measuring processes and corresponding materials properties.

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### **Ion Implantation for Formation of Nanosize Structures**

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The review presents the results of creation of nanosize elements and structures with the help of ion beams exposure for nanoelectronics and nanophotonics.

### **X-Ray Methods of Nanostructure Parameters Investigation**

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Using the method of relative X-ray reflectometry, the parameters of a discrete laminate structure were measured for the first time. There is given a description of X-ray optical measurement system utilizing two wavelengths, the conditions of the method application and the algorithm of data processing are substantiated. The results of measurements are presented of a test object obtained by magnetron deposition of Ta on a Si substrate through a mask with openings arranged in a regular manner. It was shown that the developed scheme may be used in semiconductors engineering for control of nanosize-thick instrument structures parameters.

### **The new Method of Radiation Modification of Materials – the Formation of Cluster Structures**

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The nonequilibrium states with unusual properties are formed in metallic materials under ion fluxes irradiation. These materials (model or technical alloys and also some intermetallics) were irradiated to high level of radiation damages. The phenomenon was observed within some range of radiation parameters (doses, the target temperatures and ion flux intensities).

The main signs of the states are the following:

- specific nanocluster morphology (the initial matrix contains the little – 3-4 nm – clusters which occupy the significant part of the volume;
- characteristic changes of X-ray diffraction pattern;
- anomalously large changes in material properties.

It can be supposed the effect is of the universal type as this is observed in numerous metallic alloys including the alloys of the nuclear power engineering (based on the Fe-Cr-

Ni, Ni-Cr, Fe-Cr, V-Ti-Cr systems) and also in the some intermetallics (in the Heisler alloy Cu<sub>2</sub>Al Mn).

The nonequilibrium states appearance is accompanied by strong changes of material properties – the microhardness increases several times in the metallic alloys and also the magnetization increases three times in the Heisler alloy.

X-ray diffraction analysis, Moessbayer researches and also computer experiments by the molecular dynamics method permit us to propose the idea of the local restructuring of the crystal lattice in a vicinity of the radiation defect (a vacancy). As a result the cluster with the symmetry different from a matrix forms. Such clusters have the icosahedral symmetry in the metallic solid solutions with the FCC crystal lattice.

So it is possible to obtain the structure by the radiation modification in which the initial matrix is pierced by the clusters consisting of same atoms but having the different from matrix symmetry.

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### **Radiation-Ion Technology of Formation of 1-, 2- and 3-D Nanostructures**

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The paper gives the results of experimental works carried out in the Flerov Nuclear Reactions Laboratory along the lines: oriented basic research of mechanisms and features of formation of ion tracks in semiconductors (Si), methodology of formation of 1-D single-crystal semiconductor plates; metal 2-D nanostructures (nanowires, submicron tubes); 3-D monodisperse nanosize structures (precipitate phases) of extremely high concentrations on the surface and in the bulk of a solid.

### **Dynamics of the Propagation of Extended Defects in Metals during Ion Implantation and Deformation**

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In studies of deformation processes, the destruction of materials, and the evolution of a defect structure under the action of ion implantation, the computer simulation makes it possible to obtain both qualitative and quantitative information on the going physical processes. It allows the use of the simulation not only for the solution of fundamental physical problems but for a search for optimal manufacturing processes of ion implantation. In the present work a comparative analysis of the propagation of a microcrack and a

micropore in metals with different types of lattices (bcc, fcc, hcp) under uniaxial tension prior to and after ion irradiation was made by the method of molecular dynamics. The aim of this work is to study the microscopic mechanisms of surface hardening under ion irradiation and the dynamics of behaviour of microdefects under deformation.

The computer experiment was performed with the use of a program based on the method of molecular dynamics. Potentials of the pairwise interaction calculated from the Heine–Abarenkov–Animalu pseudopotential were used to describe the interaction between atoms. The initial configuration of atoms of a crystallite was a three-dimensional film of 20 atomic layers in thickness. Defects were simulated by the removal of part of atoms from the model crystallite. The relative deformation in tensile strain was  $\varepsilon = 5\%$ , 10%, 15%, 20% and 25%.

In pure (impurity-free) metals Fe, Al, Ti, Ni a microcrack and a micropore are rebonded during ion irradiation. Experimental results show that mechanisms and the degree of rebonding of microcracks during ion irradiation in metals depend primarily on the type of the crystal lattice. An increase in strength of a crystallite is observed for all of the irradiated samples.

From the obtained results it may be suggested that the process of rebonding of microdefects during ion irradiation plays a leading role in the change of the strength of surface layers of metals. In this case rebonding of microcracks and micropores occurs at the final stage of the development of the collision cascade at the mouth of a crack or at the boundary of a pore. The development of the collision cascade (at the stage of the heat spike) results in disappearance of microdefects in the case of their small sizes and increases the radius of the curvature at the mouth of a crack if microdefects are of a relatively large size.

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### **The Change of Metal Foil Surface Layers on Photon Irradiation**

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Recently, the influence of various fields on the structure of materials is the subject of wide discussion in literature. In this work the changes of a metal foil on light irradiation are discussed. The aim of the work was the comparative analysis of processes accompanying irradiation of rolled foils from different materials with light beams.

Rolled foils 40%Ni – 60%Cu, Cu, Ta of 30-50  $\mu\text{m}$  in thickness were investigated. Samples were irradiated with a 100 W halogen lamp of in the air at a fixed distance between the lamp and the sample ( $R = 70$  mm). For increasing the heat removal, the sample under study was fixed on the massive metal plate. The microhardness was measured with the help of the device PMT-3 (the load on indenter was 20 g). The depth distribution of components in surface layers prior to and after irradiation was studied by the secondary ion mass spectroscopy (SIMS) with the use of the surface sputtering by argon ions with the energy of

4,5 keV and the current density of  $20 \mu\text{A}/\text{cm}^2$  (the rated speed of sputtering was 3 nm/min) and the X-ray electron spectroscopy.

The experimental results show the change of the structure and the chemical composition of the irradiated foils. Irradiation results in the change of the microhardness values of samples.

These changes depend largely on the type of an irradiated material. The SIMS results show that light irradiation of studied foil surfaces causes the redistribution of elements in surface layers. The electron structure of atom inner levels was studied by the XES method. Some differences observed on these levels point to the change of the character of the interatomic interaction between elements involved in the composition of the studied alloy. The role of atoms adsorbed on the surface (partly, atoms of oxygen) was shown.

Mechanisms of observed phenomena are discussed on the base of the experimental results and the data from literature.

The work is sponsored by the Integration project of Physical-Technical Institute of Ural Branch of RAS and Institute of Physics of Material Strength of Siberian Branch of RAS

### **The Change of Mechanical Properties and the Surface Composition of the St3 Carbon Steel Depending on Ion Irradiation Parameters**

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The surface hardening of relatively inexpensive steels and alloys for producing parts of machines and mechanisms that operate under severe conditions is one of the trends in the present-day machine building [1]. This enables savings in expensive high-alloy steels and alloys and makes a product cheap. Methods of ion and ion-flame treatment by strong-precise beams are used and studied widely [2]. But in a number of cases it is impossible to use intense ion or ion-plasma treatment because of the high temperature condition arising from this action. At the same time both in the world science and the native one there are a lack of systematic and complex investigations of the comparative study of the influence of poor-precise beams on service characteristics of metals and alloys.

We have studied before the effect of radiation modes (radiation dose, ion current density) and the type of ions on fatigue strength of OT4-1 and OT4 pseudo- $\alpha$ -titan alloys [3,4]. Microhardness and fatigue strength have been found to change non-monotonically depending on the type of ions, the radiation dose and the ion current density. The increase of fatigue strength of the OT4 titan alloy is due to increasing the presence of defects in the surface layer structure and smoothing the surface in ion implantation. This determines the change of the mechanism of the crack initiation and the crack propagation that occurs not from the surface like in initial samples but in the depth of 10-30  $\mu\text{m}$ .

In this work the results of the comparative study of fatigue strength, microhardness, the surface layer composition, and morphology of sample surfaces of the St3 carbon steel,

which have been radiated by  $\text{Ar}^+$  and  $\text{N}^+$  ions ( $E = 40 \text{ keV}$ ,  $j = 10 \text{ } \mu\text{A}/\text{cm}^2$ ) in terms of the radiation dose ( $D = 10^{15}$ ,  $10^{16}$ ,  $5 \times 10^{16}$ ,  $10^{17} \text{ ion}/\text{cm}^2$ ).

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### The Change of the Chemical Composition of Ni-Cu Foil Surface Layers in Ion Implantation

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An effect of the implantation of boron and argon ions at different ion current densities on the chemical composition and the defect structure of Cu-Ni foils has been studied by X-ray photoelectron spectroscopy, X-ray crystal analysis, and micro hardness measurement methods.

The  $\text{Ni}_{20}\text{Cu}_{80}$  rolled foil of 40  $\mu\text{m}$  thick has been used as an object under study.

An analysis of the chemical composition of samples radiated by the X-ray photoelectron spectroscopy method has shown that the nonmonotonous oscillating relation between the alloy composition and the depth concentration of implanted ions is formed in radiated samples. Implantation of the chemically active element, Br, results in higher Cu contents in the subsurface layer of 20 nm thick. Analysis of X-ray spectra has revealed the presence of metal borides. Investigations of micro hardness of implanted foils as the structure-dependent parameter of a material have revealed strengthening of both the radiated foil side and the nonradiated one.

It is believed that the results we have obtained testify that the system under study is the nonequilibrium one and its consideration must not be limited by the classical thermodynamic.

The explanation of the observed composition separation due to induced radiation segregation and predominant binding of atoms of the definite sort with flows of defects are suggested. Besides, the defective structure of rolled foils is the initially very nonequilibrium, stressed structure with the high density of dislocations. Ion implantation causes the transformation of the defective structure that tends to the local gradients of mechanical stresses. Their presence is one more cause that gives rise to flows of defects and hence atoms of a matter.

The revealed formation of the oscillating nonmonotonous relationship of the Ni/Cu concentration ratio and the Br concentration in surface layers is attributable to the change of the defective structure of foils under radiation giving rise to local gradients of mechanical

stresses that in its turn is due to the diffusion redistribution of components of the system. The formation of the oscillating distribution of components of the system in surface layers from the nonirradiated side can be connected with the extended change of the defect structure of the foil with the result of the effect of shock waves, which form under ion bombardment.

### **Review of the effects of dose rate on swelling in austenitic alloys and its essential mechanism**

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In this presentation, we will present the summary of our previous studies of the experimental evaluation of the dose rate effects on microstructural evolution and swelling in austenitic alloys. Several kinds of model austenitic alloys were neutron-irradiated at several positions in FFTF/MOTA, which enabled us to obtain two orders of magnitude difference in dose rate. In the pure Fe-Cr-Ni ternary and in the Ti-modified quaternary alloy, low dose rate irradiation strongly enhanced swelling by shortening the incubation dose for the onset of the steady state swelling rate. It is surprising that the incubation dose was found to be linearly proportional to the dose rate. On the other hand, the steady state swelling rate of 1%/dpa is not affected by the difference in dose rate. When 0.05% C is added to the Ti-modified alloy, the dose rate effects disappear, and the swelling is only a function of the cumulative dose.

Based on these findings, we present the essential mechanism to evaluate the dose rate effects and show that the dose rate effect arises primarily in the Frank loop population, not in the void population.

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