

Восьмой Международный Уральский Семинар

# РАДИАЦИОННАЯ ФИЗИКА МЕТАЛЛОВ И СПЛАВОВ

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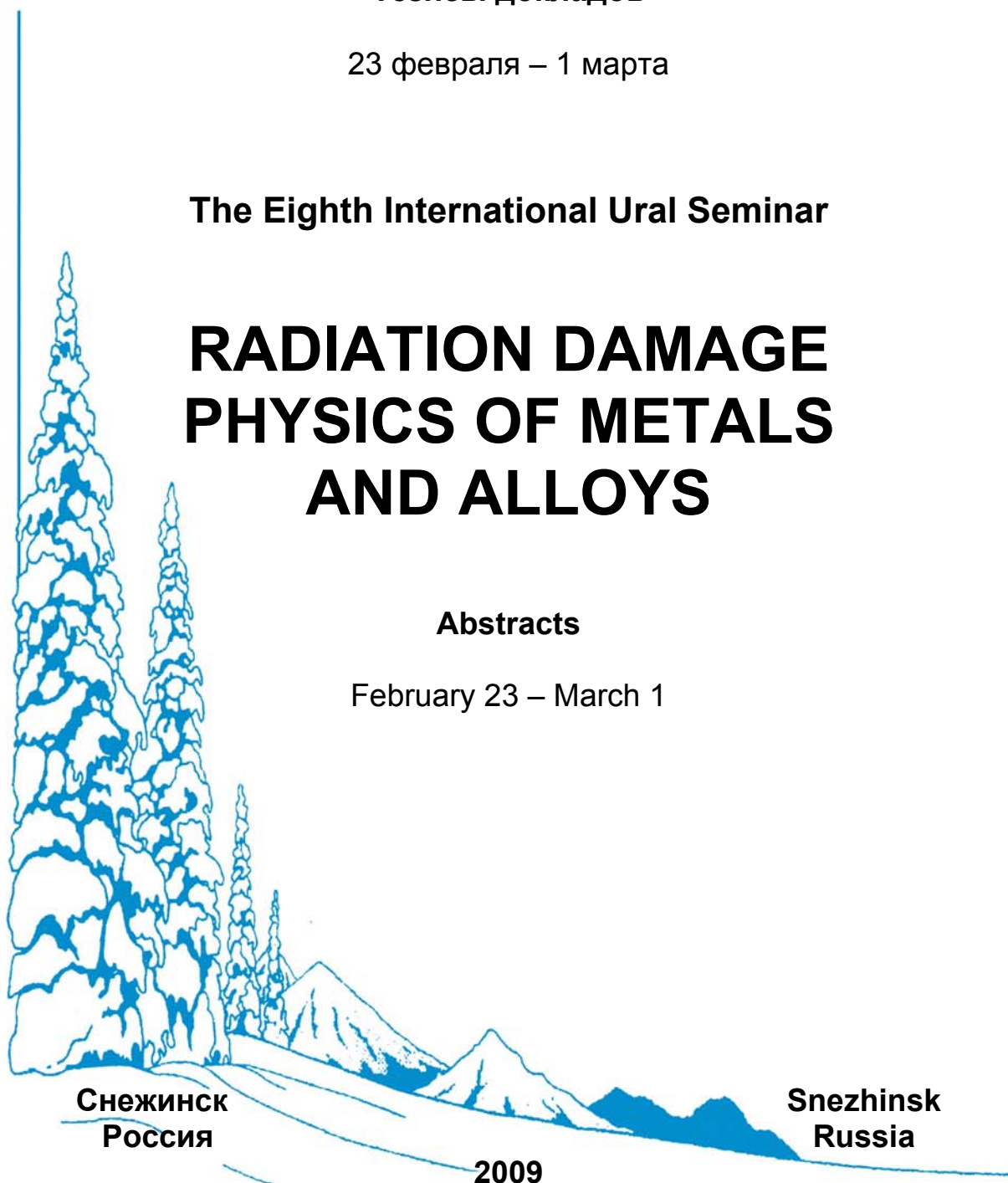
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The Eighth International Ural Seminar

# RADIATION DAMAGE PHYSICS OF METALS AND ALLOYS

Abstracts

February 23 – March 1



Снежинск  
Россия

Snezhinsk  
Russia

2009



**The Eighth International Ural Seminar**

**RADIATION DAMAGE  
PHYSICS OF METALS  
AND ALLOYS**

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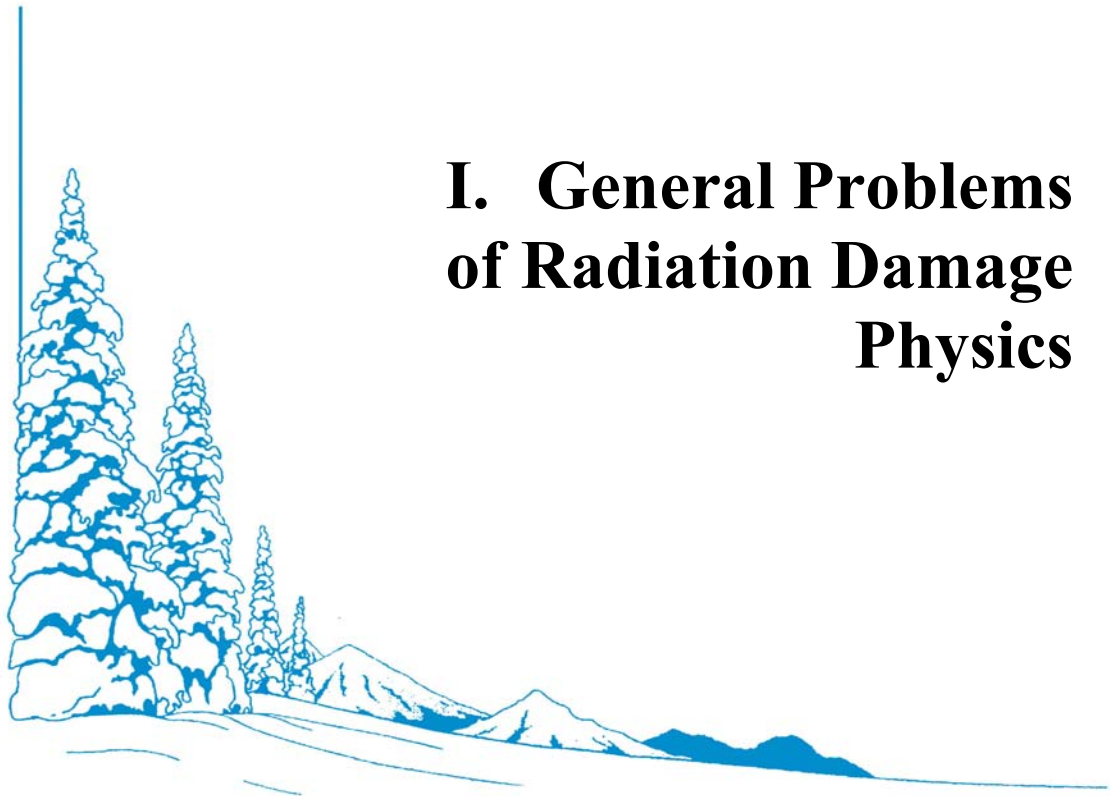
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# I. General Problems of Radiation Damage Physics

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, vacancy pores 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.



## **Temperature Dependence of Radiation-Induced Processes in Fe-Ni and Fe-Ni-Si Alloys with Different Microstructures**

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The method of residual resistivity was used to study processes of radiation-induced separation of the solid solution in the Fe-34.7 at.% Ni (Fe-Ni) alloy and the solid solution decomposition, which was followed by formation of pre-precipitates, in the Fe-33.1 at.% Ni-5.6 at.% Si (Fe-Ni-Si) alloy at different temperatures and in different initial states under electron irradiation. The alloys were made by vacuum melting of their pure components. The alloys were compared in the following states: quenched from 1373 K; aged at 780 K; deformed to 40%; deformed, but annealed at 573 K to remove vacancy clusters. Dose and temperature dependences were determined. The alloys were subjected to isochronal and isothermal annealing. It was shown that concentration inhomogeneities in the matrix of the aged Fe-Ni alloy were not significant sinks of point defects. Deformation considerably suppressed processes of the radiation-induced separation of the solid solution in the deformed Fe-Ni alloys. This was explained by a large concentration of point defect sinks mostly arising from the dislocation structure. Vacancy clusters of the deformation origin and radiation-induced clusters dissociated over one and the same temperature interval of 350-500 K in both alloys. It was shown that the decomposition of the solid solution in the Fe-Ni-Si alloy, which showed up as the growth of the electrical resistance, was thermally activated at temperatures of 600-900 K. At lower temperatures the solid solution in this alloy decomposed either under irradiation by means of migrating point defects or during annealing after irradiation or deformation because of the dissociation of vacancy clusters. Similarly to the Fe-Ni alloy, the radiation-induced decomposition of the solid solution in the deformed state was considerably suppressed on account of a high concentration of point defect sinks represented by dislocations. The analysis demonstrated that the variation of the electrical resistance is described in terms of the statistical model for the Fe-Ni alloys and the matrix model for the Fe-Ni-Si alloys.

This study was supported by the Project ISTC 3074.2, RAS Program № 01.2.006 13394, RFBR (projects No. 07-02-00020-a and No. 07-02-96052-r\_ural\_a).

## **The Effect of Alloying Elements on the Vacancy Defect Evolution in Electron-Irradiated Austenitic Fe-Ni Alloys**

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The vacancy defect evolution under electron irradiation in austenitic Fe-34.2 wt.% Ni alloys containing oversized (aluminum) and undersized (silicon) alloying elements was investigated by positron annihilation spectroscopy at temperatures between 300 and 573 K [1]. It is found that the accumulation of vacancy defects is considerably suppressed in the silicon-doped alloy. This effect is observed at all the irradiation temperatures. The obtained

results provide evidence that the silicon-doped alloy forms stable low-mobility clusters involving several Si and interstitial atoms, which are centers of the enhanced recombination of migrating vacancies. The clusters of Si - interstitial atoms also modify the annealing of vacancy defects in the Fe-Ni-Si alloy. The interaction between small vacancy agglomerates and solute Al atoms is observed in the Fe-Ni-Al alloy under irradiation at 300 - 423K.

This study was done within RAS Program (project № 01.2.006 13394), with partial support of Russian Foundation for Basic Research (project Nos. 07-02-00020 and 07-02-96052).

### References:

1. Druzhkov A.P., Perminov D.A. and Davletshin A.E. *J. Nucl. Mater.* to be published (2009).

## In-situ TEM and Ion Irradiation of Ferritic Materials

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We will review several ongoing experimental programs on the irradiation response of ferritic materials: pure Fe, Fe-Cr alloys, and ODS (oxide dispersion strengthened) alloys. These programs utilize the IVEM-Tandem user facility at Argonne National Laboratory to perform TEM on in-situ ion irradiated samples at controlled elevated temperatures. Direct real time observations of defect formation, motion, and coalescence into extended microstructures will be illustrated.

## Features of Structural State of Radiation-Resistant Construction Materials

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The principal factor damaging the material of fuel element shells is a neutron flux. The neutron flux causes appearance of vacancies, interstitial atoms and their complexes in the metal crystal lattice. The rate of radiation-induced defects accumulation may be attained through acceleration of recombination of vacancies and interstitials on sinks (grain boundaries, dislocations, excessive phases, etc.).

The paper is dedicated to investigation and analysis of a mechanism of efficient acceleration of the process of recombination of vacancies and interstitials on distortions created in the structure of transition metals in the course of formation of a nano-domain short-range order (Figure).

In the alloy Ni-41Cr-1Mo featuring a short-

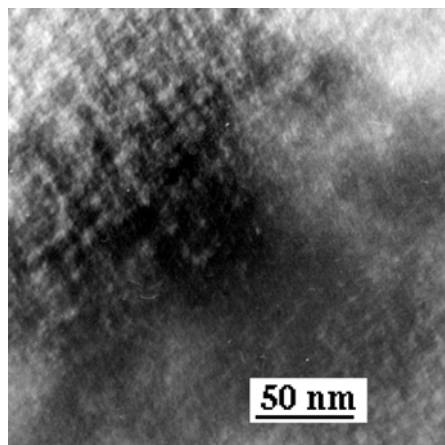


Fig. Short-range order in alloy Ni-41Cr-1Mo after quenching and ageing at 450 °C during 9000 h



range structure, when irradiated to  $>32$  dpa at  $\sim 350$  °C, the number of vacancies and interstitials was by an order of magnitude smaller than in the alloy with a stable structure, and the total elongation was 3 times larger.

Investigation of radiation damage under irradiation with electrons by the positron annihilation method also showed that the rate of vacancies and interstitials recombination in the alloy forming a short-range order becomes equal to the rate of their generation.

### **Difference Approach to Analysis of Resistivity Recovery Data in concentrated alloys undergoing the short-range order formation**

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A resistivity study of point defect properties in electron-irradiated concentrated Fe-Cr alloys has been started from Cr4 and Cr9 elsewhere (Phil. Mag. **31**, 4847, 2007). Commonly, the short-range order (SRO) formation enhanced by defect migration is the main factor complicating fundamentally the analysis of the resistivity recovery (RR) data in concentrated alloys as compared with that in pure metals and dilute alloys. In contrast to the rest of Fe-Cr alloys, the signs of the SRO formation were not pronounced in RR of Cr4 and Cr9. This favourable moment allowed analysing their RR data in a simplified manner neglecting any complicating effect of the SRO formation.

The analysis of the RR spectra in the samples of both alloys did not reveal any peaks shifting their temperature positions with a change in the initial defect concentration. The latter feature is a signature of stage III (the onset of vacancy free migration) and its missing implies the absence of a peak of stage III in the RR spectra. At the same time, the signs of vacancy migration were clearly detected by the positron lifetime spectroscopy in the same and close alloys.

To clarify this contradiction, a new approach to the analysis of RR data was proposed. A new quantity was introduced, the so-called difference RR (DRR), i.e. the difference between RR dependencies of two similar samples having different initial defect concentrations:

$$\delta R(T) = R(T)_H - R(T)_L,$$

where  $R = \Delta\rho/\Delta\rho_0$  and  $\Delta\rho$ ,  $\Delta\rho_0$  are the current and initial resistivity increments,  $T$  is annealing temperature, indexes  $H$  and  $L$  refer to the samples having *Higher* and *Lower* initial defect concentrations. It has been demonstrated that the stages of free migration manifest themselves in the DRR plot by the emergence of a specific peak, called the DRR peak, which corresponds in physical terms to a partial (separated) RR peak of the related stage of free migration. The other stages having the different origin do not give rise to any peaks.

Features of the DRR peaks revealed in the analysis of the RR data of Cr4 and Cr9 indicated that the stage III peak had never been observed in the conventional RR spectra of Fe-Cr alloys before (see, e.g., Mater. Sci. Forum **15–18**, 1263, 1987). The partial RR rate in stage III turns out negligible in Cr4 and even inverted (i.e. negative) in Cr9, while only peaks

characterised by a non-negligible positive partial RR rate are taken into consideration in the conventional RR method. The DRR data allowed revealing the actual structure of the RR spectra and finding the position of a stage of the onset of long-range migration of vacancies at  $\sim 205$  K and that of interstitial atoms at 220 K. The abnormal character of stage III was assigned to the anomalously large specific resistivity of immobile di-vacancies formed in the stage.

It becomes clear that because of the abnormal character of stage III the analysis of RR data in the rest of Fe-Cr alloys liable to the SRO formation becomes improper beyond the context of the DRR approach. That is why we undertake an effort to analyse and formulate the regularities of RR in alloys undergoing the SRO formation in terms of DRR. In our reasoning we based on the regularities of resistivity variations induced by the SRO formation found before (J. Phys. F: Met. Phys. **14**, 793, 1984; Acta Metall. **33**, 1887, 1985 and references therein).

The results of the analysis and obtained formulations are illustrated by the analysis of the available RR data of Fe-16Cr-20Ni and Fe-4Cr in terms of DRR. A small contribution ( $\sim 2-3$  %) to RR of Fe-4Cr induced by the SRO formation not resolved before was revealed within the DRR approach. It has been demonstrated that even such small SRO-induced contribution to RR is sufficient within the DRR approach to detect and identify stage III, which is not seen by other experimental means in Fe-4Cr.

### **Resistivity Recovery, Short-Range Order Formation and Defect Migration in Low-Cr Fe-Cr Alloys**

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This work is a continuation of the study on resistivity recovery (RR) fulfilled in electron-irradiated Fe-Cr4 and Fe-9Cr (Phil. Mag. **31**, 4847, 2007). The original RR data of Fe-6Cr and Fe-4Cr doped with carbon ( $\sim 0.1$  at. %) after low temperature ( $< 80$  K) electron irradiation (5 MeV) at several initial defect concentrations are presented.

The RR spectra of Fe-6Cr are qualitatively similar to those obtained earlier in Fe-Cr9. In contrast to RR data in Fe-4Cr and Fe-9Cr, the systematic divergence between RR curves of samples having different defect concentrations is observed above 240-250 K. This feature gives evidence on the short-range order (SRO) formation in the course of post-irradiation annealing.

Carbon addition to Fe-4Cr leads to the suppression of a RR peak at  $\sim 190$  K earlier attributed to correlated vacancy migration and does not affect the stage at  $\sim 220$  K interpreted earlier as the onset of long-range migration of interstitial atoms (IAs). Since the vacancies, most probably, should be captured by carbon atoms, the carbon effect on RR does not contradict the previous interpretations. A new stage is formed around 350 K attributed to a release of trapped vacancies from carbon atoms, i.e. almost at the same temperature as in pure iron.

Signature of the SRO formation of the type corresponding to the earlier data (I. Mirebeau et al. Phys. Rev. Lett. **53**, 687, 1984) is observed in the temperature range 210-300 K, while at 300-400 K, i.e. within the vacancy dissociation stage, the SRO formation inverts its type from “ordering” to “clustering”.

The analysis of the RR data within the difference (DRR) approach allows finding the following. The vacancy clustering throughout stage III (~205 K) in Fe-6Cr leads, like in Fe-9Cr, to the increase of defect-induced resistivity (i.e. resistivity induced by the presence of defects). This rise of the defect-induced resistivity is accompanied with the onset of the SRO formation. Both these effects give evidence that vacancies are the fastest partner of a Frenkel pair.

In Fe-4Cr doped with carbon, the onset of the SRO formation is slightly shifted towards higher temperatures (by ~15 K) as compared to that in non-doped and Fe-6Cr alloys. The position of this onset coincides with the stage at 220 K. Since vacancies are immobilised at carbon atoms up to 350 K, the onset of the SRO formation and stage at 220 K can be assigned to long-range migration of IAs only.

Within the temperature interval of the stage at 350 K, two processes are distinguished. The first one corresponds to dissociation process and is associated with the vacancy release. Combined interaction of vacancies, chromium and carbon atoms leads to the decoration of the latter with the former due to chemical affinity. Formation of chromium atom pairs at the lattice sites nearest to carbon is equivalent to short-range clustering. The second one is associated with free migration of some species. Since atoms of carbon are the only species, which can migrate freely in the considered case, it may be supposed that this process corresponds to decoration of vacancy clusters with carbon atoms.

### **Positron Spectroscopy Investigation of Effect of Intermetallic Nanoparticles on Accumulation and Annealing of Vacancy Defects in the Electron Irradiated Fe-Ni-Al Alloy**

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Austenitic stainless steels and alloys are one of the basic structural materials for reacting regions of fast reactor. A main factor limiting the use of austenitic steels in fast reactors is a vacancy swelling. Essentially to suppress a swelling it is possible by application of ageing alloys in which there is a formation of dispersed intermetallic precipitates of type Ni<sub>3</sub>Ti(Si,Al). However for today there is no common opinion about the mechanism of influence of coherent precipitates on behavior of point defects.

Alloy Fe-34.5mas.%Ni-5.4mas.%Al was investigated. A part of samples of an alloy were aged at temperature 823 – 923 K during 3 – 35 h, and also at temperature 1023 K during 6 h. All samples were irradiated to 5 MeV electrons on the linear accelerator at temperatures 300 K, 423 K and 573 K. The method of angle correlation of an annihilation radiation (ACAR) was used for examination of the defect structure. ACAR allows

determining concentration and type of vacancy defects and also providing information about local chemical environment of a positron trapping site.

In this study it has been shown that presence of homogeneous distributed nanosized (1-8 nm) precipitates ( $\gamma'$ - and the iron-rich phases) in Fe-Ni-Al alloy result in to reduce of accumulation of vacancies in several times in comparison with an alloy quenched on solid solution. Thus quantity of this effect strongly depends on density, size of particles  $\text{Ni}_3\text{Al}$ , and also their type. Besides, this effect increases then irradiation temperature increase. Also it is revealed, that in the aged at 1023 K alloy there is a radiation-induced nucleation of intermetallic particles of type  $\text{Ni}_3\text{Al}$  during irradiation at 573 K.

This study was done within RAS Program (project № 01.2.006 13394), with partial support of Russian Foundation for Basic Research (projects Nos. 07-02-00020 and 07-02-96052)

### **Change of Interatomic Interaction in Metals in Conditions of Exposure to Ionizing Radiation**

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The action of ionizing radiation on metals causes change in the potentials of interatomic interaction due to appearance of ionized atoms and conduction electrons excitation. For investigating this effect a method is required that would allow first-principle calculations to be performed with account for changes in the respective characteristics of solid-state structures. In this connection, we studied the interaction potentials in the presence of ionized states using the pseudopotentials method, where the parameters of such states were defined in compliance with the method of quantum defects by the free ions spectroscopic terms.

We made an attempt to find out the effect of Fermi surface thermal diffusion on interatomic interaction potentials by way of direct calculations which showed that temperature has a weak influence on the screening properties of conduction electrons. At the same time, the screening properties strongly depend on their concentration.

### **Influence of Electronic Structure Features on Segregation of Substitution Elements on Grain Boundaries. First principles Calculation**

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Segregations of dissolved atoms on grains boundaries (GB) have the considerable influence, both on phase stability and on physico-chemical properties of alloys. Though thermodynamic principles of segregations formation are known for a long time, the microscopic mechanism of this phenomenon continues to remain a subject of discussions. The model approaches, viewing dimensional misfit of ionic radiuses as a principal cause of

interaction of a solute element with GB, are not capable to explain the the scale of segregations observed for substitution impurities. According to existing concepts, one of the factors, determining formation of segregations on GB, is charge transfer between impurity and matrix atoms (chemical interaction). Therefore research of segregations by molecular dynamics method with the use of model interatomic potentials is not capable to provide reliability of obtained results.

For clarification of electronic effects role in formation of segregations by methods of the electronic density functional theory (PAW-VASP) with account of nuclear relaxation, calculation of electronic structure and of total energy of a crystallite, containing special GB and atom of an alloying element in various positions relative to GB, has been carried out. As an example, tilt GB  $\Sigma\{112\}$  in Al was considered doped by Mg or Si. It was shown, that interaction of substitution atoms with special GB is short-range and is determined by reconstruction of electronic structure in GB nodes with the atomic coordination distinguished from bulk. Thus Mg atom substitution in GB with smaller number of valence electrons, than the matrix atoms have, is accompanied by the considerable gain in energy ( $\Delta E_{segr}^{Mg} = -0.3$  eV) while segregation of Si atoms (with bigger number of valence electrons, than Al), is energetically unfavorable ( $\Delta E_{segr}^{Si} = 0.04$  eV). Considering valence electrons distribution and local density of states it was determined, that the major role in interaction of an alloying element with GB is played by charge transfer between it and a matrix. The conclusion was made, that nonequilibrium GB, for which crystallographic nuclear coordination is broken in wide adjacent area, have increased tendency to formation of alloying elements segregations.

### Characteristics of Dose in Structural Materials of WWER and BN

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For an analysis and forecasting of radiation-induced phenomena in structural materials of WWERs, PWRs and BN reactors the fast neutron fluence is usually used (for structural materials of the reactor core and internals the fluence of neutrons with energy  $>0.1$  MeV, for WWER vessel steels the fluence of neutrons with energy  $>0.5$  MeV in Russia and East Europe, and with energy  $>1.0$  MeV in USA and France).

Displacements per atom (dpa) seem to be a more appropriate correlation parameter, especially, for structural materials of thermal reactors. The use of dpa allows to compare results of irradiation of materials in different neutron energy spectra or with different types of particles (neutrons, ions, fast electrons). Calculations of energy spectra of primary knocked atoms (PKA) and “effective” dpa, which are introduced to take into account the point defect recombination during the relaxation stage of a displacement cascade, are of both scientific and practical interests.

In this work the results of calculating fast neutron fluxes with different threshold energies, dose rates and "effective" dpa for a number of positions in the WWER-440 and WWER-1000 reactors, including the core, internals, positions of surveillance samples, and at internal and external vessel surfaces are presented. The calculations were carried out for pure iron for all positions considered as well as for zirconium in the core and for structural materials of internals, surveillance samples and vessel. Similar calculation were performed for a number of pure metals (Fe, Ti, Zr, W) and structural materials (steels ChS-68, EP-450) in the core of BN-600 reactor. Energy spectra and mean PKA energy were calculated for metals in all reactors. Characteristics of dose in pure iron and steels are compared. Effect of neutron spectrum on dose is studied. The characteristics of dose can be used for an analysis of radiation-induced phenomena in structural materials, and also for the development of physical models of these phenomena.

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### **Phase Transitions in the High-density Cascades of the Atom-Atom Collisions**

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The origin of the high-density cascades of the atom-atom collisions under neutron or ion irradiation are due to the essentially nonlinear processes and the atom displacements are placed within compact regions. As a result the valuations of the radiation damage by dpa (displacements per atom) similar to TRN-standard often turn out to be unfounded for the metallic materials and ever inapplicable for dielectrics.

In the paper the experimental evidences of the structural phase transitions in the high density cascades are shown. The phase transitions of the solid-liquid type are identified by the radiation changes of the nanostructure of the quartz glasses, by the radiation induced resolution of the nanoparticles in the metallic alloys, by the redistribution of the ions between the different positions in the complex spinel structures under radiation. The polymorphous phase transition, the high-temperature phase  $\beta$ -chrysoberyl formation, takes place in the high density cascades in the alexandrite single crystal.

The dynamics of the high-density cascades of the atom-atom collisions is described. The microscopic nonequilibrium state is appeared in an initial stage of the atomic displacements, all energy of the high-density cascade (1-2 eV per atom) is localized in the compact area for a time until  $10^{-11}$  s. There is the effective diffusion mixing - the atomic jump frequency is  $\sim 10^{12}$  Hz. The diffusion shift of the each of the  $10^4$  atoms of the cascade region is  $\sim 1$  nm. That is why the kinetic possibilities of the phase transitions - dominant causes of radiation damaging, takes places. It is shown that the probability of the high-density cascade origin is 0.2 - 5 %.

## Hardening due to and absorption of $\frac{1}{2}\langle 111 \rangle$ and $\langle 100 \rangle$ dislocation loops via interaction with dislocations in bcc Fe and Fe-Cr alloys

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The microstructure of neutron-irradiated ferritic alloys, at sufficiently high doses, typically consists of dislocation loops of interstitial-type with Burgers vector  $\mathbf{b}$  equal to either  $\frac{1}{2}\langle 111 \rangle$  or  $\langle 100 \rangle$ . Their presence obstructs motion of dislocations, leading to an increase in the yield stress and reduction in ductility. However, the ability of dislocations to absorb these loops assists in the formation of 'clean' channels, possibly causing plastic instability. Thus, the mechanisms determining the absorption and factors controlling the process must be rationalized.

Molecular dynamics simulations were used to investigate reactions between screw and edge dislocations with  $\mathbf{b} = \frac{1}{2}\langle 111 \rangle$  and interstitial dislocation loops with  $\mathbf{b}$  equal to either  $\frac{1}{2}\langle 111 \rangle$  or  $\langle 100 \rangle$  at different locations with respect to the slip plane. The loop size was varied from 0.5 nm (invisible in a TEM) up to 10 nm (easily resolvable), and ambient temperature was varied from 1K (quasi static simulations) up to 600K. Two different types of loading where (i) the thin foil conditions and (ii) interaction with an infinite row of loops in the bulk were performed.

The results show that small loops (with size up to 1 nm) are easily absorbed on the dislocation, independently of their  $\mathbf{b}$ . Large  $\frac{1}{2}\langle 111 \rangle$  loops are found to glide towards the dislocation such that an energetically-favourable reaction occurs on contact. It results in formation of a  $\langle 100 \rangle$  segment, whose motion controls the product of reaction and subsequent release of the pinned dislocation. Depending on temperature and strain rate conditions, release occurs with either complete or partial absorption of the pre-existing loop. Large  $\langle 100 \rangle$  loops exhibit a variety of reactions, including shearing, complete absorption, transformation into  $\frac{1}{2}\langle 111 \rangle$  loop (with partial absorption) and transformation into immobile interstitial complexes consisting of conjoined loops with different  $\mathbf{b}$ . The critical stress for unpinning from  $\langle 100 \rangle$  loops depends strongly on the geometry of interaction and the orientation of  $\mathbf{b}$  of the loop. Some reactions are complex, but all can be described in terms of conventional dislocation reactions in which Burgers vector is conserved. The fraction of interstitials absorbed varies from 0 to 100%. The nature of these reactions and of those requiring high applied stress for dislocation breakaway has been identified.

## **IAEA Activities in the Areas of Fuel Performance Analysis and Advanced Radiation-Resistant Materials Development**

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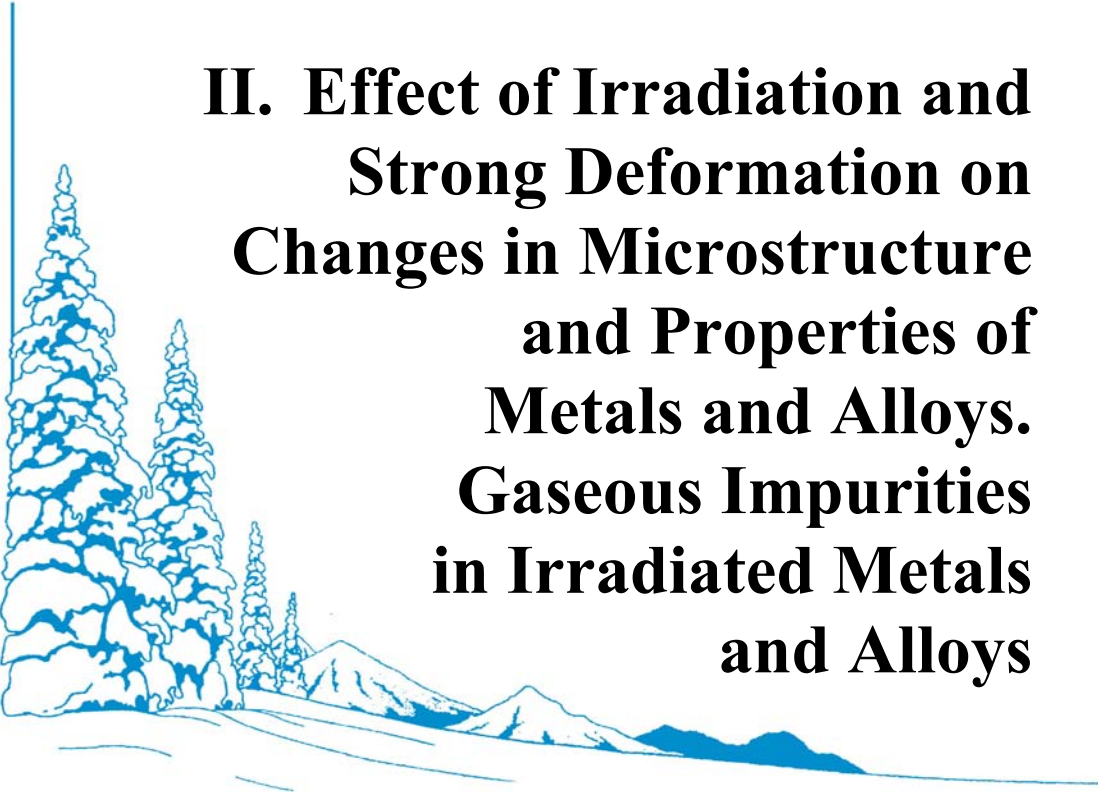
The focal IAEA entity that deals with nuclear materials is the Nuclear Fuel Cycle and Materials Section (NFCMS) in the Department of Nuclear Energy. It coordinates the subject activities including the Review of Fuel Failures in Water Cooled Reactors, IAEA Zirconium Book, Coordinated Research Projects (CRP) on fuel modeling, delayed hydride cracking in Zr alloys and optimization of water chemistry, as well as the IAEA Database on PIE Facilities and the joint IAEA-OECD/NEA Irradiated Fuel Performance Experimental Database. The Agency also supports education oriented activities, e.g. joint ICTP/IAEA workshops on basic radiation materials science held in Trieste, Italy.

Since 2001 the Agency implements a large interdisciplinary project on innovative nuclear reactors and fuel cycles (INPRO). A part of INPRO works related to advanced fuel and structural materials issues is coordinated by the NFCMS. To assist these activities the Departments of Nuclear Energy and of Nuclear Science and Applications agreed to implement a joint CRP on “Accelerator simulation and theoretical modeling of radiation effects” with the aim of supporting development of materials, which will be able to operate reliably up to very high burn-ups and temperatures with acceptable swelling and mechanical properties changes. Synergies with future fusion material development are also taken into account.

The paper presents details of the above-mentioned activities, illustrates the IAEA project implementation tools and describes how work is initiated, organized and carried out. This information might be useful for potential participants and applicants for IAEA projects.







## **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 and their complexes, with each other, 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 causes 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. Attention at the Seminar will be drawn to analysis of structural-phase transformations caused by point defects generation under strong 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.





### Radiation Defects and Deuterium in Two-Phase Austenitic-Martensitic Steel under Low-Temperature Neutron Irradiation

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Two-phase steels of the austenitic-martensitic type are highly radiation resistant and, also, are a good model material for investigations into the role of interfaces as point defect sinks and hydrogen traps.

The two-phase austenitic-martensitic **X16H9M3** steel was studied. Samples of the two-phase steel underwent the following thermal treatment operations before examination: heating to 1323 K (holding for 30 min) and water quenching (1); post-quenching cooling to 190 K, holding for 3 hours, and heating to room temperature(2); annealing at 843 K (1,5 h) after the low-temperature treatment and cooling with the furnace to 300 K (3). The  $\alpha$ -martensite was formed after the second treatment operation. The reverse  $\alpha \rightarrow \gamma$  transformation (48% martensite and 52% austenite) was partially realized after the third operation. Some of the samples treated by this method were saturated with a deuterium-tritium mixture from the gas phase to a level of 300 at.ppm. Along with other techniques, tritium was added for measuring the hydrogen concentration. The saturated samples were kept in liquid nitrogen. Neutron irradiation was realized in an IVV-2M reactor at 80 K to a fluence of  $1.5 \cdot 10^{19} \text{ cm}^{-2}$ . The residual resistivity and the mechanical properties were measured at 4.2 K and 77 K respectively. Isothermal and isochronal annealing treatments were performed.

Results of the research into the effect of neutron irradiation at 80 K on physics and mechanical properties of this steel with and without the deuterium impurity have been described. Results of post-radiation annealing have been analyzed. A method has been proposed for separating the contributions of radiation defects in the austenitic and martensitic phases.

The work was done with support of the RAS Program № 01.2.006 13394, RFBR Project № 07-02-00020-a.

### Gases in Metals

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In many papers dedicated to investigation of behaviour of construction materials (CM) in reactor irradiation field, the changes in the properties of CM are attributed to the effect of helium. However, analysis of the results of investigations prompts that the element helium exerts no influence on macroscopic properties of materials (such as swelling, embrittlement). The effects of reactor irradiation are influenced by the structural-phase state of the material.

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As it follows from the overall array of data found in the literature, the presence of helium (as well as of other inert gases) in the absence of radiation damage does not vary the properties of any material: such variation takes place only as a consequence of radiation-induced structural and phase changes. For this reason, the vector of our efforts should be directed to investigation of the structure and properties of the materials proper, but not the helium in them.

### Phase and Structural Features of ODS Alloys with BCC and FCC Lattices

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In recent years the mechanically activated synthesis (MAS), which involves an intensive deformation of powders in ball mills and high-pressure shear, has been used increasingly in radiation materials science for development of oxide dispersion strengthened (ODS) steels. This promising method can provide new practically significant materials with improved heat resistance characteristics for use in fast reactors.

It was shown for a variety of alloys and compounds that the process of deformation-induced dissolution of oxides in metal matrices represents the competition between nonequilibrium and equilibrium formation of supersaturated solid solutions and secondary phases [1-4]. The duality of the process largely determines the mechanism and the kinetics of MAS, as well as the phase composition of the structure being formed. The thermodynamic impetus to phase transformations plays the main role in the "metal-oxygen" system where oxides with ionic bonds are formed. Strengthening oxides are distributed more uniformly in the structure of ODS alloys synthesized in mills rather than during experiments on pressure shear. This is due to better mixing and a wider temperature interval.

The MAS process involving unstable iron oxides  $Fe_xO_y$  and matrices of BCC or FCC alloys (Fe-Cr-W-Y-Ti, Fe-Y-Ti, Fe-Ni-Ti, Fe-Ni-Zr) is realized through mechanochemical reactions, which are connected with decomposition of initial oxides and formation of new iron oxides having a lower valence (wustite, maghemite, etc.), solid solutions (including oxygen in metal), and compounds of reactive elements (titanium, yttrium, and zirconium) with oxygen. The electron microscopy analysis, which was performed in combination with Mössbauer and X-ray structural investigations, demonstrated that preliminary alloying of steels with Ti, Y and Zr provides conditions for formation of strengthening oxides of the corresponding elements during MAS.

This study was supported by the RAS Presidium program on nanomaterials (project No. 7), the "Intels" Foundation (project No. 45-07-02), and the Foundation for promotion of national science (programs "Outstanding scientists – Candidates of Science'2008" and "Outstanding postgraduates'2008").

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### The Influence of deformation on changes in structure and physical and magnetic properties of the austenite steel of the type X18H10T

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The austenite steels of the type X18H9 and X18H10T are widely used for in-core components of national and international thermal neutron reactors. Neutron irradiation induces changes in the structure and phase content of these steels, in particular it leads to the formation of  $\alpha'$ - phase, which results in a changing of physical and mechanical properties of materials.

Changes in the microstructure and physico-mechanical properties can also result from the impact of deformation, cold work in particular, on the steels. In order to differentiate the contributions from the neutron irradiation and the cold work deformation to the steels of this type one needs to know the processes proceeding during these impacts and their influence on the changes in the physico-mechanical properties.

The changes in micro structure and several physico-mechanical properties of the X18H10T austenite stainless steel induced by the deformation impact have been studied. The results of the investigations of the microstructure, magnetic properties, electrical resistance, Young's modulus, micro hardness of the austenite steels specimens after 0 to 45% cold work deformation are presented in this paper.

During the deformation an increase of dislocation density, a formation of micro-twins and b.c.c.  $\alpha'$ -phase were observed. While the deformation was increasing to 45% the volume fraction of  $\alpha'$ -phase was changing from 0 to ~20 %, Young's modulus decreased by ~10 %, and the electric resistivity increased by ~10 % and micro hardness increased, too.

The changes in the physico-mechanical properties of the deformed material is attributed to the occurrence and growth of the volume fraction of the deformation martensite and the increase in the dislocation density.

These results can be used to compare the influence of the cold work and neutron irradiation on the physical properties of unstable austenite steels.

**Effect of Chemical Composition on Amorphization of Titanium-Nickelide-Based Alloys with Fast Neutrons**

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The structural state of a  $\text{Ti}_{50}\text{Ni}_{47}\text{Fe}_3$  single crystal irradiated with fast neutrons ( $F = 2.5 \times 10^{20} \text{ cm}^{-2}$ ) at a temperature of 340 K was studied using thermal neutron diffraction. The alloy of this chemical composition was chosen in searching for a radiation-resistant shape memory material. It is established that this alloy retains its crystalline state after irradiation, whereas the  $\text{Ti}_{49}\text{Ni}_{51}$  crystal studied previously is completely amorphized after similar irradiation. A detailed analysis of the structural state of the irradiated ternary alloy allowed us to discover the main physical causes of its radiation resistance.

**Modeling of Radiation-Induced Segregation in Alloys under Neutron and Ion Irradiation**

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Radiation-induced segregation (RIS) leads to significant changes in alloy composition near main microstructural features: grain boundaries and sample surfaces, dislocations, precipitates, voids and strongly effects the precipitate phase composition, swelling, embrittlement and other radiation phenomena. When developing new radiation-resistant structural materials one should study the RIS and formation of the radiation-induced phases in these materials.

In the present work experimental data and mechanisms of RIS in Fe-Cr-Ni and Fe-Cr alloys are considered. Physical models and a computer code are developed for modeling of RIS near flat (grain boundaries and sample surfaces), cylindrical (dislocations ) and spherical (precipitates and voids ) point defect sinks in ternary substitutional alloys. In particular, it allows to calculate RIS of alloying elements in Fe-Cr alloys. Calculations of RIS near these sinks are performed in Fe-Cr-Ni and Fe-Cr alloys under neutron and ion irradiation. An informative express method of investigating RIS is the irradiation of alloys with heavy ions and subsequent research of the segregation by methods of SIMS, X-ray photo-electron or Auger spectroscopy.

This work was supported by the Russian Foundation for Basic Research under the Project # 07-02-01353.



**Influence of Dislocation Mobility on the Size of Nanocrystal Grains of Metals at Shear Under Pressure**

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A lot of nanostructures for the transition d-metals, alloys and steels were prepared by shear under a high pressure technique. Using the above, both influence of dislocation mobility and evolution of relaxation processes on grain size of nanostructures were summarized. The analysis of experimental data both on requirements of formation parameters, and metric features of the deformation metallic nanostructures is carried out. Both the transition single phase and polymorphic metals and alloys were studied; higher strains (7-9 units of true logarithmic scale) were used at pressures 8-14 GPa.

Correlation between a final average size of crystal grains  $d_{av}$  and mobility of dislocations in hardly deformed metal under pressure is determined. The size of crystal grains is in inverse relationship with the mobility of dislocations which is in turn defined by a series of interdependent parameters, such as a shear modulus, Pierls barrier, stacking faults energy, homology temperature, alloying level, a phase state, the route of a straining, etc.

The data gained on various metals and alloys has allowed to lead generalization of effects and to accompany concrete instances. The low-temperature (80 K) deforming of various metals leads to smaller (in 1.3-1.5 times) size of crystal grains in comparison with the cold deformation at 300 K. Refractory metals such as Mo, W, Ir, Re after shear under pressure at 300 K had more dispersed structure ( $d_{av} \sim 20$  nm) in comparison with Nb, Fe, Pd, Cu, etc. metals which have smaller melting temperature. High-carbon steels U12, 110G13, 120G4, etc. after shear under pressure had the linear average size of crystal grains about 10-20 nm, witch is essentially smaller in comparison with medium-carbon steels ( $d_{av} \sim 40-80$  nm). Behavior of palladium and its alloys is a dramatic example of crystal grain size dependence on mobility of dislocations. The biggest shear deformation of pure palladium at 300 K results in a size of crystal grains about 60 nm, the low-temperature shear deformation at 80 K leads to  $\sim 30$  nm grain size, shear deformation under pressure of the hydrogenated palladium ( $\alpha$ -PdH solid solution) results in 15-20 nm grain size, while  $\beta$ -hydride ( $\beta$ -PdH) with even smaller mobility of dislocations results in 3-5 nm grain size. The nanostructure of palladium remained unchanged after the full degassing of hydrogen.

**Evaluation of Vacancy Bobility from Trapping of Deuterium by Initial and Radiation-Induced Traps**

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The exposure of hydrogen-containing materials to ions leads to the radiation-induced segregation (RIS) of hydrogen. The RIS intensity depends on many factors including the

## **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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initial structure of targets, the possibility of diffusion redistribution of hydrogen and radiation-induced defects (vacancies and self interstitial atoms) in the targets, etc. The study of the RIS formation and evolution revealed that if the radiation damage of the irradiated target is distributed nonuniformly, hydrogen is trapped in stages: initial traps are filled first and then hydrogen is captured on radiation-induced traps.

A scheme relying on this fact was proposed for RIS studies in metallic materials using the nuclear reactions method. By this scheme it is possible to separate the contributions to the segregation by the capture of deuterium on initial and radiation-induced traps and determine the capacity of the initial traps, as well as the type and stability of traps formed under irradiation.

Data on the capture of implanted deuterium in targets with substantially different vacancy mobility (vacancies are practically immobile at room temperature in a nickel target; vacancies are mobile in an austenitic steel target) have been given. The total capacity of deformation vacancy traps was several times higher in nickel than in steel, with the degree of cold plastic deformation being equal. From the analysis of the results obtained in this study and the literature data it follows that the large capture of hydrogen on deformation traps in nickel is due to a low mobility of vacancies in this material and, hence, the absence of deformation vacancy clusters in this target at the initial stage of implantation. It was found also that the removal of irradiation was followed by the decrease in the segregation intensity in nickel and its growth in steel. These observations were explained by different spectra of hydrogen traps and different mobility of vacancies in the compared targets.

It has been proposed to evaluate the mobility of vacancies in metallic materials by comparing the total capacities of deformation hydrogen traps during implantation of hydrogen ions and the post-implantation evolution of RIS.

The work was done with support of RAS (Program № 01.2.006 13394), RFBR (Project № 07-02-00020)

### **Radiation-Induced Segregation of Deuterium in Titanium**

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The deuterium segregation induced by ion irradiation was studied in titanium samples. The segregation was induced at room temperature by implantation of  $D^+$  ions having the energy of 700 keV and was analyzed using the  $D(d,p)T$  nuclear reaction. The segregation was induced and measured with one and the same deuteron beam. The concentration of deuterium in the near-surface region was determined from the energy spectrum of protons (products of the nuclear reaction between deuterons and implanted atoms of deuterium in the irradiated target) using a standard sample containing a constant concentration of deuterium.

The objects of study were polycrystalline (VT1-00) and monocrystalline (~10 appm oxygen) samples of pure titanium. Depth and dose dependences of the concentration of implanted deuterium in the ion-irradiated region were measured for these samples.

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Information was obtained for the target volume bounded by sections of the ion beam 1 mm across and 5  $\mu\text{m}$  deep.

It was found that the depth distribution of implanted deuterium is a maximum at a depth of  $\sim 4.8$   $\mu\text{m}$ . The deuterium concentration increased throughout the analyzed zone during implantation. Dose dependences of the average deuterium concentration in the analyzed volume (the segregation intensity) were almost linear.

Stability of the deuterium segregation induced by ion irradiation was studied. It was found that the segregation intensity increased once the implantation was resumed after a long-time interruption. The analysis of the depth distribution of implanted deuterium in the monocrystalline samples showed that the segregation intensity increased due to the redistribution of the implant from the bulk to the surface of the target.

Results of the study have been discussed considering the mobility of radiation defects, efficiency of their sinks, and the nature of deuterium traps in ion-irradiated titanium. A possible variant of the evolution of the defect structure in the samples during implantation and the pause in the ion exposure has been proposed.

The work was done with support of RAS (Program № 01.2.006 13394), RFBR (Project № 07-02-00020)

### **Effect of Neutron Radiation on Dissolution and Precipitation of $\text{Ni}_3\text{Me}$ Intermetallics in Steels**

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Low-temperature (340 K) irradiation of FCC Fe-Ni-Ti alloys with fast neutrons leads to dissolution of the intermetallic  $\gamma'(\text{Ni}_3\text{Ti})$  phase in displacement cascades and enrichment of the matrix with nickel. It was shown that the dissolution rate decreases as the initial size of particles increases and the irradiation temperature is elevated. The process of radiation-induced dissolution of intermetallic particles is accompanied by radiation-accelerated aging. This fact explains the kinetic parameters of dissolution of  $\text{Ti}_3\text{Ti}$  intermetallics as the irradiation temperature rises to 540 K, leading to predominant precipitation of  $\text{Ni}_3\text{Ti}$  particles.

Nonequilibrium dissolution of intermetallics was observed in Fe-Ni-Me (Ti, Al, Zr, Si) alloys during severe cold plastic deformation (SCPD) by compression shear in Bridgman anvils. The obtained data suggested a dual character of deformation-induced phase transitions including processes of nonequilibrium dissolution and defect-accelerated formation of intermetallic phases. Thus, similar kinetic regularities were established for radiation-induced (in displacement cascades) and deformation-induced (SCPD) dissolution of intermetallic particles in Fe-Ni-Ti matrices.

To understand the influence of radiation-induced point defects on the aging process, we conducted studies on low-temperature cascadeless electron irradiation of FCC Fe-Ni-Me (Ti,

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Al, Zr, Si) alloys. The experiments revealed active radiation-induced aging in the alloys with Ti and Al, a lower rate of aging in the alloy with Si, and little, if any, aging in the alloy with Zr, which has low diffusion mobility in the Fe-Ni matrix.

Results of neutron irradiation (analogously to SCPD) pointed to the competitive development of alternative processes: nonequilibrium dissolution and equilibrium precipitation of particles. In the case of SCPD the dissolution rate in displacement cascades depended on pre-aging conditions and showed up as the increase in the transformation rate with decreasing size of the intermetallics in the Ti and Al alloys. In the Zr- and Si-alloyed compounds the variation kinetics of the Ni concentration in the matrix depending on pre-aging conditions was "sluggish". As to the aged alloys with Zr and Si, this was explained, first, by large dimensions of the intermetallic particles and, second, by the enhancement of the competing radiation-induced aging of the Zr alloy over the irradiation temperature interval.

This study was performed under the topic "Structure" with a partial financial support from RFBR (grant No. 07-02-00020) and in line with the RAS Presidium program on nanomaterials (project No. 7).

### Study of deformation-induced segregation in Fe-Cr-Ni alloy

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In the present work the theoretical analysis of results of experimental work [1] is given in which dependence of nickel concentration of deformation temperature on grain boundary in an alloy was investigated at deformation-induced segregation. It is shown in [1], that concentration of nickel on grain boundary decreases monotonously on a temperature interval (400-600) K. Besides enrichment of grain boundary by nickel is not observed at temperature 600 K. For an explanation of the given experimental fact and of deformation-induced segregation character in the specified interval of temperatures generally it has been considered, that rate of generation of point defects upon deformation strongly goes down at rise in temperature [2] as a result of decreasing of generating defects dislocations quantity. It has been shown in work [2] also, that movement of screw dislocations with steps of the nuclear sizes has big influence on generation of point defects upon deformation. Let us consider now also the fact, that annihilation rate of screw dislocations upon deformation depends essentially on temperature [3]. As a result expression for generation rate of point defects depending on temperature was suggested which described correctly qualitative features of experiment.

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## Moving “Waves” of Plastic Deformation Observed in Highly Irradiated 12Cr18Ni10Ti Stainless Steel

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Mechanical tests were conducted on miniature 12Cr18Ni10Ti stainless steel specimens that were cut from the faces of hexagonal shrouds of BN-350 fuel assemblies irradiated to 13-55 dpa in the range 280-430°C. Using a video marker extensometry technique the strength and plasticity were defined for both engineering values and true strain – true stress values.

Whereas 3-7 % engineering strain is usually observed in this steel after exposures of  $\leq 15$  dpa in this temperature range, a new deformation phenomenon was found at higher doses, involving a moving wave of plastic deformation which precludes the onset of necking and thereby significantly extends the deformation band down the length of the specimen. This phenomenon leads to unusually high values of engineering plasticity, 25-50 % and possibly more.

The first post-irradiation examination results (microscopy and magnetic techniques) show that radiation-induced enhancement of the martensitic  $\gamma \rightarrow \alpha$  transformation is the principal mechanism driving this phenomenon. Compared to deformation-induced martensite in unirradiated specimens the martensitic transformation in highly irradiated steel is very high, with volume fractions reached on the order of 30-40%. As the local martensite fraction reaches these levels the resultant hardening limits further local deformation and the deformation shifts to adjacent, not-so-deformed, softer regions, producing a continuously moving wave traveling along the specimen at a speed approximately five times greater than the engineering deformation rate.

To check this martensite-related hypothesis a series of tensile test experiments were conducted in the range of  $-100$  to  $+200^\circ\text{C}$  at deformation speeds ranging from  $8.3 \times 10^{-3}$  to  $8.3 \times 10^{-5} \text{ s}^{-1}$ . Deformation waves were not observed at  $\geq 100^\circ\text{C}$ , which for 12X18H10T corresponds to the critical temperature ( $M_d$ ) at which martensite does not form. For the miniature specimens used in this study, high strain rates were unable to elevate the temperature significantly and therefore no effect of strain rate was observed. The influence of specimen size and geometry on occurrence of deformation waves was estimated, however. It is shown that high-speed deformation and deformation-induced self-heating of larger specimens will probably lead to suppression of the deformation wave phenomenon.

**Energy Balance of Plastic Deformation of Irradiated Metals and Alloys**

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According to modern concepts the plastic deformation of a material is accompanied by transformation of mechanical energy into various other types of energy such as thermal (70-90 %), latent (30-10 %), acoustic ( $\ll 1$  %) and other very minor forms ( $\ll 1$  %). If insignificant energy forms are ignored, one can describe the energy balance within the limits of the first law of thermodynamics:  $A = Q_{\text{def}} + E_{\text{sdef}}$ , i.e. mechanical energy delivered from the outside (A) partially dissipated as heat ( $Q_{\text{def}}$ ), and is partially stored in the material as latent energy ( $E_{\text{sdef}}$ ) residing in a variety of deformation-induced defects.

Results of calorimetric experiments are presented here involving the energy balance following deformation of pure metals (Armco iron, nickel, copper, vanadium, and niobium) and industrial alloys (12Cr18Ni10Ti and 03Cr20Ni45Mo4Nb2) in both unirradiated and irradiated conditions. Irradiation was conducted in the WWR-K reactor at  $<80^\circ\text{C}$  to fast fluences up to  $2 \cdot 10^{20}$  n/cm<sup>2</sup>.

During deformation an excess of heat release over the deformation work ( $Q > A$ ) was observed in irradiated specimens. After reaching a material-specific critical fluence (for example,  $\sim 10^{19}$  n/cm<sup>2</sup> for Fe, and  $\sim 10^{20}$  n/cm<sup>2</sup> for Ni), excess heat release becomes a common feature. This excess heat is assumed to result from interaction of moving dislocations with radiation-induced black spot defects, a process that sweeps out these small defects, giving rise to additional heat generation  $Q_{\text{sweep}}$ . Also, it was observed that during deformation of metastable steels the martensitic  $\gamma \rightarrow \alpha'$  transformation is also accompanied by an additional heat release  $Q_{\text{ga}}$ , also leading to a excess heat condition where  $Q > A$ . Martensite formation is also known to be accentuated in these steels by irradiation at low temperature.

Based on these two mechanisms a new extended model of energy balance has been developed for calculation of thermal emission during deformation of highly irradiated and/or metastable materials. Disagreements between experimental and calculated values of heat release are on the order of  $\sim 10$ -12 %. The model is especially applicable to mechanical tests conducted in absence of thermo-vision cameras or calorimetric measurement techniques.

**The Effect of Low Dose Neutron Irradiation on the Tensile and Impact Properties of a Series of Titanium Alloys**

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Structural materials used in the first wall of fusion reactors or in any type of nuclear power device will become activated and will represent an environmental hazard during their storage. Titanium is the natural metallic element having the fastest radioactive decay. Binary or ternary titanium alloys can be designed to keep this basic property and at the same time

## 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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improve the mechanical properties of pure titanium. The most widely used titanium alloy is Ti6Al4V. This metastable alpha-beta alloy suffers from structural instabilities when exposed to radiations. Vanadium precipitates are generated deteriorating the ductility and inducing brittleness. [1, 2]. The situation is better with alpha alloys. A few available results indicate that the resistance to irradiation is improved compared to alpha- beta alloys [3]. Nevertheless the unique industrial alpha alloy available Ti5Al2.5Sn suffers also from phase instabilities and is not an excellent low activation material due to the presence of aluminium [2].

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

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

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

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## Mechanical Characteristics and Corrosion Spallation of CAB-1 Low Aluminum Alloy after Neutron Irradiation in WWR-K Reactor

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The experimental data on mechanical strength and corrosion resistance under permanent load of CAB-1 low aluminum alloy after its irradiation with neutrons are discussed.

The processes of corrosion spallation were investigated by the methods of accelerated materials testing in an aggressive medium under conditions of permanent load and static tension. A comparison was carried out of mechanical characteristics of CAB-1 alloy: irradiated and unirradiated, under exposure to an aggressive medium and without it. Analysis of the obtained results points has brought to a conclusion that sensitivity of CAB-1 low aluminum alloy to corrosion spallation under strain is determined by the initial structure of the material, the irradiation dose and the rate of load application.

### **Electromagnetic Irradiation of Metal Melts**

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A discussion of the effects of strong nanosecond electromagnetic pulses on the properties of molten metals and alloys is presented.

The advantage of the proposed method of treatment is in its simplicity, relative safety and an opportunity to directly control the irradiation parameters.

### **Radiation-Induced Modification of Dislocation Density and the Dynamics of Radiation Creep of the Materials**

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It is well known, that the creep of materials under irradiation is provided by dislocation's movement through its glide planes. Free length of dislocation is limited by the presence of different structural defects in the materials (impurity atoms, dispersed particles, grain boundaries, radiation defects).

The purpose of these papers was to investigate the influence of radiation-induced changes of the dislocation density on the creep's dynamics of the materials which contain dispersed particles.

The creep's dynamics was described within the framework of dislocation "climb-glide" model [1]. It was taken into account that the concentration of vacancies and interstitial atoms increased due to generation by external irradiation; and decreased due to flowing by dislocations or non-dislocation traps. The dislocations density increase due to transformation of Frank's loops and tetrahedron stacking faults into gliding dislocation; and decreased due to the dislocation going out grain boundaries. Also it was taken into account that dispersed particles can lock dislocation glide (the immobilization factor is more than 1) or they can be as obstacles, which generate dislocations [2].

Stationary states, their stability, criterions of appearance of several stationary states, and corresponding creep rate change are obtained.

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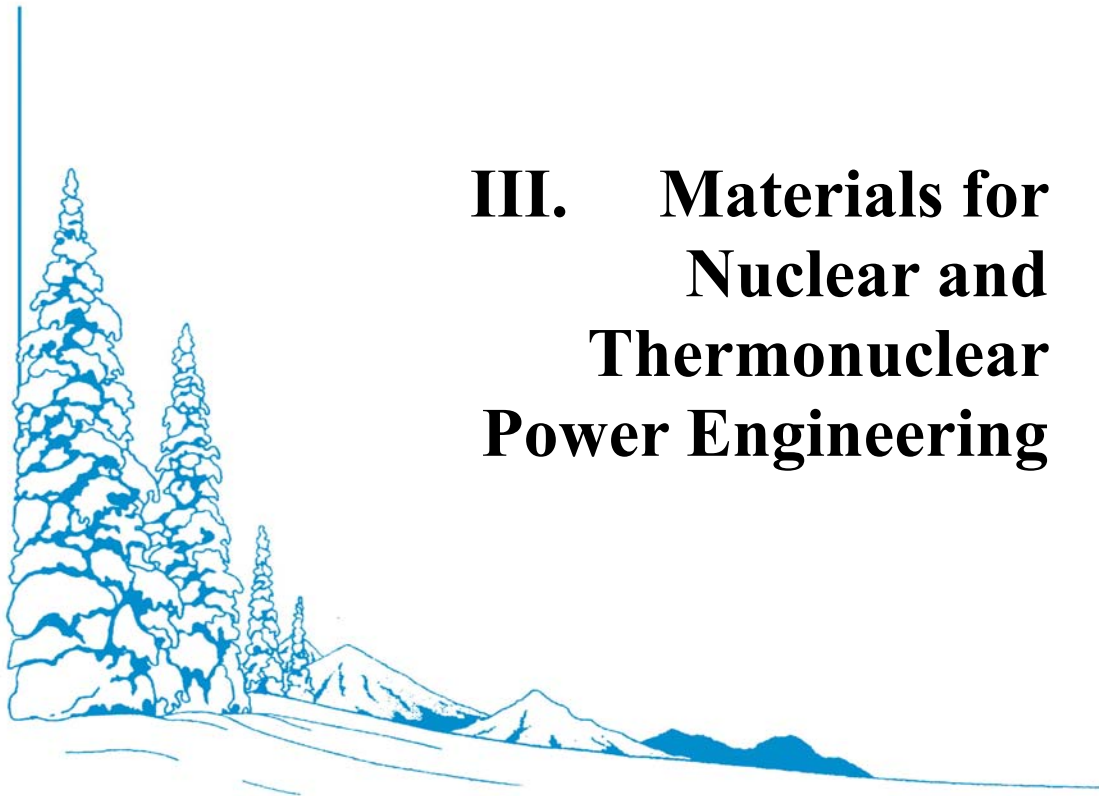
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### **Irradiation effects on the tensile behavior of Al-6061**

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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 molten-salt 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.





### Tomographic Atom-probe Nanocharacterization of Irradiated VVER-440 Weld

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There are several VVER-440 reactors with annealed vessels in Russian Federation that are still functioning. High temperature annealing has extended life-time of these reactors, however, safety operation of these units requires reliable forecast of mechanical properties behavior under re-irradiation for the weld, located in front of the core. That is the element of construction, which limits radiation safe life of the whole reactor pressure vessel (RPV). Previous investigations have shown that degradation of mechanical properties of VVER-440 RPV steels correlate with formation of copper enriched precipitates (clusters), with typical size about several nanometers [1,2].

In this work nanoscale investigation of RPV weld material were performed. For nanostructure characterization of this steel TAP technique was used, as it allows to reconstruct a 3D image of investigated volume and to determine chemistry simultaneously. Four different states were investigated: initial (unirradiated), irradiated ( $6 \times 10^{19} \text{ cm}^{-2}$ ), annealed and reirradiated ( $8.5 \times 10^{19} \text{ cm}^{-2}$ ). All samples were irradiated in surveillance channels of Rovno NPP unit 1 and 2 (Rovno-1,2). The first results of tomographic atom probe investigation of weld material with high phosphorus concentration (0.038%) were obtained. Two types of clusters: enriched in phosphorous and copper were discovered. Typical size of each type were estimated to be less than 2 nanometers. Besides P-Cu clusters two types of carbide segregations were found.

Concentration profiles of different chemical elements for matrix and structure features (clusters, carbides) are presented.

This work was partly supported by RFFI (07-02-13655-офи\_ц and 08-02-01448-a)

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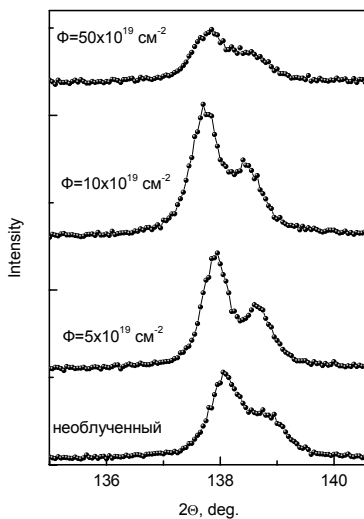
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### Effect of Preliminary Treatment on Structural State of Fe-Ni-Ti Intermetallic Compounds Irradiated with Fast Neutrons

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The development of nuclear power engineering calls for creation of materials with a number of properties necessary for operation in radiation fields at different temperatures, often under loads and after different types of preliminary treatment, e.g., materials of drawn

fuel element tubes with different types of stresses. To create materials with the required properties, their structural states after irradiation with fast neutrons need to be studied first, both on model alloys and on real materials. Iron-nickel-titanium alloys were selected for experiments. The samples presented either thin foils or massive blocks ( $5 \times 5 \times 65 \text{ mm}^3$  parallelepipeds). First all samples were annealed at  $1100 \text{ }^\circ\text{C}$  during 30 min. Part of the samples were quenched in water or oil. Some foils underwent plastic deformation, others were aged at  $650 \text{ }^\circ\text{C}$  during 30 minutes. Then all samples were irradiated to different fast neutron fluences  $\Phi$  (up to a maximum value of  $5 \times 10^{20} \text{ cm}^{-2}$ ) at  $80 \text{ }^\circ\text{C}$ .



The structural state of both the initial and the fast neutron irradiated samples at room temperature was studied by the methods of X-ray and neutron diffraction pattern analysis.

The Figure presents details of X-ray patterns of a foil sample first quenched in water and then irradiated to different fast neutron fluences. There is observed narrowing of a diffraction line before  $\Phi \sim 5 \times 10^{19} \text{ cm}^{-2}$ , which normally takes place at defects annealing. This means that, at this stage, radiation-stimulated annealing of defects initially present in the quenched sample prevails over the process of new defects formation. However, with further

fluence increase, considerable growth of radiation defects becomes the prevailing process (reflection widening is again observed). Further in the talk, somewhat different processes going in other samples under fast neutrons irradiation will be discussed.

Work carried out under programme of RAS (Theme No. 01.2.006 13394, code “Impuls”), with partial support of Programmes of Basic Research of Department of Physical Sciences of RAS “Neutron Diffraction Investigation of Structure of Materials and Fundamental Properties of Matter” (Project No. 33, UB RAS), Presidium of RAS “Influence of Atomic-Crystal and Electronic Structure on Properties of Condensed Matter” (Project No. 21, UB RAS), State Contract No. 02.518.11.7026 and RFBR Project No. 07-02-00259.

### The Accounting of the Overlap of Cascade Regions in the Description and Evolution of Radiation Clusters in Austenite Steels under Cryogenic Neutron Irradiation

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Substantial changes in physico-mechanical properties are observed in the metals and alloys irradiated to damage doses as low as  $\sim 0.01$  at cryogenic temperatures. These changes must be taken into account in the application of metal materials in the installations operating under neutron irradiation at cryogenic temperatures. The strong irradiation effect is attributed

to the formation of radiation clusters which can not be annealed at cryogenic temperatures. The adequate description of the formation and accumulation of the radiation clusters in the metals under cryogenic temperature irradiation is required for a quantitative estimation of their influence on the characteristics of strength, plasticity, elasticity and other physical properties of the metals.

The paper presents the quantitative model of the formation and evolution of the radiation clusters in the f.c.c. metals under cryogenic neutron irradiation. The model takes into account the overlap of the cascade regions, i.e. the cases when a new displacement cascade develops in the location of the “single” cluster formed earlier. The relationship was obtained for the single and “double” (resulted from the overlap of the cascade regions) radiation clusters at the initial irradiation stages when the triple and multiple impositions are of minor importance.

The obtained data were compared with the results of the electron microscopy studies of the X16H15M3T austenite steel specimens irradiated in the IVV-2M reactor to damage doses of 0.0016 and 0.016 dpa at a temperature of 77 K.

#### **Effect of Fast Neutron Dose rate on PWR Pressure Vessel Steel Embrittlement**

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The effect of fast ( $E \geq 0,5\text{MeV}$ ) neutron dose rate (flux) on PWR pressure vessel steel of 48TC type embrittlement was under study.

Objects of the research were:

- base metal surveillance specimens from VVER-440/213 NPP unit after 10 years of operation under the coolant temperature of 270°C;
- base metal of the nuclear icebreaker «Lenin» reactor pressure vessel after 18 years of operation under the coolant temperature of 290°C.

The results showed that ductile-to-brittle transition temperature shifts for above mentioned materials at a flux range of  $10^{10} \div 10^{11} \text{ cm}^{-2}\text{s}^{-1}$  are 50-60°C greater than those for flux level of  $10^{12} \div 10^{13} \text{ cm}^{-2}\text{s}^{-1}$ .

The reason of neutron flux effect is assumed to be competition between the radiation-induced diffusion effect on precipitations nucleation on the one hand and precipitation decay under neutron bombardment on the other.

### Technological Aspects of Making of Nano-Oxide-Strengthened Reactor Steels

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The study deals with technological aspects of formation of steels strengthened with dispersed titanium and yttrium nano-oxides based on austenitic and ferritic matrices. A new nontraditional method for making of reactor steels was used envisaging the use of unstable Fe<sub>2</sub>O<sub>3</sub> iron oxide (hematite), which readily decomposes and dissolves during intensive cold plastic deformation, with oxygen forming heat-resistant nano-oxides of yttrium and titanium if these elements have been added to the alloy.

Powders of the X16H15M3T1 and X12B2T steels, which were mixed respectively with dispersed additions of 0.5% Fe<sub>2</sub>O<sub>3</sub> + 0.4% Y and 0.5% Fe<sub>2</sub>O<sub>3</sub> + 0.4% Y<sub>2</sub>O<sub>3</sub>, were ground in a Pulverisette-7 planetary ball mill and a high-energy vibratory mill. The deformation methods included vacuum hot pressing followed by compaction and sintering at a high temperature (1100°C) under a pressure, preliminary magnetic-pulse pressing, and hot hydrostatic extrusion. All these methods provided bulk solid samples having different structures and mechanical properties. Then the samples were subject to reduction in a press and finally were hot-rolled.

The solid samples generally were cylinders 7...10 mm long and 9...13 mm in diameter. The method of hot hydrostatic extrusion of the (X12B2T + 0.5% Fe<sub>2</sub>O<sub>3</sub> + 0.4% Y<sub>2</sub>O<sub>3</sub>) system provided a cylindrical rod 450 mm long and 12 mm in diameter.

The structure was analyzed by the method of transmission electron microscopy. It was shown that the proposed processing conditions allowed forming a structure with grains 0.1...2 μm in size. Redox reactions led to formation of iron and titanium oxides. The oxides had a sufficiently uniform distribution over the volume of the samples. Particles of FeO, TiO<sub>2</sub>, Y<sub>2</sub>TiO<sub>5</sub>, YTiO<sub>3</sub>, and Y<sub>2</sub>O<sub>3</sub> resided mostly in the grain bulk, while their size varied between 5 and 30 nm. Coarse oxides 150...500 nm in size occurred both at grain boundaries and in the grain bulk.

HRC hardness of the sample of the oxide-loaded X16H15M3T1 steel, which was made by vacuum hot pressing under a pressure of 1 GPa, was 37 units. Hardness increased to 42...43 units after hot rolling. HRC hardness of the same sample was 10 units in the case of magnetic-pulse pressing and additional reduction at 1200°C without the high pressure applied and 25 units after rolling at 1200°C; HB increased from 1760 to 2550 MPa, while the ultimate strength was ~840 MPa. A drawback of the samples sintered without a pressure applied was their low density (6...6.5 g/cm<sup>3</sup>) because of a large concentration of pores. The hydrostatic extrusion treatment allowed improving the density up to 7.5 g/cm<sup>3</sup>. The values of  $\sigma_B$  and  $\sigma_{0.2}$  were 1025 and 967 MPa respectively, while the relative elongation  $\delta \approx 10\%$ .

This study was performed on the topic "Structure" and supported by the RAS Presidium program on nanomaterials (project No. 7), the "Intels" Foundation (project No. 45-07-02), and the Foundation for promotion of national science (programs "Outstanding scientists – Candidates of Science'2008" and "Outstanding postgraduates'2008").



### **Investigation of the Influence of Strain on Radiation Swelling and Creep Deformation in X18H10T Steels**

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The problem of substantiating the long design service term of type WWER nuclear reactors and its extension necessitates investigation of the laws governing changes in the structure and properties of construction materials exposed to neutron irradiation and operating under characteristic conditions of service of devices inside WWER reactor vessels.

The present paper offers a discussion of the influence of compression and tensile stresses on radiation swelling and creep deformation in X18H10T steels.

### **The Mechanism of Formation of Pores in ChS ЧС-68 Austenite Steel under Neutron Irradiation**

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Radiation swelling of structural steels substantially limit the life time of fuel element assemblies of fast neutron reactors. The available data prove a complex effect of the fuel composition and irradiation conditions of this process. One of the factor inducing a formation of pores is accumulation of transmutation gases. Thus, the investigation of porosity development and a revealing of the conditions initiating the radiation pore formation are contributing to both the application of the steel and the description of the physics of the process.

The paper describes the mechanism of pores initiation in ЧС-68 austenite steel under neutron spectra irradiation in fast neutron reactors. The mechanism is based on the bubble formation from the vacancy complexes and transmutation gas atoms during their migration. The bubbles, being the nucleators of pores, absorb the gas–vacancy complexes and grow and reach a critical size, after that the growth becomes statistically beneficial owing to uncompensated flow of vacancies into the pores. The expression for the critical diameter of pore nucleators was obtained by using the defect migration model [1].

The obtained data are comparable with the electron microscopy study results on the ЧС-68 steel irradiated as fuel claddings in the fast reactor BN -600 to different doses at temperature of 370 to 580°C.

The relation of radiation–induced phases and pores [2] observed at the later stages of swelling was explained by the suggested mechanism.

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## **The Influence of Neutron Irradiation on Changes in Structure Characteristics of ЧС -68 Austenite Steel**

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The austenite chromium–nickel steel ЧС-68 (08X16H15M2Г2ТФР) used as fuel cladding material in the fast neutron reactor BN–600 is compliant to vacancy swelling when being in a cold work condition. This leads to changes in geometrical sizes and a decrease in the operation characteristics of the fuel cladding material.

In the cladding material under reactor operation conditions complex processes in the changes of the structure occur, i.e. accumulation of radiation defects (including pores), structure-phase transformations, a change in internal stresses level and etc. All these phenomena are related to the initial condition of the structure of the ЧС-68 steel and the irradiation conditions.

The results of the ЧС-68 structure characteristics study are presented in this paper. For the study the tubular specimens were prepared in both conditions before irradiation and after neutron irradiation to within a temperature range of ~ 370°C to ~ 600°C to doses of ~ 70 dpa. New data were obtained in the range of 370 to 460 °C and damage doses from 1 to 58 dpa. Substantial changes in the crystal lattice parameter as dependent on dose and irradiation temperature were observed after the operation of the material in the reactor. A relation between the crystal lattice parameter and micro stresses with the evolution of the dislocation structure and phase changes under irradiation was analyzed. Some peculiarities in the changes of the level of micro stresses, and package defects concentration, characteristics of cellular dislocation structure were noted as well as the occurrence of package defects on the dislocation loops and phase transformations related to carbide precipitations on grain boundaries and twins, and G–phase in the grain body.

## **Radiation effect on changes in the structure, physical, mechanical and magnetic properties of X18H9 steel**

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Numerous data on the neutron irradiation effect on the mechanical properties and dimension stability of austenite steels were obtained for the conditions of high density of neutron flux of fast reactors. Many in-core components of nuclear reactors operate at lower displacement rates. The available data on long-term irradiation of structure components at low neutron flux density are scarce.

The steel X18H9 (Cr-18 %, Ni-9 %, Fe-base) equivalent to AISI 304 has been investigated. The specimens were taken from the X18H9 steel tube of the external diameter

95 mm and thickness 20 mm. The tube has been exploited in the BN-600 reactor core at a temperature of 370-375 °C for 22 years. The specimens were made of parts of the tube located at different positions in the core; the parts of the tube were irradiated to damage doses from 1.5 to 21 dpa at displacement rates from  $3 \cdot 10^{-9}$  to  $4 \cdot 10^{-8}$  dpa/s.

The irradiation resulted in radiation swelling of the steel about 3 %. In addition to swelling some other structural changes take place under these irradiation conditions, they are a formation of second phases,  $\alpha'$ -phase in particular, and changes in the crystal line matrix content due to the precipitation of carbides.

The dose dependencies of the changes in density, electrical resistivity, elasticity modulus, short-term mechanical properties and magnetization of the material under study were determined. The changes in the electrical resistivity and elasticity characteristics was no more than 2 % of the corresponding values from the initial condition and their dependence on damage dose was non monotonous. Radiation strengthening takes place in the material in the dose range of 0 to 14 dpa; the ultimate strength and relative yield stress are increasing and plasticity is decreasing with damage dose. It is found that the magnetization of the material grows with dose due to the occurrence of the b.c.c.  $\alpha'$ -phase. It is shown that there is a relation of the changes in the physico-mechanical properties with the evolution of the steel micro structure under neutron irradiation.

#### **Effect of Long-Term Post-Radiation Annealing on Structure and Properties of 12X18H10T Steel Irradiated with Neutrons to 53.4 dpa in BN-350 Reactor**

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The results of examination of changes in the structure and properties of type 12X18H10T stainless steel irradiated in BN-350 reactor to high damaging doses (~53.4 dpa) after annealing under different conditions are given. Investigations were carried out by the methods of metallography, electron microscopy and X-ray phase analysis, and changes in the mass, density and microhardness of samples were evaluated.

#### **Set of Investigations of HFR Fuel Rods in Justification of Their Serviceability and Safe Operation**

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RIAR perform works on the upgrading of the SM high-flux research reactor core. The main objective of these works is to improve effectiveness of the reactor utilization by increasing a scope of experiments with a high density of neutrons intended for isotopes

accumulation, irradiation of structural and other materials, as well as for the performance of different kinds of experiments. The objective may be achieved by the arrangement of irradiation channels within the SM reactor core. The resulting reactivity losses can be compensated in two ways: by increasing of  $^{235}\text{U}$  loading into the fuel assemblies or using new fuel rods with a less cross-section of neutron absorption. At the initial upgrading stage it was decided to use standard fuel rod designs with the uranium loading increase by 20 percent in order to compensate the reactivity losses.

Standard fuel rods of the SM reactor are made on the basis of dispersion fuel composition  $\text{UO}_2 - \text{Cu}$ , while the fuel rod claddings are made of steel EI-847. The fuel rod section is cross-shaped. The SM standard fuel rods are successfully operated for 40 years. They have shown high serviceability and operation safety at a high density of the heat flux from the surface of  $15 \text{ MW/m}^2$

However, current commissioning of the new or modified nuclear fuel provides for a performance of the required set of tests and investigations in justification of its serviceability. In this connection, RIAR implement a program for a study of serviceability and safe operation of the modified SM fuel rods with the increased uranium loading.

A set of works presented in this paper incorporates the results of calculations and investigations of the experimental fuel rods tested under specially performed reactor experiments with a simulation of a wide range of neutron-physical and thermal-physical irradiation parameters on the fuel rods. Simulated parameters of the reactor tests of the fuel rods changed within the following ranges: fission density ( $4,42 \cdot 10^{14} \text{ 1/cm}^3 - 1,08 \cdot 10^{15} \text{ 1/cm}^3$ ), thermal flux density ( $6,62 \text{ MW/m}^2 - 15,7 \text{ MW/m}^2$ ), temperature ( $295^\circ\text{C} - 582^\circ\text{C}$ ), and concentration of the fission products up to  $1,1 \text{ g-fr./cm}^3$ . Reactor test conditions different in temperatures and thermal loadings allowed to obtain detailed data on the behavior of fuel rods in general and of their components in particular.

The paper describes a connection between neutron-physical and thermal-physical parameters of the reactor tests and a change in the properties of the fuel rod components. The following regularities of the change in macro- and microstructures of the fuel column were revealed: further sintering and its influence on the porosity in the fuel particles and matrix, migration of voids and fuel particles in the fuel columns. Peculiarities of the radial and local swelling of the fuel rods, as well as the swelling at different fuel rod elevations, were revealed. Dependencies of the radial swelling on the accumulation of fission products, fission density, heat flux density and testing temperature were plotted. The dependencies allow for a qualitative evaluation of the fuel rod swelling at different stages of operation and different values of the thermal-physical parameters, as well as for a selection of safe parameters subject to the ultimate swelling of the fuel rods and technical conditions.

### Nanoscopical processes of radiating embrittlement of cases steels of water-water nuclear reactors

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Main reason of radiating embrittlement of steels of pressure water-water nuclear reactors (WWER, PWR, BWR) and increases of temperature of fragile / viscous transition is formations of precipitates of phosphorus on borders of grains and borders of the unit of formed second phases and matrixes.

Consecutive transformation into nuclear reactions of transutetion atoms of impurity Mg, Al, Si, P, S each other (and further in Cl and Ar) in a stream of neutrons of reactor WWER-1000 and formation their equilibrium concentration during generation and burning out depends on initial concentration of parent elements, sections of nuclear reactions, a eergy spectrum and density of a stream of the neutrons, falling on an internal surface of the case of pressure.

In a circuit of transformations Si (concentration 0,17 ... 0,37 weight. %) in P (concentration of  $\leq 0.025$  weight. %) and further P in S it is revealed, that generation of phosphorus from silicon in  $\sim 100$  times exceeds burning out of phosphorus that leads to to increase of its equilibrium concentration [1]. Processes of segregation of impurity atoms depends on their size in relation to the size of atom of a matrix, concentration and solubility. The thermodynamic analysis of processes of formation of complexes vacancy - impurity atom, case that in conditions of an irradiation steels of water-water nuclear reactors of division by a stream of neutrons solubility unpersize atoms of phosphorus goes down, that the CuMnNi/matrix, and to increase in shift of temperature of fragile - viscous transition  $\Delta T_k$  leads to to formation of second phase  $Fe_{2P}$  on borders of grains and borders of the unit of a phase.

The method of mathematical statistics carries out analysis of existing experimental data of change  $\Delta T_k$  as functions of concentration of components steels cases of pressure of water-water reactors and on a minimum of a residual dispersion the kind of regressive function is determined. The received analytical expression with reliability 0.9 is inside confidential intervals of values of concentration of the basic components сталей and impurity for degrees of freedom of distribution available in this case quantels of Fisher.

Analytical expression for criterion function  $\Delta T_k$  multilinearly (contains products of concentration Ni, Cu and P).

In connection with complexity of the form of surfaces of an equal level on phosphorus

$$\Delta T(C_P) = f(C_{Ni}, C_{Cu})$$

The method of the numerical decision has been used. Surfaces of an equal level of concentration Ni and Cu in space of variables depending on concentration of phosphorus

find out rotation of curves  $\Delta T_k = f(C_P)$  in different directions at change of concentration Ni or Cu. It defined a direction of search of such criterion function  $\Delta T_k$  which would not depend on concentration of phosphorus in connection with its increase while in service a nuclear reactor. Optimization of the decision (i.e., a choice of the parameters providing optimum value of criterion function  $\Delta T_k$ , for example, its constancy) it was made by a method of consecutive iterations.

Results of optimization find out correlation of mutual influence of concentration of impurity on  $\Delta T_k$ , connected with redistribution and localization of these elements in volume of an irradiated material.

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#### **The Analysis of Radiation Dissolution of ODS-Particles in Ferritic/Martensitic Steels**

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The analysis of radiation dissolution of the oxide particles insert by a method of a mechanical alloying (MA) in ferritic-martensitic steel, in the neutrons flux with high energies ( $E_n=14$  MeV) is lead. The nuclear reaction of transmutation and elastic (inelastic) scattering of neutrons leads to diffusion of boundaries lattice/oxide particle and to occurrence of a stratum of introductions of atoms-yields of these processes in a matrix of steel. The Monte-Carlo method calculates lateral views of introduction of atoms of dispersion-strengthening oxide- particles behind their limits in a matrix of steel. The breadth of a band of introduction depends on the energies transmitted by neutrons in processes of interaction to atoms of an oxide particle. Radiation dissolution leads to decreasing of their size (i.e., to decreasing of their integrated density of a sink of radiation defects) and depends on a dose of an irradiation. The size of introduction depends on energy allocation of a flux of the atoms-resultant leaving an oxide- particle, and its size. Depending on the size of ODS-particle and their concentrations (that defines medial distance between oxide-coated particles in steel MA ferritic-martensitic class) became possible essentially various two lateral views of introduction of atoms-resultant in a matrix in space between particles because of superimposition of lateral views. In case of superimposition of lateral views of atoms of introduction in a matrix concentration increases.

The analysis of processes transmutation of atoms ODS-particles in nuclear reactors with neutrons has found out, that the dimensional volumetric factor of atoms- resultant of these

particles exceeds the dimensional factor of parent atoms of ODS-particles, - yttrium, hafnium, vanadium, the titan, etc. The subsequent processes of formation of educations from radiation -formed atoms- resultant in area between ODS - particles create new sinks of flaws of a radiation origin, that practically cancels diminution of density of sinks of ODS-particles in connection with their radiation dissolution. Process of neutralization of a loss of initial sinks by radiation sinks formed precipitates depends on dynamics of radiation dissolution of ODS - particles, formation new and is defined by radiation requirements of operation of a material, - dose rate of radiation damage (dpa/sec) and temperature of an irradiation.

#### **Swelling and Irradiation Creep of D9 Stainless Steel Cladding and Ducts in FFTF Driver Assemblies after High Neutron Exposure**

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The Ti-modified stainless steel designated D9 was developed for the U.S. fast reactor program to replace higher-swelling 316 stainless steel. This alloy was used to produce wire-wrapped fuel pins with 217 pins contained in a hexagonal duct, where cladding, wire, end caps and duct were all in the 20% cold-worked condition.

This report focuses on the swelling and creep behavior of the cladding and ducts of three fuel assemblies designated C1, D9-2 and D9-4 which were irradiated in the FFTF fast reactor to maximum exposures of 15.8, 25.3 and 21.4 x 10<sup>22</sup> n/cm<sup>2</sup> (E>0.1 MeV), respectively. The range of operating temperatures varied strongly, with the highest temperatures in C1, followed by lower temperatures in D9-2 and lower yet in D9-4. Temperature is the most important variable that determines the onset of swelling in this steel in this three-assembly comparison.

It was observed that relatively minor changes in the phosphorus level between two heats of cladding used in these assemblies had a very dramatic impact on the swelling. In both heats, the compositions fell below the specified upper limit for phosphorus but were a factor of 5 different. A similar situation occurred in a Russian fuel assembly where two heats fell below the specified silicon limit but by different amounts, leading to an equally dramatic difference in swelling. These results clearly show that specifications for “minor” elements must have both a lower and upper bound to insure reproducible behavior.

The duct operated at lower temperatures than the fuel cladding with swelling in D9-2 peaking at only 6-7% while the cladding peaked at 37-38%, with much of the pin length having attained the terminal swelling rate of ~1%/dpa.

While no failures were observed during in-reactor operation, void-induced embrittlement was observed in post-irradiation handling.

## Strong Impact of Neutron Spectra on Stress Relaxation of Inconel X-750 Springs in CANDU<sup>®</sup> Reactors

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Irradiation-accelerated stress relaxation is a well-known manifestation of irradiation creep. Frequently it is the practice to conduct irradiations in high-flux zones of a reactor and use the acquired creep relaxation data to predict the relaxation to be expected in lower-flux regions far outside the core. If the relaxation process is known to be independent of neutron flux or dpa rate such an extrapolation should be valid. If, however, there is a significant change in neutron spectra between the in-core and out-of-core locations this assumption needs to be evaluated much more carefully. Presented in this paper is a strong example where such a flux-independent extrapolation is not valid and can produce very misleading results.

During a routine maintenance outage in 2006 the adjuster rod guide tubes in a CANDU reactor that had operated for 18.5 effective full-power years at ~60°C were found to be completely relaxed. Further evaluation pointed to the relaxation of the Inconel X-750 spring at the bottom of the guide tube in the reactor core reflector region as the cause. It appeared that complete relaxation might have occurred much earlier than 18.5 years.

An in-core stress-relaxation experiment in NRU reactor indicated that the fast neutron dose required for complete relaxation of the Inconel X-750 spring was about  $1.2 \times 10^{25}$  n.m<sup>-2</sup>, E>1.0 MeV, a fluence corresponding to  $\geq 650$  years in the reflector region. A reduction in life from  $\geq 650$  years to  $\leq 18.5$  years obviously requires explanation, probably one based on some unique difference in neutron spectra between the in-core NRU position and the CANDU reflector region.

CANDU reactors are cooled with heavy water, a moderator with low absorption of thermal neutrons, but in the core absorption by fuel reduces the thermal neutron flux. Therefore the contribution to displacements by thermal neutrons is relatively small. Each (n, gamma) recoil event produces 4.9 displacements. Far out of core the thermal-to-fast neutron ratio increases strongly, however, such that the direct contribution to the total number of recoil-induced displacements by thermal neutron absorption becomes larger than that produced by fast neutrons.

Additionally, however, the time-dependent production of <sup>59</sup>Ni from <sup>58</sup>Ni in this high nickel alloy produces an even larger contribution to displacements. Most of these displacements arise primarily from the <sup>59</sup>Ni (n, alpha) <sup>56</sup>Fe reaction and secondarily from the <sup>59</sup>Ni (n, p) <sup>59</sup>Co reaction. Both of these strongly exothermic reactions have large cross-sections in highly thermalized spectra. Recoil of <sup>56</sup>Fe and the alpha particle produce 1762 displacements per event and the recoil of <sup>59</sup>Co and the proton produce 222 displacements per event. When the relative cross sections are taken into account the (n, p) reaction adds about 3.5% to that produced by the (n, alpha) reaction.



As a consequence of the thermal contributions the predicted total relaxation of >650 years based on the neutron fluence above 1.0 MeV would be reduced to 30-40 years if thermal neutron recoils are included and to only 3-4 years if the Ni-59 contributions are included.

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#### **Extensive Nano-cavity Development Observed at 33-70 dpa and 290°C-315°C in a PWR Flux Thimble Tube**

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The radiation-induced microstructure of a flux thimble tube from a currently operating PWR was examined. The tube was constructed from cold-worked 316 stainless steel. The helium and hydrogen levels were also measured. Two specimens at (33 dpa, 290°C) and (70 dpa, 315°C) were examined by transmission electron microscopy.

The original cold-worked dislocation network had completely disappeared during irradiation and was replaced by fine dispersions of Frank loops and small nano-cavities at very high densities throughout the grains. The latter appear to be bubbles containing high levels of helium and hydrogen. The presence of these cavities, especially strong on grain boundaries, is postulated to be involved in the development of strong intergranular stress corrosion cracking observed to be occurring during slow strain rate tests also conducted on this and other tubes.

The nano-cavities are best observed by electron microscopy but only when large levels of under-focus are employed. Otherwise these small cavities can easily remain undetected.

Comparison of these results with those of comparable studies on thimble tubes irradiated to lower exposures shows that hydrogen storage in cavities may be accelerating, in agreement with predictions based on earlier experimental studies.

#### **Study of Composition of Interaction Layer Formed Between Uranium-Molybdenum Fuel and Aluminum Matrix under Irradiation**

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U-Mo dispersion fuel is a candidate for those research reactors where the fuel of low U-235 enrichment is used. In the course of irradiation an interaction layer of (U, Mo) Al<sub>x</sub>-type is being formed between fuel particles and an aluminum matrix of a fuel meat, whose thickness, finally, limits a life time of fuel elements. At present time the means reducing the interaction of the fuel particles with the aluminum matrix are being developed. The data on

the kinetics and composition of the forming interaction layer are required for a solution of this problem.

For a complex study the fuel was irradiated to a maximum burn up of 53% in the IVV-2M reactor. The composition of the fuel included U-Mo alloy fuel particles of 36.3% U-235 enrichment, those particles were dispersed in the pure aluminum matrix. For testing the fuel specimens were cut from the fuel element areas at different positions.

It has been found that the thickness of the interaction layer formed between the fuel and the Al matrix increases with both the irradiation temperature and attained burn up. The interaction layer consists of uranium, molybdenum, aluminum and fission products. In the interaction layer the ratio of atoms of Mo, U and Al is  $1 \div 4 \div 20$ , which coincides with the results obtained in other investigations. According to the quantitative X-ray microanalysis data the mean atomic number of the interaction layer is equal to 25. The atomic number of the fuel structure components can be determined from an absorption current by the scanning electron microscopy. For that an experimental dependence of the absorption current against the mean atomic number was plotted for the specimens of the known composition. By this method the mean atomic number was equal to 42.

The obtained data prove a necessity of making specially prepared reference specimens for a precise determination of the interaction layer composition by X-ray spectrum microanalysis.

#### **Nnanoscale Structure Investigation of Fission Reactor Structural Materials**

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At present, considerable effort is directed toward study of nanoscale features of nuclear reactor materials. It is well known, that investigations of radiation defects in metals and modeled alloys have played significant role in understanding of degradation mechanisms [1]. Also, the process of radiation damages nucleation under irradiation is the most difficult and the most important for experimental studying. Essential information can be extracted at nano and atomic scales and it should represent not only structure changes but also chemical elements redistribution. On the other hand, the crucial detail under development of innovative nuclear reactor materials is some nanoscale structure features (different type of clusters and segregations) [2]. It is considered that these features may lead to improve long and short term mechanical properties and to increase radiation hardness. Tomographical atom probe technique used in this study makes possible to investigate nanoscale features described above. This technique allows to detect nanoscale clusters and segregations of chemical elements. Also, it helps to estimate the value of enrichment or depletion of different components in material matrix and structure defects.

In the present work nanostructured state investigation results of ferritic martensitic steels and yttrium oxide-dispersed ones are presented. Concentration of different chemical elements in the material matrix and in the nanoscale oxide clusters are shown.

Also new results of material study in course of surveillance specimen program for VVER-440 reactors are presented (it is irradiated, annealed and re-irradiated states of VVER-440 reactor pressure vessel steels weld). Significant information about redistribution of chemical elements during irradiation, annealing and re-irradiation is obtained.

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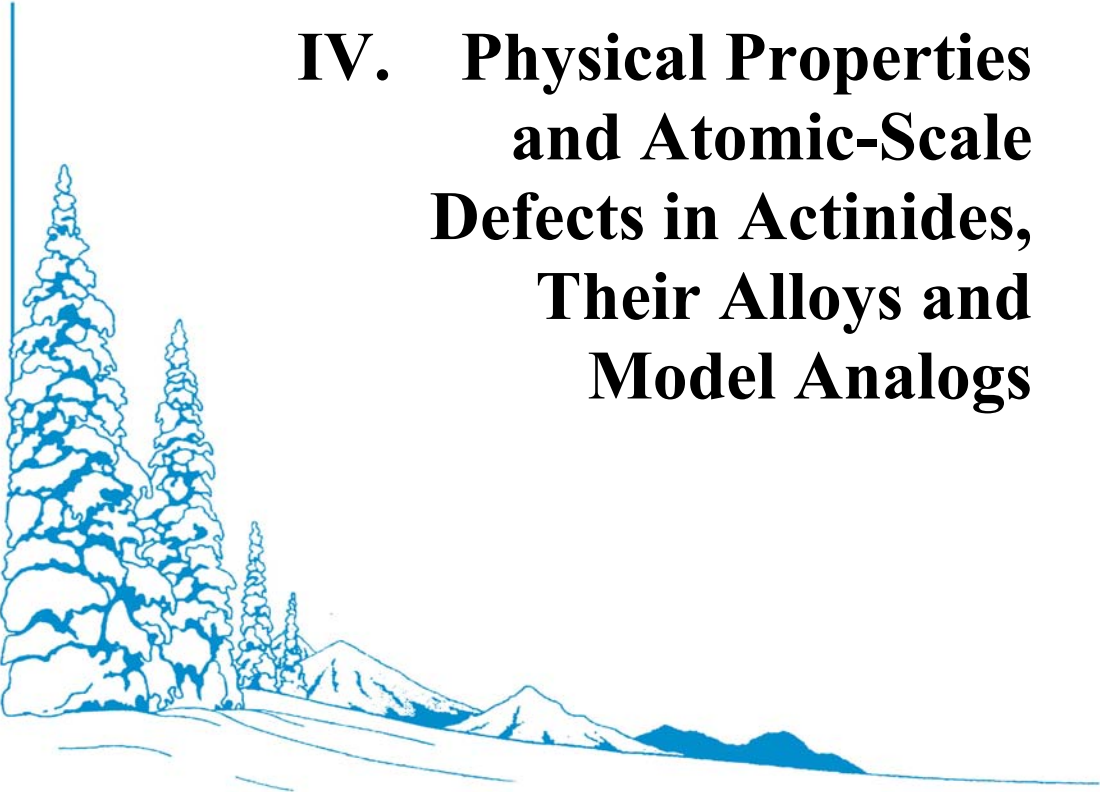
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### Phase Transformations in Alloys of Zr–Nb–Fe–Sn System

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Search for an optimal composition and state of materials for cores of water-cooled reactors with increased depletion rates calls for higher attention to alloys of the Zr-Nb-(Fe-Sn-O) systems, since components made from them show high performance characteristics under conditions of service within reactors. Investigations were carried out of the structural and phase state of shell tubes made from a series of alloys of the composition Zr-(0.6–1.2)Nb-(0–0.6)Fe-(0–1.5)Sn (% wt.) quenched from 600–940 °C for determining the temperature of phase transformations and the regions of existence of precipitates of second phases  $\beta$ -Nb,  $\beta$ -Zr,  $Zr(Nb,Fe)_2$  and  $(Zr,Nb)_2Fe$ .



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

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, physicommechanical, 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 analogs in different thermodynamic states, the mechanisms of phase transformations in them, to revealing the features of their electronic states and the interrelation of their crystalline structure, the electronic and magnetic properties of actinides and their compounds, the problems of their ageing, radiation stability, and response to external dynamic and shock actions.





## High-Temperature Superconductivity in Iron-Based Layered Compounds

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The author presents an overview of main experimental data on a new class of high-temperature superconductors: type *REOF*EAs ( $RE = \text{La, Ce, Nd, Pr, Sm, ...}$ ),  $A\text{Fe}_2\text{As}_2$  ( $A = \text{Ba, Sr, ...}$ ),  $A\text{FeAs}$  ( $A = \text{Li, ...}$ ) and FeSe(Te) iron-based layered compounds.

In a very a short period of study of the new superconductors, an impressive progress has been made in investigating their basic properties. The author offers a discussion of the electronic spectrum structure, including the role of correlations< the spectrum and the role of collective excitations (phonons, spin waves), and the principal models describing the possible types of magnetic ordering and Cooper pairing in these compounds.

There are quire justified hopes for more new systems to be discovered in the immediate future. Undoubtedly, the already performed works have expanded our understanding of the nature of high-temperature superconductivity, although we are still quite far from working out concrete “recipes” of search for new high- $T_c$  superconductors.

## Pressure Effects in CeNi

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The intermetallic compound CeNi is known as a classical intermediate-valence system. CeNi crystallizes in the CrB-type orthorhombic structure (space group *Cmcm*). The pseudo-binary solid solution  $\text{Ce}_{1-x}\text{RE}_x\text{Ni}$  ( $\text{RE} = \text{La, Lu, Y}$ ), having the same crystal structure, can be synthesized for a wide range of  $x$ , allowing thus to study the effects of chemical pressure on the  $f$ -electron properties of CeNi. Moreover, CeNi undergoes a pressure-induced first-order structural phase transition accompanied by a volume jump [1]. Thus, investigation of both the chemical and external pressure effects in CeNi can contribute to the understanding of the pressure-temperature phase diagrams and the nature of the magnetic-structural phase transitions connected with the  $f$ -electron instability and strong electron correlations [2].

We discuss the results of specific heat and magnetic susceptibility measurements for the  $\text{Ce}_{1-x}\text{La}_x\text{Ni}$ –CeNi– $\text{Ce}_{1-x}\text{Lu}_x\text{Ni}$  series. It is shown that the chemical compression increases the  $f$ -electron hybridization in the system [3]. The effective Ce valence is estimated to vary

from 3.11(1) in  $\text{Ce}_{0.9}\text{La}_{0.1}\text{Ni}$  to 3.14(1) in  $\text{CeNi}$  and 3.23(2) in  $\text{Ce}_{0.6}\text{Lu}_{0.4}\text{Ni}$ . We conclude that the "Kondo physics" dominates the behavior of the system under chemical compression.

By measuring the magnetic susceptibility under external pressure we extended the pressure- temperature phase diagram of  $\text{CeNi}$  and showed that only two  $\text{CeNi}$  phases exist within the  $P$ - $T$  domain up to  $P = 2$  GPa and  $T = 300$  K [3]. The volume jump at the room temperature structural transition is estimated to be 6.5%. The pressure-induced first-order structural phase transition is also observed in the chemically compressed composition  $\text{Ce}_{0.9}\text{Lu}_{0.1}\text{Ni}$  at room temperature. The results obtained allow to compare the effects of external and chemical pressure in the  $\text{CeNi}$  system.

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### Kondo Universality and Energy Scales in Plutonium

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The macroscopic properties of  $\alpha$  and  $\delta$ -plutonium were analyzed within the Fermi-liquid approach as well as the properties of a few model rare earth-based systems. The following major parameters in Pu were estimated within the single-site approximation: the characteristic Kondo energy, the  $f$ -electron shell occupation number, the effective degeneracy of the ground  $f$ -multiplet, the crystal field splitting energy. The ground state in  $\delta$ -plutonium is a quantum superposition of atomic states [1] According to its macroscopic physical properties  $\delta$ -Pu at low temperature is very close to the intermediate valence regime [2]. The temperature dependence of the static magnetic susceptibility in plutonium was calculated. The absence of magnetism issue in plutonium [3] is discussed in a quantitative manner on the basis of the estimated major parameters. Since the universal Wilson criterion and the Kadowaki-Woods universal relation are valid in  $\delta$ -Pu it can be considered as a Kondo system, while the position of  $\alpha$ -plutonium in the general classification of solids remains a puzzle. The plutonium homology issue is discussed.

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**Magnetism of Uranium in Strong Ferromagnetic Matrix  
(uranium magnetic materials - is it possible?)**

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Uranium intermetallics, where the 5f electrons play dominant role in formation of magnetic properties, are subject of intensive studies. The most of interesting properties of most of U intermetallics, however, are manifest at low or very low temperatures. „Pulling out“ the f-electron magnetism to elevated temperatures by employing the f-3d intersublattice exchange interactions, which was very successful in the 4f rare-earth compounds, seems to fail in the case of the 5f electrons. In the U intermetallics with 3d metals (T), the strong 5f-3d hybridization delocalizes the 5f electrons. Consequently, the U magnetic moment is either zero or strongly reduced. Simultaneously, the 5f-3d hybridization reduces (up to zero) the magnetic moment of T atoms. Nevertheless, in contradiction with this general trend, several U-T intermetallics are known in which both U and T are magnetic even at room temperature. This will be shown on the example of  $U_2(T,Si)_{17}$  and  $UT_{12-x}M_x$  ( $M = Al, Si$ ) compounds with following conclusions:

1. Magnetic ordering of U and 3d-metal can coexist in the same intermetallic compound.
2. Inter-sublattice interaction can pull the U magnetism to rather high (room and higher) temperature.
3. Nevertheless, perspectives of real magnetic materials with uranium look currently rather pessimistic from several viewpoints.

### Spin-State Polarons in Lightly Hole-Doped $\text{LaCoO}_3$

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Physical properties of nanostructured magnetic materials are extensively studied because of their fundamental interest and potential applications. A naturally occurring analog

to the artificially fabricated heterostructures are hole-doped cobaltites  $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$  with intrinsic inhomogeneities, i.e. with a spatial coexistence of magnetic clusters in a nonmagnetic matrix.

In this work, we elucidate the mechanism of how already the light hole doping  $x \sim 0.002$  dramatically affects magnetic properties of  $\text{LaCoO}_3$ . Using inelastic neutron scattering (INS) data, obtained with and without external magnetic field, we find that the charges introduced by substitution of  $\text{Sr}^{2+}$  for  $\text{La}^{3+}$  do not remain localized at the  $\text{Co}^{4+}$  sites. Instead, each hole is extended over the neighboring  $\text{Co}^{3+}$  ions, transforming them to higher spin state and thereby forming a magnetic seven-site (heptamer) polaron. Spin-state polarons behave like magnetic nanoparticles embedded in an insulating nonmagnetic matrix. The present data give evidence for two regimes in the lightly hole-doped samples: i)  $T < 35$  K dominated by spin polarons; ii)  $T > 35$  K dominated by thermally activated magnetic  $\text{Co}^{3+}$  ions. Additional charge carriers increase the number of such spin-state polarons, which form a percolative network resulting in a metallic state

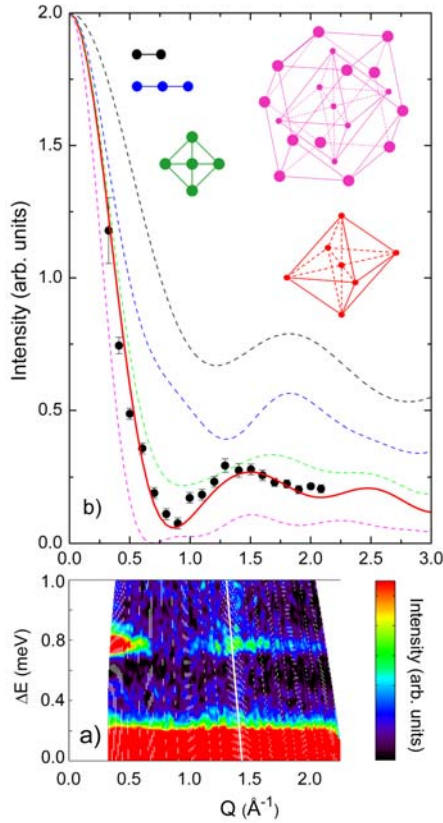


Fig. 1. a) Excitation INS spectrum from  $\text{La}_{0.998}\text{Sr}_{0.002}\text{CoO}_3$  at  $T = 1.5$  K. b) Circles: experimental  $Q$  dependence of the intensity of the peak observed at 0.75 meV. Lines: Calculated  $Q$  dependence of the neutron cross section for different Co multimers (visualized in the Figure) in the cubic perovskite lattice of  $\text{LaCoO}_3$  and for  $|\text{Si}\rangle \rightarrow |\text{Si}\rangle$  transitions

with long-range ferromagnetic order at the critical concentration  $x_c = 0.18$ .

### Actinide Compounds: From Heavy Fermions to Magnetically Ordered Systems

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This lecture will concern the physical properties of light actinide (An) intermetallic compounds, which are closely connected to the particular nature of the  $5f$  states. Their energy close to the Fermi level and relatively large spatial extent are responsible for a considerable hybridization of the  $5f$  states with the valence states of neighbouring atoms in the crystal lattice, and hence for their participation in bonding. Moreover, a strong spin-orbit interaction frequently exists, which provides a powerful connection between the direction of  $5f$  magnetic moments and the crystal structure. All this leads to a large diversity in physical behaviour, as the nature of the  $5f$  electrons can be strongly affected by external variables like the crystal structure, nature of the nearest-neighbour atoms, magnetic field, temperature, pressure, etc.

The correlation between magnetic behaviour and the An interatomic spacing was first pointed out by Hill [1], who proposed that the  $5f$  overlap between An neighbour atoms determines whether the actinide atoms are magnetic or non-magnetic. Most of U-based compounds behave as Hill expected: for  $d_{U-U} \leq 3.4 \text{ \AA}$  the  $5f$  wave functions overlap and form a broad band with relatively low density of states at the Fermi level, and thus the lack of magnetism and conventional superconductivity are favoured. For larger U-U spacing there is very small direct  $5f$  overlap and U magnetic order can be expected. However, because the Hill's rule does not take into account the details of the bonding and resulting density of states, frequently determined by the  $5f$ -ligand hybridization, there are several exceptions to this rule. Heavy-fermion U superconducting compounds are one of the clearest exceptions: the  $f$  electrons condensate into a superconducting state although the spacing between the U atoms is fairly large. The existence of U magnetism in compounds with  $d_{U-U}$  lower than the Hill limit is much rarer and examples will be discussed in detail. Until recently,  $\text{UNi}_2$ , crystallizing in the hexagonal  $\text{MgZn}_2$ -type structure (Laves C14), was the only clear exception to this rule, as  $d_{U-U} < 3.2 \text{ \AA}$  and it exhibits a weak itinerant ferromagnetism below approx. 20K [2]. Very recently, a new magnetic compound,  $\text{U}_2\text{Fe}_3\text{Ge}$ , crystallizing in the  $\text{Mg}_2\text{Cu}_3\text{Si}$ -type structure, an ordered variant of the C14, was reported by two independent groups [3,4], stressing once more the fundamental role of the nature and ordering of ligands on the physical properties of An intermetallic compounds. Pressure studies, changing the An-An distances, are expected to give additional information on the character of the  $5f$  states.

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### Valence Fluctuations in Actinides

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The long-standing issue of non-magnetic  $\delta$ -Pu, which could not be captured by conventional Density Functional Theory, triggered lot of interest in fundamentals of electronic structure of actinide systems. At present, the non-magnetic state can be obtained already by various types of calculations, although this does not help to distinguish, which types processes are dominant when forming or suppressing magnetic moments in Pu based systems and in general in light actinides. Recent DMFT calculations [1] deduce the Kondo effect responsible for the lack of magnetism in  $\delta$ -Pu. On the other hand, static LDA+U calculations [2,3] also lead to a non-magnetic state if the  $5f$  occupancy exceeds approximately 5.3.

This situation requires to refer carefully to various regimes know from anomalous rare earths (Ce, Yb, Sm, Eu, Tm), which exist on the path between the localized, atomic,  $4f$  states on one side and band states on the other side. Here, besides the Kondo regime, acting close to integral  $4f$  occupancy (i.e. weak  $4f$  instability), a regime of valence fluctuations occurs. In this case, the average  $4f$  occupancy is significantly different from integer, but the fluctuations  $4f^n \rightarrow 4f^{n-1} + 1$  cond. el. are still relatively slow, with the characteristic timescale interfering with the scale of thermal fluctuations. The charge fluctuations are the reason for the destruction of magnetic moments without invoking any spin-compensation mechanisms.

It is very difficult, in case of Pu systems, to obtain a direct insight in the dynamics of charge fluctuations e.g. by neutron scattering. But the valence fluctuators are characterized by very specific bulk properties and photoelectron spectra. Here we will discuss analogies between properties of Pu compounds and rare-earth valence fluctuators from the point of view of spectroscopies and occurrence of other specific markers as a negative value of elastic coefficient  $c_{12}$ .

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### Spontaneous and Field-Induced Magnetic Transitions in $\text{RBaCo}_2\text{O}_{5.5}$

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A detailed study of magnetic properties of cobaltites  $\text{YBaCo}_2\text{O}_{5+x}$  has been performed in high (up to 35 T) magnetic fields and under hydrostatic pressure up to 0.8 GPa. The oxygen content  $x = 0.49 \pm 0.01$  was chosen close but slightly lower than the stoichiometric value  $x = 0.50$ . The composition shows phase separation into two perovskite-based phases, which is supposed to be the immanent feature of this compound. Contrary to the previously studied sample  $\text{YBaCo}_2\text{O}_{5+x}$  with  $x = 0.52 \pm 0.02$ , where the two-phase state was realized only below 190 K, these phases coexist in the studied composition already at room temperature. Measurements of temperature dependence of magnetization revealed both the phase separation and a set of spontaneous magnetic transitions between para- ferro- and antiferromagnetic states. In the temperature range of existence of the AF phase, application of high magnetic field induces the metamagnetic AF-FM phase transition. In contrast to the rare earth compounds  $\text{RBaCo}_2\text{O}_{5+x}$  with magnetic R ions, in the studied system with yttrium even the field of 35 T is not enough to completely transform AF phase into FM state at low temperatures. Analysis of the high-field susceptibility in the field-induced FM phase shows that strong magnetic field results in the change of the Co spin state in these compounds.

We determined pressure derivatives for the temperatures of magnetic transitions. The observed behavior is in agreement with the available data on the transition temperatures and unit cell volumes for the compounds with different R atoms. We also observed the effects of magnetic viscosity (likely due to the delay of the motion of the narrow domain walls separating AF and FM phases). In order to check whether the observed effects are common for the underdoped cobaltites, we synthesized the compounds  $\text{RBaCo}_2\text{O}_{5.5}$  for  $R = \text{Tb}$  and  $\text{Pr}$  with the same oxygen content, using the same sample preparation technique. Magnetic measurements for these samples reveal the effects of phase separation, very similar to those observed for the Y-containing sample.

#### Acknowledgements

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### The Advancement of Work on Inert Matrix Fuel

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Recent aims to destroy plutonium in the most effective way have led to the development of a uranium free fuel based on zirconia concepts for this so-called "inert matrix fuel-IMF." IMF is a non-fertile oxide fuel consisting of  $\text{PuO}_2$  diluted in inert oxides such as stabilized  $\text{ZrO}_2$ , its main advantage is that it does not produce new plutonium during irradiation, as it does not contain uranium (U-free fuel). An addition of thoria in the matrix (thoria-doped fuel) may be required for coping with reactivity feedback needs. Several ceramics (ceramic + ceramic) and cermets (ceramic + metal) are good candidates for plutonium fuels without uranium or for the targets related to the heterogeneous recycling of minor actinides. Those ceramics are actinide compounds ( $\text{PuO}_2$  or  $\text{AmO}_2$ ) dispersed in inert matrix such as  $\text{MgO}$  or  $\text{MgAl}_2\text{O}_4$ . All U-free fuels are envisaged to be operated under a once-through cycle scheme - the spent fuel is supposed to be spent directly to the final disposal in deep geological formations without requiring any further reprocessing treatment. The objective of this paper is to discuss the advancement of work on inert matrix fuels and discuss the damage sources of inert matrix fuels for transmutation of minor actinides or for burning excess Pu, which are the following:

1. Thermal or fast neutrons.
2.  $\alpha$ -decay (5-6 MeV He-ions, 100 KeV daughter recoil atoms).
3. Fission fragment impact (70-100 MeV heavy ions of elements between Ga and Dy).
4. Intense  $\beta$ - and  $\gamma$ - radiation.

### Physics of the Kondo-Insulators: Neutron Spectroscopy Study

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The extended study of the magnetic and atomic excitations in valence unstable system based on rare earth ions of Sm [1,2], Yb [3] and Eu [4], have been performed by the thermal neutron spectroscopy method. The results obtained allow to get characteristics of the ground state for  $\text{SmB}_6$ ,  $\text{Sm}(\text{Y})\text{S}$ ,  $\text{YbB}_{12}$ ,  $\text{EuCu}_2\text{Si}_2$ , to establish the features of the temperature driven transition from the nonmagnetic regime to the spin fluctuative state at temperatures above 50...100K, which has been studied in fine details for Kondo-insulator  $\text{YbB}_{12}$  for the first time. The interesting feature for the systems like  $\text{Sm}(\text{Y})\text{S}$  and  $\text{YbB}_{12}$  is the clear evidence of the cooperative character of the excitations for the magnetic subsystem at low temperature, it appears as the dispersions of energy and intensity in the q-space. The physical models are

analyzed suggested to be responsible for the properties of the systems. Special attention is paid to the problem of the low energy excitation (in analogy with “resonance mode” in HTSC materials). The peculiar interplay between the number of basic interactions in such systems with f-electrons (exchange, crystal field, etc. [1-5]) is discussed.

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### Investigations of Spin Excitations in High-Temperature Superconductors by the Neutrons Scattering Method

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The paper traces the development of investigations into excitations of the spin nature, first identified as “magnetic resonance” in the YBa<sub>2</sub>Cu<sub>3</sub>O<sub>6+x</sub> system [1], which had attracted increased attention by the fact of its emergence at the temperature of superconducting transition. These works make use of all the advantages of the neutron spectrometry method developed on stationary nuclear reactors and enriched by application of “optical” focusing and analysis of neutron beams polarization.

The gathered experimental data are discussed in terms of development of representations on the nature of conducting states in the investigated systems.

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### Nonlinear Charge Transport in the Magnetite/Semiconductor Structure: Effects of Magnetic Field and Optical Radiation

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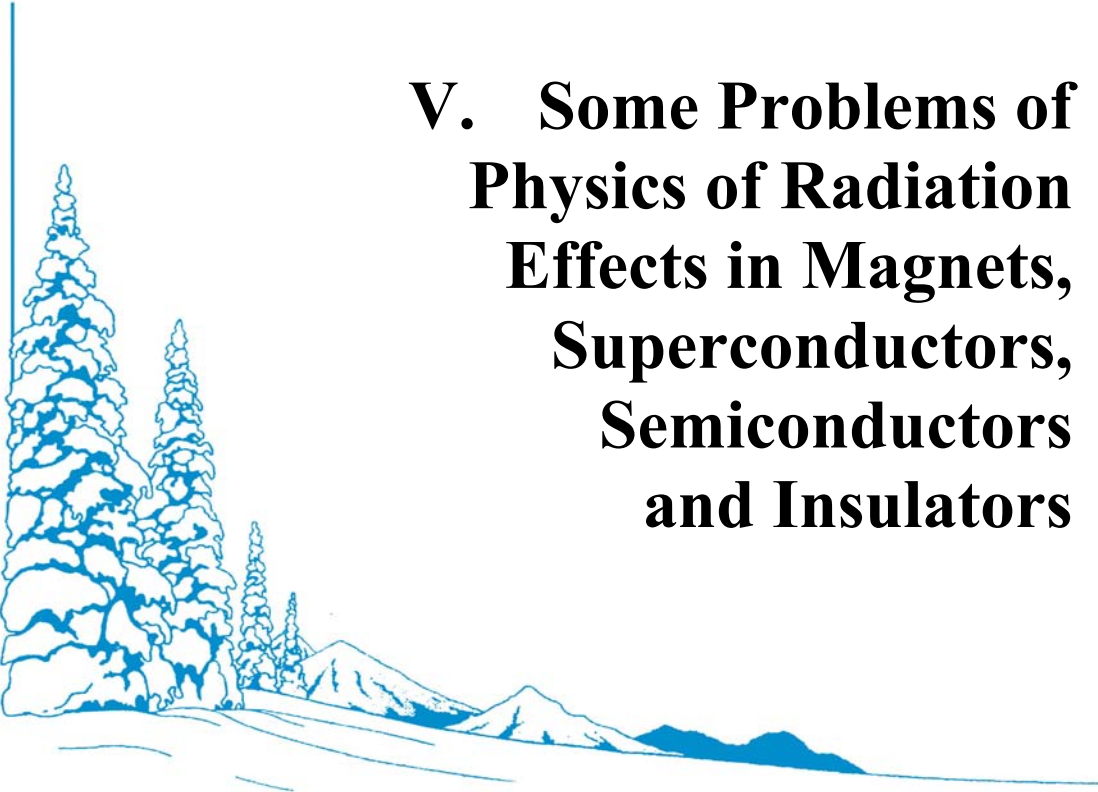
Recently, research interest has turned to the fabrication and study of the hybrid manganites/semiconductor structures where classical Si- or GaAs-based semiconductors are used. On the one hand, such manganite-based devices are found compatible with current semiconductor technology (for example, CMOS technology), on the other hand, they can be expected to exhibit novel characteristics which might be useful for spintronics applications.

The tunnel manganite/depletion layer/Si:Mn structure was fabricated to study characteristic transport and magnetotransport properties using CIP (current in plane) geometry. The manganite is  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ , the manganite depletion layer in the structure constitutes the potential barrier sandwiched between two conducting manganite and Si:Mn layer. Voltage-current characteristics of the structure show a nonlinear behavior, that is caused by conducting channel switching from the upper manganite film to the bottom more conductive Si: Mn layer at increasing of the current applied to the structure. The current bias assists the tunneling of the carrier across depletion layer establishing a low resistance contact between current-carrying electrodes and bottom layers. Below 30 K, both conducting layers are in ferromagnetic state (magnetic tunnel junction), this open up possibility to control the resistance of the tunnel junction and, consequently, the switching of the conducting channels by the magnetic field. The latter, in fact, defines the novel mechanism of the magnetoresistance realized in the magnetic layered structure with the CIP geometry. The magnetoresistance for structure studied achieves almost 300 % in magnetic field less than 1 kOe. Positive value of the observed magnetoresistance is related to peculiarities of the spin polarized electronic structures in the manganite and the silicon doped with manganese.

In addition, we have detected the effect of optical radiation ( $\lambda=1 \mu\text{m}$ ) on the transport and magneto-transport properties of the film. This effect is not heat in origin. The strongest effect of optical radiation is observed below 30 K. The applied magnetic field suppresses photo-induced changes in the transport properties of the structure. We suggest that electron-hole pairs are generated in the manganite and Si:Mn layers as a result of influence of the irradiation. The photogenerated carriers tunnel through depletion layer contributing to the photoelectric effect. Since the tunneling probability strongly depends on the magnetic state of the structure, the external magnetic field can effectively control the observed photoelectrical effect.







## 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.





### Polaron States in Electron-Irradiated $\text{LaMnO}_{3+\delta}$ Manganite

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The interest in manganites arises from the effect of colossal magnetoresistance (CMR), which is observed above and below the Curie temperature  $T_C$ . Stoichiometric  $\text{LaMnO}_3$  only contains  $\text{Mn}^{3+}$  ions and is an A-type antiferromagnet with  $T_N = 140$  K.  $\text{Mn}^{4+}$  ions appear if the composition becomes nonstoichiometric or  $\text{La}^{3+}$  ions are replaced by  $\text{A}^{2+}$ . The ferromagnetic  $\text{Mn}^{3+}$ - $\text{Mn}^{4+}$  superexchange and double exchange modify the magnetic order. Depending on the temperature and the  $\text{Mn}^{4+}$  concentration, the  $\text{LaMnO}_3$  manganite can have different crystal symmetries: orthorhombic  $O'$  ( $c/\sqrt{2} < a < b$ ) and  $O^*$  ( $a < c/\sqrt{2} < b$ ), rhombohedral and monoclinic symmetries. The feature of manganites is their internal structural and charge inhomogeneities: the spatial separation of the AF and FM regions.  $T_C$  is insensitive to the crystal symmetry, but the effect of structural transitions on the paramagnetic properties is poorly understood. According to the new temperature scale, orbital-correlated polarons exist at  $T_C < T < T^*$ , while uncorrelated Yahn-Teller polarons are present at  $T^* < T < T_{\text{pol}}$ . Orbitally ordered regions, which play the key role in the CMR effect, were observed only in the  $O^*$  phase [2]. Uncorrelated polarons are present in all structures.

This study deals with the effect of electron irradiation on paramagnetic susceptibility of  $\text{LaMnO}_{3+\delta}$  with the  $O^*$  lattice symmetry at  $T = 300$  K. The lattice parameters were  $a = 5.515$  Å,  $b = 5.532$  Å, and  $c = 7.798$  Å. Irradiation was performed at  $T = 423$  K. Magnetic measurements were made using the Faraday balance at temperatures of  $77 \text{ K} \leq T \leq 600 \text{ K}$ . The Curie temperature was determined from the dependence  $\chi_{\text{dc}} = f(T)$  in a weak magnetic field. The temperature dependences of the paramagnetic susceptibility can be divided into several regions. In the initial sample the Curie-Weiss law with  $\theta = 252$  K and  $\mu_{\text{eff}} = 4.68\mu_B$  is fulfilled starting from  $T > 440$  K. At  $300 \text{ K} < T < 440 \text{ K}$   $\mu_{\text{eff}} = 5.36\mu_B$ , while at  $T < 300 \text{ K}$   $\mu_{\text{eff}} = 6.41\mu_B$ , which is much larger than the theoretical value  $\mu_{\text{eff}} = 4.91\mu_B$  for  $\text{Mn}^{3+}$  ions. Large values of  $\mu_{\text{eff}}$  can be explained by formation of polarons with a high magnetic moment near defects. The polarons are in the paramagnetic state as indicated by the linear field dependences of the magnetization. The Curie temperature decreases from  $T_C = 153$  K to  $T_C = 149$  K on exposure to the fluence  $\Phi = 5 \cdot 10^{18} \text{ cm}^{-2}$ . The paramagnetic polarons vanish at lower temperatures  $T < 320$  K. A two-phase ( $O^*$  and R) state is realized at  $T > 450$  K [3]. Irradiation causes structural transitions and weakening of ferromagnetic interactions.

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## Magnetic Susceptibility of Electron-Irradiated Copper Monoxide

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The effect of radiation defects on magnetic properties of a CuO polycrystal and nanoceramic was studied. The copper monoxide has a monoclinic lattice and is a low-dimensional (1D) antiferromagnet, which transforms into the 3D collinear state at  $T < T_N = 230$  K. The magnetic order is determined by superexchange interactions of  $\text{Cu}^{2+}$  ions ( $S = 1/2$ ) via oxygen ions. The antiferromagnetic (AF) superexchange along the  $[10\bar{1}]$  axis is most intensive. The ferromagnetic (FM) exchange is much weaker in all the other directions.

The magnetic energy of the materials includes the exchange, anisotropy, magnetoelastic and magnetostatic energies. Any inhomogeneities can change the magnetic ground state. The exposure to electrons produces point defects, which can modify the exchange coupling distances and angles, similarly to surface defects in nanoparticles. Anomalous for 3D antiferromagnetics the increase in the susceptibility  $\chi$  was detected in samples irradiated with electrons to the dose  $\Phi = 5 \cdot 10^{18} \text{ cm}^{-2}$  as the temperature decreased to  $T < 150$  K. The values of  $\chi$  in the nanoceramic were larger than the corresponding values in the polycrystal. This was due to surface defects of the crystallites in addition to their radiation defects. The analysis of possible reasons for the increase of  $\chi$ , the field dependences of  $\chi$ , and the appearance of the FM moment demonstrated that the presence of paramagnetic  $\text{Cu}^{2+}$  and  $\text{Cu}^{3+}$  ions in the AF matrix could not lead to the observed behavior of  $\chi$ . The anomalous magnetic properties were not connected with the superparamagnetism either as its distinctive feature, coincident magnetization isotherms in the coordinates  $M = f(H/T)$  at different  $T$ , was not fulfilled. The most probable reason was an inhomogeneous magnetic state, namely the presence of the FM polarons in the AF matrix. Whatever the nature of point defects in CuO, they probably caused local changes in the exchange parameters and formation of regions with FM ordered  $\text{Cu}^{2+}$  ions near defects. The start temperature of the FM polaron formation was close to 150 K. Small values of the spontaneous magnetic moment were due to the small size and a low number of polarons. As the temperature increased to  $T > 150$  K, the FM exchange weakened and it was more favorable in energy terms for the copper monoxide to pass to the homogeneous AF state. The time for relaxation of radiation defects is much longer than that of elastic stresses in the nanoceramic [1]. In 5 years the susceptibility of the irradiated CuO samples does not reach the value characteristic of the initial state.

This study was supported by the program of RAS "New materials and structures" and scientific program of Far-Eastern Branch and Ural Branch RAS.

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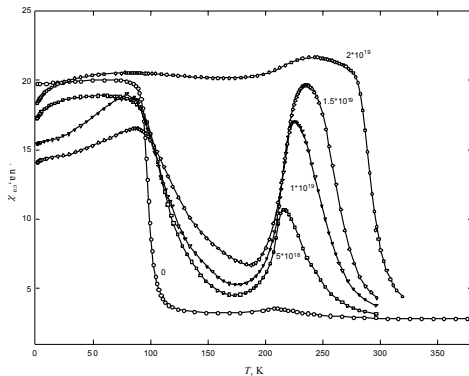
## Low-Temperature Investigation of Magnetic and Crystal States of Radiation-Disordered $Ce_2Fe_{17}$

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In our earlier works [1-2], the effect of high fast neutron fluences ( $\Phi \geq 2 \times 10^{19} \text{ cm}^{-2}$ ) on the magnetic and structural states of the intermetallic compound  $Ce_2Fe_{17}$  was investigated. It was found that under irradiation with fast neutrons a disordered crystal state emerges, being accompanied with significant atom displacements from their regular lattice sites, and further such state goes amorphous under a maximum fluence. At that, transition of a complex magnetic state (antiferromagnetic at  $T_N \sim 205 \text{ K}$  and ferromagnetic at  $T_C \sim 94 \text{ K}$ ) to a ferromagnetic state took place over the entire temperature range below  $T_C \sim 300 \text{ K}$ .



In the present work we successively irradiated  $Ce_2Fe_{17}$  with small fluences of fast neutrons ( $5 \cdot 10^{18} \text{ cm}^{-2} - 2 \cdot 10^{19} \text{ cm}^{-2}$ ) in order to follow the process of magnetic state transformation (AF-F). Under such fluences, strengthening of the antiferromagnetic peak of susceptibility and its shift towards the higher

temperature range are observed (see the Figure). Model calculation of

Temperature dependences of temperature susceptibility of initial and irradiated  $Ce_2Fe_{17}$  samples

temperature dependence of magnetic susceptibility of a two-sublattice antiferromagnet at different values of inter-sublattice exchange interaction (ISEI) has shown that Neel point susceptibility grows with ISEI decrease, which is manifested by growth of the magnetic susceptibility peak under irradiation. This conclusion is in agreement with disappearance of the ordering AF under high fluences, when the exchange between sublattices ceases and the AF disintegrates. In support to this conclusion, neutron diffraction investigations were conducted.

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## Research of neutron radiation influence on properties OF GaN/InGaN - structures

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Recent interest of leading industrial nations to GaN-technologies was a precondition of this study what is defined by expected applicability of GaN-based devices in UHF systems under high temperatures and intensive radiation fields.

In this study, consideration was given to the influence of 1 MeV [1] and 14 MeV [2] neutrons on electrical, structural, and optical features of GaN film, GaN/InGaN-structures, and GaN/InGaN-based light-emitting diodes (LEDs) made using MOCVD.

Dependencies of concentration and mobility of free charge carriers in GaN-film on the neutrons fluence were obtained. Gamma-neutron radiation influence on LEDs voltage-current characteristics was studied.

Geometry of quantum wells before and after irradiation was investigated with the help of XRD.

Dependencies of photo-, and electroluminescence intensity of heterostructures on neutrons fluence were obtained. The recovery of irradiated LEDs luminescence intensity immediately after irradiation by pulsed gamma-neutrons was studied. The broadening and biasing effect of light emitted by irradiated structures was observed.

The model [3] describing major experimental results was suggested. This model is capable of predicting sensitivity of GaN- structures to neutron radiation.

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### Electronic Structure of CVD Diamond Irradiated with Fast Neutrons

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Investigation was carried out of the effect of disordering induced by irradiation with fast neutrons at  $(325 \pm 10)$  K on the behaviour of resistivity  $\rho$ , magnetic susceptibility  $\chi$ , heat capacity  $c_V$ , Raman scattering and structural state of samples ( $4 \cdot 2 \cdot 0.5$  mm<sup>3</sup> slabs of polycrystalline CVD diamond synthesized in microwave plasma), in the high fluences range  $\Phi = (1-5) \cdot 10^{20}$  cm<sup>-2</sup>. Neutron diffraction pattern measurements show that the crystal structure remains stable in this fluences range without noticeable widening of structural reflections. Increase of crystal lattice relative volume is well expressed by a simple dependence describing the effects of saturation at defects formation:  $\Delta V/V \sim 1 - \exp(-\Phi/\Phi_0)$ , where  $\Phi_0 = 1.2 \cdot 10^{20}$  cm<sup>-2</sup>. The value of  $\rho$  significantly decreases under irradiation and displays an activation-type temperature dependence with energy  $E_a \approx 0.3$  eV. The Raman scattering data point to complete suppression of one-phonon optical mode at  $1332$  cm<sup>-1</sup> and emergence of an extended spectrum in the range of  $(100 - 1700)$  cm<sup>-1</sup> corresponding to phonon state density. Irradiation leads to suppression of the type Curie-Weiss paramagnetic contribution  $\chi = C/(T + T_0)$ ,  $T_0 \approx 3$  K,  $C \approx 10^{-3}$  K, and also to a strong (by 4 orders of magnitude) increase of  $c_V$  in the low temperatures range with a dependence characteristic of a multilevel electron system of the Schottky anomaly type (Fig. 1). Thus the region in the vicinity of the radiation defect carries an effective electrical charge, a magnetic moment, and forms a multilevel electron system with the splitting scale of the order of units of meV.

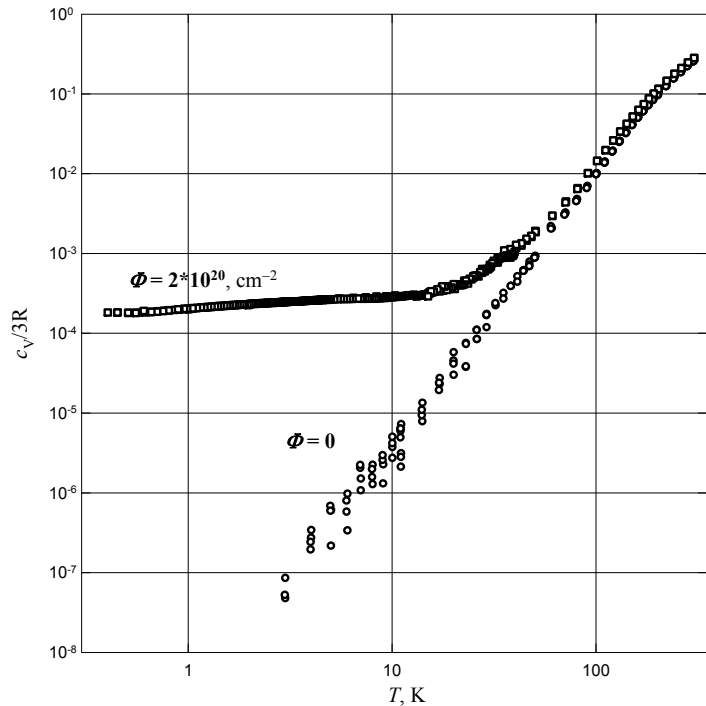


Fig. 1. Temperature dependences of heat capacity of initial and irradiated CVD diamond samples

## **Crystal-to-Amorphous Solid Transformation under Irradiation with Fast Neutrons: Laws and Mechanisms**

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On the basis of analysis of physical properties of compounds with garnet and perovskite structure irradiated to different fluences of fast neutrons, the principal laws governing radiation amorphization were established. It was shown that the factor causing radiation amorphization is statistical redistribution under irradiation of  $3d$ -  $4f$ - cations of transition metals with significantly differing ion radii over nonequivalent crystallographic positions (i.e., formation of antisite defects)). Significant nonuniform static displacements of ions appearing in this process inevitably result in a loss of translational symmetry. Analysis of the results of investigation shows that coordination polyhedrons characteristic of the initial crystal structure are preserved in the amorphous state formed under irradiation (clearly, in a distorted state), while mean Me-O inter-ion distances and Me-O-Me bond angles are close to similar values in the crystal. The amorphous and crystal states in this case actually differ only in the values of ions displacement from the equilibrium position and in their directional properties. It is essential that with such “distortion”-type mechanism of amorphization, the chemical composition and continuity of the sample actually remain the same.

Based on analysis of experimental results, correlations between the following values were established: fluence  $\rightarrow$  antisite defects concentration  $\rightarrow$  nonuniform static ions displacement value. Critical concentrations and displacements at which amorphization takes place were estimated. The results of investigation of local atomic and magnetic structures of radiation-amorphized compounds are given.

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### **Radiation Stability of Nanostructures**

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Radiation stability of materials used in nanoelectronics in the manufacture of electronic and optical components is considered from a general viewpoint.

There is given a detailed review of works testifying to the fact that increased radiation stability presents a general feature of nanosize structures both in relation to degradation of functional parameters and in relation to the rate of introduction of radiation-induced structural disturbances. Experimental results are discussed.

A model of this effect explaining the effect of increased radiation stability of nanocrystalline materials is proposed and discussed. The fields of application and prospects for use of nanoelectronic materials with increased radiation stability are considered.

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### **Structural-Phase Transformations in Carbon Materials under Gamma-Irradiation**

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The influence at different temperatures of 60-Co and nuclear reactor gamma-irradiation of the structure of carbon material;s (fullerene soot, graphite, synthetic diamond) was investigated. The DRON plant with improved beam collimation and higher resolution, including small-angle scattering, was used. Under certain conditions there were detected structural-phase amorphous carbon – fullerene – graphite – diamond transformations by the nucleation mechanism. The influence of the size of nano-inclusions on material properties was found out.

The work was supported by the Committee of Science and Technology Development of Uzbekistan, Project F2-068.

### **Radiation-induced modification of the semiconductors electronic properties as the process of self-compensation: modeling calculations and experiment**

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Rigid radiating influence causes the change of the electronic properties of semiconductors and the shifting of the Fermi - level  $F$  in the limiting position  $F_{lim}$ , which is the characteristic value for the each material. In the theoretical models, the  $F_{lim}$  identifies with the "initial" charge neutrality level  $F_{in}$  of a crystal that allows a-priori to calculate the electronic properties of the defective semiconductors [1, 2]. As supposed the driving force causing the change of the electronic properties of a material at an irradiation is the process of the self-compensation as it for the first time has stated at a qualitative level [3]. However, these authors could not explain the corresponding change of properties of so-called "narrow gaped" semiconductors using the classical model of self-compensation [4].

By us it has been shown, that the model of the self-compensation can be used for calculation of the irradiated semiconductors electronic properties if as for the forbidden gap to accept not the minimal gap  $E_g$  of the crystal, but an average energetic interval  $\langle E_g \rangle$  between the conduction and the valence zones of a crystal [5]. Then irrespective of  $E_g$  –

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

value the position of  $F_{lim}$  – level in the irradiated semiconductor will be identical as  $\langle E_g \rangle / 2$  (Table)

Table. The numerical calculated values of  $E_g$ ,  $F_{in}$ ,  $\langle E_g \rangle / 2$  and the experimental values  $F_{lim}$  in some semiconductors. (Count of the values from a ceiling of a valence zone, eV). The experimental and the prognosis\* electro physical parameters of the irradiated semiconductors are submitted.

Semicond	$E_g$	$F_{in}$ [1]	$F_{in}$ [2]	$\langle E_g \rangle / 2$ [5]	$F_{lim}$	Physical properties (n, i, p)
C	5.45	1.96	2.16	2.22	1.71 **	(i-p)-type *
Si	1.20	0.39	0.47	0.37	0.39	(i-p) type ( $\rho \approx 10^5$ Ohmxcn)
Ge	0.78	0.18	0.26	0.06	0.13	p-type ( $p \approx 10^{16}$ cm <sup>-3</sup> )
BAAs	1.82	0.00	0.14	0.08	-	p <sup>+</sup> -type*
AlAs	2.24	0.88	1.07	1.07	1.04	i-type *
GaAs	1.51	0.63	0.77	0.70	0.6	(i-p) type ( $\rho \approx 10^8$ Ohmxcn)
InAs	0.39	0.51	0.53	0.50	0.52	n <sup>+</sup> -type ( $n \approx (2-3) \times 10^{18}$ cm <sup>-3</sup> )
BP	1.99	0.71	0.95	0.80	-	(i-p)-type *
AIP	2.48	1.20	1.37	1.31	-	i-type *
GaP	2.37	1.00	1.16	1.03	1.1	i-type ( $\rho \geq 2 \times 10^{13}$ Ohmxcn)
InP	1.49	0.89	1.03	0.90	1.0	n-type ( $n \approx (3-6) \times 10^{12}$ cm <sup>-3</sup> )
BN-wz	7.86	3.35	4.01	3.85	-	i-type *
AlN-wz	6.04	3.33	3.73	3.51	3.55 **	i-type *
GaN-wz	3.53	2.73	2.80	2.64	2.66	(n-i) type
InN-wz	1.17	1.65	1.63	1.84	1.6	n <sup>+</sup> -type ( $n \approx 10^{21}$ cm <sup>-3</sup> )
AlSb	1.74	0.47	0.63	0.45	0.5	p-type ( $\rho \approx 10^6$ Ohmxcn)
GaSb	0.87	0.14	0.24	0.00	0.02	p <sup>+</sup> -type ( $p \approx 3 \times 10^{18}$ cm <sup>-3</sup> )
InSb	0.14	0.12	0.17	0.05	~0.0	p <sup>+</sup> -type ( $p \approx 7 \times 10^{17}$ cm <sup>-3</sup> )
3cSiC	2.55	1.30	1.42	1.10	1.05	i-type

Thus the degree of compensation of such material is close to 100 % even if it has n<sup>+</sup>-or p<sup>+</sup>-type of conductivity after an irradiation.

Reaction of the semiconductor to an irradiation could describe as two interconnected processes

$$\Delta G = \Delta G_{AT} + \Delta G_{EL},$$

here  $\Delta G$  – the change of free energy  $G$  of the semiconductor upon irradiation.

$\Delta G_{AT}$  - the atomic component of the free energy, which includes the energy of the broken chemical bonds, the elastic pressure energy and the entropy contribution.

$\Delta G_{EL} = (\Delta G_{free} + \Delta G_{bond} + \Delta G_{cl})$  - the electronic component of free energy, which includes:

$\Delta G_{free}$  – the contribution of the free carriers;

$\Delta G_{cl}$  – the contribution of the accidental electrostatic potential created with defective clusters, for example, at the irradiation by the fast neutrons;

$\Delta G_{bond}$  - the contribution of the charge localized on the radiation-induced defects.

As at an irradiation the semiconductors become not only high-resistive, but can get n<sup>+</sup> (InAs, InN ...) - type or p<sup>+</sup> (GaSb, InSb ...) – type conductivity, thus the contribution of the  $\Delta G_{free}$  – value in the general change of  $\Delta G_{EL}$  – value is not determining Moreover, the  $\Delta G_{free}$  – value at an irradiation aspires to stationary value  $\Delta G_{free}(F_{lim})$ . At the presence of the defective clusters in the crystal the Fermi - level  $F \sim F_{lim}$  in the area of the cluster, and  $F \sim F_0$  in the crystal matrix, where an initial chemical doping level sets  $F_0$ . It results in the formation of the accidental "potential of the radiating damage"

$$\Delta\varphi(r)_{\max} \sim (F_0 - F_{lim})$$

and the corresponding electrostatic field intensity

$$\xi_{\max} \sim -\text{grad}(\Delta\varphi(r)_{\max}).$$

In such material the position of a Fermi - level will depend on the coordinate  $r$

$$F = F(r) - q \Delta\varphi(r).$$

To growth of a doze there is "alignment" of  $F$  – level (in the all sample  $F \sim F_{lim}$ ), that corresponds to transition of a material in the macroscopic homogeneous state  $\Delta\varphi(r) = 0$ .

The contribution of a member  $\Delta F_{bond}$  is determining in the heavy irradiated semiconductor, as it is responsible for the performance of a neutrality condition, which can be provided with a neutrality of all broken chemical bonds (with a partial neutrality), or with the mutual compensation of the radiation-induced donor-type and acceptor-type defects. These both conditions work on the shifting  $F$  – level close to  $F_{in}$  - position. Thus in the semiconductors, for which it is characteristic covalent (or strongly covalent) chemical bonding, the process of the self-compensation according to the classical model [4] is poorly effective because of the small  $E_g$  – value and because of the big energy of the chemical bonds. Therefore, "the radiating self-compensation" in the semiconductors can be carried out due to the formation as a result of an irradiation the amfouteric initial lattice defects, the centers with negative correlation energy (the Anderson's centers) or the low symmetrical complexes of the lattice defects and the chemical impurities. As a whole, the reaction of an electronic sub-system of a semiconductor crystal to the rigid radiating influence is a display of the compensator mechanism of Le Shatelie for the systems, which are taking place in the thermodynamic balance with an environment.

It is possible to note, that parameter  $F_{in}$  plays the important role in the processes of the chemical impurities doping of the semiconductors for the impurities with high solubilities. The limits of chemical impurities doping level depends on the values

$$\Delta F \approx |F_{PIN}^n (F_{PIN}^p) - F_{in}|,$$

which are determined by the character of the chemical bonds of a crystal lattice. Here  $F_{PIN}^n (F_{PIN}^p)$  – the limiting (pinning) positions of the Fermi - level due to the donor-(acceptor) – type chemical impurities doping.

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### Formation of dynamically stable structures during heavy-ion implantation of insulators

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Use of optical diagnostics during irradiation (the methods of radiation photonics) allows researchers to overcome the conventional approach, according to which the properties of

materials are examined solely before and after irradiation. We used the methods of radiation photonics to study evolution of metal phases during fabrication of metal-nanoparticle composites by heavy-ion implantation into optical materials.

The radiation photonics helps us in finding the ranges of dynamic equilibrium between phases under irradiation and allows us to draw the non-equilibrium phase diagrams. An important feature of the dynamic balance of phases is the formation of dynamically stable structures in materials under irradiation. Although the formation the dynamically stable structures diminishes the efficiency of ion implantation for further accumulation of implants as in nanoparticles, the properties and mechanisms of formation of these structures deserve much attention in the radiation materials science, in particular in the problem of radiation-resistant materials.

In the report, we show results of the optical measurements conducted during heavy-ion implantation into various optical substrates, including radiation-resistant materials and soft non-linear-optical crystals. Non-equilibrium phase diagrams are presented. HTEM and AFM images of as-implanted nanostructures are presented. In many cases, a drastic transformation of metal-nanoparticle composites accompanied by abrupt changes of metal phase concentration, size and depth distributions of metal nanoparticles precedes the saturation behavior. After irradiation to fluences corresponding to formation dynamically stable structures, surface structures and bimodal (size, depth) distributions of metal nanoparticles in the bulk are observed.

The dynamically stable structures form because of the competition between the incoming ions and the surface recession due to sputtering which causes steady-state depth distributions of implanted ions to form. Formation of the dynamically stable structures is also affected by radiation-stimulated diffusion and selective energy transfer from incoming ions to atoms of metal phase.

### **Phase stratification in yttrium cuprate under irradiation and deformation**

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Yttrium cuprates (YBCO) are known to belong to perovskite-like structures and transit into the superconducting state at  $T > 77$  K (HTSC). Possible mechanism of HTSC relates with phase stratification like a stripe superstructure of alternating dielectric and metallic strips being in phase coherence each to other. Earlier we found the texture created under proton irradiation of YBCO due to a faster decreasing of the oxygen content in the subsurface layer than in the grain volume. This paper presents the results of diffraction study of phase stratification of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  under effect of radiation and deformation. Ceramic samples of  $\text{YBaCuO}$  ( $\delta \approx 0.17$ ) had a density of  $5.2 \text{ g}\cdot\text{cm}^{-3}$ , lattice parameters  $a = 3.824$ ,  $b = 3.884$  and  $c = 11.676 \text{ \AA}$ , the transition at  $T_c \approx 87$  K. We studied the shape of (004) and (007) reflections from the basis plane at 77 K at the X-ray diffraction spectrometer DRON-UM1 with the selected  $\text{Cu}_{K\alpha 1}$ - radiation and the unit URNT-180. Irradiation with protons of 18 MeV was

done at the cyclotron U-150 in the INP AS RUz at 300 K, beam current 20 nA to doses within  $10^{14}$ – $10^{15}$  cm<sup>-2</sup>. Mechanical stress was induced in the samples with the screw mounted in the sample holder of the cryostate. For the non-irradiated samples the reflection (007) had a symmetric singlet shape with the width  $7.56 \times 10^{-3}$  rad. In the samples irradiated to a small dose ( $\sim 10^{14}$  cm<sup>-2</sup>), the peak shape became asymmetrical. And after the dose of  $8 \times 10^{14}$  cm<sup>-2</sup> the reflection got a bell shape, i.e. another narrower reflection appeared on the peak maximum. After separation of these peaks their widths were  $\beta_1 = 10.2 \times 10^{-3}$  and  $\beta_2 = 12.2 \times 10^{-3}$  rad, respectively. Using Selyakov-Sherrer formula, the characteristic sizes of scattering centers were determined  $l_1 \approx 68$  and  $l_2 \approx 37$  nm. The samples were layered into structure HTSC domains with practically equal oxygen index, but differing sizes, between which insulator strips are possibly formed, not disturbing the coherence of HTSC domains. Equality of the ratios  $\beta(007) / \beta(004) \approx \text{Cos}(004) / \text{Cos}(007)$  indicates that each domain consists of tiny fragments. Generation of mechanical deformation in the samples turned out less effective than the irradiation induced ones. In the both cases the phase stratification is suggested to happen by the mechanism of formation of local dislocation structure in the yttrium cuprate ceramics.

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### **Structure and kinetic properties of $\text{Ni}_{50+x}\text{Mn}_{25-x+y}\text{Ga}_{25-y}$ alloys with shape memory**

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Studying the physical properties of the stoichiometric  $\text{Ni}_2\text{MnGa}$  compound is of great interest due to the observed magneto-operated shape memory. We present results of kinetic coefficient experiments and structure studies in  $\text{Ni}_{50+x}\text{Mn}_{25-x+y}\text{Ga}_{25-y}$  ( $x=0$  and  $y=0$ ;  $x=4$  and  $y=0$ ;  $x=0$  and  $y=3.5$ ) alloys at temperatures  $T \ll T_M$ ,  $T_C$  ( $T_M$  is the temperature of the martensitic transition and  $T_C$  the temperature of the magnetic (Curie) transition, which are close to room temperature). The magnetic, galvanomagnetic and electrical properties were measured in the temperature interval from 2 to 80 K and in magnetic fields of up to 15 T. Two types of samples were investigated: cast samples with a grain size of about 100-500  $\mu\text{m}$  and quenched samples with a grain size of about 300-500 nm.

We find that the deviation from the stoichiometric composition leads to an increase of the structural and magnetic disorder and, hence, to changes in the low-temperature kinetic properties. Similar effects are observed as the result of a quench treatment.

This work was supported by the Russian Foundation for Basic Research (projects 06-02-16695, 07-03-96062, 08-02-00844) and the Austrian Academy of Sciences.

**Atomic-Force Microscopy Investigation of Post-Radiation Changes of Silicon  
Surface**

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





## **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-rate 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, Zr and Ge in particular, and the features of microdefects accumulation in metals and alloys under shock-wave loading will be analyzed.**



**About the influence of parameters of ion irradiation on formation of atomic structure and composition of surface layers of Fe-Cr alloy**

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It is known that under ion implantation degree of defect state of irradiated materials depends on parameters of implantation. Two of these parameters, which influence on processes of organization of surface layers during ion implantation, are dose and velocity of gaining the dose. Local increase of defect density in area of cascade leads to increase of radiation damage taken by implanted area, up to, in a number of cases, amorphization. In its turn, radiation defects, presented in the target, stimulate many diffusion processes, related with formation of chemical composition and atomic structure of modified layers.

In this paper influence of irradiation dose ( $1\text{-}5 \times 10^{17}$  ion/cm<sup>2</sup>) and ion current density (10-50 mA/cm<sup>2</sup>) of B<sup>+</sup> and P<sup>+</sup> ions with 40 keV energies on composition and atomic structure of model alloy Fe-10 at.%Cr are studied.

It is shown that increase of implantation dose leads to transition of atomic structure from amorphous (less than  $1 \times 10^{17}$  ion/cm<sup>2</sup>) with depth of up to 10 nm to formation and growth of crystalline phase. Implantation with dose of  $5 \times 10^{17}$  ion/cm<sup>2</sup> forms textured polycrystalline structure with a texture period of 0,270 nm and axis perpendicular to target surface, that consists with the vector of internal stresses gradient. Obviously, clusters (Fe)Cr-P-C, formed under small doses, with increase of the dose transform to FeP and Cr<sub>2</sub>C<sub>3</sub>. Also, concentration maximum of P slightly increases and moves to the target surface.

Increase of B<sup>+</sup> implantation dose doesn't change the structure of surface layer, it remains amorphous. Modified layer contains clusters (Fe)Cr-B, corresponding to amorphous Fe-Cr-B alloy. Concentration profiles of boron are consistent with calculations, with increase of concentration maximum to 60 at.% and increase of depth.

Changes of the dose gaining velocity Изменения плотности набора дозы were made by increase of ion current density (10-50) mA/cm<sup>2</sup> with constant dose of  $1 \times 10^{17}$  ion/cm<sup>2</sup> and ion energy of 40 keV.

At starting P<sup>+</sup> ion current density amorphous structure is formed in surface layers of the target, increasing ion current density to 30 mA/cm<sup>2</sup> and higher fixes polycrystalline structure and formation of metal-metalloid compounds takes place. Irradiation with B<sup>+</sup> ions leads to amorphization of surface layer under all ion current densities studied.

Changes of concentration profiles of implanted admixtures, the general one being the decrease of their complete concentration, are found. To determine the mechanisms of the effect, described above, a physical-mathematical model is proposed. This model is based on the next assumptions: 1) implanted atoms forms microscopic clusters with atoms of the target; 2) the behavior of the clusters is determined by the theory of redistribution of macroscopic inclusions in crystals placed in temperature, elastic and gradient-vacancy fields [1]; 3) Brownian motion of clusters is neglected; 4) amorphous alloys tend to accumulate

quasi-vacancies and to form their gradient; 5) presence of high gradients according to theory of macroinclusions' movement leads to high velocities of redistributions of components.

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## Wide-Application Efficient Detectors of Ionizing Radiation on the Basis of Single Crystals and Nanostructures Ceramics of Oxygen-Deficient Aluminum Oxide

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The TLD-500 thermoluminescent detectors of ionizing radiation developed in the University on the basis of anion-deficient single crystals of aluminum oxide ( $\text{Al}_2\text{O}_3:\text{C}$ ) display a complex of unique properties: high sensitivity, wide range of registered doses, low energy dependence of thermoluminescence output with the possibility of its compensation.

Traditionally, detectors TLD-500 are used for individual monitoring of ionizing radiation at nuclear power stations, radioactive waste processing and storage plants, in universities, scientific and research centers, medical institutions, for radiation situation monitoring in residential buildings and for control of construction materials with high level of natural radiation background, in geology and on oil production sites.

Radiation detectors operating on the basis of nanostructural ceramics  $\text{Al}_2\text{O}_3$  open new dosimetric opportunities: they may be used in high-dose (up to 10 kGy) dosimetry at elevated temperatures, which is important for radiation monitoring of technological rooms of nuclear reactors.

## The Post-Irradiation Examination of Fission Products Distribution along Multi-Component Electro-Generating Channel

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The method of the direct thermo-emission conversion of heat energy produced by U-235 nuclear fission is one of the attractive means of generating electric energy in space vehicles. Space nuclear reactors based on electro-generating channels (EGC) can work simultaneously as a board power sources and power-driven engines. In order to predict the EGC performance capability it is necessary to study the processes proceeding in fuel and structure elements of the EGC components under the conditions close to the operation ones.

At the Institute of Nuclear Materials, Zarechny, the EGC are tested in the IVV-2M reactor and undergo post-irradiation materials-science investigations in the Hot Cell laboratory in order to obtain the information on the influence of operation conditions on thermo-emission properties of the EGC and performance capability of its structure materials and, consequently, to determine the ways of increasing a life time of the power installations of this type.

After the high temperature in-pile tests primary post-irradiation examinations of the EGC include the gamma-spectrometry, which is the NDT method, to obtain data on a distribution of the fuel inside the EGC components and in between them at different positions along the EGC.

This paper presents the investigation of the distribution of gamma-emitting radionuclides:  $^{95}\text{Zr}$ ,  $^{95}\text{Nb}$ ,  $^{144}\text{Ce}$ ,  $^{137}\text{Cs}$ ,  $^{106}\text{Ru}$  at the positions along the multi-component EGC irradiated in the IVV-2M reactor.

The obtained data show that

- the fuel migrates from the center to the sides of the electro-generating components;
- there is no fuel release from the electro-generating components because the radionuclides (which are fuel fission products) have not been detected in the gap between the electro-generating components;
- there is no significant change in both the length of the fuel meats of the electro-generating components and the general length of the fuel column of the EGC after irradiation in the IVV-2M reactor.

### **Effect of Type and Dose of Implanted Ions on Change of Mechanical and Tribological Properties and Composition of Carbon Steel Surface**

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Surface hardening of comparatively cheap steels and alloys used in the manufacture of components for heavy-duty machines and mechanisms presents one of the trends in machine engineering of today.

Investigation was carried out of the effect of irradiation with  $\text{N}^+$  and  $\text{Ar}^+$  ions with energy of 40 keV to doses from  $10^{15}$  to  $5 \cdot 10^{16}$  ion/cm<sup>2</sup> and  $\text{Mn}^+$  ions with energy of 45 keV to doses from  $2 \cdot 10^{16}$  to  $10^{17}$  ion/cm<sup>2</sup> on the mechanical properties and morphology of surface, and on the composition of surface layers of carbon steel St3sp. There was revealed non-monotonous variation of microhardness and fatigue strength depending on the type of irradiation and the irradiation dose, and surface flattening under ion bombardment.

Work was carried out with the financial support of the integration project of FTI UB RAS and IMP UB RAS.

### **Cable Transducers Application for Measuring Spectral-Angular Electron Distributions of Powerful Accelerators**

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Cable transducers (CT) [1] are used as primary measuring devices within the technique of investigation of the angular electron distributions of pulsed powerful accelerators [2]. This technique was applied for investigation into spectral composition of electrons emitted by

accelerating tube (AT) of accelerator IGUR-3 [3] under various angles as described in current paper.

Measurements are based on matching amplitude-time profiles of electrons recorded by each CT to amplitude-time profile of voltage on AT. The first yields the number of electrons recorded in a certain time range and the second yields their energy in the same range. Various factors influencing on the error of spectrum reconstruction are taken into account during measurements. These factors include distortion of amplitude-time profiles of signals when passing through long communication line (~ 40 m), voltage drop on AT, accuracy of time profiles superposition.

The software was developed for experimental data processing. Obtained results are presented as bar graphs illustrating spectral-angular distribution of electrons. As this technique [2] allows investigation into spatial distribution of electrons as well, the information about spatial-angular distribution may be eventually obtained taking into account dependence of electrons spectra on the angle of escape from AT. This is illustrated by recorded spectra of electrons escaping from AT under angles  $0^\circ$  and  $22^\circ$  with regard to its axis. We see that alongside with electrons deviation angle growth the spectrum becomes more 'soft' that confirms credibility of developed technique.

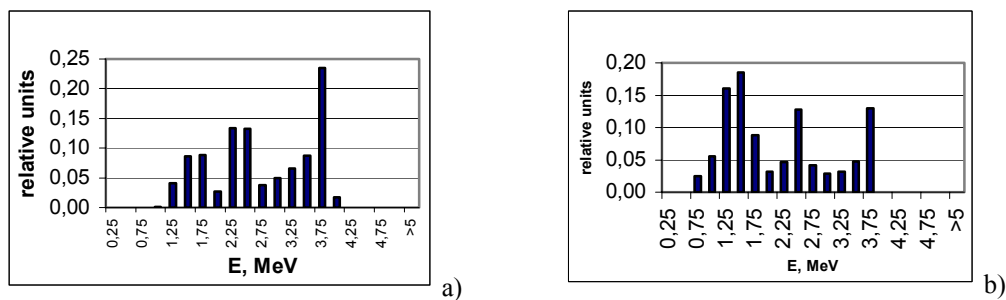


Figure. Bar graph of distribution of electrons escaping from AT under angle  $0^\circ$  (a) and  $22^\circ$  (b). Duration of electrons pulse in this shot was  $\approx 100$  ns.

Spectral data combined with angular distribution of electrons provides more comprehensive data on escaping electron beam that is of interest both for investigation into AT operation and for application in electron beam irradiation experiments.

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## Crystal Monochromators Application in Circuits with X-Ray Radiation Sources

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The task of x-ray irradiation monochromation emerges in solving a broad range of problems related to investigation of the structure of both crystal and non-crystal materials. This task is most often resolved with the application of crystal monochromators.

It was shown in this work that combination of basic parameters of an x-ray beam, such as intensity, monochromaticity, divergence and linear dimensions, in x-ray experiments of different types requires application of different crystal monochromators. Depending on the nature of a specific problem being solved, it makes sense to use crystal monochromators with different degrees of crystal structure perfection and different surface finish quality. At that, in the majority of cases, the task of obtaining such crystals may be well resolved in laboratory conditions.

## Ion Irradiation and Recrystallization of Metals

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Diffusion processes, which substantially depend on temperature of heating and mobility of atoms, play an essential role in formation of structure and, hence, structurally sensitive properties of metal materials at recrystallization (migration of crystal boundaries) and magnetic annealing. It is known, that the ion irradiation anomalously raises the low-temperature mobility of atoms in alloys.

Investigation is lead on a single crystal of soft magnetic technical bcc-alloy Fe-3% Si with orientation (110) and thickness of 0,5 mm. Treatment: deformation (D) 80% of rolling in a direction [001]; polygonization (P) 520°C – 30 min; an ion irradiation (II) by accelerated ions  $Ar^+$  (30 keV) by doze  $10^{15}$  ion/sm<sup>2</sup>; usual annealing (UA) with a gradient of temperatures from 20°C up to 620°C; magnetic annealing (MA) in the magnetic field with frequency of 50 Hz, amplitude of 10 kA/m and a gradient of temperatures, as at UA.

The ion irradiation and/or presence of an external magnetic field change temperature of the beginning  $t_r^b$  and the end  $t_r^e$  of recrystallization; size (d), the form and perfection (estimated on change of coercive force  $H_c/H_{c\text{ initial}}$ ) of recrystallization grains; componental composition of crystallographic texture ( $S_{(110)}/S_{(310)}$ ), S-areas of peaks); see the table.

Treatment	Structure of specimen*	d, μm	S <sub>(110)</sub> /S <sub>(310)</sub>	H <sub>c</sub> /H <sub>c initial</sub>
1. D + UA		20×22	0,47	0,05
2. D + P + UA		20×23	1,32	0,20
3. D + II + UA		22×22	1,75	0,07
4. D + P + II + UA		18×19	1,26	0,22
5. D + MA		20×22	0,24	0,05
6. D + P + MA		20×23	0,31	0,12
7. D + II + MA		18×19	0,35	0,05
8. D + P + II + MA	 $t_r^b$ $t_r^e$ Temperature →	22×23	1,71	0,21

\* Black zone – deformation (treatments: 1, 3, 5, 7) or polygonization (treatments: 2, 4, 6, 8), grey – incomplete recrystallization, white - full recrystallization.

Influence of II and MA on structure- and texture-formation at primary recrystallization can be caused by influence of these treatments on process of deformation ageing. Thus, apparently, inhibitors are formed on subgrain boundaries, it changes a parity of growth rates of recrystallization nucleuses with various orientations.

The work was done within RAS Program (project № 01.2.006 13391), with partial support of RFBR (grants № 07-02-96032, 08-02-00327) and the integration project with the Siberian Branch of the Russian Academy of Science.

### Metallographic Examination of Structure of Aluminum Alloys after Ion-Beam Treatment

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This paper presents the results of metallographic examination of the structure of cold-rolled sheets of alloy 1441 of the system Al- Li-Cu-Mg (degree of deformation 72 %, sheet thickness 1 mm) and alloy AMg6 of the system Al-Mg (degree of deformation 35 %, sheet thickness 3 mm) in the *initial cold-worked* state, in the state *after intermediate annealing* used in the cold rolling process to remove cold-work, and *after irradiation* of cold-worked samples with accelerated Ar<sup>+</sup> ions with the energy of 20-40 keV at ion current density of 150 μA/cm<sup>2</sup>. The irradiation dose varied from 10<sup>15</sup> to 10<sup>17</sup> cm<sup>-2</sup>.

Irradiation with continuous Ar<sup>+</sup> ion beams was carried out in the PULSAR ion-beam implanter with a cold hollow-cathode glow discharge source. The maximum temperature of samples heating did not exceed the temperature of respective intermediate annealing performed to remove cold work.

Metallographic examination was carried out under the Neophot-30 optical microscope in a section square to the irradiated surface.

It was established that, in the course of alloy 1441 irradiation with Ar<sup>+</sup> ions to a dose of 5.6·10<sup>16</sup> cm<sup>-2</sup> (irradiation time 60 s, T < 260 °C), there takes place formation of new small grains 4 to 7 μm long and ~ 2 μm wide stretched in the direction of rolling, and equiaxial grains 2-5 μm in size. With further irradiation dose increase to 1·10<sup>17</sup> cm<sup>-2</sup> (irradiation time



107 s,  $T < 370$  °C), grains extension actually disappears completely. Grains acquire equiaxial shape with considerable increase in size: up to  $\sim 30\text{-}40$   $\mu\text{m}$ .

In alloy AMg6, irradiation to a dose of  $10^{17}$   $\text{cm}^{-2}$  (irradiation time 107 s,  $T < 280$  °C) causes formation (in separate sections of the sample) of new equiaxial grains 15  $\mu\text{m}$  in size. With dose increase to  $1.3 \cdot 10^{17}$   $\text{cm}^{-2}$  (irradiation time 135 s,  $T < 320$  °C), formation of equiaxial grains of 35-40  $\mu\text{m}$  size goes in the whole bulk of the sample.

Thus it was established that irradiation of cold worked alloys 1441 and AMg6 with  $\text{Ar}^+$  ions facilitates the processes of recrystallization in the whole bulk of the samples (1 and 3 mm thick, respectively), similar to the processes taking place at usual annealing. At that, recrystallization goes within just a few tens of seconds, which is significantly shorter than in case of usual annealing (1 – 2 h), and in the absence of high-temperature exposure.

### **Modelling of Developed Surface of Metal by Irradiation with High-Energy Heavy Ions**

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It is known that heavy high-energy ions bombardment creates in a metal areas with the changed structure of a material ("tracks"). The area of the track covers the ion trajectory and usually has axial symmetry. Each incident ion creates his own track which sizes depend on not elastic and elastic losses of energy of an ion. Thus it can be formed both quasicontinuous, and a interrupted track. For example, interrupted tracks are formed in  $\text{NiZr}_2$  when speed of allocation of energy on electronic excitation exceeds  $40\text{KeV} \cdot \text{nm}^{-1}$  [1]. Effective diameter of the track can reach value of several nanometres (for example, *Pb*

incident ion creates area of modified structure of a material of the spherical form. Position of these areas in a material was modelled in a random way with use of a Monte-Carlo method. Were thus considered both modelling, and realistic distributions of areas of the modified substance generated by heavy ions on depth of the sample.

When in use of irradiation all new areas which can be overlapped and form, thus, the complex branched out structure (nanoclusters) are created. The beam of bombarding particles relied to be homoenergetic, speeds of all particles are parallel and normal up to irradiated surface. Calculations were fulfilled at various values of parametres of model: sizes of energy of incident ions, density of their stream and values of cutsets of elastic and not elastic losses of energy.

As a result of the realized simulating is search out dependence of the effective area of a surface irradiated sample from irradiation fluence. It has been found that in process of a stock up fluence dynamics of change of effective square of a surface of the irradiated sample can have various stages.

a) At small fluence irradiations slow growth of effective square of an irradiated surface (the so-called incubatory period) is observed. The square increase occurs at the expense of occurrence of disrupt defective nanoclusters (areas of the modify substance of the spherical form) near to a surface of the sample. In the interior of the specimen nanoclusters, which yet are not wedging out, are generating and storing.

b) Sharp increase of effective square of an irradiated surface at the expense of germination and wedging out nanoclusters which were generated in the interior of the specimen owing to overstrike of the areas created by ions with the changed structure of substance.

c) Steady (quasistationary or periodic in time) a mode linked to processes of elastic and inelastic sputtering of substance as a result of the irradiation.

Conditions of implementation of these modes are received, their characteristics are defined. Dependences of growth of effective square of an irradiated surface from the characteristic sizes of a track are found.

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### **Investigation of Collision Cascades in Two-Layer Substrate by Molecular Dynamics Method**

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This paper presents the work on molecular-dynamic modeling of atomic collision cascades initiated by 50, 100 and 500 eV ions falling normal to surface of a two-layer crystal.

Data are given on formation of vacancies, radiation-absorbed and interstitial atoms in a cascade. The effect of collective deceleration of ions as a result of simultaneous interaction

of an ion with 2-3 knocked-on atoms of the substrate displaced by it from their equilibrium positions is discussed.

Work was carried out with the financial support of the integration project of FTI UB RAS and IMP UB RAS.

### **Ion Irradiation Effect on Mechanical Properties and Microstructure of Deformed Alloy VD1 (Al-Cu-Mg)**

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Investigation was carried out of the effect of irradiation with medium-energy (20 – 40 keV) argon ions on the mechanical properties and structural-phase state of cold-deformed commercial aluminum alloy VD1 of the system Al-Cu-Mg.

Irradiation of samples with continuous Ar<sup>+</sup> ion beams was carried out in the PULSAR ion-beam implanter with a cold hollow-cathode glow discharge source. In the course of irradiation, the following parameters varied: ions energy, ion current density (100 – 400  $\mu\text{A}/\text{cm}^2$ ) and irradiation dose ( $10^{15}$  –  $10^{17}$   $\text{cm}^{-2}$ ).

Already at alloy VD1 exposure to low doses of  $1 \cdot 10^{15}$  and  $1 \cdot 10^{16}$   $\text{cm}^{-2}$  (irradiation time  $\sim$  1 and 10 s), formation of a developed subgranular structure took place, with subgrains misorientation not exceeding  $10^\circ$ . This results in some loss of strength characteristics and gradual increase of relative elongation. With further irradiation dose increase up to  $1 \cdot 10^{17}$   $\text{cm}^{-2}$  and higher, a homogeneous coarse-crystal granular structure is formed with grain size over 10  $\mu\text{m}$ , similar to the structure of the same alloy in the recrystallized state after annealing at 400 °C during 2 hours.

Besides, irradiation initiates accelerated solid solution decomposition in the precipitation hardening alloy VD1, with formation of equiaxial particles of  $\theta'$  ( $\text{CuAl}_2$ ) phase 10 to 20 nm in diameter. A simultaneous course of two competing processes, recrystallization and solid solution decomposition, causes non-monotonous variation of mechanical properties under high irradiation doses.

It was also established that irradiation of cold-worked sheets of aluminum alloy VD1 with Ar<sup>+</sup> ions leads to gradual elimination of the rolling texture, and formation, at doses over  $5 \cdot 10^{16}$   $\text{cm}^{-2}$ , of the recrystallization texture, similar to that formed in case of furnace annealing.

The registered structural and phase transformations under irradiation go at a high rate (their completion takes from units of seconds to several tens of seconds of irradiation) and in the whole bulk of  $\sim$  3 mm thick samples (with mean ions projected range being only several tens of nanometers).

The obtained results testify to the fact that variation of irradiation regimes allows the structure and properties of the alloy to be influenced, thus giving an opportunity for controlled variation of service properties of aluminum alloys.

### **Electrochemical Behaviour of Samarium in Alkali Metal Chloride Melts**

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Investigation was carried out of electrochemical behaviour of samarium ions in NaCl(0.35)-CsCl(0.65) eutectic melt and equimolar mix of NaCl-KCl with application of the methods of linear and cyclic voltammetric measurements in the interval of 0.1 to 1 V/s potential sweep speeds. Melts containing 0.4–1.4 mol% SmCl<sub>3</sub> were examined. It was shown that the process of cathodic reduction of Sm(III) also includes a recharging stage: Sm(III) + e ↔ Sm(II).

The diffusion coefficients of complexes formed by tri-valent samarium ions and conditional formal oxidation-reduction potentials  $E^*Sm^{3+}/Sm^{2+}$  in the named salt solvents were calculated. Comparison with available literature data was made.

### **Ion modification of surface properties of rolled Cu-Ni foils**

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An effect of the implantation of bore and argon ions at different ion current densities on the chemical composition and the defect structure of rolled 40-μm-thick Cu<sub>80</sub>Ni<sub>20</sub> foils has been studied by X-ray photoelectron spectroscopy, X-ray crystal analysis, and micro hardness measurement methods.

Analysis of the chemical composition of samples radiated by the X-ray photoelectron spectroscopy method has shown that implanted ions are present on the non-irradiated side of the foil and that the nonmonotonic relation between the alloy composition and the depth concentration of implanted ions is formed in radiated samples.

As foil thickness is three orders higher than calculated depth of ion insertion, we suppose that in our experiment long-range segregation effect takes place.

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 unirradiated one.

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 nonmonotonic 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 unradiative 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.

### **Nanochemical Analysis of Ferritic/Martensitic Steels EK-181 by Tomographic Atom Probe**

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Heat resistant low activation steels are most promising structure materials for new generation fusion and fission nuclear reactors. The main interest in Russia is applied to 12%-Cr ferritic martensitic steel EK-181 development. This steel has an increased heat resistance, which overcomes foreign analogues at temperatures higher than 650°C. This improvement of mechanical properties is connected with nanometer sized structure features formation (different types of clusters and segregations) during temperature treatment. Transmission electron microscope studies of EK-181 steel phase state have showed a presence of nanometer particles [1, 2]. These particles seemed to be carbides or carbonitrides of V and Ta and chromium carbides. In our study nanoscale characterization of EK-181 steel was performed by tomographical atom probe. This technique allows to receive 3D structure of a specimen with atomic resolution and to determine the chemical nature of each detected atom [3]. 3D reconstructions of investigated volumes were obtained in result of TAP analysis. Size of the data volumes was about 9x9x30 nm<sup>3</sup>, number of events ~ 10<sup>5</sup>. Detailed nanochemical analysis allowed to obtain the information about different chemical elements redistribution in bulk. Some nanocluster (areas with a size of about 3 nanometers) enriched with V and N atoms were found. The ratio of elements V to N is about 2:1 in these areas.

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## Confinement of positrons in vacancies and complexes of vacancies in metals and alloys

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Defects of vacancy type in metals and alloys are the effective centers of capture of positrons (phenomenon of confinement) [1,2]. For example, time positron annihilation spectra allow to define thus fundamental "operational" parameters of metals:  $N_v$  - concentration and  $H_v$  - enthalpies of formations of vacancies. The analysis positron annihilation spectra is based on the decision of system of diffusion-annihilation equations [3]. Below the probability  $P$  of capture of positrons by spherical defects in metals and alloys of vacancy type in metals in approach of the modified theory of capture [4,5] is calculated. Dependence of probability of capture of positrons on temperature in the aluminium containing spherical defects – traps of positrons is investigated. At calculation following values of sizes and parameters [4] have been used:  $S_v = 4\pi(r_0 + \lambda)^2$ ,  $r_v = 250 \text{ \AA}$ ,  $R = ((3/4)\pi N_v)^{1/3}$ ,  $V = \frac{4}{3}\pi R^3$ , where  $N_v = 2 \cdot 10^{14} \text{ cm}^{-3}$ ,  $\lambda_0 = 5,9 \text{ ns}^{-1}$ ,  $L = (D\tau_0)^{1/2} = 1,02 \cdot 10^{-5} \text{ cm}$ ,  $D = 0,6 \text{ cm}^2/\text{c}$ ,  $R^3 = 1,2 \cdot 10^{-15} \text{ cm}^3$ ,  $(r_0 + \lambda)^3 = 1,76 \cdot 10^{-17} \text{ cm}^3$ .

As follows from calculations the satisfactory consent of experimental and theoretical values of probability  $P$  of capture of positrons by spherical defects in the aluminium, irradiated by fast neutrons is observed. Thus, the analytical decision of diffusion-annihilation equations equations has allowed to receive expression for probability of capture of positrons spherical defects (the phenomenon of confinement) in metals and alloys depending on temperature.

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## Study of nanoobjects in the irradiated metals and alloys by method of positron annihilation spectroscopy

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Positron annihilation spectroscopy (PAS) [1,2], allowing to define both electronic structure of the perfect crystals, and various imperfections of especially small sizes in solids and porous systems, such as vacancies, vacancy clusters and free volumes up to one cubic nanometer includes itself in the basic three methods: studying of time distribution of

annihilation photons (TDAP), angular distribution of annihilation photons (ADAP) and dopler of width of annihilation lines with energy 0,511 MeV (DWAL) [1]. Method TDAP gives data on electronic density in a annihilation place of a positron, and methods ADAP and DWAL give the information on distribution of electron impulses and about a chemical compound of the environment surrounding nanoobjects in a annihilation place аннигиляции. (see, for example, [1-5]).

On the basis of ADAP method we develop one of effective modern methods of definition of the sizes of nanodtftcts (vacancies, vacancion clusters), free volumes of pores, cavities, emptiness and their concentration and a chemical compound in a anniyilation place in the irradiated metals and alloys and other technically important materials. On ADAP method the sizes and concentration of nanodefcts are certain a chemical compound of environment in a annihilation place: in wafers of silicon [3] and in the monocrystals of quartz [4] irradiated by protons, in porous silicon [5], and also in powders of quartz.

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### **Radiation Methods of Materials Treatment with the Use of Radiation-Dynamic Effects**

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In the talk, analysis of different aspects of the effect of ionizing radiation on materials is presented. A specific role of radiation-dynamic influence on metastable media is substantiated. It is noted that instantaneous energy release in the region of dense atomic collision cascades may reach the values of  $\sim 0.5$  eV per atom and higher, with characteristic process times of the order of  $\sim 10^{-12}$  s. As a result, irradiation may initiate structural-phase transformations in metastable media (at the front of shock waves emitted by the cascade regions). Such transformations explain the nature of long-range dynamic effects in nonequilibrium condensed media under corpuscular irradiation (low-dose irradiation including). The nature of the effect of “radiation annealing” of materials is discussed, and examples are given of its use as an alternative to furnace annealing (at low temperatures during 5-30 s). Methods of improvement of electrical, magnetic and mechanical properties of materials with the use of radiation-dynamic effects are proposed.

## **Manufacture and Study of Electrical Characteristics of Copper Nanowires Obtained by Matrix Synthesis Method**

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The ever growing interest to nanosize objects calls forth the search for new methods of their manufacture. One of such methods is matrix synthesis. The method is based on polymer films irradiation with subsequent filling of formed pores with the required material. Thus an opportunity is provided for fast and economic manufacture of a large number of nanowires with controlled parameters.

## **Structure and phases transformation of metals and alloys by ion implantation**

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For the first time, application of field ion microscopy in investigation of irradiation phenomena in metals and alloys initiating interaction of low-energy gas ions with a surface has allowed structural phase states in modified subsurface volume (600 nm) to be studied with atomic resolution.

As a result it was established that ion implantation is able to not only cause deformation effects in pure metals (Ir) but also to cause phase transformations of the order - disorder type (Cu<sub>3</sub>Au), in subsurface layers of atom-ordering alloys, and also, under certain conditions, the break-up of precipitates in the solid solution (PdCuAg).

On an atomic scale, "the long-range effect mechanism" is experimentally shown.

Quantitative characteristics of this phenomenon are found out, and analysis is carried out of the defects of various types arising due to ion implantation and cascades of atomic displacement and proceeding by processes of stimulated-radiation diffusion and segregation.

## **About Piezoceramics Applicability to Research of Pulsed Stress Generated by Electron Beam in the Sample**

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Quartz gauges are usually used for registration of thermomechanical stress in pressure range 2 to 3 GPa generating in material, as a result absorbing of energy of pulsed electron beams [1]. However to study materials having small value of Grüneisen parameter (~ 0.01



to 0.1) on needs piezoelectric transducers having essentially higher pressure sensitivity due to small values of occurring stresses. Applicability of piezoceramics TsTS-19 (PZT- 52/48) for this purpose is analyzed in the current study.

Studied sample was a layered structure of the plates glued together: carbon-carbon composite - absorber of electrons energy, acoustic delay of pulsed thermomechanical stress and piezoceramic TsTS-19. Acoustic delay is necessary to enhance interference immunity of measurements.

In this study we demonstrate that application of materials with high atomic number and value of Grueneisen parameter for acoustic delay of stress pulse and piezoceramic of recording results in occurrence of background signals caused by heating up by accompanying bremsstrahlung radiation of accelerator. This radiation is always present at operation of powerful accelerator both in the mode bremsstrahlung radiation generation and in the case of electron beam output to the atmosphere.

Application of fused silica of KV grade for acoustic delay (Grueneisen parameter of silica is equal to 0.03 [2]) has allowed to get rid of parasitic signals virtually completely if the thickness of sample is more than the mean free path of electrons in it. Pressure pulse associated with electrical breakdown in silica is observed at less thickness of silica. Simultaneously the plate of fused silica 20 mm thick noticeably shields the transducer from the effect of bremsstrahlung radiation and due to this reduces piezo-effect occurring in the transducer due to heating by radiation.

It has been demonstrated that the influence of pyroelectric effect in piezoceramics due to accompanying bremsstrahlung radiation may be neglected at applied dimensions of TsTS-19 (diameter 30 mm, thickness 12 mm).

Electrical circuit of transducer switching into the circuit for signal measurement under load was analyzed. It was demonstrated that unlike quartz gauges operating for short-circuited load [1], piezoceramics requires differential scheme of switching taking into account signal amplitude growth at stress pulse propagation along the transducer thickness.

The figure illustrates oscillation pattern of signal recorded by TsTS-19 under such load on carbon-carbon composite with application of acoustic delay of fused silica with a thickness slightly less than electrons path. The first pulse of negative polarity is associated with transducers heating by bremsstrahlung radiation, the first pulse of small duration of positive polarity is associated with electrical breakdown in fused silica and negative pulse with two extreme values is the pulse of thermomechanic stress.

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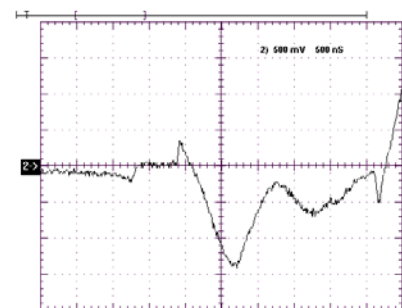


Fig. Oscillation pattern from transducer at electrons energy absorption in carbon composite. Sweep 0.5  $\mu$ s per unit, sensitivity 0.5 V per unit

## Evolution Mechanisms and Properties of Nonequilibrium Nanocrystal Solid Solutions for Limited Solubility Systems

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The evolution features and the properties of non-equilibrium solid solutions from the initial powder elemental mixtures by shear under high pressure technique were studied. Basically, the elemental mixtures with the positive enthalpy of mixing were investigated [1]: Fe-Cu, Fe-Cd, Fe-Bi, Co-Cu, Cu-Ag, Ti-N, Cu-C, Cu-C<sub>60</sub> (fullerenes), etc. Different stages of structural changes for the above mixtures were taken into account. Due to complex flow processes development and mechano-diffusion non-equilibrium-in-concentration solid solutions formation occurs; the typical size of crystallites was 10-50 nm. Elements with the same crystal structure constitute single-phase solutions. When initial elements are different in structure, the formation of solid solution with the crystal structure of dominating element in concentration takes place. For intermediate concentrations close to equiatomic ones two-phase solid solutions were observed. Binary systems from the elements which have a large difference in shear modulus constitute the solid solutions. The concentration of such solutions considerably exceeds the equilibrium one. However, it is impossible to obtain a solid solution for all concentration range. A limiting factor is the content of “soft” component in the range 8-15 at.% which acts as lubricant during mechanical processing. In that case, there are two ways for obtaining high concentration of solid solutions: a repeated shear under pressure with the increasing of “soft” component concentration or lowering the deformation temperature down to 80 K. As a result, the homogeneous-in-color close to steel tone metastable “solid solution-composition materials” with nanostructured pulverization were obtained. Dissolution in such systems occurs during heating at 100 °C or after several hundred of hours at standard environment. In such a case, both surface regions and edges of samples are coated with the amorphous carbon modification. At the same time, separate regions attain the color close to original copper one.

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## R-States Formation in Metallic Materials under Irradiation

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The specific R-states are formed under high dose ion irradiation within certain narrow range of irradiated parameters (doses, target temperatures and ion flux intensities). It turns out that these states have nanoclusteric morphology with the cluster size of several

nanometers and also the properties which significantly different from the initial material properties and from the properties of the materials irradiated at the other irradiation conditions. This effect is evidently of universal character as it was observed by us in the various solid solutions of the Fe-Cr-Ni, Ni-Cr, Cu-Ni, Fe-Cr, V-Ti-Cr and also in the pure metals Ti and Zr after irradiation by the ions of different natures and energies.

Some experiments were fulfilled to elucidate the nature of these states, namely, the determination of the activation energy of the defect structure annealing, the calculations of the stationary defect (of vacancies) concentration in the range of the R-state existence in the various materials and also some computer experiments. It turns out that the formation of the R-states in the various materials occurs at the certain threshold vacancies concentration which corresponds to the distance between vacancies equal 10 nm. It can be suppose that the mechanism of the defect interaction changes at these conditions.

The model is proposed – the clusters which have the symmetry different from the matrix one are formed in a vicinity of the radiation vacancies (the clusters of the icosahedral symmetry are formed in the FCC materials). These clusters reinforce the matrix and as a result the nanoclusteric composite arises.

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### **Influence of Deformation on Structure and Properties of Compounds Prepared by High-Temperature Self-Propagating Synthesis**

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The aim of the present work is to study the room temperature compressive deformation at High Temperature Self-Propagation (HTSP) synthesized alloy Ti-48Al-2Nb-2C (at. %) based on titanium aluminides. The objective is to improve technological properties of the material. Therefore, possible phase transformations by different compression ratio were investigated. The cell parameters of  $\gamma$ -TiAl crystal lattice after HTSP synthesis were determined. It was observed, that during compression the processes of intragrain hardening as well as intragranular deformation and transcrystalline destruction take place. The empirical formulas for the hardness – deformation and density – deformation dependences were established. The residual strains and stresses in bulk of the material were calculated.

## Application of Cellular Automata for Describing the Kinetics of Martensite Transformation during Deformation of Metastable Cr- Ni Steels

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The features and peculiarities of martensitic transformation in unirradiated and irradiated 12Cr18Ni10Ti steel that arise during deformation are analyzed. Various physical models and equations describing the kinetic curves of “martensite volume fraction  $V_a$  vs. strain  $\varepsilon$ ” are considered. Certain restrictions of each of the available models when applied to neutron-irradiated steels are discussed.

A new approach based on some aspects of the theory of “cellular automatic machines” is offered. A number of assumptions are included in this approach:

- The probability of martensitic transition in some volume  $\Delta V$  can be described by a probability of phase transition  $P$ .
- The value of  $P$  depends on the instantaneous local flow stress, and the martensitic transformation begins only upon reaching a critical value of stress  $\sigma_K$ .
- The value of  $P$  depends on a parity of phases in the space surrounding the chosen volume  $\Delta V$ .

A numerical model constructed with use of the above assumptions is presented, allowing us to describe the curve  $V_a - \varepsilon$  by means of two free parameters ( $P$  and  $\sigma_K$ ), using true stress– true strain measurements on irradiated specimens. The calculated curves based on this model are compared with experimental data published for steel 12Cr18Ni10Ti and similar materials.

## Analyses of Nano-Structured States in Ion-Irradiated Pt

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This paper represents the results of field ion microscopy (FIM) study of subsurface volumes of pure (99.99 %) platinum irradiated by accelerated up to 30 keV  $\text{Ar}^+$  ions. The FIM method makes it possible to carry out direct precise investigation on an atomic scale of defects of crystal lattice of a material on an atom-pure surface. At the same time the method allows to analyze the object of research in volume by controlled and sequential removal of surface atoms by an electric field at cryogenic temperatures.

The irradiation of accelerated up to 30 keV ions  $\text{Ar}^+$  the tip-samples, preliminary certified in a field ion microscope, was spent with fluencies -  $10^{16}$ - $10^{18}$   $\text{cm}^{-2}$  and density of an ion current  $j=150$  (T=343 K), 200  $\text{mA}/\text{cm}^2$  (T=473 K). Bombardment was made in a direction parallel to an axis of the tip-sample.

As a result: the effect of formation nanocrystalline structures in subsurface volumes of platinum in result of implantation accelerated up to 30 keV positive ions of argon ( $F = 10^{16}$ -

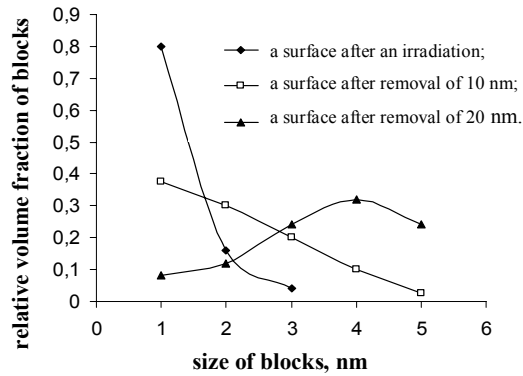


Fig. 1. Distribution of nanoblocks in the sizes on surface irradiated by  $\text{Ar}^+$  Pt ( $F=10^{17} \text{ sm}^{-2}$ ), depending on depth subsurface volume

nanoblocks varied within the limits of 1-5 nanometers with increase in distance up to 20 nanometers on depth. Formation such nanostates can appear perspective for reception of high surface properties of a material.

In the process of studying nanostates in platinum at various regimes of an irradiation the threshold of formation of pores has been found out.

Work is executed with support of the Russian Fund of Basic Researches (the project N 07-02-00722-a). Also it is supported by the Urals Division of the Russian academy of sciences (the Program of presidium of Russian Academy of Science OFN-5 "New materials and structures" and the grant of young scientists and post-graduate students).

### Neutron-Generating Target of Accelerating Source of Epithermal Neutrons

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At present, construction of a pilot accelerating source of epithermal neutrons [1] is being completed at the Institute of Nuclear Physics of Siberian Branch of the Russian Academy of Sciences. The source will render opportunities for introducing the methods of boron-neutron-trapping therapy (BNTP) of malignant tumors [2] in clinical treatment practice. The results obtained in clinical tests conducted on nuclear reactors [3, 4] are promising.

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## Nickel-Based Alloys Structuring Induced by Heavy Ion Irradiation

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It was found that the specific structures form in metals under heavy ion irradiation within a narrow range of radiation parameters such as doses, target temperatures and ion flux densities. Further experiments estimate that these specific radiation-induced states are characterized by nanoclusteric morphology and extremely large changes of material properties. The formation of nanoclusteric structures accompany by significant changes of X-ray diffraction.

There are two types of metallic alloys was studied – Ni-Cr-Mo solid solutions with different Cr concentration and Ni-15Cr-10Fe-2Ti-3Al-3Mo (aging type alloy). The specific radiation-induced state was found in the alloys of the both types. This state exhibits unusual X-ray diffraction pattern – splitting of diffraction peaks and specific nanoclusteric morphology. These changes accompanied by the extremely high microhardness increase. All these specific changes take place in the narrow range of target temperatures and disappear after postirradiation annealing.

Such transformations observed in both the solid solution alloys and in aging alloys. However it was found that disperse participations appearance formed during aging particularly suppress radiation-induced transition and the specific states formation.

## Structure of Ti-TiN Coating Obtained by Method of Condensation with Ion Bombardment

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Results are presented of electron-microscopy and x-ray examination of the structure of Ti-TiN multilayered cermet coating.

Examination of the coating surface by the method of scanning electron microscopy proved to be useful in analysis of dependence of the coating structure on the technology of its application.

Determination of granular structure and dimensions of the regions of coherent scattering, elemental composition and phases crystal structure allowed continuity and homogeneity of the coating layers to be evaluated and the ways of improvement of the technology of coating application to be identified.

### Bimetallic Iron-Nickel Nanoalloy: Synthesis and Characterization

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Synthesis of nano-sized Fe-Ni permalloy of the composition 25 wt% Fe and 75 wt% Ni took place by the relatively low temperature thermal decomposition of co-precipitated [Fe(Bipy)<sub>3</sub>] and [Ni(Bipy)<sub>3</sub>] in inert atmosphere of dry argon gas. The decomposition temperature was decided with the help of TGA. Elemental ratio was confirmed from SEM with EDAX analysis. All peaks were indexed successfully on the basis of fcc structures with XRD. The resulting nanoalloy is found stable in air, single phase, spherical in shape and 15-30 nm in size. The size and shape of the particles confirmed with AFM. Magnetic properties were investigated at room temperature using a VSM with an applied field -5000Oe H 5000Oe. The magnetic hysteresis loop is the typical loop of soft magnet.

### Two-pulsed response of bismuth samples to pulsed electron beam irradiation

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Within the study of thermoacoustic response of several metals [1] we have also made measurements of bismuth samples. Specimens were irradiated with EMIR-M accelerator [2], operated in the mode with electron beam output to the atmosphere. Samples diameters 60 mm and 3 to 12 mm thick were made in laboratory by melting bismuth (impurities concentration less than 0.02%) in aluminum mold in ambient conditions with subsequent machining. Thermoacoustic signal generated by electron beam in the sample was recorded by quartz transducer connected to the back side of irradiated sample.

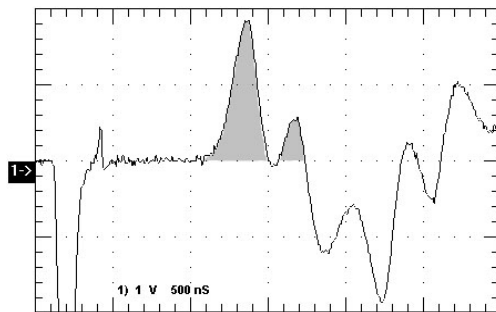


Figure 1. Oscillation pattern of signal from quartz sensor connected to bismuth sample, scales: X – 500 ns/unit, Y – 1.0 V/unit

2.48	2.51	2.51	2.44	2.38	2.23
2.47	2.48	2.48	2.48	2.12	2.22
2.36	2.45	2.45	2.40	2.06	2.05
2.26	2.48	2.41	2.04	2.04	2.08
2.29	2.11	2.10	2.04	2.04	2.13
2.14	2.04	2.05	2.05	2.06	2.10

Figure 2. Map of measured sound speeds on the back side of the sample, km/s.

Figure 1 illustrates oscillation pattern of signal recorded by oscilloscope TDS-2014 (under load 75 Ohm). In this test we irradiated bismuth sample ~3 mm thick. In the beginning of the curve we see the signal of electromagnetic interference of negative polarity

associated with the radiation pulse. After the time corresponding to time of mechanic stress passage through the sample thickness we recorded the signal of thermoacoustic response consisting of two pulses (highlighted with grey in Fig. 1). Roughly in 0.8  $\mu$ s after that the pressure wave reaches the back side of quartz sensor and we see signals of similar shape and reverse polarity on the curve.

We made ultrasonic probing of one sample with the help of thickness gauge (T-MIKE E). The grid with mesh size 5×5 mm was put on the sample for this purpose. The field of the instrument radiation was 4×4 mm in this test. The map of measured speeds on the back side of the sample is illustrated in Fig. 2. The circle here designates the area covered by quartz transducer. We see that the values of measured sound speeds are around two values  $\sim 2.4 \pm 0.1$  and  $\sim 2.1 \pm 0.1$  km/s. The same values of speed of acoustic signal propagation were also found from oscillation pattern illustrated in Fig.1. The sites with different values of speed have similar areas that possibly define comparability of signals measured at irradiation. Noteworthy that amplitude of the second positive pulse in Fig. 1 is undersized as it is superposed on rarefaction wave that goes immediately after the first compression pulse and has reverse polarity.

Results of performed experiments allow suggestion that formation of domains with different sound speed at crystallization is the reason of this observed effect of 'abnormal' thermomechanical response of bismuth samples. This phenomenon is confirmed by visual examination of the samples: specimens with differing sound speed differ in terms of light reflectivity.

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## Composition and Properties of Surface Layers of Cu<sub>50</sub>Ni<sub>50</sub> Foils with Al Coating after Laser Treatment

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Methods of metallic materials treatment with concentrated energy streams presents one of most promising parts of modern material science.

In particular, recent rapid development of laser technology contributed to application of lasers for treatment of metals, aimed to directionally change elemental composition and structural-phase state of their surface layers.

Viability of such technology is due to possibility to create various new surface structures with improved physical-chemical-mechanical properties, using relatively simple devices at simple technological processes. It opens the possibility to create such a different non-equilibrium surface alloys, that are impossible to make with traditional methods of metalworking [1].



In this paper changes of surface layers composition and mechanical properties of Cu<sub>50</sub>Ni<sub>50</sub> foils with Al coating after laser treatment using the methods of XPS, probe microscopy and microhardness measurements are studied.

It is shown that laser energy (10 A, 13 A) influences the elemental redistribution in surface layer both on irradiated and unirradiated sides of foils. At the same time changes of microhardness and surface topography of Cu<sub>50</sub>Ni<sub>50</sub> foils are observed.

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### High-temperature tests of fine-grained high-dense graphite for neutron target converter

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Tests and investigations of samples of fine-grained high-dense graphite MPG brande, CGD and POCO AF5(ZFX-50) have been performed to predict lifetime at high temperatures (~2000°C). Tests are heating of samples by the electric current up to sample destruction temperature. Investigations include electron microscopic, X-ray, electrophysical and other measurements of samples. Rentgenography has been executed for initial samples, results was compared with X-ray photography for graphite of a domestic production MPG-class. The raster electronic microscopy was executed both for initial and heated samples. The forecast of lifetime was based on application of classical Zhurkov formula. It is shown, graphite of CGD and POCO brand has structural characteristics relatives to of MPG graphite, its durability as well as lifetime at high temperatures are close to MPG lifetime.

1. *E.I.Zhmurikov, A.I.Romanenko, P.V.Logachev, K.V.Gubin, O.B.Anikeeva, and Tecchio Luigi.* High-temperature Tests of Fine-Grained Dense Graphite Composites to Predict the Lifetime of a Neutron Target Converter // Journal of Surface Investigation. X-ray, Synchrotron and Neutron Techniques, 2007, Vol.1, No1, PP.60-63

### Modeling of Nanostructured State in Fe-18Cr-8Ni Alloy Subject to Ion Implantation and Severe Cold Deformation

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This study deals with phase transformations and nanostructurization in metastable stainless Fe-18Cr-8Ni steel exposed to a beam of nitrogen ions and then to severe cold plastic deformation (SCPD) by compression shear (CS) in Bridgman anvils. Similarly to ion

deposition of nitrides on the surface of iron, nitriding of iron alloys and compounds doped with nitride-forming elements (Cr, Ti, etc.) can lead to formation of nitride phases (Cr<sub>2</sub>N, CrN, TiN, etc.) in the alloy matrix.

Ion-plasma nitriding of the Fe-18Cr-8Ni steel leads to saturation of the austenite with nitrogen, formation of the CrN nitride, and destabilization of the structure relative to the  $\gamma \rightarrow \alpha$  transformation. It is known that ion beam nitriding forms submicron-sized mixtures of nitride phases in surface layers of the alloy matrix. Nanostructurization of nitrided iron included deformation-induced dissolution of nitrides ( $\gamma'$ -F<sub>4</sub>N, TiN) during CS, leading to additional refinement of the structure. This was followed by formation of a supersaturated solid solution of nitrogen in the iron matrix and precipitation of secondary extremely dispersed nitrides.

SCPD modification of the structure is of practical significance since nitrogen-hardened surfaces of steel parts work in conditions of intensive deformation (friction, impacts, etc.), which, like CS, can induce similar phase transitions and nanostructurization processes in surface layers.

This study was supported by the RAS Presidium program on nanomaterials (project No. 7) and the program "Outstanding scientists – candidates of science'2008" of the Foundation for promotion of national science.

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**Production of Micro- and Nanopoints and Their Emissive Properties**

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**Manufacture of Polymer Matrices with Tapered Pores and Their Replication**

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**Destruction of Graphite Shell by Electrohydroimpulse Methods in Water**

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**Modeling of Neutrons Transport in Type WWER-1000 Nuclear Reactors**

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## **VII. ISTC Working Seminar**

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



**Neutron diffraction study of internal stresses in materials for nuclear reactors.  
ISTC Project 3074.2**

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The Project ISTC 3074 was updated within a framework of PDG-grant up to the 3074.2 version. It was approved and started in September 2007.

The Project objective is investigation of micro- and macrostresses in samples of radiation-resistant steels used in nuclear reactors. The purpose of such investigations is to improve safety of the already existing nuclear power plants (in particular, by more reliable extension for their life expectancy) and development of new advanced materials for nuclear power engineering. Special emphasis will be made on internal stresses in samples of welds of nuclear reactor structural materials presenting critical elements from the point of view of safety of such structures. The Project work envisages a comprehensive study of samples of the test materials. The main method will be high-resolution neutron diffraction analysis providing measurements of both microstresses, which arise under irradiation or during decomposition of solid solutions (followed by the formation of intermetallics, carbides, radiation clusters and other precipitates) in a preset volume of samples, and stresses in welded joints. Besides, neutron diffraction analysis is envisaged of the details of formation and the morphology of precipitates under external uniaxial load. The central task of this Project will be measuring internal microstresses in the bulk of samples from radiation-resistant ageing alloys in the process of formation, growth and coagulation of second-phase disperse particles noticeably influencing pores formation.

The implementation of the Project work will provide new fundamental data concerning the distribution of microstresses near different types of precipitates. This is extremely important for prediction of the radiation damageability of materials.

The proposed Project will pull together the efforts of participants from Institute of Metal Physics UB RAS (Ekaterinburg), the Laboratory of Neutron Physics at Joint Institute of Nuclear Research (Dubna), and Russian Federal Nuclear Center – All-Russia Scientific-Research Institute of Technical Physics (Snezhinsk), who have already accumulated a wealth of experience in studies of the radiation damageability of constructional materials and determination of internal stresses by the neutron diffraction method. Foreign collaborators: Hahn-Meitner Institute, Berlin Neutron Scattering Center (Dr. Rainer Schneider); Institute for Energy, Petten, Netherlands (Dr. Carsten Ohms); Neutron Physics Department of Nuclear Physics Institute of Czech Academy of Sciences (Dr. Pavol Mikula).

### **Radioresistance of structural elements of a carbon target irradiated by deuterons**

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A two-staged target for producing short-lived radioactive isotopes is being developed under the SPIRAL-2 project. At the first stage, deuterons interact with carbon, producing fast neutrons. At the second stage, the fast neutrons cause fission of uranium-238, producing neutron redundant fission fragments. The paper presents results of theoretical research into the radiation effects of deuterons and neutrons on a carbon (graphite) target and its structural elements. The molecular dynamics method was used to predict displacement energies in graphite and stainless steel. These data were used in Monte Carlo calculations which were done to predict the spatial distribution of defect generation rate in the carbon target irradiated by 40-MeV deuterons. The resulted data can be used to evaluate radioresistance of structural elements that defines the operating life-time of the target.

### **Radiation induced enhancement of hydrogen sorption by carbon soot**

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Atom-hydrogen energy was proclaimed as the main prospective in this century. Nanostructure materials based on carbon and oxides are good for safe storage of fixed hydrogen, however it is still a serious problem. This paper presents the results on radiation induced nano-phase transformations in carbon soot and hydrogen sorption from water (radiolysis) under separated gamma-irradiation in the WWR-SM type nuclear reactor at the INP AS RUz during the regular prophylactic shut-down. Structure was studied at the X-ray diffraction spectrometer DRON- UM1 with the selected  $\text{Cu}_{K\alpha 1}$ - radiation with the improved resolution. Dry carbon soot samples contain some ultra disperse fullerene and graphite inclusions and have low conductivity. Under the irradiation there appear carbon nanotubes. Temperature dependences of electric conductivity and dielectric losses measured with DC and AC techniques demonstrated a significant growth of conductivity with a broad peak at 270-300 K. The activation energy of proton conductivity decreases strongly at temperatures close to the ice-water phase transition in the nanopores of fullerite or at nano-tubes, when radiolysis becomes more efficient.

The work was carried out under the grant F2-068 from Committee of Science and Technology Development of Uzbekistan and was partially supported by STCU project Uzb23j.

## **RFNC-VNIITF Research into Interaction of Hydrogen Isotopes with Structural Materials for Termonuclear Facilities**

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The paper presents a number of methods used to study deuterium and tritium interaction with structural materials for nuclear facilities. For vanadium alloys V-10Cr-10Ti and V-4Cr-4Ti and some other materials the paper gives radiometric measurements of the amount of tritium captured in the alloys as the result of sample contact with tritium-containing environment and follow-on vacuum annealing. The hydrogen penetration method is discussed in term of its capabilities to determine coefficients of tritium diffusion, penetration and solubility using as examples Russian-made chromium-nickel-titanium and chromium-nickel-molybdenum-titanium steels and steel SS316L. The method of autoradiography is shown to determine tritium localization in the structure of chromium-nickel-molybdenum-titanium steel which was subjected first to heat treatment and then to long term high-temperature annealing.

## **Radiation Dynamic Effects at Irradiation with Neutrons, Ions, Fission Fragments, Non-traditional Methods of Material Properties Modification and the Problem of Nuclear Reactors Safety**

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The goal of the ISTC Project under the same title is comprehensive experimental and theoretical investigation of radiation-dynamic effects presenting fast processes and phase transformations initiated by irradiation with neutrons, fission fragments and heavy ions, being similar to combustion and detonation phenomena [1], going in metastable metals and alloys, including radiation-resistant steels.

Radiation-dynamic (RD) transformations [1,2] take place at the front of nanoshock waves emerging in materials under irradiation as a result of evolution of dense atom-atom collision cascades. Depending on the type of RD transformations taking place, they may, in some cases, ensue formation of unique electrical, magnetic, tribological, corrosion and other properties of materials, while in other cases, they may significantly deteriorate the service properties of structural materials, particularly those used in nuclear power engineering. For this reason, investigation of such processes is necessitated, on the one hand, by the requirements of safety of nuclear and thermonuclear reactors, and on the other hand, by the need for development of principally new radiation-resistant materials and radiation methods of modification of properties of different materials.

As a positive example of RD effects application, there may be named the methods of improving electrical, magnetic, mechanical and other properties of condensed

media by their treatment with accelerated ion beams and the technology of cold radiation annealing of aluminum alloys (see, e.g., [2]).

In characterizing the effect of radiation, such parameters as the number of displacements per atom and the rate of radiation damage accumulation are mainly used. The fine structure and spatial distribution of the formed radiation defects are analyzed to a lesser degree. And, as a rule, fast radiation-dynamic effects connected with propagation of shock and elastic lattice waves generated at the final stage of development of atom-atom collisions cascades are altogether ignored.

Taking these effects into account is particularly important in case of unstable (metastable) media with high stored energy.

The authors of the project (IEP U BRAD and RFNC) observed fast RD phase transformations in amorphous and crystalline metals and alloys, fissionable materials including.

Regretfully, today the laws governing RD processes remain underinvestigated.

The organizations submitting the Project (IEP and IMP of UB RAS, RFNC) are the leading Russian developers working in the sphere of design of unique research reactors, charged particles accelerators, and occupying the leading position in the sphere of investigation of the effect of ionizing radiation on materials and creation of new radiation-resistant materials. RFNC is the leading designer of nuclear weapons in Russia.

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### **Vanadium Alloy with Ferritic Stainless Steel Cladding: A Material for Fuel Element Shells of Fast Neutron Reactors**

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The involvement of fast neutron reactors in the fuel cycle of nuclear power engineering calls for design of fuel elements with respective service characteristics. The key problem is creation of a construction material with a complex of mechanical and technological properties, compatibility with heat carrier and fuel, and stability of its properties under neutron irradiation. The recent investigations have opened the prospects for application of vanadium-based alloys in plants operating at elevated working temperatures and high neutron fluences.

The purpose of this investigation was the development of a method of cladding of semi-finished products from vanadium alloy V-4Ti-4Cr with type 12X17 corrosion-resistant ferritic steel.



**Computational and Experimental Investigation of Molten-Salt Fluoride Systems  
for Innovative Nuclear Power Engineering**

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The nuclear-power systems on the basis of fluoride melts feature a number of potential advantages over the traditional solid-fuel nuclear systems, such as a good neutron balance due to a minimum number of parasitic absorbers, a significantly smaller amount of fissionable material loading as compared with fast neutron reactors; a higher degree of fuel depletion and an ability of the reactor to operate on fuel with different nuclide compositions; higher performance ratio, and a relative simplicity of fuel regeneration and implementing a closed fuel cycle.

In 2007, the ISTCC Project #1606, which was realized in close cooperation with the MOST European Project, was completed. The principal results of works performed under the ISTC Project # 1606 will be presented.



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