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The Fifth International Ural Seminar

RADIATION DAMAGE
PHYSICS OF METALS
AND ALLOYS

Abstracts

Снежинск
Россия

Snezhinsk
Russia

2003
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The Fifth International Ural Seminar

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Contents

I. General Problems of Radiation Damage Physics

Molecular Dynamics Simulation of a Swift Ion Track in NiAl
C. Abromeit, A.R. Kuznetsov 3

Physical Principles of Radiation Resistance of Alloys
Structural Self-Organization
V.V. Bryk, I.M. Neklyudov 3

Radiation Defects Annealing in Austenitic Stainless Steels with Different Microstructures
S.E. Danilov, V.L. Arbuzov, K.V. Shal’nov, V.A. Pavlov 4

Correlation Effects in Cascades of Atomic Collisions
Yu.N. Devyatko, S.V. Rogozhkin and V.M. Chernov 5

Effect of Intermetallic Precipitates on Evolution of Radiation-Induced Defects in Austenitic Alloys
A.P. Druzhkov, V.L. Arbuzov, D.A. Perminov, K.V. Shal’nov, V.A. Pavlov 5

Theory of Damage Accumulation under Cascade Damage Conditions
S.I. Golubov, B.N. Singh and H. Trinkaus 6

Calculation of Track Transverse Sizes in a YBa$_2$Cu$_3$O$_{7-x}$ Single Crystal Taking into Account Energy Transfer Velocity Dependence on Temperature of Excited Electrons
I.N. Goncharov, B.F. Kostenko, J. Pribiš 7

Long-Range Effect. The Theory and Experiments
V.S. Khmelevskaya, V.G. Malynkin 7

Theory of Irradiation Hardening of Metals and Alloys Based on the Energy Concept of Ductility
Yu.V. Konobeev, V.A. Pechenkin, F.A. Garner 8

Effect of Initial Dislocation Structure on Accumulation and Annealing of Radiation Defects in Austenitic Alloys
D.A. Perminov, A.P. Druzhkov, V.L. Arbuzov, V.A. Pavlov 9

Cascades of Atomic Collisions and Point Defect Accumulation
S.V. Rogozhkin, Yu.N. Devyatko and V.M. Chernov 10

Modeling Complexes of Defects in Solid Solution Using Neutron Spectroscopy
V.V. Sumin 10
Sputtering of Sub-atom Carbon Films on Surfaces of Metals by Low-Energy Gas Ions

Theory and Computer Simulation of Radiation Processes in Multicomponent Materials
Yu.V. Trushin, D.V. Kulikov, V.S. Kharlamov, P.V. Rybin

Effect of Irradiation on Electronic Properties of Pure Compensated Metal Single Crystals at High Magnetic Fields
V.V. Marchenkov, V.E. Arkhipov, A.L. Suvorov and H.W. Weber

Quantitative Measurement of Electron Diffuse Scattering by Single Manometer-Sized Defects in Au and Mo
M. A. Kirk, M. L. Jenkins, R. D. Twesten

II. Effect of Irradiation on Changes in Microstructure and Properties of Metals and Alloys

NDT Measurements of Irradiation Induced Void Swelling in EBR-II Materials
I. I. Balachov, F.A. Garner, I. Isobe, H. T. Tang

Peculiarities of Martensitic Transformation Kinetics in a Monocrystal of Cu-Al-Ni Alloy Being Irradiated by Reactor Neutrons in Low-Temperature Helium Loop
S.P. Belyaev, R.F. Konopleva, I.V. Nazarkin, V.A. Chekanov

Dose Dependences of Mechanical Properties and Composition of Titanium Alloy Surface During Si⁺ and Ar⁺ Ion Irradiation

Atomic Mechanisms of the Microcrack Propagation in Titanium During Ion Irradiation

Disordering of LaMnO₃-Based Compounds by Fast Neutrons

The influence of displacement rate on void swelling in model Fe-Cr-Ni alloys irradiated with either neutrons or nickel ions
F. A. Garner, T. Okita, T. Sato and N. Sekimura

Radiation-Enhanced Amorphism: Comparison of Materials and Influence Techniques
N.N. Gerasimenko
Phase Transformations in Ion Irradiated NiTi Thin Films
T. LaGrange, R. Schäublin, D.S. Grummon, C. Abromeit and R. Gotthardt

Radiation-Induced Enhancement of Critical Current Density in YBa$_2$Cu$_3$O$_{7-x}$ Superconductors
E.M. Ibragimova, M.A. Kirk

Monitoring an Insulator-Metal Transition in Icosahedral AlPdRe by Neutron Irradiation

Martensitic Transformations and Shape Memory Effect in TiNi Alloys in the Process of Neutron Irradiation in Low-Temperature Helium Loop
R.F. Konopleva, S.P. Belyaev, A.E. Volkov, I.V. Nazarkin, V.A. Chekanov

The Percolation Model of Radiation Porosity Influence on Austenitic Steel Strength
A. V. Kozlov, I. A. Portnykh, S. V. Bryushkova, E. A. Kinev

Temperature Dependence of Radiation Swelling of ChS-68 Steel Analyzed by Different Models
A.V. Kozlov, Yu. V. Konobeev, I. A. Portnykh, E. A. Kinev

About the Surface Erosion Mechanisms under the Pulsed Ion Beams Irradiation
G.A. Bleikher, V.P. Krivobokov, O.V. Paschenko

Formation of Nanoscale Intermetallic Phases at High-Intensity Ion Implantation

Vacancy Clusters in Titanium Nickelide Irradiated with Fast Neutrons
V.D. Parkhomenko, S.F. Dubinin, S.G. Teploukhov, E.Z. Valiev

Swelling and Microstructure of EP-753 and Nimonic PE-16 High Nickel Alloys after Neutron Irradiation to High Doses
S. I. Porollo, Yu. V. Konobeev and F. A. Garner

Long-Range Effect in Ion Implantation-Ordered Alloys
N.N. Syutkin, V.V. Alehin
A Short Range Order In The Amorphous Ribbon of Ti$_{0.5}$Ni$_{0.25}$Cu$_{0.25}$ Irradiated by Fast Neutrons
V. D. Parkhomenko, S. F. Dubinin, S. G. Teploukhov 33

Structural Phase Transitions in Intermetallic Compounds Y$_2$Fe$_{17}$ and Ce$_2$Fe$_{17}$ after Irradiation with Fast Neutrons

Comparison of the Effects of Radiation- and Strain-Induced Defects on the Phase Transformation in Iron Alloys
V.V. Sagaradze, A.R. Kuznetsov 35

Long-Range Effect in Metal Foils under Ion and Photon Irradiation

III. Implantated and Transmutational Gaseous Impurities Behavior in Irradiated Metals and Alloys

Determination of Hydrogen Permeation Reduction Factor (PRF) for Different Protective Coats on Vanadium
S.E. Afanasev, T.V. Kulsartov, V.P. Shestakov, E.V. Chikhray, D. Smith 39

Gas Bubble Evolution Peculiarities in Ferritic and Austenitic Steels and Alloys under Helium Irradiation

Hydrogen Accumulation and Retention in Materials of Reactor VVER-1000 Vessel Components
I.M. Neklyudov, B.A. Shilyaev, A.N. Morozov, A.A. Parkhomenko, V.V. Bryk 40

Investigation of Concentration and Temperature Dependences of Processes of Hydrogen and Helium Accumulation and Desorption in Steel 08Cr18Ni10Ti
V.V. Bryk, O.V. Borodin, V.N. Voyevodin, I.M. Neklyudov, V.V. Ruzhitsky, G.D. Tolstolutskaya 40

Hydrogen Thermodesorption from Helium Ions-Implanted Steel 08Cr18Ni10Ti
A.N. Morozov, I.M. Neklyudov, V.V. Bryk, V.G. Kulish, V.I. Zhurba 40

Influence of Interstitial and Substitution Elements on Helium and Hydrogen Behavior in FCC and BCC Metals
I.I. Chernov, B.A. Kalin 41
Investigation of Tritium Permeation Process Produced in Lead-Lithium Eutectic Pb83Li17 Through Tube Steel Sample F82H- in Reactor Irradiation Conditions
T.V. Kulsartov, V.P. Shestakov, E.V. Chikhray, E.A. Kenzhin, A.N. Kolbaenkov, Hiroshi Kawamura and Masaru Nakamichi 42

Investigation of Helium-3 Buildup Operation in Structural Materials by “Tritium Trick” Method
I.L. Malkov, V.G. Klevtsov, A.A. Yukhimchuk, S.V. Zlatoustovski 42

Hydrogen Permeation Through Thin Coatings
A.A. Kurdyumov 43

Development of Helium Bubbles near Grain Boundaries in Nickel-Carbon Alloys
I.V. Reutov and V.F. Reutov 44

Radiation Microstructure, Helium, Hydrogen in Reactor VVER-1000 Vessel Structure Elements
V.N. Voyevodin, V.V. Bryk, O.V. Borodin, B.A. Shilyaev, I.M. Neklyudov 45

Formation and Growth of Hydrides in Irradiated Zirconium Alloys
A.A. Shmakov, A.G. Ioltukhovsky, B.A. Kalin, E.A. Smirnov 45

Structural Factors Influence on Hydrogen Extraction from Irradiated Construction Materials
A.G. Zaluzhnyi, V.P. Kopytin, A.L. Suvorov, D.M. Uspenskyi 46

Specific Features of Interaction of Heavy Hydrogen Isotopes and Radiation-Resistant Cr16Ni15M03Ti1 and SS316L Steels
Yu.N. Dolinsky, Yu.N. Zouev, I.A. Lyasota, I.V. Podgornova, I.V. Saprykina, V.V. Sagaradze 47

Mechanism by Which Titanium Influences Formation of Radiation-Induced Segregation of Deuterium in Cr16Ni15Mo3(Ti1) Steel
G.A. Raspopova, V.L. Arbuzov, V.V. Sagaradze, N.L. Pecherkina, V.A. Pavlov, K.V. Shal’nov, A.P. Druzhkov 47

The In-Pile Experiments on the Reactor IVG.1M
<table>
<thead>
<tr>
<th>Title</th>
<th>Authors</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>IV. Radiation-Enhanced and Radiation-Stimulated Phenomena</td>
<td></td>
<td></td>
</tr>
<tr>
<td>The Radiation-Enhanced and Radiation-Induced Phenomena in Fe-16Cr Based Alloys</td>
<td>A.L. Nikolaev</td>
<td>52</td>
</tr>
<tr>
<td>Theory of Solute Segregation in the Boundary of Finite Size Grains in Polycrystals</td>
<td>V.V. Slezov, L.N. Davydov, O.A. Osmayev</td>
<td>53</td>
</tr>
<tr>
<td>Acceleration of the Rate of Short-Range Layering in Alloy Fe+15at%Cr under Irradiation with Gaseous and Metallic Ions</td>
<td>V.V. Ovchinnikov, B.Yu. Goloborodsky, N.V. Gushchina, L.S. Chemerinskaya, V.A. Semyonkin, E. Wieser, W. Möller</td>
<td>54</td>
</tr>
<tr>
<td>The Laws Governing Radiation-Dynamic Effects in Alloy Fe+8.25at%Mn under Combined Electromagnetic and Ion Irradiation</td>
<td>V.V. Ovchinnikov, B.Yu. Goloborodsky, N.V. Gushchina, L.S. Chemerinskaya</td>
<td>55</td>
</tr>
<tr>
<td>Effect of Ion Irradiation on Oversaturated Solid Solution Decomposition in Alloy Al-4%Cu</td>
<td>V.V. Ovchinnikov, N.V. Gushchina, B.Yu. Goloborodsky, L.S. Chemerinskaya</td>
<td>56</td>
</tr>
<tr>
<td>On the Laws and Nature of Long-Range Dynamic Effects under Corpuscular Irradiation</td>
<td>V.V. Ovchinnikov</td>
<td>56</td>
</tr>
<tr>
<td>Mössbauer Effect Observation on $^{57}$Fe Isotope in $\alpha$-Iron under Ion Bombardment (in situ)</td>
<td>V.V. Ovchinnikov, B.A. Semyonkin, B.Yu. Goloborodsky</td>
<td>57</td>
</tr>
<tr>
<td>Modeling of Accelerated Embrittlement in Reactor Pressure Vessel Steels at High Neutron Fluences</td>
<td>V.A. Pechenkin, Yu.V. Konobeev, I.A. Stepanov, Yu.A. Nikolaev</td>
<td>58</td>
</tr>
<tr>
<td>Significance of Radiation-Induced Segregation in Alloy Properties Alteration under Irradiation</td>
<td>V.A. Pechenkin, Yu.V. Konobeev</td>
<td>58</td>
</tr>
</tbody>
</table>
Grain Shape Effect on the Solute Segregation in Elastic Stressed Polycrystal
R.V. Shapovalov, V.V. Slezov, O.A. Osmayev 59

The Effect of Impurities and Grain Boundaries on Radiation-Enhanced Diffusion in Zirconium
E.A. Smirnov, A.A. Shmakov 60

Kinetics of Intragranaular and Intergranular Radiation-Induced Phosphorus Segregation in Reactor Pressure Vessel Steels
I.A. Stepanov, V. A. Pechenkin and Yu. V. Konobeev 60

Modeling of Radiation-Induced Segregation in Fe-Cr-Ni alloys
I. A. Stepanov and V. A. Pechenkin 61

Redistribution of Phosphorus in Reactor Pressure Vessel Materials During Operation
O.O. Zabusov 62

Radiation-Induced Crystallization of Amorphous Materials
I.A. Antoshina, V.S. Khmelevskaya 63

Materials for Nuclear and Thermonuclear Power Engineering

Uranium Containing Active Layers Behavior under Irradiation at RFNC-VNIITF Pulse Fission Reactors
S.I. Abramenko, I.L. Svyatov, A.E. Shestakov, L.N. Lokhtin 67

Atomic Vibrations and Polymorphism of Actinides

Basic Results of Forced Tests of Core Structural Materials in Fast BN-600 Reactor
V. V. Chuev, A. N. Ogorodov, V.V. Maltsev 68

Commercial Fast Reactor MOX Irradiation Validation
V. V. Chuev, A. N. Ogorodov, V. V. Maltsev, F. G. Reshetnikov, N. B. Sokolov, O. S. Korostin 68

Studies on Modification of Some First Wall Materials Using 3-6.8 MeV He Ions
B. Constantinescu 70

Recent developments concerning void swelling in PWR austenitic internals at relatively low irradiation temperatures
F. A. Garner 71
Corrosion and Electrochemical Properties of EP - 450 Steel Exposed to High-Dose Irradiation in BN - 600 Reactor
O.A. Golosov, V.B. Semerikov, T.L. Kuzina, A.N. Ogorodov, E.A. Kozmanov, E.A. Medvedeva

Irradiation-Assisted Intergranular Stress Corrosion Cracking of Austenitic Stainless Steel in Steam-Water Mixture

X –ray Investigation of Fine Structure of Alloy Zr –2.5% Nb after In –pile Irradiation at ~ 80 °C
V. E. Kalachikov, V. A. Tsygvintsev, N. V. Gloushkova

Changing of Physical and Chemical States of Iron Atoms in Zirconium Alloys Oxide Films During Neutron Irradiation
V.P. Filippov, A.B. Bateev, R.N. Pugachev, G.P. Kobyljansky

Variation of Isothermal Kinetics of α→β-Transformation of Unalloyed Plutonium over Different Times of Samples
Self-Irradiation

Radiation Phenomena in Austenitic Steels Irradiated in SM, BOR-60, VVER-1000 Reactors at 280-400 °C
V.S. Neustroev, Z.E. Ostrovsky, V.K. Shamardin

Peculiar Features of Structure and Behavior Changes in Some Zirconium Materials at Damage Doses up to 50 dpa

Microstructure Changes in EI-847 Steel Exposed to Long-Term Irradiation at 300 -390 °C in Fast Neutron Reactor
I.A. Portnykh V. L. Panchenko, E. N. Shcherbakov , A. V. Kozlov

Influence of Radiation Swelling on Thermal Expansion Coefficient of Steel ChS-68
E.N. Shechrbakov, A. V. Kozlov, I. A. Portnykh, O. I. Asiptsov, V. L. Panchenko, L. A. Skryabin

Influence of Structure-Phase State Zr-Nb- Containing Alloys on Irradiation-Induced Growth
Damage of Beryllium under High-Dose Neutron Irradiation

The Mechanisms of Uranium Metal Fuel Radiation Creep and Its Temperature Nonlinearity
V.D. Rusov, V.A. Tarasov

Modification of Plutonium Dioxide Properties under Exposure to Radiation
A.V. Troshev, S.I. Abramenko, V.N. Kordyukov, A.M. Lyasota

The Investigation of Distribution of Erbium in Uranium-Erbium Oxide Fuel of RBMK by X-ray Analysis
V.A. Tsygvintsev, A. N. Timokhin, V. R. Kuznetsov, V. Ivanov

Comparison of Irradiation Conditions for Fusion Materials in IFMIF and ESS
P.V. Vladimirov, A. Möslang

New Structural Material for Fusion Reactor Based on Ferritic Stainless Steel-Clad V-Ti-Cr Alloy

Study of the Effect of Tritium and Reactor Irradiation on Properties of Materials in the First Wall of Fusion Reactors
Yu.N. Zouev, B.N. Goshchitskii, V.V. Sagaradze

Effects of Actual Aging (Self-Irradiation) of Delta-Stabilized Pu Alloy with Gallium on Microstructure, Mechanical Properties, δ–α′–Phase Transformation Macrokinetics in Weak Shock-Waves

Effects of Prolonged Self-Irradiation on Shear Strength of Unalloyed Pu under Explosive Loading

ix
Features of Microstructural Changes in Unalloyed Coarse-Grained Anisotropic Depleted Uranium after Shock-Wave Loading in the Range of 20-50 GPa at Loading Impulse Duration of 0.5-1.3 µs

Structural Changes in Reactor Alloy of the Hastalloy Type during Long-Time High-Temperature Exposure
I.I. Kositsyna, V.V. Sagaradze, V.G. Subbotin, Yu.N. Zouev 86

VI. Facilities and Techniques of Experiment

Neutron Diffraction Studies of Internal Stresses in Structural Materials for Nuclear Reactors
A.M. Balagurov 89

Investigations of Thermomechanical Effect in Metals on Impulse Electron Accelerators EMIR-M and IGUR-3
V. Afanas’ev, V. Bychkov 89

Research Center of Simulation and Modeling of Irradiation Phenomena in State Scientific Center of Russian Federation “Institute of Physics and Power Engineering”
A.V. Gulevich, Yu.V. Konobeev, V.A. Pechenkin, V.A. Romanov 90

Scanning Probe Microscopy of Radiation Damages of Proton Absorbers
M. Kozodaev, A. Drozdovskyi, O. Makeev 91

Investigation of Behavior of Model Heat Producing Rods for Ignition Pulse Reactor under Pulse Neutron Heating at YAGUAR Reactor

Miniature Scanning Tunnelling Microscope for Intrareactor Investigations
A.L. Suvorov, B.A. Loginov, M.A. Kozodaev, O.N. Makeev 92

Monte Carlo Simulation of Neutron Yield from Be, B, Li and C Targets Irradiated with 10-MeV Proton Beam
V.V. Plokhoi, Ya.Z. Kandiev, S.I. Samarin, G.V. Lukin, D.G. Modestov, G.N. Malyshkin, A.A. Malakhov, S.G. Spirina 93

Ion Track Nanotechnology
V.F. Reutov, S.N. Dmitriev 94
Neutrino Method of Fuel Burning-Out and Radiation Creep Rate Monitoring
V.D. Rusov, V.A. Tarasov, I.Yu. Shaaban, D.A. Litvinov 94

Mass-Spectrometric Facilities for Isotope and Chemical Analysis of Multi-component Mixtures
A.V. Kozlovsky, S. N. Markovsky, G.L. Saksagansky, I. L. Fedichkin 95

Automated Hydrogen Measuring System at IVG.1M reactor
E. Chikhray, V. Shestakov, T. Kulsartov, E. Kenzhin 96

Calorimeter for Intense Electron Beams Dosimetry
A.P. Stepovik, D.V. Khmelnitsky, V.N. Afanas’ev 97

Implanter for Ions of Hydrogen Isotopes Based on Accelerator of NG-12 Device

Proton Accelerator-Based Intense Source of Radioactive Ions

Source of High-Energy Resonance γ-Rays Based on Proton Storage Ring with Electron Cooling
V.A. Vostrikov, K.V. Gubin, V.A. Mashinin, V.V. Parkhomchuk, V.G. Shamovsky 99

Elastic-Plastic Crack Propagation under Irradiation and Cyclic Loads Caused by Thermoacoustic Instability of Heat-Carrier in Active Zone
V.D. Rusov, V.I. Skalozubov, V.A. Tarasov 99

Reactor Antineutrino Cross-Correlation Spectrometry
V.D. Rusov, V.A. Tarasov, D.A. Litvinov 101

Development of Measurement and Governing Systems Meant for Study with Hydrogen Isotope Mixtures
Yu.I. Vinogradov, A.V. Kuryakin, V.S. Aryutkin, S.V. Fil’chagin 101

Synthesis, Study and Application of Aza- and Diazabicyclononanes as Complexing Agents and Preparations for Radiation Hygiene and Medicine
V. A. Dokichev, R. I. Yusupov, E.P. Magda, Y.A. Kulinich, A.G. Tsvetokhin, Y.V. Tomilov 102
VII. Some Aspects of Physics of Radiation Phenomena in Semiconductors and Insulators

Effect of the Nanoparticle Size and Structural Defects on Magnetic Properties of Antiferromagnetic and Ferromagnetic Oxides
T.I. Arbuzova, S.V. Naumov, B.A. Gizhevskii, V.L. Arbuzov, K.V. Shal’nov, E.A. Kozlov

Quantitative Model of Irradiated Semiconductor
V.N. Brudnyi, S.N. Grinyaev, N.G. Kolin

Fermi-Level Pinning in Irradiated Non-Metallic Materials
V.N. Brudnyi, S.N. Grinyaev, N.G. Kolin

$^{11}$B and $^{25}$Mg NMR Estimates of Electron Density-of-States in Superconducting MgB$_2$ Disordered by n$^1$-Irradiation

Long-Lived Radiation Defects on Implanted Silicon
N.N. Gerasimenko (junior), N.N. Gerasimenko, V.Yu. Troitskii

Investigation of Defectiveness of Nanocrystal Ceramics CuO by Positron Annihilation Method

Neutron Diffraction Studies of Structural State of Mn$_3$O$_4$ after Shock Wave Loading

Development of Compacting Technology and Processing of Non-Metal Materials Using Spherical Explosive
E.A. Kozlov, B.A. Gizhevskii, S.V. Naumov

Effect of Indexed-Spectrum and 14-MeV Neutrons on Arsenide - Gallium Schottky Field-Effect Transistors
V.T. Gromov, O.V. Tkachov, V.P. Shukaylo, S.V. Obolensky

Effect of Reactor Neutron Irradiation on InP Single Crystal Structure
N.G. Kolin, V.T. Bublik, M.I. Voronova, D.I. Merkurisov, K.D. Scherbachev

Influence n-$\gamma$ Irradiations on Physical Properties of SiC-Based Diodes
N.G. Orlov, V.T. Gromov, V.P. Shukaylo, A.A. Lebedev, A.M. Strelchuk, N.S. Savkina
Charge Stability of Submicron Integral CMOS-Transistors
Based on Silicon-on-Insulator Structures
V.P. Shukaylo, V.T. Gromov, I.V. Vorozhtsova, T.N. Krushinsky,
V.P. Popov, N.V. Sapozhnikov, A.A. Frantsuzov

Ge/Si Nanostructures with Quantum Dots
Grown by Ion-Beam Assisted Heteroepitaxy
A.V. Dvurechenskii, Z.V. Smagina, R. Groetzschel, A.K. Gutakovskii

Radiation Processes in Si Single Crystals
L.S. Smirnov

Properties of P-Channel Transistors on Silicon Gate Oxide
Created Using BF₂⁺ Ions for Drain/Source Areas
A.N. Tarasenkov, N.N. Gerasimenko, E.V. Kuznetsov, E.Yu. Denisenko

Investigation of Solid-Phase Processes in Insulator and Semiconductor
Materials under Influence of Powerful Electron Irradiation
A.P. Surgikov, A.M. Pritulov, V.V. Peshev, T.S. Frangulian,
A.V. Chernyavski, R.U. Usmanov, B.B. Moyzes, S.A. Gyngazov

Defect Detection in Binary Zinc-Blende Compounds –
Experimental (part-2)
A. Kisiel, B.V. Robouch, E. Burattini, A. Marcelli, M. Cestelli Guidi,
P. Calvani, A. Nucara, E.M. Sheregii, J. Polit, J. Cebulski

Authors Index
I. General Problems of Radiation Damage Physics
Molecular Dynamics Simulation of Swift Ion Track in NiAl

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The aim of the work is to simulate the formation of a track in a metallic alloy and phase transformations induced by irradiation with swift ions. The extremely strong electronic excitations inside a track due to the electronic energy loss during the slowing down process can result in a significant modification of the solid-state structure, e.g., martensitic, and order-disorder phase transformations (in ordered alloys and intermetallics) are possible. Examples for such transformation are Ni-Al alloys where martensitic transformations are well documented. In this study we chose stoichiometric and non-stoichiometric NiAl (which can undergo B2-L\textsubscript{1\text{0}} martensitic transition, order-disorder transition and also amorphization under irradiation). We used the molecular dynamics method (with many-body interatomic potentials). The method is very suitable for study of such fast processes, as track formation, to directly simulate at the atomic level the process of track formation and possible transitions in it.

We showed that swift ions form tracks in NiAl and induce the following microstructural modifications: melting and crystallization, order-disorder and martensitic transformations. Disordering induced in the track is kept after crystallization of the track. Equilibrium (martensitic L\textsubscript{1\text{0}}) phase cannot be reached in ion tracks in the first stage, when diffusion in a solid does not play a role. Martensitic transition commonly begins from the development of lattice instability. The sine-like lattice oscillations are transformed into a sequence of kink-like excitations – “soliton lattice”. The character of the formed domain structure and the kinetics of the transition can depend on the presence of a track. A track can change the scheme of the martensitic transition, i.e., it can be started near the track without freezing of the long-wavelength phonon.

Physical Principles of Radiation Resistance of Alloys
Structural Self-Organization

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Point defect interactions and radiation-induced structural and phase transformations in the Fe-16%Cr-15%Ni-3%Mo steels has been investigated by residual resistivity and scanning tunneling microscopy (STM).

Stainless steels non-doped and doped by titanium at the level of 0.6% and 1.0% have been smelted from high purity components. After cutting the samples of this steels has been quenched from 1373 K in cold water. The existence of the only one austenitic phase has been suggested by X-ray analysis. The part of the investigated samples were annealed at 773 K for short range ordering (SRO) and other part were annealed at 923 K for the formation of $\gamma'$-phase precipitates. The steels were irradiated by 5 MeV electrons at 80 K, 320 K and 573 K.

The isochronous annealing after low temperature irradiation (80K) of Ti doped steel indicate the annealing peaks at 110 and 220 K. The migration energy of self interstitials estimated as 0.65 eV. It is not observed the influence of Ti content and the microstructures to the annealing of interstitials.

The migration of vacancies begin near 300 K with migration energy over 1.0 eV. The decomposition of vacancy complexes occurs in the range 400-550 K with decomposition energy over 1.4-1.5 eV.

The ordering processes characteristic of these alloys take place at temperatures more than 300K owing to the migration of vacancies during irradiation and at the annealing. The titanium doping suppresses the ordering. The resistivity of irradiated and nonirradiated steels higher than 750 K settles to the equilibrium dependencies of resistivity via temperature. This dependencies is altered in according with the concentration of titanium.

The radiation-induced formation of $\gamma'$-phase precipitates with size of 2 – 3 nm with the concentration over $10^{18}$ cm$^{-3}$ has been detected at 573 K electron irradiation by means STM investigation. The thermal formation of such precipitates occurs at temperatures higher than 800 K. The influence of microstructures on the radiation-induced processes is considered.

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Correlation Effects in Cascades of Atomic Collisions

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The defect formation in metallic materials under reactor irradiation takes place due to cascades of atomic collisions. Only long-term effects of these microscopic processes can be investigated by an experimental way. That is why the main methods of the research of the initial radiation damage via cascades formations are theoretical or computational. Being irradiated in a reactor, materials is non-equilibrium medium with strong fluctuations. The average concentrations of defects in such medium are by far more than their equilibrium thermodynamic values. The fluctuations of the defect concentration resulted from the cascades are not thermodynamic. Their values vary depending on the energy of a primary knocked atom (PKA). Moreover, the PKA energy having released in the region of the cascade excites strong non-equilibrium fluctuations of temperature. All these circumstances must be taken into account to describe the process of point defect accumulation inside the medium correctly. However, the consequent procedure of averaging is not applied, and the role of fluctuations is neglected. The effective source of defect formation is much less than the value determined by the cascade function.

One of the aspects in which fluctuations play especially important role is the void size distribution. Usually (follow Lifshitz and Slezov) the void formation are considered as a phase transition in oversaturated solution of vacancies. In this approach, the Fokker-Planck equation should be used to describe the void size distribution. In fact this means that random forces presented in the void growth equation are -correlated. It is cannot be used for cascade fluctuations. The input of these fluctuations significantly modifies the void growth equation. The critical radius of a void sharply decreases while the amplitude of defect density fluctuations rises. Thus, the defect creation in atomic collision cascades needs the correlation function of random forces to be calculated very accurately, because it defines the diffusion in the space of void sizes. To sum up, the correlation function is another important characteristic of the defect formation process in cascades.

The Effect of Intermetallic Precipitates on the Evolution of Radiation-Induced Defects in Austenitic Alloys

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The projected degree of the atomic fuel burnout in fast-neutron atomic reactors imposes stringent requirements on the radiation resistance of constructional austenitic steels. In this connection, the search for efficient methods of suppressing the vacancy swelling of these steels represents one of the key problems of the radiation materials science. Some researchers proposed to reduce swelling of austenitic alloys by producing elastically distorted microscopic regions re-
lated to coherent boundaries of fine precipitates of secondary phases. In particular, it was hypothesized that point defects are recombined quickly on coherent interfaces in precipitation-hardening alloys.

To verify this hypothesis, we studied the accumulation of radiation defects under electron irradiation in controlled conditions at an early stage of radiation damage ($10^{-4} \cdot 10^{-3}$ dpa) of Fe-36%Ni-2%Ti model alloys, which were quenched and aged beforehand. The samples were quenched and aged at 923 K for 0.5 to 50 hours. The size and concentration of coherent Ni$_3$Ti intermetallic precipitates was 2 to 15 nm and $10^{17}$ to $10^{15}$ cm$^{-3}$ respectively depending on the aging time. The irradiation temperature was varied between room temperature and 573 K. The accumulation of vacancy-type radiation defects in the form of three-dimensional clusters or nuclei of loops was checked by the positron annihilation spectroscopy.

It was found that the precipitates affected the accumulation of vacancy-type defects at an irradiation temperature of 573 K. The density of vacancy loops in aged Fe-36%Ni-2%Ti alloys decreased several times as compared to the density in the Fe-36%Ni alloy irrespectively of the size of the precipitates. Radiation-accelerated precipitates, which also inhibited the accumulation of loops, appeared in the quenched titanium-loaded alloy with increasing radiation fluence.

Vacancies interacted with titanium atoms in the solid solution at lower temperatures. This interaction made it difficult to determine the role of the precipitates.

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**Theory of Damage Accumulation under Cascade Damage Conditions**

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In recent years, a considerable amount of effort has been spent on establishing a new model called the Production Bias Model (PBM) originally proposed by Woo and Singh (1990, 1992). The model takes into account the consequences of intracascade recombination, spontaneous formation of clusters of vacancies and self-interstitial atoms (SIAs) and one-dimensional diffusion of the SIA clusters. While diffusing, these clusters are likely to interact with all other sinks present in the crystal. Over the years, these features of the primary damage production and the ensuing consequences have been incorporated in the production bias model (PBM). During the last ten years or so, various aspects of defect accumulation and microstructural evolution under cascade damage conditions have been treated both analytically and numerically within the framework of the PBM. It has been shown that the PBM explains many features of damage accumulation under cascade damage conditions, which could not be explained in terms of the SRT and BEK types of models. Thus the model allows rationalizing high swelling rate at low dislocation density, the grain boundary/size effects, the decoration of dislocations with SIA loops,
void lattice formation and saturation of void growth. The present talk will, first of all, briefly review the progress and the main results. Recently, the dose and temperature dependences of microstructural evolution including SIA loops, stacking fault tetrahedra (SFTs) and voids have been evaluated. The main results of these calculations will be presented and compared with experimental results. Implications of these results to further calculations, for example, of microstructural evolution in alloys will be discussed.

Calculation of Track Transverse Sizes in a YBa$_2$Cu$_3$O$_{7-x}$ Single Crystal Taking into Account Energy Transfer Velocity Dependence on Temperature of Excited Electrons

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High resolution electron microscope measurements of track transverse sizes in high temperature superconductor single crystals testify to a relatively small scatter of their values (for the specific type of heavy ions) and sharp borders of the internal amorphous region. These facts point out to a dominant role of superfast melting-hardening processes in track creation (thermal spike model). The effective times of electron-atom energy relaxation ($\tau_{\text{eff}}$) in a YBa$_2$Cu$_3$O$_{7-x}$ single crystal were calculated in our paper [1]. They are in satisfactory agreement with the experimental data, obtained using short laser pulses for the same material, and with the Allen theory which has predicted a linear $\tau$ dependence of excited electron temperature (see references in paper [1]).

Taking into account the foregoing, we have calculated the transfer sizes of heavy ions with $dE/dx=20\sim40$ KeV/nm in YBCO in the frame of the thermal spike model.

References

Long-Range Effect. The Theory and Experiments

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Long-range effect (effect of deep and superdeep penetrating of a radiation damage front into a matter) is a subject of thorough studies and wide discussions for a long time because this is a phenomenon of a great interest for both fundamental and applied needs. Present models of irradiation-matter interaction are based on a consideration of isolated collisions of fast particles with atoms of a matter and following diffusional relaxation of damages. This predicts a very small depth of radiation-changed layer (of 100 – 1000 A for middle energies of ions). By the way experiments give significantly larger values – microns or even tens and hundreds of
microns. In the frame of present models two explanations are proposed to this disparity. It is, firstly, an appearance of considerable strains within a subsurface layer; secondly, an influence of radiation-stimulated diffusion. However, due various reason a totality of experimental facts are not explained adequately by these. It seems to be that some models based on supposition of a co-operative interaction are more perspective ones [1,2].

A straight evidence of long-range effect existence is obtained by us. We observed “radiation white layers” on cross-sections of the Fe-Cr-Ni, the Fe-Cr, the V-Ti-Cr alloy samples (the layers of changed etching capability) of 20-30 μm depth. A radiation-induced hardening and changes of other properties extend for the same depth. Similar layers were observed in various materials including hard alloy (VK8) where it with difficulty can be supposed deep spreading of strains. Additionally it was observed a disintegration of supersaturated solid solution (the alloy of the nimonic type) and an appearance of particles of other phases within a clearly limited subsurface layer of approximately the same depth.

A field of the long-range effect manifestation in metal alloys studied corresponds to the field of dissipative structures formerly found by us [3,4]. These are nonequilibrium states arising in open systems within nonlinear range. It is known that such structures are a result of a co-operative response of a system to an external influence. So the results of the experiments described are the confirmation of the models of the co-operative mechanism of the long-range effect.

References

Theory of Irradiation Hardening of Metals and Alloys
Based on the Energy Concept of Ductility

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The irradiation induced hardening of metals and alloys is determined by their microstructure (density of dislocation network, sizes and concentrations of dislocation loops, voids, precipitates, etc.). The conventional approach in estimating irradiation hardening consists in evaluation of the critical shear stress for a dislocation segment bowing between two identical barriers. Total hardening by all microstructural components is obtained as the sum of separate contributions or as the root square of the sum of squared separate contributions.

In the present work a new method of calculating the yield strength of metals and alloys is proposed, based on energy concept of ductility. This method allows to calculate barrier harden-
ing constants in an explicit form within the framework of isotropic elasticity theory. Expressions for hardening by the dislocation network, voids and precipitates are derived. In the case of dislocation hardening, the tensile yield strength depends as the root square of dislocation density, the hardening constant is close to unity. This result coincides with well known result based on the forest dislocation theory. In the case of hardening by voids a distinct from widely known result is obtained: hardening constant is determined by the void surface energy, but not by the shear modulus. In the case of hardening by spherical misfit precipitates in isotropic and homogeneous medium a general expression for the yield strength is derived. This yield strength is proportional to product of the misfit and root square of the volume occupied by precipitate particles. The last result means that the precipitate hardening should not change under irradiation beginning from the stage of coalescence or coagulation of precipitates.

Effect of Initial Dislocation Structure on Accumulation and Annealing of Radiation Defects in Austenitic Alloys

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Chromium-nickel stainless steels are among main constructional materials of active zones in fast-neutron reactors. Vacancy swelling represents a significant phenomenon limiting the use of these steels. Preliminary cold deformation may be used as one of the methods for inhibiting the vacancy swelling of the constructional materials. Dislocations, which are formed during cold deformation, serve as sinks of point defects. The accumulation of radiation defects may also be reduced by introducing alloying impurities, such as silicon or titanium, into the steels. The goal of this study was to determine the effect of the dislocation structure and impurity titanium atoms on the accumulation and annealing of vacancy-type defects in model austenitic alloys at an early stage of radiation damage ($10^{-4}$-$10^{-3}$ dpa).

Fe-36.5 mass % Ni and Fe-36.5 mass % Ni-2 mass % Ti alloys, which were pre-deformed (40%) at room temperature, were studied. The samples were irradiated with 5-MeV electrons at 300 K and 573 K in a linear accelerator. Isochronal annealing was performed in a vacuum of $10^{-5}$ Pa at an average heating rate of 1 K/min. The positron annihilation spectroscopy, which is highly sensitive to vacancy defects and dislocations, but is insensitive to interstitial atoms and their agglomerates, was used in the study.

It was found that the accumulation and annealing of point defects in the austenitic alloys depended on the initial microstructure of the samples. When the initial density of dislocations in the Fe-Ni samples was large, the accumulation of vacancy clusters decreased several times as compared to the samples subject to solid-solution hardening. Alloiyor the compound with titanium caused a considerable change in the behavior of vacancy defects and dislocations during irradiation and annealing. Titanium enhanced clusterization of vacancies both in quenched and deformed samples at room temperature and retarded dissociation of vacancy clusters during annealing. Ni$_3$Ti precipitates, which were formed on the dislocation lines in the titanium-loaded
alloy, pinned the dislocations. Consequently, initial stages of recrystallization were decelerated. The same precipitates impeded the capture of positrons by dislocations.

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Cascades of Atomic Collisions and Point Defect Accumulation

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Cascades of atomic collisions play an important role in the defect structure formation. However, as they last very short time (about several picoseconds), there is no detail understanding how they work. Computer simulation and some analytical approaches allow to bring to light a number of peculiarities of the cascade and to distinguish some stages in the process itself. The evolution of a defect structure in the irradiated material is usually described by models based on macroscopically averaged characteristics (for example, the concentrations of point defects). The non-linear interaction of the defects in a cascade area needs additional attention to calculating their average concentration. To note, the description using the average concentration completely misses the peculiarities of defect creation in cascades. The interaction between bombarding particles and atoms of the target is a probabilistic process. Therefore, defect creation is a random process. For the quantitative description of point defect accumulation it is necessary to use the stochastic equations and to average them.

In this work we have completed a careful sequential procedure of averaging the kinetic equations describing point defect accumulation. We have managed to take into account the fluctuations of the defect formation process in material under irradiation. An effective source of point defect creation is obtained. The calculations were carried out for the case of pure iron irradiated by fast reactor neutrons with the energy 0.6 MeV. The values $Q$ turned out to depend upon the density of sinks in the medium. The obtained values $Q$ are far less than those that can be found in the literature for the source of defects under neutron irradiation $\\sim 1\text{ dpa/s}$. Thus, it should be noted that the damage calculation based on the only dpa parameter results in overestimations.

Modeling Complexes of Defects in Solid Solution

Using Neutron Spectroscopy

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A new method for the calculation of splitting of the fundamental local modes was proposed. It takes account of the relaxation of metal atoms around the defects and the elastic inter-
action of the deformation fields. This approach allows one to explain the neutron spectroscopy results, which was impossible in case of the force constant approach.

This method will be useful in interpretation of neutron spectroscopy data in interaction between radiation point defects and interstitial (O, C, N, H) atoms.

Sputtering of Sub-atom Carbon Films on the Surfaces of Metals by the Low-Energy Gas Ions

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An earlier developed original experimental technique for measuring and analyzing the parameters of low-frequency fluctuations of field-electron current of metal-film systems was used in the present work to measure sputtering yield $Y_f$ of carbon films (degree of covering $\theta$ from 1 to 5) on $Fe$, $Nb$, $Ta$ and $U$. The measured values proper were amplitudes $\Delta I_n$ of anomalous field-electron current fluctuations arising due to single bombarding ions hitting the film. Sputtering yield $Y_f$ evaluation was carried out with the use of an analytical expression of the developed theoretical model. Measurements were made of coefficients $Y_f$ of carbon films sputtering with ions $H^+$ and $He^+$ with energies $E_i$ ranging between 2.0 and 10.0 keV. For each type of ions, the energy dependence of $Y_f$ were obtained for fixed values of $\theta$; besides, for certain fixed ion energy values $E_i$, for each type of ions, there were obtained the dependence of $Y_f$ on $\theta$. The measured values of $Y_f$ in all cases noticeably exceed the same in case of pure carbon.

Another original technique combining field-ion microscopy and fine measurements of light characteristics of local areas of field-ion microscopy images was used to determine energy thresholds $E_{th}$ of the beginning of sputtering of carbon films on metal surface. Energy distributions of $Y_f$ for different values of $\theta$ in the threshold energy range were measured.

Theory and Computer Simulation of Radiation Processes in Multicomponent Materials

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The basis of the radiation processes in the multicomponent materials is presented briefly [1-4]. The physical features of the cascade processes in the materials with impurities are exam-
ined as well as processes taking place on the diffusion and sink evolution stages and their influence on the changes of macroscopic properties of the materials.

The problems of computer simulation of the radiation processes in the multicomponent materials are viewed [2,3]. The features of Monte-Carlo computer codes' constructing with accounting of cascade regions' overlapping when irradiating with high fluences are noted [2,3]. Several results of the ballistic simulation for specific materials are presented [2,3,5,6]. The attention is put on the describing of the physical principles of diffusion simulation in the irradiated materials. The physical results for computer simulation for the materials with different composition and structure under irradiation are presented [2,3,7,8].

References

Effect of Irradiation on Electronic Properties of Pure Compensated Metal Single Crystals at High Magnetic Fields


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The effect of 5 MeV electron irradiation on the crystal structure, transverse magnetoresistivity $\rho_{xx}$ and Shubnikov – de Haas effect in pure tungsten single crystals was investigated. The structure of tungsten was studied by field-ion microscopy. The electronic properties were measured in magnetic fields up to 15 T and over the temperature range 2 – 55 K in tungsten single crystals having a residual resistivity ratio $\rho_{293K}/\rho_{4.2K} \approx 75000$ prior and after irradiating up to fluences of $1\cdot10^{18}$ els/cm$^2$ and $2\cdot10^{18}$ els/cm$^2$ near room temperature. It was shown that the radiation-induced defects strongly affect the transverse magnetoresistivity and the amplitude of Shubnikov – de Haas oscillations. It was observed that the radiation-induced defects lead to substantial changes in the temperature dependence of the magnetoresistivity at
low temperatures, i.e., the exponential dependence changes to the power one. The possible reasons of such behavior of $\rho_{xx}$ are discussed. The field-ion microscopy study showed the presence of the isolated vacancies and their compact complexes in irradiated crystals. Namely these types of radiation-induced defects lead to the substantial changes in the electronic properties.

**Quantitative Measurement of Electron Diffuse Scattering by Single Manometer-Sized Defects in Au and Mo**

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Elastic diffuse scattering of electrons by single nanometer-sized defects in ion irradiated Au and Mo has been measured quantitatively. Huang scattering from isolated single defects is separated from Bragg scattering at a weakly excited diffraction peak (usually 2 g, with near 5 g excited). The asymmetry in the Huang scattering immediately reveals the interstitial or vacancy nature of the defect. Results will be compared among various dislocation loop geometries and with calculations. Comparison to similar measurements on necessarily broad distributions of defect types and sizes by diffuse x-ray scattering will be made. Contributions of inelastic electron scattering to defect images and diffraction data are also measured and will be discussed.

Work is supported by DoE.
II. Effect of Irradiation on Changes in Microstructure and Properties of Metals and Alloys
Irradiation-induced void swelling has been postulated as a potential degradation mechanism for core internals of commercial Pressurized Water Reactors (PWR). While evidence of void swelling in PWR materials has not been directly obtained, similar materials from fast breeder reactors show swelling under PWR relevant temperatures and dpa. US DOE and EPRI sponsor an effort to develop an in-situ void swelling inspection technique based on non-destructive examination of core internals for the US Nuclear Industry.

The principle of in-situ detection of void swelling [1] is based on the previous work performed in the US and Russia where almost linear correlation between swelling and measurable material parameters (electrical resistivity and Young’s modulus) have been observed. Important limitation in practical application is that the inspected component (such as a baffle plate) may be accessed only from one side. This limitation eliminates several attractive techniques (for example, resonance ultrasonic technique) from the list of candidates for field use. Initial screening of the candidate techniques included (1) eddy current, (2) Barkhausen noise, (3) electropotential or four probe resistivity measurements, (4) guided waves, and (5) conventional ultrasonic technique. Proof of concept was performed on a set of surrogate materials with elastic and electrical properties changed by power metallurgy technology and cold work. The following three techniques have been downselected for testing on irradiated materials (1) ultrasonic, (2) eddy current and (3) electropotential technique [1]. These three techniques have been tested on irradiated materials with known amount of swelling (determined by immersion density measurements in the previous work). The set of materials included 26 disks punched from a hexagonal sheath of the EBR-II reflector. Nominal dimensions of disks are 19 mm in diameter and 1 mm thick. Temperature range is 378 to 414 °C, dose varied from 4 to 35 dpa, which covers a range of void swelling from 0.128 to 2.808%. Most of the specimens (21 out of 23) have been irradiated at temperatures 378 to 390°C. The exposure is represented relatively uniformly between 4 and 35 dpa. Distribution in swelling was shifted towards lower swelling in order to estimate sensitivity threshold of the void swelling sensors.

Most encouraging results have been obtained by using UT technique. First, a reliable correlation between sensor signal and void swelling was observed. Second, the sensitivity of the method defined in experiments with EBR-II disks seems to be sufficient for practical applications. Success with UT technique indicates that dominant changes in elastic properties of EBR-II disks have been induced by formation of voids, which is the expected situation for SS304 material exposed to PWR conditions.

Results of EC and EP method are more difficult to interpret. Both methods produced extremely repeatable results, which indicates to adequate design of sensors and experimental conditions. At the same time, measured electric properties of EBR-II disks seem to have no
correlation with void swelling. The major questions are: «What kind of inclusions change the electrical properties of EBR-II disks in addition to voids?» and «Are these inclusions in EBR-II disks caused by operating conditions of the EBR-II reactor relevant to PWR operating conditions?» The answers to this questions have to be obtained yet from examination of microstructure and microchemistry of EBR-II materials.

References


Peculiarities of Martensitic Transformation Kinetics in a Monocrystal of Cu-Al-Ni Alloy Being Irradiated by Reactor Neutrons in Low-Temperature Helium Loop

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The temperature kinetics of the martensitic transformation in a monocrystal of the Cu-3.4%Al-5%Ni alloy have been investigated with the use of technique of in-reactor measurements of resistivity in the process of irradiation in the low-temperature helium loop of the WWR-M nuclear reactor at the temperatures of 120-350 K. In the experiments, the isothermal irradiation has been alternated with thermal cycles through the martensitic transformation temperature interval. So the temperature dependencies of the resistivity have been obtained in various irradiation periods. The isothermal irradiation temperatures have been selected so that they should correspond to various structural state of the material (austenite, martensite or two-phase).

It has been established that in the area of small fast neutron fluences (<6·10^18 cm^2), the resistivity of the Cu-Al-Ni alloy rises linearly with the fluence growth. The resistivity growth rate significantly depends upon the structural state of the material: in the austenite state it is more than by five times as less as compared with the martensite state. Upon irradiation in the two-phase or martensite state, there is a considerable (by 25-30 K) growth of temperatures of the reverse transformation “martensite→austenite”. Such growth is observed once only in the process of the first heating being implemented directly after the isothermal irradiation and proportional to the integral neutron dose. At subsequent thermal cycles, the martensite transition occurs at lesser temperatures, and the hysteresis temperature dependence of the resistivity shifts towards lower temperatures with the growth of fluence. So the radiation effect leads to a stable decrease of critical transformation temperature and to a single (unstable) growth of the reverse transformation temperatures. The first of these effects is caused by the disordering of the crystal lattice at the reactor irradiation, similar to the situation, which is also observed in TiNi-based alloys. The single effect is perhaps connected with the violation of coherence of the interphase
and interdomain boundaries in the course of irradiation. The boundary mobility, in this case, decreases and an additional thermodynamic stimulus is required for the transformation development. Special experiments indirectly prove this supposition.

**Dose Dependences of Mechanical Properties and Composition of Titanium Alloy Surface During Si⁺ and Ar⁺ Ion Irradiation**

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A comparative study of fatigue strength, microhardness, the compositions of the surface layers and microstructures of ruptures of the Ti-2,2Al-0,6Mn alloy samples irradiated with Ar⁺ and Si⁺ ions (E = 40 KeV, j = 10 μA/cm²) has been conducted depending on the radiation dose (D=10¹⁵, 10¹⁶, 2×10¹⁶, 3×10¹⁶, 5×10¹⁶ ion/cm²).

Fatigue strength tests were performed by means of the console bending method at maximal alternate bending stress over the section (9 x 2 mm²) \( \sigma_{max} \approx 24 - 45 \text{ kgf/mm}^2 \) and at frequency of 22,5 Hz. The composition of the surface layers was studied with the use of the mass-spectrometer MC-7101M. Microhardness was measured by means of a conventional technique with the use of PMT-3 with loads of 20 and 100 g. Rupture microstructures were studied by means of the scanning electron microscope SEM-200. Additionally, the X-ray structure analysis and microstructure investigations were conducted.

The microhardness of the samples exposed to silicon and argon ions increases by 10-30% as compared to the initial value. A maximum increase of the microhardness (the applied load on the indentor is 20 g) is attained either after the silicon or argon ion irradiation with the dose 10¹⁶ ion/cm².

The fatigue tests show that the samples irradiated with silicon and argon (with the dose 10¹⁶ – 3x10¹⁶ ion/cm²) have endured a much larger number of cycles before they rupture than the initial samples at the same stresses over the section. The maximal increase of the fatigue strength takes place at the 10¹⁶ ion/cm² dose just as the maximal increase of the microhardness does. When exposed to the Ar⁺ ions with the dose 10¹⁵ ion/cm² the sample ruptures prematurely.

The analysis of the chemical composition of the implanted samples after the rupture shows a significant enrichment of the surface layers with aluminium and manganese atoms. Besides, the transformation of the concentration profile of the interstitial silicon atoms can be observed. At the rupture itself, large amounts of aluminium, manganese and silicon and only traces of titanium can be found. It may point to the precipitation of the alloying components along the rupture boundary and the silicon atom migration to the surface during the fatigue tests.

The results of the X-ray structure analysis do not reveal any changes in the structure-phase states in the bulks of the samples irradiated with Si⁺ and Ar⁺ ions as compared to the initial samples. Consequently, the fatigue strength increase of the irradiated samples is due to the changes of the structure and composition of the surface layers caused by the ion implantation.
Atomic Mechanisms of the Microcrack Propagation in Titanium During Ion Irradiation

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The process of the microcrack propagation in the model crystallite with the close-packed hexagonal lattice at the uniaxial tension has been studied after the implantation of inert gas ions with the use of the molecular-dynamic simulation; titanium is used as an example. The attempt has been made to clarify the influence of ion implantation on the microcrack propagation in model metals with the hcp-lattice. The comparative study of the microcrack propagation in metals with different types of lattice at the uniaxial tension has been conducted.

The computer-based experiment is performed with the use of the MMDYN programme, which allows to solve the set of many-particle equations of Newton by means of numerical methods. The potentials of pairwise interaction are used to describe the interatomic interactions; these potentials have been calculated based on the model Heine-Abarenkov-Animalu pseudopotential. Energy removal from the system is realized by introducing viscous forces. The initial atomic configuration of the crystallite was a three-dimensional film with the thickness of 20 atomic layers. The surface (001) was free; on the surfaces (100) and (010), the cyclic boundary conditions were superimposed. A system consisting of 4000 atoms was calculated in each computer experiment. The crack was simulated by the removal of a certain number of atoms from two neighboring planes at a depth of 10 atomic layers.

In the area of the mouth of a crack a heat peak was simulated for the period of $8000 \times 10000 \tau$; the thermodynamic parameters of the system, i.e., temperature $T$ and potential energy $U$, were stabilized near a certain value. After the crystallite had reached a stable state over the range of the times under study, the uniaxial tension along [100] direction was performed. The relative strain was $\varepsilon = 5\%, 10\%$ и $15\%$.

The analysis of the atomic structure of the crystallite shows that the microcrack does not recover after the ion irradiation; it transforms into a micropore. As this takes place, the propagation of the region enriched with defects deep into the crystallite does not occur. The transition from the microcrack to the micropore occurs in accordance with the vacancy mechanism. The propagation of the microcrack deep into the crystallite is not observed at the 5% and 10% uniaxial tension of the irradiated crystallite. The widening of the microcrack can be observed; migrations of separate atoms are possible only along the boundaries of the microcrack. The atomic rearrangements caused by the ion irradiation lead to the strength increase of the crystallite. The peculiarities of microcrack propagation in metals with different types of lattice (bcc, fcc, hcp) have been studied. It has been shown that the propagation of the microcrack in pure metals has a viscous character.

The work has been financed by the Russian Foundation for Basic Research (project № 02-0216670).

Disordering of LaMnO$_3$-Based Compounds by Fast Neutrons
The perovskite –type compound La$_{0.85}$Sr$_{0.15}$MnO$_3$ was exposed to fast neutrons ($F = 2 \times 10^{19}$ cm$^{-2}$, $T_{irr} = 340$K) and then the structure of this monocrystal was studied by the thermal neutron diffraction method at 300°C. Areas of small size were found in the crystal lattice of the oxide affected by fast neutrons; the structure and the size of the areas being related to the peculiarities of branching of the atomic cascade collisions in the compounds of this class. For the first time it was found, that anti-site defects (~15% of diamagnetic cations transferred into the sites of manganese sublattices) had been formed in a crystal of the oxide exposed to fast neutrons. It was shown by the example of the La$_{0.8}$Ba$_{0.2}$MnO$_3$ exposed to $F = 3 \times 10^{20}$ cm$^{-2}$ at $T_{irr} = 340$K that, a defect pattern of the perovskite crystal does not considerably depend on the element of the dopant.

The Influence of Displacement Rate on Void Swelling in model Fe-Cr-Ni Alloys Irradiated with Either Neutrons or Nickel Ions

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The results of two companion experiments exploring the effect of dpa rate and composition on void swelling will be presented. These experiments used simple Fe-Cr-Ni ternary alloys irradiated over a wide range of dpa rates to determine the influence of dpa rate on both microstructural evolution and macroscopic swelling.

The first experiment involved irradiation of three simple alloys (Fe-15Cr-16Ni, Fe-15Cr-16Ni-0.25Ti, and Fe-15Cr-16Ni-0.25Ti-0.04 C) in FFTF at ~400°C over a range of dpa rates, $8.9 \times 10^{-9}$ to $1.7 \times 10^{-6}$ dpa/sec. The results show very clearly that the effect of increasing dpa rate is to extend the transient regime of swelling for the two carbon-free alloys, but without any change in the steady-state swelling rate of ~1%/dpa. The microstructural origin of this process arises primarily from the sensitivity of Frank loop formation, interaction and unfaulting to dpa rate. The primary requirement to attain steady-state swelling is the resultant formation of a mobile dislocation network. The addition of carbon suppresses the flux sensitivity, however, and the microstructural origins of this response are currently under investigation.

The second experiment involves nickel ion irradiation of Fe-15Cr-16Ni only, but at three temperatures, 300, 400, 500°C, and three dpa rates between $1 \times 10^{-4}$ and $1 \times 10^{-3}$ dpa/sec.

At these higher dpa rates the transient regime is longer than that observed in the neutron irradiation experiment, and increases further as the dpa rate increases. Once again the most
important results are the sensitivity of Frank loop behavior to dpa rate, and the decrease of swelling at every tested temperature as the dpa rate increases.

The implications of these findings on the interpretation of other experiments conducted on neutron-irradiated commercial alloys will be discussed. In general these latter studies also show that swelling decreases as the dpa rate increases, primarily due to an extension of the duration of the transient regime.

**Radiation-Enhanced Amorphism: Comparison of Materials and Influence Techniques**

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In this work an attempt is made of analytic comparative review of experimental results and theoretical conceptions about amorphism in semiconducting materials (monatomic and multicomponent) and metals. The following problems are considered:

1. Is it possible to get amorphism during irradiation by:
   - fast-moving electrons;
   - protons;
   - neutrons.
2. The difference in carbon and silicon amorphism processes.
3. The existing models of amorphism for ion bombardment of silicon. Accumulation of defects critical concentration: spatial separation of internodal and vacancy defects (either by means of different mobility or due to dividing agents – boundary surfaces, force fields, getters and so on).
4. Statements about amorphism processes in metals. Does critical concentration of defects exist?
6. Phase transition monocrystal – amorphous state and vice-versa. Consideration from the synergetic position. Could one expect self-organization under such transitions?

Offered report does not have one-valued answers on majority formulated problems and its aim is drawing attention on the set of known experimental results and approaches to their description. An attempt is made to compare known approaches of description between semiconducting and metal materials.

**Phase Transformations in Ion Irradiated NiTi Thin Films**

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SMA thin films have an anomalously high strain energy storage output and large transformational strains which have stimulated a current interest for their use as active elements that require large force and displacement outputs. We have studied a novel processing technique using ion irradiation to develop a thin film micro-actuator. The main experimental approach has been to irradiate one side of martensitic TiNi thin film that has been pre-deformed in tension. With sufficient irradiation dose, the martensitic transformation can be suppressed in the beam-damaged layer, whose thickness can be on the order of half the film’s thickness. During heating to austenite phase, only the un-modified martensite structure can recover and contract to its original length, and the differential strain between the damaged and unmodified layers causes the film to bend out of plane. The partial energy stored in the damaged layer by the prior heating transformation is available to deform the martensite on subsequent cooling, which thus causes an uncurling of the film and thereby creates a two-way motion during thermal cycling.

Grummon et al [4] noted the dependence of the TiNi film’s thermomechanical properties (after a 5 MeV Ni$^{2+}$ ion irradiation) with the irradiation fluence, and they also observed an unusual and unpredictable motion in the samples irradiated with a low fluence ($1 \times 10^{13}$ ions/cm$^2$). In a similar study by LaGrange et al [3], they observed monoclinic and BCC nanocrystallites surrounded by an amorphous matrix in this damaged layer with an irradiation temperature of $30^\circ$C. Barbu et al. have performed ion irradiations in electron stopping regimes and concluded that martensite has undergone transformation to amorphous and austenite phase in the tracks[3]. We have explored the electron stopping effects using 350 MeV Au ions. Amorphous and crystalline phase transformations were observed within the tracks and due to the electron excitations by HRTEM, and in addition, BCC TiNi phase nanocrystalline particles were observed, which were surrounded by an amorphous matrix within the track. The deformed $1 \times 10^{12}$ ion/cm$^2$ diffraction spectrum and microstructure was very different as compared to the undeformed $1 \times 10^{12}$ ion/cm$^2$. Thus, there was an effect of the predeformation on irradiation effects, which has not been previously observed. Whether this crystalline structure has undergone a reserve martensitic phase transformation due to irradiation itself or due to the stress surrounding crystal imposed by newly nucleated amorphous material, is still unclear.

References

Radiation-Induced Enhancement of Critical Current Density in YBa$_2$Cu$_3$O$_{7-x}$ Superconductors

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The application of high temperature superconductor materials in industrial energy is hindered because of an exponential decrease of the critical current density with increasing temperature up to 77 K and the magnetic field up to 2 Tesla (working field of motors). This paper reviews successful attempts for significant improvement of magnetization and transport critical current densities ($J_c$ and $I_c$) of YBa$_2$Cu$_3$O$_{7-\delta}$ (YBCO) single crystals, ceramics, films and tapes by means of irradiation-induced modification of structure. The irradiation with moderate doses of fast neutrons and charged particles was found to result in such an effect at magnetic fields of 1-5 T because of generation of flux pinning centers. The temperature dependence of $J_c$ became weaker, but the initial $T_c$ and the conductivity decreased because of carrier localization at the irradiation induced defects. The irradiation also caused the decrease of initial anisotropy of structure and $J_c$. Most researchers ascribe the strongest pinners to linear and columnar defects, and weaker ones to point defects. Most data are related to improvement of $J_c$ in bulk – intra-granular magnetization critical current, which is much higher than the real inter-granular transport critical current $I_c$ limited by weak contacts. However, the effects of granularity and anisotropy were also mentioned. The problem is to form the particular texture ensuring enhancement of the inter-granular contacts by minimizing both off-plane and in-plane grain misorientation during synthesis. Much less studied is the effect of irradiation on the inter-granular critical current $I_c$ and conductivity. Several authors found some increase of current carrying ability of YBCO ceramics, crystals and films after X-ray, gamma-ray, oxygen plasma, electron and proton irradiations. It would be attractive for industry application because of no induced radioactivity. Magnetization in strong fields at 10 K proved to depend on granularity, and that at 77 K - on the texture degree of YBCO ceramics sintered in gamma-field. The transport $I_c$ at 77 K in self-field increased with the texture degree growth. Highly ionizing 18 MeV proton irradiation at 300 K caused anisotropic stress and texturing of YBCO ceramics, both depending on the dose rate, and the conductivity along the exposed surface increased.

The second generation of HTSC tapes are based of less anisotropic coated YBCO thin film which has much higher $J_c$ ~1 MA/cm$^2$ at 77 K at several Tesla, though its disadvantage is improper texture. The latest experiment of the authors on 9 MeV proton irradiation of the films at elevated temperatures (150-300 C) demonstrated a noticeable increase of the transport $I_c$ at 77 K and a significant increase of the normal state conductivity, while the initial value of $T_c$ decreased just by 2-3 K.

**Monitoring an Insulator-Metal Transition in Icosahedral AlPdRe by Neutron Irradiation**


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Icosahedral i-AlPdRe samples can be made with low-temperature resistivity $\rho$ and resistance ratios $R = \rho(4.2 \text{ K})/\rho(295 \text{ K})$ varying over wide ranges of values. Already early studies of high-$\rho$ samples with $R \sim 100$ suggested insulating properties. It is only recently, however, that more direct evidence of a metal-insulator transition (MIT) in i-AlPdRe has been found. The magnetoresistance (MR) at large $R$ was found [1] to obey variable-range hopping (VRH) theory for electron transport in insulators. From the metallic side, MIT is suggested both from analyses of the metallic MR at small $R$ [2] and from a scaling approach to the conductivity $\sigma(T)$ of metallic samples at temperatures $T > 400 \text{ mK}$. These different investigations all give estimates that an MIT occurs in the region $R \sim 20–30$. However, the most perspicuous results on an MIT are normally expected for low-temperature $\sigma(T)$, but such results for i-AlPdRe have remained controversial. Descriptions in terms of VRH theories have given widely different results. A major difficulty is the saturation of $\sigma(T)$ observed at very low temperatures ($\leq 20 \text{ mK}$ in high-$R$ samples) and incompatible with VRH. Recent results indicate that a finite $\sigma(0 \text{ K})$ is a property of the icosahedral phase, which may suggest that at large $R$ and low $T$ two channels contribute to $\sigma(T)$, e.g., VRH and quantum tunneling. We therefore started with an insulating sample and irradiated it by fast neutrons at $T_{\text{irr}} = (330 \pm 10) \text{ K}$ with a series of doses $D$ up to $7 \times 10^{18} \text{ cm}^{-2}$. Samples had nominal composition $\text{Al}_{70.5}\text{Pd}_{21}\text{Re}_{8.5}$ were prepared by melting in an arc furnace followed by annealing at 940 and 600–650 °C. Details of these preparation techniques and structural characterizations have been given previously [2]. Using a single sample focuses on the effect of varying intrinsic disorder. For increasing $D$ the icosahedral phase was preserved, peak x-ray intensities (volume of the coherent icosahedral phase) and $R$ decreased, and an insulator-metal transition was traversed. The MR vs $R$ across the MIT was found to be independent of sample history.

References


Martensitic Transformations and Shape Memory Effect in TiNi Alloys in the Process of Neutron Irradiation in Low-Temperature Helium Loop

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The investigations of martensitic transformations, shape memory effect and peculiarities of kinetics of the radiation damage of equiatomic TiNi alloys in the process of irradiation in the low-temperature helium loop of the WWR-M nuclear reactor at the temperatures of 120-350 K have been performed. The method of resistivity measurement has been used, which is sensitive
both in respects to radiation defects of the crystal lattice, and in respect to martensitic phase transitions in crystals. The shape memory effect has been investigated by means of a spring sample loaded by an elastic element. The deformation has been measured by a variable-induction pickup.

It has been established that the resistivity of the TiNi alloy grows proportional to the fast neutron fluence. The resistivity growth rate in the martensitic state is by 3-4 times as much as in the austenitic state. Such behaviour has been caused by different contribution of radiation defects into the resistivity and by fragmentation of the polydomain martensite structure under irradiation. The critical temperatures of martensite transitions in TiNi alloys decrease with the fluence growth according to the following law: \[ \Delta T_{ph} = d \exp\left(\frac{-\Phi}{\Phi_0}\right) - 1 \] where \( \Delta T_{ph} \) is the increment of the temperature corresponding to the structural transformation; \( \Phi \) is the fluence; \( d \) and \( \Phi_0 \) are constants. The temperature hysteresis of the transformation rises. These regularities can be detected under irradiation both in the martensitic state and in the austenitic state. A short-term temperature rise for the samples irradiated by the fluence of \( 7 \times 10^{18} \text{ cm}^2 \), up to temperature of 450 K leads to the return of the resistivity and the temperature kinetics of martensitic transformations. The activation energy of the property return has been determined.

It is assumed that the change of transformation kinetics under irradiation and subsequent annealing are connected with the change of the degree of long-range order \( S \) of the ordered solid solution. The evolution of the long-range order degree is proposed to describe by the equation:
\[ \frac{dS}{dt} = a(S_{\infty} - S)I + b(S_0 - S)\exp\left(-\frac{U}{kT}\right) \] where \( t \) is the time, \( T \) is the temperature, \( S \) and \( S_0 \) is the long-range order parameter at the fluence \( \Phi \to \infty \) and at \( \Phi = 0 \) respectively, \( I \) is the neutron flux, \( U \) is the activation energy of the radiation defect annealing, \( a \) and \( b \) are constants.
The Percolation Model of Radiation Porosity Influence on Austenite Steel Strength

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The model of the influence of radiation porosity characteristics on a strength of austenite steels is presented. It accounts for the mechanical strains induced by pores and other defects and for a probability of a pore alignment along the surface, where conditions of damage are being fulfilled. The model was used to estimate the changes of ultimate strength of the austenite steel ChS–68 (20% c.w.), which had operated as fuel claddings in the BN–600 reactor and accumulated different damage dozes. Ultimate strength values were calculated by using the results of quantitative electron microscopy study and compared to the experimental data obtained from short–term mechanical tests on tension of ring samples. The samples were made of fuel claddings irradiated in the BN–600 reactor.

The main statements of the model are as follows:
- the pores, which are forming under neutron irradiation, can randomly align along some surface, where the damage conditions are being fulfilled;
- vacancy pores induce mechanical strains in the steel due to surface tension and, hence, the external loads needed to fulfill the damage conditions are smaller.

The expression for the strain, $\sigma_B$, produced by the external load and with which the damage proceeds by the above mechanism was obtained

$$\sigma = (\sigma_g - \sigma_v) \cdot (1 - P/P_{th})^{2/3},$$

where $\sigma_g$ is the strain in a matrix induced by pores due to surface tension and which can be described through the characteristics of the pore assembly in the material;

$P$ and $P_{th}$ are porosity and its threshold value, respectively, at which self damage occur, i.e. with no external load applied. The quantitative microstructure investigations of the samples of the fuel claddings made of ChS–68 and irradiated to damage doses in the BN–600 reactor were made by the electron microscopy methods. The ultimate strength was calculated from these results and by formula (1). Then ring samples were cut out of the same areas for short–term mechanical tests. For different ranges of irradiation temperatures the experimental dependencies of ultimate strength values were plotted against swelling. The dependencies were compared to the values calculated by the model. It was found that the manner of the swelling dependence of calculated and experimental values of the ultimate strength were similar, however, the experimental data (corresponding to swelling higher than 8 %) are decreasing with the growing swelling faster than the calculated ones.

It is shown that the observed difference is owing to the additional influence of two factors, i.e. a preferential aligning of radiation pores along twins and a presence of corrosion cracks on the internal side of the fuel elements, the cracks are strain concentrators in the mechanical test performance.
Temperature Dependence of Radiation Swelling of ChS-68 Steel Analyzed by Different Models

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The fuel claddings made of ChS-68 steel (20% c.w.) were irradiated in the BN-600 reactor, swelling of specimens of the claddings was measured and the data obtained were used for examination. The data were divided into two groups with respect to the fuel irradiation in different modifications of the BN-600 core, which differed in a height of the core and a maximum speed of attaining a damage dose. In different groups the composition of the cladding ChS-68 steel differed in the micro alloying dopants: boron and vanadium. Three alternative models were used for a quantitative description of swelling. In the empirical model A swelling $S$ was given by the equation.

$$S=K_1 \cdot G^{K_2} \cdot D^{K_3}$$

where $G$ is the speed of attaining the dose in the units of $10^{-6}$ dpa/s; $D$ is the damage dose; $K_1$ and $K_2$ are the parameters independent on irradiation temperature; $K_3$ is the temperature dependent $T$- function

$$K_3 = a_3 + a_4 \cdot T + a_5 \cdot T^2$$

where $a_3$, $a_4$, and $a_5$ are constant coefficients.

In the model B equation (1) was used, where $K_1$ and $K_2$ were the parameters independent on irradiation temperature and $K_3$ was given by equation

$$K_1 = \exp(a_3 + a_4 \cdot T + a_5 \cdot T^2)$$

Model C describes swelling as

$$S = A \cdot (D - D_0) \cdot (1 - \exp(-p \cdot (D - D_0)))$$

where $A$ - is the speed of swelling at a stationary stage; $D_0$ - is the incubation swelling dose; $p$ - is the coefficient characterizing the speed with which the condition of stationary swelling is attained.

The parameters in the equations of radiation swelling dependence on irradiation conditions were determined by an optimization with the criteria $\chi^2$. In order to estimate an adequacy of the descriptions by the above models the obtained values of $\chi^2$ were compared to the reference value $\chi^2_{0.05}$, which usually corresponded to the applied level of significance of 0.05. The results obtained by the three models were compared. The data obtained was used to plot a temperature dependence of swelling against different levels of damage doses. The dependence of peak swelling temperature on accumulated damage dose and a possible existence of two local temperature peak of swelling were analyzed.
About the Surface Erosion Mechanisms under the Pulsed Ion Beams Irradiation

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Irradiation of solids by high intensity submicrosecond ion beams is accompanied by active surface erosion. It was developed yet at the first stages of the investigation of their interaction with matter that every pulse is able to remove from surface the layer of near micron thickness. Thus, the erosion coefficient can reach up to $5 \times 10^5$ atoms/ion. It is near five orders more than under continuous irradiation. The estimation of partial contributions of atoms that are sputtered by quick particles and evaporated as a result of surface heating is one of unexplored questions. The first group of ions landed the surface interacts with cold solid and sputters it. Then the irradiating surface is heated quickly (with the rate up to $10^{11}$ K/s) up to and above the boiling temperature. The intensive evaporation is able to arise on such surface.

Therefore we divided the stream of atoms leaving the surface to two parts: quick (concerned with collision processes) and heat (caused by evaporation). Accordingly, total erosion coefficient $D$ is equal to the sum of two coefficients: sputtering ($S$) and evaporation ($Q$) ones ($D=S+Q$).

The aim of present work was to study the features of irradiated surface erosion under the power pulsed ion beam, in particular, the calculation of erosion coefficient $D$ taking into account the surface temperature change and phase state, as well as the calculation of the partial components $S$ and $Q$.

As a result of this work the following conclusion was formulated. In the range of power density of pulsed ion beams up to $(1..5) \times 10^6$ W/cm$^2$ sputtering is the main mechanism of erosion. In this case the erosion coefficient does not exceed several tens. It the range of $(0,5..1) \times 10^6$ W/cm$^2$ and more the erosion is concerned mainly with atoms evaporation as a result of surface heating by beam particles up to boiling temperature and above. In this case the erosion coefficient achieves $10^4..10^5$ atoms/ion (for $E \leq 2$ MeV). The intermediate range of power density ($\sim 5 \times 10^6 .. 5 \times 10^7$ Vt/cm$^2$) is not yet well studied. We expect that the contribution from sputtering and evaporation occurred on activation mechanism can be comparable. But the total erosion coefficient is below in orders there than under the intensive evaporation at boiling point.
Formation of Nanoscale Intermetallic Phases at High-Intensity Ion Implantation


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The high intensity ion implantation of metal ions using the vacuum-arc ion-beam and plasma flow source “Raduga-5” uncloses wide perspectives for modification of constructional metal materials. Major advantages of the source “Raduga-5” are the high dose rate of the implanted metal ions, plasma-assisted deposition, heating-up of a target by ion beam up to necessary temperature and opportunity of forming the modified ion beam surface layers, the thickness of which equal to several micrometers. The vacuum - arc ion-beam and plasma flow source “Raduga-5” can be applied for formation of nanoscale intermetallics phases in the surface layer of the micrometer thickness with the purpose to increase the wear resistance and heat resistance of constructional metal materials.

The aim of the paper was the investigation of an opportunity of the formation surface layer, thickness of which exceeds the ion projected range on some orders, with the nanoscale intermetallic phases by the method of the high intensity implantation of the Al ions in polycrystalline nickel and titanium.

It was established that the fine dispersed intermetallic precipitates are arisen in the nickel and titanium surface alloyed layers. Compositions of these intermetallic precipitates are closed to NiAl, Ni3Al Ti3Al and TiAl phases which are characterized by a set of unique properties as high melting temperature, high heat resistance and wear resistance. Besides the solid solutions of aluminum in nickel and titanium are formed. The intermetallic phases on the sizes of grains 30-100 nm are the nanoscale phases.

The increase of the target temperature both by the increase of the ion flux density and the ion treatment duration allows us to increase the modified surface layer thickness from one micrometer up to several micrometers. One should expect that such surface layers with the high density of the nanoscale intermetallic phases would have not only the high temperature strength but also the high wear resistance.

Vacancy Clusters in Titanium Nickelide Irradiated with Fast Neutrons

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Amorphous alloys of titanium nickelide of 2 types (one was prepared by affecting with fast neutrons, second was obtained from a melt) investigated for homogeneity by the methods of small angle scattering, dilatometry and electronic microscopy.
It was found that the effect of small angle scattering takes place only in the titanium nickelide amorphized with fast neutrons. Then small angle neutron scattering was measured in the irradiated alloys. The measurement results were processed with the assumption that microareas rich in vacancies are formed in titanium nickelide under irradiation.

The observed experimental situation can be explained admitting that the scattering object is a cluster, which is a three – dimensional fractal object in mathematics. In the above case the fractal cluster is understood as the microarea, which is rich in vacancies, has a rough surface and numerous thin barriers.

The cross sections of the neutrons scattered with the vacancy clusters were calculated, the calculation data in a log–log scale are shown as solid lines in Figs. 1, 2. The full profile of the calculated curve in a regular scale is in the figure insertion.

As based on the combined analysis of the data, obtained by the three methods, the inhomogeneity of the titanium nickelide (amorphized with fast neutrons) was attributed to the formation of the microareas with a high concentration of vacancies under irradiation at 340K. The sizes and the volume fraction of the clusters rich-in vacancies were also determined.
Swelling and Microstructure of EP-753 and Nimonic PE-16 High Nickel Alloys after Neutron Irradiation to High Doses

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The temperature dependence of swelling in the majority of pure metals and alloys looks described by a bell-shaped curve, the position of maximum on which is determined by both the type of a material and irradiation conditions. However, data are available on a more complicated temperature dependence of swelling, when the swelling temperature curve has two and even three maxima. Several swelling maxima were observed in austenitic steels, nickel alloys, and also in some pure metals irradiated with neutrons, ions and fast electrons.

For the present, mechanisms responsible for occurrence of several swelling temperature peaks are not established exactly that is presumably due to a lack of experimental data on the microstructure of materials demonstrating several swelling maxima.

In this report data on the swelling temperature dependence and microstructure for two high nickel alloys EP-753 (1X20H40M5B) and Nimonic PE-16 (6X17H44M4IOT) irradiated with neutrons up to maximum dose of 44 dpa are presented. The alloys were irradiated as pin cladding structural in the BOR-60 fast reactor (Dimitrovgrad) in the temperature range of 320-600°C. Data on pin diameter measurements and TEM-data give evidence about the existence of two swelling maxima located at temperatures of 390 and 590°C in the EP-753 alloy and at temperatures of 420 and 585°C in the Nimonic PE-16 alloy. The appearance of the second, high-temperature maximum is, probably, a result of formation and growth of voids with elevated helium content due to accumulation of helium in nuclear reactions and its migration to the voids at temperatures above 500°C.

Long-Range Effect in Ion Implantation-Ordered Alloys

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The long-range effects, i.e. the possible phase transformation in the material and the formation of structural defects at depth which the implanted ions did not reach, were observed in the some cases.

The radiation doses for the Cu3Au and PdCuAg alloys were 10¹⁵-10¹⁸ argon ion/cm², j=100-300 μA/cm², E=20-40 keV.

A region of the ordered materials lying at a depth of more than 0.5 μm from the irradiation surface were examined in detail.
It was revealed that implantation leads to the formation of a large number of defects identified in the ion micrograph as a regularity interruption in the annular pattern of the superstructural low-index poles.

A Short Range Order In The Amorphous Ribbon of Ti$_{0.5}$Ni$_{0.25}$Cu$_{0.25}$ Irradiated by Fast Neutrons

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It was shown by the authors in Refs [1,2] that the type of a short atomic order in intermetallics and compounds, amorphized under fast neutron irradiation, is relevant to the type of an initial crystal lattice. It was especially noted, that the type of the short range order of the amorphous substances, prepared by quenching a melt, was relevant to a random close pack of atoms and its main features were independent of an atomic content.

In order to investigate an effect of neutron irradiation on the structural state of the amorphous alloy prepared by the fast quenching of a melt the amorphous ribbon of Ti$_{0.5}$ Ni$_{0.25}$ Cu$_{0.25}$ was exposed to fast neutrons, $\Phi=1 \times 10^{20}$ cm$^{-2}$, at 340 K. The packet was investigated by the neutron diffraction method before and after fast neutron irradiation. The obtained diffraction patterns are shown in the Figure. They revealed some portion of a crystal phase in the unirradiated sample. In the pattern the crystal phase portion is absent because the crystal phase has been amorphized by irradiation. The type of a short range atomic order determines a diffusion peak at $K$~$2\AA^{-1}$. Since the form of the peak has not changed it may be concluded that the fast neutron irradiation has no effect on the type of the short range order in the alloys amorphized by quenching a melt.

References
Structural Phase Transitions in Intermetallic Compounds \( \text{Y}_2\text{Fe}_{17} \) and \( \text{Ce}_2\text{Fe}_{17} \) after Irradiation with Fast Neutrons

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In earlier papers [1-3] we showed that change of magnetic properties of intermetallic compounds \( \text{R}_2\text{Fe}_{17} \) (\( \text{R} = \text{Y}, \text{Ce}, \text{Lu} \)) at their doping with transition elements is caused by lattice transformation. A dependence of the Curie temperature on interatomic distance was found. The main difficulty in the experimental study of doped systems is that the process changes the sample composition, and hence, the set of parameters depending on composition, such as: effective ion valence, ion sizes, their quantity, etc. The radiation exposure method permits to avoid the majority of these difficulties, since the irradiated sample preserves its original stoichiometry. As experiment shows, the cell size and the ions local environment in the lattice usually vary at irradiation with fast neutrons. Such distortions must affect the magnetic properties of the irradiated materials as well. Really, at irradiation with fast neutrons, antiferromagnetic ordering in \( \text{Ce}_2\text{Fe}_{17} \) is suppressed, and the compound turns into a ferromagnet with \( T_C = 300 \) K, and in \( \text{Y}_2\text{Fe}_{17} \) the Curie temperature grows significantly. The purpose of this work was investigating the crystalline structure of compounds \( \text{Ce}_2\text{Fe}_{17} \) and \( \text{Y}_2\text{Fe}_{17} \) after irradiation with fast neutrons with an energy over 0.1 MeV, and finding the structure – magnetic properties correlations. Irradiation was carried out in the vertical irradiation channel of reactor IVV-2M (Zarechny) at 370 K in a broad range of fluences up to \( \Phi = 3 \times 10^{20} \) cm\(^{-2} \). Quite unexpected was the Curie temperature drop from 300 to 170 K at fast neutrons fluence increase to \( \Phi = 3 \times 10^{20} \) cm\(^{-2} \). Crystalline structure was investigated using X-ray and neutron diffraction methods at room temperature. It was established that change in the symmetry of crystal lattices took place under intermediate fluence, while under maximum fluence, amorphous state was realized in the bulk of the material.

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References
Comparison of the Effects of Radiation- and Strain-Induced Defects on the Phase Transformation in Iron Alloys

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It was analyzed on the comparative basis [1-4] how point defects of different origins (radiation- and strain-induced defects) influenced the progress of anomalous phase transformations in steels and alloys at low temperatures (200-400 K). It was shown that atomic decomposition of Fe-Ni, Fe-Cr-Ni, Fe-Ni-Ti, and Fe-Cr-Ni-Ti solid solutions took place irrespective of the origin of point defects, which were formed both during a severe cold deformation and under irradiation with high-energy particles. A relatively stable ultrafine-grain structure with grains ~100 nm in size appeared during a severe cold deformation. Boundaries of those fine grains were preserved during a strong deformation and they served as efficient sinks of point defects. As in the case of irradiation, the reverse Kirkendale effect led to enrichment of the boundaries with nickel, which was attested to by a large rise of the Curie temperature in the near-boundary regions in the Fe-Cr-Ni austenite. Profiles of the near-boundary segregation of nickel in the Cr12Ni30 alloy caused by diffusion of strain-induced vacancies to sinks were calculated in [1].

Low-temperature dissolution of strengthening particles (intermetallics, etc.) in the austenitic matrix during an intensive cold deformation was discussed in [2]. This is analogous to a certain extent to dissolution of fine particles in displacement cascades under low-temperature irradiation [3]. It should be noted that the deformation effect differs from irradiation since not only defects, but also dislocations are formed in the former case. Dislocations complicate considerably the character of structural and phase transformations. Calculation [4] demonstrated that dissolution of fine particles of secondary phases under cold deformation (200-300 K) can be explained by strain-induced transfer of atoms of the particles to interstices and subsequent migration of the interstices to the stress field of moving dislocations.

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References
The Long-Range Effect in Metal Foils under Ion and Photon Irradiation


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The review of the original studies on the long-range effects in metals under various type (ions, photons and so on) irradiations is presented. The essence of the effect is the change in the material properties over distances by several orders of magnitude exceeding the thickness of an ultimate energy deposition region. Unlike the other phenomena, where the change can be explained as due to the strong heating of the region, such heating does not play a significant role in our case. Moreover, the phenomenon considered (low-dose long range effect - LDLRE) differs from the other “long-range effects” investigated by various groups under ion irradiation: the changes take place at significantly lower doses and at the doses exceeding some critical one the effect becomes less pronounced.

LDLRE is characterized by the following anomalous features: the non-monotonous dose dependence, the existence of the threshold ion energy (independent on the ion type, dose, and dose rate), the ability to penetrate through the interfaces between the foils and to redistribute the alloy species and impurities at super-deep distances, etc.

Recently, anomalously deep change in the metal mechanical properties was found at certain conditions also under light (photon) irradiation of metal foils having several μm (and higher) thicknesses. The effect regularities, their similarity and difference from the LDLRE were investigated. It has been demonstrated that the necessary condition of the phenomenon manifestation is the existence of thin dielectric layer (natural oxide) on the irradiated surface of the foil but not the heating. The phenomenon was named “photo-memory of metals – PMM”.

The PMM is characterized by the complicated non-monotonous dependence of mechanical properties (microhardness) on the dose, by a relaxation in the changes after stopping irradiation and by a number of unusual specific features. The possible mechanisms of all mentioned phenomena are discussed from the single point of view.

The new encompassing concept is suggested concerning the interaction of energy flows with solids having a strongly non-equilibrium structure. The problems concerning the non-observation in more early experiments of the considered effects and their application in practical purpose are discussed.
III. Implanted and Transmutational Gaseous Impurities Behavior in Irradiated Metals and Alloys
Determination of Hydrogen Permeation Reduction Factor (PRF) for Different Protective Coats on Vanadium

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Selection of the structural material for liquid-metal system as for another system and constructions of nuclear energy plant must be carried out and based on appointed request depending on conditions of functioning this materials.

Their compatibility with liquid metals is a specific request. The pile creation with liquid-metal heat-transfer (Li, PbLi) reproducing tritium requests additional clause to structural materials. This creation of structural material or protective barrier with minimum acceptable value of tritium permeation through them or with maximum permeation reduction factor- PRF.

Vanadium and vanadium alloy planed to use as a blanket structural material like nuclear energy plants.

Workable on a first stage vanadium must have stability characteristics at temperature 800°C hydrogen influence.

Given work shows experiments results by testing protective coats on vanadium- glass-ceramic coating which based on CaO. Permeation reduction factor (PRF) for every coating and its changing depending on thermocapacity of vanadium sample with coating was determinate by method of hydrogen permeation.

The experiments results will be used at the development of pile protection outlines with liquid-metal heat-transfer.

Gas Bubble Evolution Peculiarities in the Ferritic and Austenitic Steels and Alloys under Helium Irradiation


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Austenitic and ferritic-martensitic steels of Cr13 type are considered as candidate structural materials for the fusion reactors first wall. A significant quantity of helium will be accumulated in the structural materials via \((n, \alpha)\) or other transmutation reactions, as well as it may be implanted from plasma through the first wall surface. Helium can have a pronounced effect on the radiation damage of materials and often may be an important reason in catastrophic degradation of their properties and shortening of the useful life of reactor constructional elements. In this connection considerable attention has been given to the helium problem in fission and fusion materials. One of these problems is the role of helium in enhancement of radiation swelling.
The aim of this paper is to investigate the peculiarities of gas swelling at high helium concentration and to compare the gaseous swelling level for materials with ferritic and austenitic structures.

By means of transmission electron microscope the gas bubble development in model alloys of Fe-C system, ferritic-martensitic steels of Cr13 type, nickel and austenitic steels has been investigated after 40-keV helium-ion irradiation up to a fluence of $5 \times 10^{20}$ m$^{-2}$ at the temperature of 650°C.

It was shown that helium-ion irradiation at high temperature in Fe and ferritic-martensitic steels Cr12MoWSiNbVCeB and Cr12MoWSiNbVCeNB resulted in formation of bubbles with a greater size and a smaller density, than those in nickel and austenitic steel Cr16Ni15Mo2Mn2TiV. Thus the gas swelling of ferritic-martensitic steels may be high and over the gas swelling for some austenitic steels. In Fe-C alloys as well as in ferritic-martensitic steels, large gaseous bubbles in ferritic component are uniformly distributed in grains body. The bubbles with higher density and smaller size than those in ferritic component are formed in martensitic grains of steels and Fe-C alloys with a high carbon content ($N_C > 0.01\%$), which leads to a small level of swelling of martensitic in comparison with ferritic. In addition, the bubbles in martensitic grains have a tendency to ordered distribution.

The standard heat-treatment (quench + temper) of ferritic-martensitic steel results in an increase of gas swelling that significantly exceeds the swelling of martensitic grains. In this, the swelling of ferritic grains is several times higher than that of martensitic grains.

**Hydrogen Accumulation and Retention in Materials of Reactor VVER-1000 Vessel Components**

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**Investigation of Concentration and Temperature Dependences of Processes of Hydrogen and Helium Accumulation and Desorption in Steel 08Cr18Ni10Ti**

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**Hydrogen Thermodesorption from Helium Ions-Implanted Steel 08Cr18Ni10Ti**

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Influence of Interstitial and Substitution Elements on Helium and Hydrogen Behavior in FCC and BCC Metals

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The main experimental results obtained by authors during the last 20 years on ion-implanted helium and hydrogen behavior and microstructure evolution under different irradiation conditions and post-irradiation treatment of structural steels and alloys, as well as of model alloys of Ni-C, Ni-$X$ ($X = \text{Be, Si, Mo, W, Al, Ti, Ta, Sn, Zr}$), Fe-C, V-Ti systems, are presented.

It was shown that the helium thermodesorption (TDS) peaks are displaced to higher temperatures and the effective activation energy for helium desorption grows with increasing carbon concentration in metals by changing of bubbles migration mechanism from surface diffusion in pure metals to volume diffusion in alloys because of carbon segregation in the bubbles near-surface volume. Influence of carbon on deuterium behavior is similar. During post-irradiation annealings small-sized and high density bubbles are formed in the alloys of Fe-C system than that in Ni-C alloys owing to higher binding energy of helium-vacancy complexes in iron. In contrast, after high-temperature (650 °C) implantation by helium large bubbles with small density are formed in iron and ferritic-martensitic steels of Cr13 type than bubbles forming in nickel and austenitic steels.

Decrease of matrix atom self-diffusion coefficient in Ni-Al alloys can result in a decrease in bubble migration rate and shift of TDS peaks to the higher temperatures with increasing Al concentration. To the contrary, in the V-Ti alloys the TDS peaks are displaced to lower temperatures with increasing Ti content because of increase of vanadium atom self-diffusion coefficient by titanium doping. However in both systems of alloys the effective activation energy for helium desorption grows with alloying element concentration due to the change of bubble migration mechanism from surface diffusion (for pure metals) to an increase of the role of volume diffusion (for alloys) because the volume diffusion coefficient is greater than that for surface diffusion.

It is found that the most thermally stable complexes of the He$_m$Me$_k$V$_n$ type (where He and Me are helium and alloying element atoms, V is a vacancy) are formed along with simple complexes of He$_m$V$_n$ type in the case of positively discrepancy between matrix atoms and atoms of alloying elements in the substitution alloys. As the result, small bubbles and high density are formed in the alloys during post-implantation annealing.

The obtained regularities of gas swelling for Ni-$X$ alloys under helium-ion irradiation are in a good correlation with data for alloying influence on swelling by neutron or heavy ion irradiation, namely, the swelling has maximum if the number of bonding $d$ electrons in the alloy is equal to three.
Investigation of Tritium Permeation Process Produced in Lead-Lithium Eutectic Pb83Li17 Through Tube Steel Sample F82H- in Reactor Irradiation Conditions

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The problem of hydrogen permeation structural materials (KM) of fusion reactor arise and exist as a result of using tritium in future fusing reactors, which has diffusion leakage and as calculations show, they are great and make line of constructional hardship and will request great expense as for defense personnel and environment as for cleaning a heat-transfer from tritium.

Lithium-lead eutectic (Li\textsubscript{17}Pb\textsubscript{83} further denote as Li-Pb) is one of more useful material of heat-transfer as applied to shell of tritium breeder due to its high-level of tritium reproduction, low reactivity and workability as a heat-transfer. It is very important to study the process of tritium ejection by breeder material and process of leakage through structural material, surrounding alloy.

Low-activity alloy F82H suppose to use as a main structural material of system by accumulation tritium of the future fusion plants.

In this work were produced the experiments by study of permeation tritium process through F82H steel from PbLi eutectic at reactor irradiation.

The experiments were made by hydrogen permeation method on IVG1.M NNC RK reactor. Sample of F82H steel was produced by Japan institute of nuclear energy. The tritium flow dependence through sample were got during reactor irradiation. By them were made model, which describe the process of permeation tritium from lead-lithium eutectic.

Investigation of Helium-3 Buildup Operation in Structural Materials by “Tritium Trick” Method

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The investigation of the effects of dissolved helium-3 and hydrogen isotopes on physical and mechanical properties of metals is defined as one of the fundamental lines in metal physics and has practical importance.

In RFNC-VNIIEF, the accelerated buildup of the helium-3 concentrations of \(~100-500\) appm (\(~8-40\) \(\text{cm}^3\text{He}/100 \text{ g Me}\)) was performed by “tritium trick” through the exposure the samples of the investigated material in gaseous tritium at higher temperature and pressure. For the future radiation safety research of the effects of dissolved helium-3 on material structure, mechanical properties and hydrogen permeability the samples should be detritiated.

42
The setup of the detritiation was implemented and the method of the samples detritiation was selected.

The tritium and helium-3 tests in the structural materials were carried out by the setup developed on basis of the gas-analyzer “ELTRA”. The experimental results of helium-3 test were in reasonable agreement with the calculated helium-3 concentrations. Dependence of the tritium desorption rate on the detritiation time was obtained. It was found that the process of the detritiation has some stages.

Hydrogen Permeation Through Thin Coatings

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The main idea of this research is based on the fact that high activation energy of adsorption should be an effective barrier for hydrogen transport in the case when gaseous molecular hydrogen is in temperature equilibrium with a membrane. If in addition to sufficiently high adsorption barrier (several eV), hydrogen dissolution in the bulk of material is hindered, this material can be used as a thin lightweight coating, which will be as effective as a massive fusion claddings of materials having activation energy of permeation of about a fraction of eV.

The necessary properties, which are governed by a low density of states on Fermi level and low concentration of free carriers, can have some composite materials or nonmetal materials - semiconductors and insulators. In contrast to well-studied metals, hydrogen interaction with these materials has not been adequately investigated. The complex of techniques, which includes the method of analysis of permeation isotherms and concentration pulses method in permeation mode, has been created in our laboratory. These techniques allow to obtain information about hydrogen interaction with thin films from the data obtained from the studying of two-layer systems: thin film on the permeable substrate.

Nickel oxide, amorphous silicon and titanium nitride were chosen as the objects for investigation.

Nickel oxide does not adsorb hydrogen at all and hydrogen permeation occurs via diffusion along the grain boundaries. The rate of diffusion is independent on temperature and the obtained value of preexponential factor of about $1 \times 10^{-10} \text{ cm}^2/\text{s}$ is characteristic of a diffusion process along grain boundaries. Correspondingly, nickel oxide does not practically dissolve and accumulate hydrogen.

Amorphous silicon. Silicon although slowly and with great activation energy does adsorb hydrogen, which dissociates into atoms on the surface. Hydrogen migration through the bulk is also very slow. Preexponential factor of diffusion coefficient (“released” from the effects of the trapping and detraping rates) is as low as $10^{-8} \text{ cm}^2/\text{s}$, though the activation energy has a rather common value. The peculiarities of hydrogen transport in amorphous silicon could be accounted for on the assumption that hydrogen transport is accompanied with reconstruction of local bonding.
Titanium nitride. There are two principally different mechanisms of hydrogen permeation through TiN in different temperature regions. The first one is highly activated permeation at high temperature. The second one is characterized with a common for permeation process activation energy of about 0.6 eV and occurs at lower temperatures. Kinetics of the steady state flux onset for the coated membrane appears to be very slow and can not be described in the framework of an ordinary diffusion. The observed temperature dependence of permeability is characteristic of the materials having two parallel channels of hydrogen transport with strongly different activation energies.

It has been established that the high temperature part of permeation curve presents the true permeation through TiN crystallites and the rate of hydrogen adsorption on TiN surface has a marked influence on the rate of permeation under conditions of our tests. It is most likely that permeation at low temperature is due to molecular diffusion along grain boundaries.

Development of Helium Bubbles near Grain Boundaries in Nickel-Carbon Alloys

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The peculiarities of development of helium bubbles near grain boundaries in nickel with different carbon content in the process of post-irradiation isothermal annealing are studied in the present paper. The samples of nickel with carbon content of 0.002, 0.007, 0.010, 0.039 and 0.065 % have been uniformly doped with helium up to $2 \cdot 10^{-2}$ at.% concentration and annealed in vacuum at 800°C for 1, 5, 10 and 50 hours. The development of helium bubbles in near-boundary zone has been investigated by means of optical and electron microscopy (TEM).

It is stated that at this annealing temperature intensive nucleation and growth of bubbles are observed in near-boundary region whose width grows both with the increase of annealing time and carbon content. Anomalous restraining of the development nearby boundary zone with helium bubbles in nickel samples with 0.007% carbon has been noted.

The TEM investigations have shown that near-boundary zone the process of bubble growth is non-uniform: bubble size increases and their density decreases as the distance from grain boundary is increased. The effect observed is discussed from the point of view of formation of two zones with different level of swelling in a grain (nearby boundary and matrix) and, consequently, hydrostatic stress as well conditioning the flux of vacancies and helium-vacancy complexes from matrix to grain boundary.
Radiation Microstructure, Helium, Hydrogen in Reactor VVER-1000 Vessel Structure Elements

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A computational method was used to analyze the sources of transmutational helium and hydrogen in stainless steel Cr18Ni10Ti in the reactor core (reflection shield) and between the reactor core and the pit. The dynamics of H, He generation and cascade vacancy clusters formation in a standard neutron flux from the source material atoms is described with a system of connected differential equations accounting for the rates of their formation and disappearance, or transformation to other structure elements.

References

Formation and Growth of Hydrides in Irradiated Zirconium Alloys

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Zirconium-niobium fuel cladding tubes pick up about 50-100 ppm H during their service in VVER and RBMK reactors [1]. This hydrogen is quite mobile, it accumulates in the regions with high tensile stress and low temperature. When hydrogen content exceeds the terminal solid solubility on such parts, the local brittle hydride zones form in material. It creates the danger of delayed hydride cracking (DHC) in the acting and spent fuel rods [2]. In order to prognosticate the above phenomenon the calculation experiments can be especially useful.

The temperature dependence of DHC in the alloys Zry-2 and E110:

- Irradiated Zry-2, experiment [4]
- Irradiated Zry-2, our calculation
- Unirradiated E110, experiment [5]
- Unirradiated E110, our calculation

\[ \frac{\nu}{T} = \frac{1}{1000} \]

\[ \nu, \text{ m/s} \]

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Our paper has the aim to estimate the possibility and characteristics of DHC in the fuel claddings with the deep (up to 500 µm) nodular and fretting damages. The most reliable database on diffusion and solubility of hydrogen in zirconium materials, new calculation model on DHC and some of the obtained results (for example, see Figure) will be demonstrated and discussed.

Reference

Structural Factors Influence on Hydrogen Extraction from Irradiated Construction Materials

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Reseaching of holding hydrogen isotopes in metals in different heating regimes have been studied deeply recently because of need to find and work out the possible materials for first wall of thermo- nuclear reactor (TNR). Fist of all, it connects to a problem of service safety of TNR and loosing of valuable fuel (tritium)

In the present work the results of the researching of kinetic for hydrogen extraction from the samples of austenitic steal, saturated with hydrogen in reaction with deuterium plasma, and from samples earlier irradiated in water- water reactor (WWR) in temperature of 590 K till the fluence (0.4-0.9)*10^{25} m^{2} (after the irradiation the samples during 5 years were in normal temperature), are presented. We received that, being heated, the samples, irradiated in the reactor, extract a big value of hydrogen in high temperatures. It means that atoms of hydrogen in the irradiated sample were caught by traps (the energy of bounding with the traps is 0.3 eV).

During the researching of the austenitic steal, which had reacted to deuterium plasma, the anomalously big blisters with the thickness of –1 µm were obtained. The blisters contained some hydrocarbons, methane for example. The blisters were found out to be destroyed, if methane pressure (before the temperature of methane dissociation) is higher then the pressure, needed to destroy blisters. If in the temperature of methane dissociation the pressure of methane in the blisters is not enough to destroy them, than the blisters stay safe even in the further heating.
Giving the result of the researching on prior cold deformations and grain sizes on hydrogen holding in construction materials. That is why the researching of hydrogen penetration through the construction materials with the given structure characteristics in the process of bombardment by accelerated hydrogen ions, had place. It was found out that the prior cold deformation as well as decreasing sizes of grains, increase the hydrogen penetration through the studied materials during the hydrogen ions bombardment.

Specific Features of Interaction of Heavy Hydrogen Isotopes and Radiation-Resistant Cr16Ni15M03Ti1 and SS316L Steels

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The knowledge of parameters of interaction between heavy hydrogen isotopes and constructional materials of nuclear and fusion reactors is very important for maintaining their in-service properties at an adequate level and minimizing the contamination of the environment with tritium thanks to a decrease in the tritium penetration through metal walls and development of technologies for removal of tritium from used structures.

The present paper reports results of a study dealing with penetration of deuterium and tritium through samples of Cr15Ni16Mo3Ti1 and SS316L austenitic steels from the gaseous phase. The steady-state flow method was used. Experimental temperature dependences of the permeability, diffusion coefficients, and solubility, and also calculated constants of the corresponding processes have been discussed. It was supposed that titanium caused a deviation of the temperature dependences of diffusion coefficients and solubility of the Cr15Ni16Mo3Ti1 steel from the Arrhenius dependence. This supposition was confirmed to a certain extent by results of experiments with the 12Cr18Ni10Ti steel.

Mechanism by Which Titanium Influences Formation of Radiation-Induced Segregation of Deuterium in Cr16Ni15Mo3(Ti1) Steel

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A radiation-induced segregation (RIS) of hydrogen is formed in hydrogen-containing metals and alloys exposed to ion irradiation. The appearance of RIS is attested to by a high concentration of hydrogen in irradiated zones (as compared to an average concentration in the bulk) and an equal distribution of hydrogen atoms and vacancies in the radiation-damaged region. Our earlier study showed that the deuterium RIS in the Cr16Ni15Mo3Ti1 austenitic steel was unsta-
ble after an anomalous "collective" pattern: when irradiation was stopped, the segregation was enhanced thanks to an inflow of deuterium from the nonirradiated volume of the target.

It was supposed that an anomalous behavior of the segregation could be related to specific features of the composition and (or) the structure of the steel. However, RIS proved to be free of this type of instability in nickel, which is a model material of austenitic (FCC) steels, and BCC Fe-Cr alloys. The goal of the present study was to determine the role of titanium in formation and an anomalous behavior of the deuterium segregation in the Cr16Ni15Mo3Ti1 steel.

The effect of titanium on the microstructure of the Cr16Ni15Mo3(Ti1) steel was examined by the methods of transmission electron, scanning electron, and tunneling microscopy. The nuclear reactions methods combined with concurrent irradiation with deuterons and measurements of the deuterium concentration in the segregation was used to determine the role of titanium in formation of RIS in the steel.

It was found that addition of 1% Ti changed the steel structure and favored an increase in the deuterium segregation. It was shown that the solid-solution titanium initiated formation of hydrogen vacancy traps at room temperature and caused precipitation of particles of titanium-containing compounds (carbides and intermetallics), which facilitated an additional capture of deuterium atoms, during aging. An anomalous collective-type instability of the segregation (after irradiation was stopped) was observed both in the titanium-loaded and titanium-free austenitic steel.

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The In-Pile Experiments on the Reactor IVG.1M

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Given work shows experiments results by study tritium permeation which generated from lithium -lead eutectic, through steel MANET with different covers. The results by tritium and helium evolving from lithium -lead eutectic were shown.

The results of neutron-physical and thermal-physical calculations of canal experimental devices (CED) were shown in detail. The design philosophy (CED) were discussed.

The first experimental results by study tritium steam-out from lithium-titanium ceramic were adduced.
IV. Radiation-Enhanced and Radiation-Stimulated Phenomena
Segregative Manifestation of the “Long-Range Action Effect” During Irradiation of Permalloy Foil with Boron and Phosphorus Ions

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In the given paper, boron and phosphorus are used as examples for demonstrating a new manifestation of the small dose effect of long-range action, which is an anomalous penetration of impurity atoms, implanted into the permalloy foil.

A foil from permalloy-79 with the thickness of 20 μm was irradiated with B⁺ and P⁺ with the dose 10¹⁵ cm⁻². The ion energy varied over the range from 15 to 40 keV. Current density was 3 μA/cm², and the temperature of the samples was not higher than 150 °C. The sides of some samples, adjacent to a flange, were covered with chemically stable varnish (CSV). Distributions of the components from both sides were determined by the methods of X-ray photoelectron spectroscopy (XPS) and secondary-ion mass-spectroscopy (SIMS).

The most interesting result of the work lies in the fact that in the irradiated samples boron and phosphorus are revealed on both sides of the foil. It also is important that boron is present on the back surface in both uncoated samples and CSV-coated samples, though for latter ones the penetration of impurities from gas phase is very unlikely. Besides, the ratio between the back side boron concentration and the irradiated side boron concentration grows sharply at E > 30 keV, i.e., the energy threshold appears, which agrees with the data on the microhardness changes. From the results on boron profiles, it follows that boron is predominantly concentrated in thin near-surface layers (to 8 nm). In addition to the boron penetration through the foil, the “effect of long-range action” is also manifested in the influence of irradiation on the distribution of the main component, nickel, in the near-surface layers on the back side, where the energy threshold for the main component redistribution is observed as well.

The study of the small dose effect of long-range action has been conducted with the use of permalloy foils; when irradiated with phosphorous ions, a part of a sample has been shielded from the ion flux. The measurements show, that at the ion energy of 40 keV, similar processes of the redistribution of the main components of the alloy take place on both the front and the back sides of the foil in the area of irradiation.

The results obtained should be considered as preliminary ones. It is necessary to conduct further verification of these results for some other impurities and metals. The features of the phenomenon should be studied more thoroughly, especially the role of the conditions of irradiation and hold of samples after irradiation.

The work has been financed by the Russian Foundation for Basic Research (project № 02-0216670).
By means of resistivity the processes occurring under electron irradiation at 300-420 K are investigated in the undoped and impurity (Si and Cu) doped Fe-16Cr-based alloys. Two processes with clearly different contributions to residual resistivity were found to proceed in the Fe-16Cr alloy under irradiation. The first one resulting in a resistivity drop is a well-known radiation-enhanced short-range ordering (SRO). The second process leads to resistivity increase. The similar, but a more pronounced effect was found in the Fe-22Cr alloy. Thus the most probable origin of the resistivity increase is the alloy decomposition under irradiation into $\alpha$ (Cr depleted) and $\alpha'$ (Cr enriched) phases well known due to the so-called 475 °C fragility. The compatibility of such explanation with variations of the other alloy properties, sensitive to Cr concentration, had been tested and found to be satisfactory:

1. **Resistivity.** The concentration dependence of the Fe-Cr alloys resistivities at 4.2 K (residual) and 373 K was determined. Since the alloy 4.2 K resistivity was found to be almost independent on Cr concentration over a wide concentration range (9-50 at. %), the 4.2 K resistivity rise can be easily understood as due to conduction electron scattering at elastic distortions around $\alpha'$ precipitates, whereas the matrix resistivity stays nearly unchanged. The variation of the 373 K resistivity due to irradiation agrees with the solid solution depletion with Cr.

2. **Stage I of resistivity recovery.** The amplitude of stage I resistivity recovery (after low temperature irradiation) depends on the Cr concentration. It is shown that this amplitude in the Fe-16Cr alloy irradiated at 420 K increases and in our particular case is close to that in the Fe-13.4 Cr alloy.

The simple decrease in irradiation temperature from 420 K to 300 K also decreases the rate of both processes pointing to the radiation enhancement nature of the latter. But sequential alternation of irradiation temperature between 300 K and 420 K leads to abrupt increase in resistivity rise rate at 420 K. This means that the rate of the alloy decomposition is not determined only by a non-equilibrium defect concentration (that is typical for the radiation enhanced process) but also can be affected by reactions between defects such as clustering and that is the sign of the radiation-induced process.

The same processes taking place under irradiation are observed in the Fe-16Cr alloys doped with Si (0.2-1.5 at. %), although the rate of resistivity variations associated with the SRO in these alloys is strongly decreasing with the dopant concentration.

Principally different picture of resistivity variations under irradiation is observed in the Fe-16Cr alloy doped with 0.1 at. % Cu. In all alloys except the latter the resistivity decrease (associated above with the SRO) demonstrates a tendency to saturation with the irradiation dose, and this is a typical sign of the SRO, since the degree of the SRO and corresponding resistivity are moving during irradiation towards their equilibrium (saturation) values, which depend on irradiation temperature only. When the resistivity variation due the SRO becomes sufficiently small, the resistivity increase due to the Fe-16Cr solid solution decomposition can be detected. In the Fe-16Cr-0.1Cu alloy only resistivity drop without sign of the saturation is found. Such
resistivity behaviour can be interpreted only as the copper solid solution decomposition. This is natural, since the copper solid solution in our case is strongly oversaturated. By comparing resistivity change in the Fe-16Cr-0.1Cu with that in the other alloys, the value of excess resistivity drop in the copper doped alloy can be found, and efficiency of copper atom removal from the solid solution estimated. The efficiency factor turns out to be close to unity – one copper atom is removed from the solid solution by approximately one freely migrating defect. Such high efficiency almost unambiguously points to the interstitial (mixed dumbbell) mechanism of copper atom migration and the lack of defect recombination effect on the decomposition process. The latter means that copper atoms are transported to the distances sufficiently less than the mean distances between the defects. The only physically preferred distance of a such scale is the distance between copper atoms, and therefore the most probable mechanism of the copper solid solution decomposition seems to be the clustering of the neighbouring copper atoms. It is hard to classify unambiguously whether this process is solely radiation-enhanced or radiation-induced: the process is obviously accelerated by non-equilibrium defects, but the mechanism of decomposition is typical for radiation-induced phenomena.

Theory of Solute Segregation in the Boundary of Finite Size Grains in Polycrystals

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A solute segregation from a grain of a finite size into its boundary was first studied analytically in [1]. The results allowed to describe correctly the grain boundary segregation in a polycrystalline material. As shown in [1], the segregation process and the amount of the segregated solute are determined not only by the diffusivity and initial solute concentration in the grain and its boundary, but also by the finiteness of the grain size, i.e. by a limited amount of a solute present in the grain. The influence of the grain shape was also studied in [1], where a flat, spherical and cylindrical grain were considered. In [2] the case was investigated, when an essential role in diffusion processes is played by a solute being constituent of a complex (solute atom – vacancy). The general case was considered when mass transfer is due both to diffusion of "free" unbounded solute atoms, and to solute atoms bounded in complexes with vacancies. For grains of the plate, spherical and cylindrical shape the time dependence was found [2] for: (a) solute concentration profile in a grain; (b) solute concentration in grain boundaries in case of weak solution in boundary. A simple algebraic equation for solute concentration in boundary as function of time was obtained in [2], which is valid in both cases of weak and strong boundary solution of solute. A segregation of a solute from a grain of finite size into grain boundary was investigated in [3] for a case, when the supply of matter to the grain boundary is a very fast process as compared to the process of incorporation of a solute into the boundary. A kinetics of the
solute redistribution, i.e. enrichment of the boundary with a solute or its depletion (drift of a solute into the grain), was considered in [2] and [3] for both limiting processes. A segregation of one kind of solute was considered in paper [4], and the role of local (competing for a place) and non-local interaction of solute atoms was clarified. Also, general equations were obtained in [4] for the case, when different solute atoms are present. The conditions were determined when the concentration of one of the solute atoms reaches transiently a maximum.

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**Acceleration of the Rate of Short-Range Layering in Alloy Fe+15at%Cr under Irradiation with Gaseous and Metallic Ions**

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A comparative investigation of the effect of irradiation with gaseous and metallic ions on the atomic structure of alloy Fe+15at%Cr was carried out. The fact of radiation-dynamic contribution to change in the structure and properties of solid solutions under exposure to accelerated ions was proved.

For this purpose, samples of the alloy in the form of 30-μm-thick foils were quenched from 1200 ºC, underwent cold plastic deformation and were further treated under 5·10⁻⁵ mm Hg vacuum at 450 ºC as follows: 1) irradiated with a beam of light of 1-2 W/cm² power density; 2) irradiated with gaseous Ar⁺ ions at \(E=20\) keV, \(j=50-100\) μA/cm², of the same power density; 3) irradiated with metallic Fe⁺ ions at \(E=40\) keV, \(j=25-50\) μA/cm².

For irradiation with continuous Ar⁺ ion beams, the samples were suspended on thin threads of small sectional area and low heat conductivity, for heat removal to be ensured practically by irradiation only (in cases of irradiation with ions and a beam of light). For irradiation with Fe ions, the sample was placed in a cell which was more inertial in relation to heating with an ion beam than the foil suspended on thin threads. The cell was equipped with an electric heater. Combined use of the heater and the ion beam, with subsequent heater turning-off, allowed us to ultimately bring the rate of heating close to the curve of heating with the Ar⁺ ion beam.

The Mössbauer effect was used to investigate short-range ordering in alloy Fe+15at%Cr. In determining short-range order parameter \(\alpha\), use was made of the fact of linear dependence between this parameter and the mean value \(<H>\) of the effective magnetic field on \(^{57}\)Fe nucleus.

It was established that the value of short-range order parameter \(\alpha\) for a quenched alloy and for an alloy irradiated with visible light at 450 ºC during 30 min was equal to zero within the measurement error limits, while in case of samples irradiated with gaseous and metallic ions,
the value of $\alpha$ was equal to 0.12 and 0.10±0.2, respectively. The high positive values of the short-range order parameter testify to a high degree of short-range layering after irradiation with ions.

Work was carried out with the financial support of the Russian Foundation for Basic Research (grant No. Ural 01-02-96428) and the Program for government support to leading scientific schools of the Russian Federation (grant No. 00-15-96581).

The Laws Governing Radiation-Dynamic Effects in Alloy Fe+8.25at%Mn under Combined Electromagnetic and Ion Irradiation

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Investigation of the laws governing the effect of accelerated $\text{Ar}^+$ ions on structural change in quenched alloy Fe+8.25at%Mn was carried out with a view to find out which parameters, or combinations of parameters (particularly, ions energy $E$, ion current density $j$, temperature $T$, irradiation dose $D$), are critical in initiating RD-transformations, as well as to study their kinetics.

Samples of the alloy in the form of 25-μm-thick foils, after quenching in water from 820 °C (under 5·10⁻⁵ mm Hg vacuum), were irradiated with a beam of light of power density within 1-2 W/cm² during 10 min, and with gaseous $\text{Ar}^+$ ions at different values of ion beams energy ($E=10, 15, 20$ keV) and ion current density ($j=15, 40, 75$ μA/cm²). The target temperature, and hence, the intensity of thermostimulated processes, were kept stationary under ion beam energy and density varying over a broad range at the expense of a combined exposure method, when part of the ion beam power was replaced with electromagnetic irradiation.

The results of the investigations carried out confirmed the presence of radiation-dynamic effects under ion irradiation. So, radiation-dynamic $\alpha \rightarrow \gamma$ (BCC $\rightarrow$ FCC) phase transformations under irradiation with $\text{Ar}^+$ ions ($E=20$ keV, $j=75$ μA/cm²) at 480 °C occurring during 20 s (with formation of $\sim 8$ % $\gamma$-phase) were registered, while samples heated and held at 480 °C under a light beam during 10 min remained single-phase. The existence of a critical ion current density $j_c$ caused by the $\alpha \rightarrow \gamma$ transition was established, and found to be in the limits of $40 < j_c < 70$ μA/cm². Additional experiments will be required for more accurate definition of current critical density, in case of $j < j_c$, no $\alpha \rightarrow \gamma$ transformation takes place at the above temperature. For $\alpha \rightarrow \gamma$ transformation to occur under irradiation, certain values of ions energy are required; in the case under consideration, this value should be not lower than 20 keV. It is not unlikely that the product of $Ej$, i.e., power critical density, is the critical parameter. At ion current density $j \leq 40$ μA/cm², the radiation-dynamic effects manifest themselves through abrupt acceleration of the process of short-range layering. In all cases of combined irradiation, the maximum degree of short-range layering is reached already after 20 s of exposure, while in the course of light irradiation, the same result is obtained after about 10 min of exposure.
Effect of Ion Irradiation on Oversaturated Solid Solution Decomposition in Alloy Al-4%Cu

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Decomposition of oversaturated solid solution in alloy Al-4%Cu under irradiation with Ar$^+$ ions was investigated using the methods of microhardness measurement and X-ray diffraction analysis (precision evaluation of solid solution lattice parameters). The alloy initial samples in the form of 100-μm-thick foils were homogenized at 520 °C and quenched in water. Irradiation with continuous argon ion beams was carried out using a cold hollow-cathode reflecting discharge source. For irradiation, the sample was placed in a copper cell. Irradiation was carried out in the course of continuous heating and after reaching a stationary temperature. Irradiation dose varied in the course of the experiment, the energy of irradiation was 10-20 keV, ion current density was maintained within 50-200 μA/cm$^2$.

It was established that radiation-enhanced solid solution decomposition takes place in alloy Al-Cu under irradiation.

Work was carried out with the financial support of the Russian Foundation for Basic Research (grant No. Ural 01-02-96428) and the Program for government support to leading scientific schools of the Russian Federation (grant No. 00-15-96581).

On the Laws and Nature of Long-Range Dynamic Effects under Corpuscular Irradiation

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In the recent years, intensive investigations have been undertaken of radiation-dynamic (RD) processes initiated by corpuscular irradiation, not related to doping and defects formation in the ion path zones or atoms migration (i.e., to thermo- and radiation-stimulated processes), but, presumably, caused by microshock waves generation on dense cascades of atomic collisions initiating structural and phase transformation on their wavefront.

In the course of irradiation with low-energy (up to 50 keV) ions, RD-processes cause long-range effects consisting in changes in the structure and properties of materials at depths exceeding the projected ranges of ions of the said energies by several orders of magnitude. In case of neutron irradiation, primary recoil atoms act as analogs to ions.
The detected phenomena permit formation of unique electrical, magnetic, tribological and other properties of crystalline, polycrystalline and amorphous materials. Unfortunately, the laws governing RD-processes have been insufficiently studied yet.

Further investigation of the conditions of initiation and course of RD-processes depending on ions energy and mass, ion current density, as well as temperature, composition and structure of the target is needed. The talk will be dedicated to analysis of the laws and nature of RD-processes in metastable metal alloys based on the currently available and published data, as well as the latest investigations undertaken at the Institute of Electrophysics of the Ural Branch of Russian Academy of Sciences, jointly with the Institute of Ion Beam Physics of the Research Center at Rossendorf, Germany. The latter relates to the data obtained with the use of metallic ions, which corroborate and supplement the results obtained in exposure to gaseous ion beams, as well as use of combined ion and electromagnetic irradiation.

Replacement of part of the ion beam power density (0 to 100 %) with light irradiation, while maintaining unvaried the target stationary temperature, allowed us to vary the energy of ions and the ion current density over a wide range. The irradiation dose and kinds of ions also varied. The kinetics of RD-processes for gaseous and metallic ions, as well as the critical values of ions energy and ion current density initiating RD-transformations, were investigated for metastable iron-chromium, iron-manganese and iron-palladium-gold alloys.

Mössbauer Effect Observation on $^{57}$Fe Isotope in $\alpha$-Iron under Ion Bombardment (in situ)

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Using the Mössbauer effect featuring high sensitivity to changes in the electrical and magnetic superfine interactions in crystals as well as to the dynamic state of atoms, an attempt was made in this work to detect lattice (phonon) and electronic excitations under ion bombardment (in situ) in $\alpha$-iron which naturally contains nuclei of the Mössbauer isotope $^{57}$Fe.

In this investigation, use was made of a set-up on the base of the SM 2201 Mössbauer spectrometer. The spectrometer axis intersected with the ion beam axis at an angle of 45°. The center of the target sample, presenting a thermostat entrance port, was located at the point of intersection of the above axes. Irradiation was carried out with Ar$^+$ ions (E=20 keV, j=5 $\mu$A/cm$^2$).

The negative isomer shift (–0.04±0.02 mm/s) testifies to increase in electron charge density on nucleus in the course of irradiation, which may be due to redistribution of both s- and d-electrons (which do not account for density on the nucleus, but screen the s-electrons). Since the Mössbauer experiment geometry was carried out for transmission, the said effect characterizes the state of the whole bulk of the 10-μm-thick foil, and not just the zone of ions penetration (20-30 nm).

Most probably, de-localization of both s- and d-electrons in the bulk of the target takes place under irradiation. However, the contribution of de-localization in the spectrum shift is greater, which may play an important role in explaining such unusual effects as the long-range
effects under ion irradiation, since de-localization may cause increase in the atoms diffusion mobility and phase transition points shift. Exactly the latter was observed in some alloys under exposure to accelerated ions.

The presence of Zeeman sextets with quadrupole splittings of opposite sign (+0.22±0.02 and −0.26±0.02 mm/s) in the Mössbauer spectrum points to an opportunity of elastic oscillations generation in the sample: longitudinal standing waves (and respectively, regions of compression and rarefaction) as a result of radiation-dynamic influence of bombarding ions.

Modeling of Accelerated Embrittlement in Reactor Pressure Vessel Steels at High Neutron Fluences

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The shift of ductile-brittle transition temperature in irradiated VVER pressure vessel steels is usually represented by standard guides with a power dependence on fast neutron fluence multiplied by a material constant. However, from recent data on surveillance specimens for several units of VVER-440 a strong irradiation embrittlement enhancement have been revealed at neutron fluences exceeding (2-3)×10²⁰ n/cm² (E>0.5 MeV) with a significant deviation from the standard guide. This points out to possible involving an additional mechanism of irradiation embrittlement at high fluences.

In the present study it was supposed that such an additional mechanism could be the intergranular embrittlement caused by grain boundary phosphorus segregation. A threshold dose is used which accounts for that the intergranular embrittlement contribution occurs after a sufficient phosphorus segregation when a critical stress for intergranular fracture falls below one for cleavage fracture. The modeling of phosphorus accumulation on grain boundaries is performed taking into account the radiation-enhanced phosphorus diffusion via vacancy and interstitial mechanisms, the radiation-induced segregation in the matrix near grain boundaries and the Gibbsian adsorption on grain boundaries. Predictions of such a composite model are in qualitative agreement with surveillance data for VVER-440.

Significance of Radiation-Induced Segregation in Alloy Properties Alteration under Irradiation

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The report presents a review of experimental and theoretical investigations on radiation-induced segregation (RIS) in metallic alloys. Mechanisms of RIS are considered. Experimental data on RIS in base, alloying and impurity chemical elements in model alloys (Fe-Cr-Ni) and
commercial austenitic steels, ferritic/martensitic steels and reactor pressure vessel steels in PWR and VVER are discussed. The methods of RIS investigation are considered. It is concluded, that RIS effects essentially on precipitation, swelling, irradiation-assisted stress corrosion cracking and embrittlement of these materials. A conclusion is made that a necessary stage of new radiation-resistant alloys development should be a preliminary investigation of RIS phenomenon and radiation-induced formation of precipitates in charged-particle simulation studies. The irradiation of alloys with heavy ions of MeV-energy seems as most appropriate method of such investigations.

Grain Shape Effect on the Solute Segregation in Elastic Stressed Polycrystal

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Polycrystal materials are subjected to an exterior mechanical loading in many practically important cases. If the temperature is low enough (it hasn't exceed 0.4 the material melting point) and the loading is small (smaller, than polycrystal yield point) it is possible to consider a sample were in an elastic stressed state and to neglect inappreciable plastic deformation processes in these requirements. Mechanical loading and a material maintenance long-lived time (20-30 years) essentially influence on the solute diffusion processes in a single crystallite. The solute redistribution between a crystallite and an intercrystallite boundary (solute segregation) changes materials mechanical properties and, as a rule, results in deterioration of their performances. For practical purposes it is important to know how to predict a material safe maintenance time, which in many cases is defined by limit impurity concentration in a grain boundary (the importance of phosphorus in materials of atomic engineering is known), It would be more important to operate on grain boundary impurity concentration and, thus to prolong working hours of materials and articles.

The kinetics of grain boundary solute segregation in an elastic stressed material was studied in our work. Time evolution of grain boundary impurity concentration for arbitrary grain shape was found. The influence of an elastic field was considered. It was shown, that amount of an impurity diffused into a grain boundary depended on the boundary site orientation respectively an exterior loading. Processes in a spherical shape grain were explicitly investigated. It was obtained that even in case of a spherical symmetry of a grain the anisotropic loading gave rise to formation anisotropically located grain boundary precipitate. It was shown, that by means of short-term raise of temperature (in comparison with time of exploitation) the grain boundary impurity concentration could be diminished up to a safe level. The kinetics of grain boundary solute segregation in grains of the symmetric shape was devoted article [1].

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The Effect of Impurities and Grain Boundaries on Radiation-Enhanced Diffusion in Zirconium

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This paper has the goal to discuss the influence of the «impurity-vacancy» complexes (IVC) and grain boundaries on radiation-enhanced diffusion in Zirconium [1–4]. The possibility of the formation and disintegration is considered for the IVC, some kinetic equations are solved for the reducing (Sn) and accelerating (Fe) impurities, their diffusion and influence on the mutual recombination of point defects are estimated. All obtained data are compared with the experiments [3].

Using our model [3], considering the peculiarities of grain boundary diffusion (GBD) [5] and segregation effects, the coefficients of GBD are calculated for Zirconium under irradiation. At the assumption that grain boundary is the phase with the increased concentration of point defects all obtained data on radiation-enhanced GBD are analyzed in the frame of the models [1–3].

References

Kinetics of Intragranular and Intergranular Radiation-Induced Phosphorus Segregation in Reactor Pressure Vessel Steels

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Both the intragranular and intergranular segregation of phosphorus may significantly contribute to irradiation embrittlement of reactor pressure vessel steels [1]. The modeling of phosphorus radiation-induced segregation at cylindrical (dislocations), spherical (precipitates and voids) and flat (sample surfaces, grain boundaries (GB)) point defect (PD) sinks has been carried out in order to compare the kinetics and extent of segregation at various PD sinks. Dilute Fe-0.026P (at.%) alloy relevant to model and VVER-440 pressure vessel steels was considered.
Fig. 1 and Fig. 2 show the dose dependence of P accumulation on the surface of precipitate and on a GB which were carried out at a dose rate $K=3\times10^{-10}$ dpa/s, temperature $T=270$ °C and various precipitate densities $N_p$ [2] and grain sizes $L$. The time to reach steady state P concentration near dislocation or precipitate is much less than that near GB. Although the steady state P concentration near dislocations or precipitates is much less than that near GB, a “fast” P segregation at these sinks may lead to decreasing the free P content in the matrix and to reducing its subsequent accumulation on GBs at high densities of these internal sinks. A more significant effect on the kinetics of GB phosphorus segregation could be caused by the contribution of precipitates to PD sink strength.

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Modeling of Radiation-Induced Segregation in Fe-Cr-Ni alloys

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Radiation-induced segregation (RIS) effects significantly on the phase composition, void swelling, corrosion and embrittlement of alloys. In particular RIS of Fe-Cr-Ni alloy components results in nickel enrichment and chromium depletion near point defect (PD) sinks (sample surface, grain boundaries (GB), vacancy voids, dislocations, dislocation loops and others). More-
over the ferrite regions in austenite matrix and austenite regions in ferrite matrix can reveal due to RIS near these PD sinks [1].

In the present work mathematical model is proposed and calculations of Fe-Cr-Ni alloy component segregation are carried out near motionless as well as moving GB under irradiation. The binding energy of nickel atoms with iron and chromium atoms in mixed dumbbells is estimated using experimental data of low temperature segregation of Fe-Cr-Ni alloy components near GB [2] for taking into account interstitial mechanism of RIS. The modeling of RIS near GB, void and dislocation are performed in Fe-Cr-Ni alloy at various irradiation temperatures. The physical model for explanation of decreasing both RIS and void swelling in austenitic steels after doping with oversize substitutional atoms (titanium, niobium, tantalum, zirconium or hafnium [3, 4]) is proposed. The calculations of desegregation kinetic near GB in Fe-Cr-Ni alloy are carried out for isothermal post-irradiation annealing conditions.

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Redistribution of Phosphorus in Reactor Pressure Vessel Materials During Operation

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Investigations of reactor pressure vessel materials by atom probe [1] and electron microscopy [2] have shown significant changes in the microstructure of both weld and base metals occurred during irradiation, post-irradiation annealing and re-irradiation following by noticeable redistribution of admixtures and alloying elements and leading to degradation of mechanical properties of the materials. The formation of copper-enriched clusters with phosphorus segregated on its surfaces and phosphorus-enriched atmospheres both not associated with any microstructure feature in the ferrite matrix and elongated along dislocation lines could be considered as the most important consequences of such processes. As the result the significant depletion of the ferrite matrix by phosphorus takes place – from 0.03% at. to 0.01-0.02% [3]. Thus when considering the radiation-induced grain boundary segregation of phosphorus its redistribution on microstructure features of the material must be taken into account because the matrix depletion can affect the segregation process. This phenomenon was showed for model alloys [4].
The estimation of phosphorus content on microstructure features has been carried out in this work at different conditions of the material on the basis of an atom probe investigation of VVER-440 weld metal [1]. The calculation of phosphorus content on grain boundaries of the base metal through the reactor pressure vessel wall thickness has been made using the modified McLean model [5] and the kinetic model of radiation damage of steels [6] with taking into account the depletion of the ferrite matrix by phosphorus.

The calculation has shown that the phosphorus content on grain boundaries can reach its maximum in the inner part of a reactor pressure vessel wall where the fluence does not reach its highest value.

References


Radiation-Induced Crystallization of Amorphous Materials

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By the X-ray diffraction methods and also by measurements of the properties related to both an ionic and an electronic subsystems a crystallization of the amorphous materials has been studied. These materials are of the Fe-Cr-B and the Co-Fe-Si-B systems and were produced by a speed tempering from a liquid. Initial materials were in a completely amorphous state as X-ray diffraction data show. Then the alloys were annealed at various temperatures and also were irradiated in an accelerator by Ar+ ions with energies 20-50 keV up to fluences 5.10^{17} - 1.5.10^{18} ions/cm^{2} at different temperatures of a target. The comparison of these processes show that the beginning of a crystallization under irradiation are shifted to less temperatures in comparison with a thermal activated crystallization and the shift value is considerable (150 – 200°C). This
effect can be caused by the difference of processes of clusters formation due a thermal activation and under a radiation influence.

Both strong hardening and embrittlement are observed during an amorphous materials crystallization. This reaches a maximum within a narrow range of parameters near the phase transformation point (in an amorphous-crystalline material). In the some cases the maximum hardness registers before the X-ray lines corresponding to the crystalline phase appear. It can be assumed that this is connected with the crystalline clusters which can not be observed by a X-ray analysis. At the same time the considerable changes in an electronic subsystem were registered which indicate the metallization of an interatomic bonding.

A crystallization of amorphous alloys is also accompanied by an appearance of metastable phases which are not character for equilibrium states of these systems.
V. Materials for Nuclear and Thermonuclear Power Engineering
Uranium Containing Active Layers Behavior under Irradiation at RFNC-VNIITF Pulse Fission Reactors

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The report shows experimental results of study of uranium containing materials behavior under neutron irradiation at pulse fission reactors (PFR) IGRIK and YAGUAR. Subjects of study were layers of $\text{U}_3\text{O}_8$, chemically deposited on aluminum plates; uranium layers, protected by 0.5 $\mu$m Al; and layers of radiation implanted uranium containing material, fabricated by ejection of material under irradiation of uranium oxide rooks at PFR.

Techniques of scanning electronic microscopy, X-ray structural and micro X-ray spectral analysis were used to investigate specific features of layers behavior under different regimes of irradiation. Layers structure before and after irradiation was compared with the help of REM-100U scanning electronic microscope. Results of radiation stability of the layers are presented.

Atomic Vibrations and Polymorphism of Actinides

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Literature [1,2] and our experimental data on mean-square thermal vibration amplitudes for different actinide polymorphic modifications were analyzed. It was shown that the formation of many phases ($\alpha$, $\beta$, $\gamma$, $\delta$) as well as melting was found to be concerned with the value and the temperature dependence in the Debye temperature. Such temperature dependence arises from the anharmonicity and/or may be non Debye phonon spectrum.

Similarly the alloying-produced polymorphism can be concerned with different changes of the temperature dependence in mean-square thermal vibration amplitudes for different actinide polymorphic modifications. The proposed approach extends significantly the capabilities to investigate actinide metals and alloys by neutron diffraction methods. It also poses the question as to the necessity of measuring of the Debye-Waller factors during polymorphic transition investigations.

References
Basic Results of Forced Tests of Core Structural Materials in Fast BN-600 Reactor

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A review is presented of the results of regular post-reactor investigation of the laws governing change of operating working of corrosion-proof austenitic and ferritic-martensitic steels of extractable and non-extractable structures of BN-600 reactor. The most important factors limiting improvement of the working parameters of core of a high-power industrial reactor on fast neutrons are discussed.

In predicting form change of elements depending on irradiation parameters, a single methodological approach was used, and graphic and analytical dependences were developed. Analysis of form change of worked-out structures irradiated in the range of temperatures 350–700 °C to damaging dozes of about 100 dpa was carried out. The influence of the rate of damaging dose accumulation in the range of $10^{-7}$–$10^{-6}$ dpa/s on radiation-induced swelling of austenitic steels with low radiation resistance was investigated.

The empirical dependence between the irradiation-induced swelling and deterioration of the mechanical properties of austenitic alloys in the absence of corrosive interaction with the coolant and uranium oxide/MOX fuel fission products was established.

The effect of the initial condition of structural materials of the fuel element jacket, as formed during metal smelting and manufacture of tubes, on their service properties has been determined. Proceeding from this, the principal lines of improving radiation resistance of structural materials able to support increased BN-600 core lifetime from the current maximum burn-up of 10 % of h. a. to prospective burn-up of 11.2 % of h. a. have been outlined. The burn-up increase to 12.2 and 18.3 % of h. a., as determined proceeding from the requirement of optimal fuel residence time of the BN-600 commercial power unit to allow for the target load factor of about 80 %, will call for improving the available ferritic-martensitic steels and develop structural materials of a new generation.

Commercial Fast Reactor MOX Irradiation Validation

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Tests were carried out of 28 pelletized and 9 vibropacked MOX sub-assemblies irradiated in the BN-600 reactor. All test sub-assemblies remained intact throughout all the handling operations, except for one that failed when it was dismantled in the hot cell. The test pelletized
fuel sub-assemblies operated up to a peak damage dose of 79 dpa and burn-up of 11.8 % of h. a., while the vibropacked ones up to 77 dpa and 10.5 % of h. a. Peak linear rating of 48 kW/m and a fuel cladding temperature of 708°C were reached in case of the pelletized MOX sub-assemblies. The temperature parameters of the test MOX sub-assemblies were the same as those of the hottest standard uranium oxide sub-assemblies.

The post-irradiation examination of 4 test pelletized fuel sub-assemblies (~508 fuel pins) allowed the following to be found out:

- There is actually no change (below 0.4 % due to the radiation-induced swelling and creep) in the cross-sectional size of the hexagonal wrappers (originally 94.5·2.5 mm across the flats) manufactured from EP450 ferritic-martensitic steel.
- The change in diameter of the fuel pin cladding manufactured from EP172 and ChS68 cold-worked steels from the original 6.6·0.4 mm is less than 5 or 6 %, i.e. within the limits specified by the criteria of the allowable distortion and within the limits of an increase in fuel pin diameter of the standard BN-600 sub-assemblies of the second modification core. With a damage dose of 76 dpa, the bundle averages of the actual diameter, ovalization and elongation are 3.9±0.5 %, 320±90 μm and 1.4±0.2 %, respectively. With the obtained peak damage dose of 78 dpa and burn-up of 11.7 % of h. a., the operability of the cladding is not limited by the mechanical properties degradation.
- The data of the primary examination showed the operability of the pelletized MOX columns to be the same as the radiation behaviour of the standard uranium oxide fuel. The obtained spatial distribution of the fission product activity and cladding material activation is similar to the respective one with the standard fuel. The getter and the evaporable insert installed in some fuel pins did not affect the axial distribution of radioactive nuclides along the fissile part.

The examination of the test vibropacked fuel sub-assemblies showed the following:

- An increase in distance across the flats of the wrappers manufactured from steel composed of 0.05 % C, 12 % Cr, 2 % Ni and ≤1 % Mo is negligible (up to 0.6 to 0.8 % against the original 94.5·2.5 mm). They, as well as wrappers manufactured from EP450 steel, are able to remain serviceable at even higher operating parameters.
- The distortion parameters of the wrappers manufactured from steel composed of 0.08 % C, 16 % Cr, 11 % Ni, 3 % Mo and ≤1 % Ti are within the limits obtained for the same steel used in the standard BN-600 sub-assemblies of the first modification core. The lifetime of the wrapper accumulated a peak damage dose of 77 dpa and failed due to mechanical properties deterioration and brittle destruction when in the course of dismantling.
- With a peak damage dose of 77 dpa the bundle average of the distortion of the fuel pin cladding manufactured of the EP172 cold-worked steel equal to 5.2±1.0 % is higher than that obtained earlier during the commissioning of the Pervoural’sk cladding manufacturing facilities. When removing the bundle out of the wrapper the cladding of one of the pins failed in the area of maximum swelling. The lifetime of the cladding manufactured of the initial EP172 cold-worked steel made by the VNITI prototype plant nearly ended by the criteria of the distortion and degradation of mechanical properties.
- The distribution of the slightly migrating Zr-95 and Ru-106 along the core was found out to be more non-uniform (as compared with the pelletized columns). This can be caused both
The completed in-reactor tests and post-irradiation examination of the test MOX sub-assemblies allow us to conclude that they remain serviceable at least up to 10% of h. a., i.e. up to a standard burn-up of the second modification core. Tests continue of pelletized and vibro-packed fuel of standard size and standard wrapper/cladding materials with a view to further extend the lifetime of the MOX fuel sub-assemblies.

**Studies on Modification of Some First Wall Materials**

**Using 3-6.8 MeV He Ions**

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A considerable effort has been directed towards the behavior of He in metals. He is known to have a low solubility in metals, much less than 1 appm; it strongly tends to participate into bubbles, causing swelling and He embrittlement. Surface damages with formation of blisters, exfoliation and sputtering are also produced. Because very few results are reported in the MeV region, we have started a systematic investigation concerning the fluence and energy dependence of blistering induced in some useful materials for the first wall (stainless steels, Ni, Cu, Mo). They have been irradiated with 3.0, 4.7 and 6.8 MeV He ions. The irradiation effects have been investigated by means of a TEMSCAN-200-CX electron microscope and two metallographic microscopes. Irradiation phenomena as sponge- and wave-like structures, submicronic cracks, multilayer flaking, micro-conglomerates, secondary blisters, microcraters were observed. Microstructural changes as helium bubbles on matrix and grain boundaries, on loops and on TiC precipitates were also observed. An interesting effect is the amorphous phase in the Ti-modified austenitic steel 12KH18N10T, phase formatted during irradiation with 6.8 MeV He\(^+\) ions up to a dose of crater occurrence (7×10\(^{18}\) ions/cm\(^2\)), the temperature of specimens (100 µm thick) being maintained during irradiation in the range 30-60°C. The X-ray microanalysis shows the presence of the major alloy elements in the amorphous area, whereas in that embedding a microcrystallite it is to be noticed the Ni depletion and the presence of Si and Mo.

A potential explanation of the energy dependence of average blisters diameter d and blister critical dose could be stressed-induced model, because, in our cases (medium energy He ions), d~t\(^m\), where t is the blister skin thickness. For our high energy data, a m=1.8 value is relatively convenient, except Mo, where m=1.5 is more suitable. Similarly, as concern the dependence of our experimental values for the critical fluence, we can assume a relationship of the form: f~t\(^2\), therefore concluding that not only the resistance of the blister skin itself is important (that being proportional to t), but there are also other internal resistance factors playing an important role.
Recent Developments Concerning Void Swelling in PWR Austenitic Internals at Relatively Low Irradiation Temperatures

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In the last 7-8 years there has been a growing awareness of the potential of void swelling in austenitic steels used as internal components to impact the operation and economics of Western pressurized water reactors. It was originally assumed that void swelling would not occur in the 300 series steels used in the baffle-former-barrel assemblies that surround the active core zone, primarily because of the lower dpa rates and lower temperatures in PWR internals compared to those of fast reactors. It is now known that lower dpa rates actually hasten the onset of swelling, which may be further accelerated by applied stresses and the higher gas (He, H) generation and retention rates characteristic of light water reactors.

Evidence is now accumulating to show that swelling is indeed occurring in PWR internals, with the potential to influence the prospects for license extension from 40 to 60 years. A summary of experimental evidence and industry/regulatory response will be presented.

Corrosion and Electrochemical Properties of EP - 450 Steel Exposed to High-Dose Irradiation in BN - 600 Reactor

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The ferrite martensite steel EP - 450 has been employed for many years in the cores of the fast reactors BN-600, BN-350 and BOR-60; and the items made of EP-450 have demonstrated high radiation resistance. However, when spent fuel element assemblies are extracted from the reactors and stored by the wet method, their case tubes made of EP-450 are strongly affected by corrosion in water of the decay pool. Accelerated corrosion and high percentage of corrosion products release into the water call for frequent regeneration of filters in the water chemical treatment system and cause increase in the radiation dose burden on personnel. While presenting an advanced grade of corrosion resistant steel, EP-450 may become a limiting factor for using it as a fuel cladding material in fast reactors due to its insufficient corrosion resistance, which the steel reveals in the post-operation period in decay pool water.

This paper presents the results of the investigation of corrosion and electrochemical properties of EP-450 case tubes in the water with pH = 7.0, after the tubes had been irradiated to a maximum damage dose of 75.6 dpa in the BN-600 reactor.
Tests were carried out during 2000 hours with the intermediate exposures of 500 and 1000 hours. They revealed subsurface layers of lower corrosion resistance of steel to both uniform and pitting corrosion during operation in the reactor. The depth of penetration of corrosion of both types is proportional to the damage dose and decreases with irradiation temperature. However, they have different kinetics, i.e., uniform corrosion depends linearly on time, and pitting corrosion depth is a parabolic function of time. Increased steel (irradiated in liquid nitrogen) liability to pitting corrosion is attributed to a dramatic decrease in the width of the area of stable passivity of steel, which is strongly dependent on neutron damage dose but is practically independent of operation temperature. At the same time, when the dose increases, the stationary potential the steel shifts towards positive values by approximately 140 mV, and the potential of leaving the passive state deviates towards the negative values by more than 750 mV.

Irradiation-Assisted Intergranular Stress Corrosion Crackling of Austenitic Stainless Steel in Steam-Water Mixture


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Susceptibility of austenitic stainless steels (ASS) to stress corrosion cracking (SCC), which is often of intergranular type (ISCC), is one of the important problems of their usage as in-vessel devices components of nuclear reactors in water-steam environment. The reasons of this fact arise from chromium depletion of boundaries and adjoining zones in the sensitive areas near welded joints as a result of precipitation of chromium carbides of Me\textsubscript{23}C\textsubscript{6} and/or Me\textsubscript{6}C type. Neutron irradiation facilitates SCC propagation due to radiation-induced segregation and depletion processes at interphase surface.

The real in-vessel devices components, that have been operated for a long time, are the most informative material for SCC investigation under neutron irradiation. The external 0X18H10T ASS tube of the VK-50 reactor control assembly was chosen as the subject of examinations. The assembly was damaged in the welded joint area after 30 years of operation during scheduled replacement. In the place of fracture the tube was operated under alternative cooling with sub boiling water and water-steam mixture, and in the fracture area neutron flux was $3.0 \times 10^{21}$ n/cm$^2$ (E>$0.1$ MeV).

The following conclusions can be drawn according to the obtained results:

- fracture occurs mainly along the grain boundaries;
- titanium segregates on the grain boundaries, and Cr and Ni deplete them; the thickness of the segregation layer on the fracture surface is 1-1.5 µm;
- chlorine, sulfur, and copper, dangerous elements from the corrosion point of view, are available on the fracture surface. After ion etching of the examined surface the sulfur agglomerations disappear already at a depth of 3 µm.
The analysis of literature and obtained results enables us to assume the ASS IAISCC mechanism in the following way.

At the primary stage, when cracks, pores and other defects are present on the metal oxide film the rate of general corrosion process is relatively high. The generated slightly insoluble corrosion products gradually heal the defects of oxide film on the grains and decrease the area and number of anode sections. The effect of anodes gradually propagates to sensitive grain boundaries. This period is considered as incubation period. Then the process proceeds at a low rate suitable for the anode dissolution rate in passive state. The maximum rate is observed in the areas with minimum chromium concentration and as a result of it the corrosion has a nodular character. As the intergranular cracks propagate, the corrosion products obstruct the access of fresh portions of water/seam to the anode sections and removal of metal ions in the opposite direction. Moreover the access of cathode depolarizer (oxygen) is obstructed. As a result, the cathode process is transferred onto the walls of intergranular channels close to the surface [1,2]. Gradually the poisoning process of grain boundaries with sulfur and chloride released from coolant and from radioactive decay of MnS manganese sulphides becomes stronger. Copper precipitation begins on the surfaces of the generated intergranular cracks. Complex aggregations on the basis of sulfur and copper are being generated. They consist of iron, chromium, nickel, carbon and oxygen also. The effect of radiation-induced processes of grain boundaries depletion in chromium and nickel with their simultaneous enrichment in titanium, phosphorus and silicon increases. The chemical composition of boundary areas differs more and more from the matrix chemical composition. Under the effect of the residual tensile welded stresses the intergranular crack nuclei grow in width and length. At the final stage they reach the length of some tens of millimeters and form one main cross crack. The process of intergranular corrosion cracking results into the transverse fracture of the tube.

X–ray Investigation of Fine Structure of Alloy Zr –2.5% Nb after In–pile Irradiation at ~ 80 °C

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The channel (factory # C-24) of the control and protection system made of the alloy Zr–2.5 %Nb had operated in cell # 32-25 of Unit IV at the Leningradskaya NPP for 17.4 years. After that the material of the channel was investigated by the X–ray method. A calculation neutron fluence was ~ 3·10^{22} th.n./cm^2 (~1.2 dpa) and an irradiation temperature was ~80 °C during the alloy operation.

As a result, a shift, widening and changing intensity of X-ray lines of \( \alpha \)-Zr matrix were observed. A value and a sign of the observed changes in the fine structure depend on the type (prismatic, basal or pyramid) of diffraction planes. A maximum increase of inter-plane distances and a half-width of the X-ray lines was noticed for the prismatic - type planes (h00) and (hk0). A decrease of the half–width and the inter- plane distance was observed in the basal - type planes (00L). The changes of the fine structure are attributed to the oriented preferential accum-
ulation of radiation defects of interstitial type (small dislocation loops) in the prismatic planes
and vacancy defects (small clusters and double or triple vacancies) in the basal planes.

Changing of Physical and Chemical States of Iron Atoms in Zirconium Alloys
Oxide Films During Neutron Irradiation

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The oxide films of zirconium alloys (Zr-0.76%Fe-1.6%Sn-0.95%Nb and Zr-0.76%Fe-
1.6%Sn.) oxidized in autoclave and in reactor irradiation conditions were investigated by Möss-
bauer spectroscopy method.

The corrosion tests of flat specimens placed in tight ampoules from the stainless steel,
filled by distilled water, were carried out in a reactor during 37, 80 and 112 effective
days(fluency 3·10^20 n/cm², E>0.5 MeV). Similar specimens were tested in laboratory conditions
during 40, 80 and 120 day in similar ampoules and identical thermodynamic conditions (350°C,
16.8 MPa).

The comparative analysis of corrosion curves has shown, that the thickness of oxide films
obtained at reactor irradiation are almost three times more than thickness of films obtained at
corrosion in an autoclave. It testifies that reactor irradiation essentially accelerates the corrosion
process of both alloys especially 3 component alloy.

The Mössbauer researches have shown that  the spectra of both alloys oxide films, obtained
in autoclave, have the lines of solid solutions of iron ions Fe^{n+}, Fe^{n+}, Fe^{n+} in Zer_o and for some
samples – the lines of metallic precipitates of α-Fe. The spectra of both alloy oxide films, ob-
tained in reactor, have the lines of solid solutions of iron ions Fe^{2+}, Fe^{3+}, Fe^{4+} in ZrO_2 and for
some samples – the lines of compound α-Fe_2O_3. The concentrations of iron atoms in different
chemical states in oxide films are determined.

Comparison of kinetic curves of corrosion with results of Mössbauer researches has shown,
that α-Fe metal precipitates are formed only in films of the samples subjected corrosion in auto-
clave but not in all oxide films. So α-Fe is formed in oxide films when alloys have good corro-
sion resistance. In reactor irradiation conditions when alloys show low corrosion resistance, the
α-Fe is not revealed in oxide films, and formation of α-Fe_2O_3 compound is marked. As to solid
solution of ions Fe^{4+} in ZrO_2, it was seen, that in oxides of alloys with good corrosion resistance
the concentration of these ions is more greater. In cases with bad corrosion resistance of alloys
the concentrations of ions Fe^{4+} in ZrO_2 is decreased. Thus its possible to assert about correla-
tion between concentration of ions Fe^{4+} and corrosion resistance of zirconium alloys. Obtained
data confirm the conception about positive influence of metal precipitates formed in oxide film
and ions Fe^{4+} on corrosion behavior of zirconium alloys.
Variation of Isothermal Kinetics of $\alpha \rightarrow \beta$-Transformation of Unalloyed Plutonium over Different Times of Samples Self-Irradiation


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The paper presents the results of investigation of the kinetics of $\alpha \rightarrow \beta$-transformation of unalloyed plutonium under isothermal conditions over different times of samples self-irradiation. Variations in the kinetics of phase transformation vs the time of self-irradiation are discussed.

References

Radiation Phenomena in Austenitic Steels Irradiated in SM, BOR-60, VVER-1000 Reactors at 280-400 °C

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Recently study on microstructure evolution of austenitic steels at low irradiation temperatures has become one of the most acute issues that determines, to a greater degree, swelling and embrittlement of such steels at these temperatures. Different components of fast, pressurized and boiling reactors are made of the Fe-18Cr-10Ni-Ti steel types. Sometimes they operate at close irradiation condition parameters. Comparative study of these components allows for estimation of the effect of irradiation conditions on steel microstructure and properties.

For the last few years at SSC RF RIAR several components made of the Fe-18Cr-10Ni-Ti steel types have been chosen for the investigations. They operated in different reactors under irradiation conditions close to those of the VVER in-vessel facilities. They include: blanket assembly wrappers and tail-end adapter after their long-term operation (more than 22 years) in the BOR-60 reactor, guiding channel of the VVER-1000 peripheral fuel assembly, the SM material science assembly block. The damage dose and temperature range of the products under study changed from 1.5 up to 81 dpa and from 285 up to about 400 °C.

The results and analysis of the performed investigations of these components allowed the comparison of microstructure, precipitated phases, radiation swelling and mechanical properties of these steels as a function of dose accumulation rate, temperature and damage dose.

The most obvious conclusions are as follows:

There are some differences in the microstructure of the investigated steel specimens irradiated in different reactors.
A long-term operation of the blanket fuel assemblies at low irradiation temperatures (about 320°C) in BOR-60 leads to material swelling dependent on the dose accumulation rate. The investigations on radiation swelling of all above components made of the Fe-18Cr-10Ni-Ti steel types allowed the identification of the temperature threshold (300-310°C) for voids formation. The Fe-18Cr-10Ni-Ti steel specimens cut from the VVER-1000 components and irradiated in fast reactors showed strong hardening and reduction of relative elongation. This correlates well with the hardening values calculated on the basis of microstructure changes of irradiated steels.

Peculiar Features of Structure and Behavior Changes in Some Zirconium Materials at Damage Doses up to 50 dpa


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The irradiation-induced damage of zirconium materials subjected to neutron irradiation up to dose of ~50 dpa was investigated. The dose value was caused by a change of irradiation time or fast neutron flux. The specimens of unalloyed zirconium and Zr-1%Nb, Zr-2.5%Nb and Zr-1%Nb-1.3%Sn-0.4%Fe alloys were irradiated in the BOR-60 reactor dismountable devices over the temperature range 320-420 °C. The device design allowed the removal of specimens during the reactor shut-down (2-3 times per year) and the measurement of dimensional changes in the hot cells with their subsequent return to the reactor core. The dose dependence of the irradiation growth strain increases sharply in zirconium and Zr-Nb alloy specimens irradiated at the temperature of ~350 °C at doses above ~10 dpa. As for Zr-1%Nb-1.3%Sn-0.4%Fe alloy specimens it increases at doses of ~37 dpa. With increasing the irradiation temperature to 420°C, a sharp accelerated irradiation growth of the Zr-1%Nb alloy begins shifting up to about 30 dpa. As for the Zr-1%Nb-1.3%Sn-0.4%Fe alloy no change of the irradiation growth rate was observed up to the dose of 55 dpa. The onset of increased irradiation growth stage in alloys correlates with the occurrence of c-component dislocation loops. This moment coincides with a loss of coherence of fine-dispersed precipitates. The post-irradiation annealing experiments demonstrated that a delay in loop formations leads to displacement of the “break-away” beginning in the dose dependence of the irradiation growth in the direction of high doses.

The high dose irradiation results in the change of the element phase composition: hcp matrix of solid solution is depleted with niobium; a gradient of element concentrations was noted along the radius of (ZrNb)2Fe particles occurring in the Zr-1%Nb-1.3%Sn-0.4%Fe alloy structure. In investigating precipitations of about 30-60 nm in size, it was established that irradiation of Zr-1%Nb and Zr-1%Nb-1.3%Sn-0.4%Fe alloys results in increase in the average size of particles and decrease in their concentration. In the irradiated samples of Zr-1%Nb alloy the contents of atoms Nb in precipitations of a phase β-Nb decreases in comparison with the not irradiated material and at high dozes reaches about 50%.

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Microstructure Changes in EI-847 Steel Exposed to Long-Term Irradiation at 300 -390 °C in Fast Neutron Reactor

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The austenite steel EI-847 was investigated after its long-term operation as a fuel element cladding in the pulse fast neutron reactor IBR-2. Test samples taken from the areas, which had been exposed to neutron irradiation at temperatures of 300 to 390 °C. A maximum damage dose in the cladding material reached 13 dpa at an irradiation temperature of 350 °C during 16 years of operation, including 4.8 years of its performance at the reactor power of 22.5 kW/kg U.

The material was investigated by the methods of transmission electron microscopy and micro X-ray structural analysis. They revealed pores in the material irradiated at 350 °C. The diagram of pore distribution against sizes was plotted for the statistical sampling of 1000 pores. It was shown to be a unimodal distribution of the pores with a mean size of about 4 nm. The concentration of pores was ~ 2.5*10^{21} m^{-3}. As a rule, they are related to those dislocations or dislocation loops, which concentration is higher than that of radiation pores.

There were no pores in the steel irradiated at the bottom of reactor core at 300 °C (damage dose was 6 dpa), though there was a large number of radiation defects of the dislocation loop type, which formed fragments of a small-cell dislocation structure.

There were also numerous dislocation loops in the microstructure of the material irradiated at the top of the core at 390 °C to the damage dose of 6 dpa, though their concentration and size were smaller as compared to those of the material irradiated at the bottom. There were no radiation pores in the material irradiated at the top of the core.

Influence of Radiation Swelling on Thermal Expansion Coefficient of Steel ChS-68

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Fuel element claddings made of the austenite stainless steel ChS-68 were exposed to long-term irradiation in the fast neutron reactor BN-600 and then a thermal expansion coefficient (TEC) of the steel was measured. The areas, where the irradiation temperature was from 430 °C to 590 °C, were investigated. A maximum damage dose in the cladding material reached 70 dpa, which corresponded to the temperature of 530 °C.

Cladding samples were tested for linear change parameters of the irradiated material within the temperatures of 200 to 700°C. The difference in TEC values was found to be very small for the material taken from different areas of the core and heated from 200 to 530 °C.

The TEC decreased substantially within 530 to 700 °C, which was, probably, due to structure transformations. In repeated heating, the run of a TEC curve in the high temperature region was changing.
The dilatometry measurements with the isothermal exposures at 420 °C and 660 °C were carried out to study the radiation-induced defect annealing. A length of samples was decreasing at the temperatures higher than the operation temperature during isothermal exposures, the decrease being from 0.01 to 0.11 % in relative units. An initial length of the samples was also decreasing during a linear heating to the temperatures higher than the operation temperature. It was found that a shrinkage value was inversely proportional to the operation temperature of the fuel element cladding material.

The methods of the transmission electron microscopy were used for a statistical processing of radiation defect assembly. It was shown that the decrease of the sample length was related to the annealing of radiation defects and vacancy pores evolution, in particular.

Influence of Structure-Phase State Zr-Nb-Containing Alloys on Irradiation-Induced Growth

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On account of the search of optimal composition and structure-phase state of Zr alloys much attention is paid to upgrade E 110 (Zr-1%Nb) and E 635 (Zr-1%Nb-0.35%Fe-1.2%Sn) alloys that proved well in terms of irradiation-induced creep and growth, high strength characteristics, corrosion. The difference between the alloy properties is determined by their states related to their compositions.

The structure phase state of the Zr-Nb and Zr-Nb-Fe-Sn systems has been studied after heat treatment in the α- and α + β regions and the influence was investigated that is produced by this state on the irradiation-induced growth (IIG) during BOR-60 irradiation at T =315-350 °C. A substantial difference has been revealed in the deformation effected by IIG of those alloys, it is less for Zr-Nb-Fe-Sn alloy in dissimilar structure-phase states. The incubation period of the accelerated growth stage is determined by the α-matrix composition, the phase state and the initial dislocation structure. Neutron irradiation leads to a redistribution of alloying elements between the matrix and the precipitates, to changes in the α-solid solution composition and affect accumulation and mobility of irradiation defects, anisotropy and formation of vacancy c-component dislocation loops. The appearance of c-loops usually correlates with an axial direction acceleration of the IIG of tubes conforming to their texture.

The basic regularities of the phase transformation have been established: a) β-Nb precipitates in Zr-Nb alloys alter their composition to reduce the Nb content from 85-90% to ~ 50%, precipitates likely enriched in Nb are formed, b) β-Zr precipitates are subject to irradiation – stimulated decomposition, c) Laves phase precipitates change the composition (the content of Fe decreases) and the crystal structure, HCP to BCC (β-Nb), d) (Zr, Nb)2Fe precipitates having the FCC lattice retain the composition and crystal structure, e) no amorphization of secondary phase precipitates is observable under the given conditions of irradiation (T = 315 - 350 °C).
Based on the dpa, the results were compared pertaining to Zr-alloy IIG deformation vs fluence in various reactors at different energies of fast neutrons. The presented graphs enable comparison between the results of numerous experiments and prediction of Zr-material behaviour in long-term operation and at high burn-up in commercial reactors.

**Damage of Beryllium under High-Dose Neutron Irradiation**


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Beryllium is supposed to be used in structural components of the future fusion reactor as plasma facing material, blanket material and neutron multiplier. Evaluation of degradation degree of beryllium properties under the influence of the neutron irradiation in the nuclear reactor is an important problem for feasibility of the fusion reactor components. The paper describes the research results of the irradiation effect in the SM and BOR-60 reactors at 70-450°C in the neutron fluence range \((0.5-16) \times 10^{22} \text{ cm}^{-2} (E>0.1 \text{ MeV})\) for physical-mechanical properties and microstructure of several Russian beryllium grades (TE-400, TE-56, TE-30, TIP, DIP) produced by the HE and HIP technologies.

**The Mechanisms of Uranium Metal Fuel Radiation Creep and Its Temperature Nonlinearity**

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In [1] it is shown, that in uranium metal at moderate temperatures and high neutron flux densities the radiation point defect concentrations considerably may exceed thermally equilibrium, also it is submitted the theoretical model of the established radiation creep within the framework of the mechanism of dislocation gliding and climbing, based on the conception of a dislocation as a non-ideal sink for point radiation defects. Are developed the “dynamics” method of mathematical simulation of gliding and climbing of flexible dislocation interacting to obstacles of a various type (spherical centre of extension, dislocation prismatic loop and their spatially random distributions), and a computer program complex for mathematical simulation of radiation creep and radiation creep rate estimations by a method of Monte Carlo. Computer simulation of uranium metal fuel radiation creep has revealed its temperature nonlinearity [2]. These temperature nonlinearities have served the reason for search of nonlinear effects and modes in open nonlinear stochastic system which the system uranium metal fuel under an irradiation [3] is.

The generalized diagrams of radiation-thermal creep for not fissionable (constructional)
metals and non-fissionable (fuel) metals are submitted. The regions of various radiation creep theoretical models are qualitatively determined. It is shown, that the nonlinearities of curve thermal dependence of the radiation creep established rate are caused by the features of used radiation creep theoretical model based on the concept conception of a dislocation as a non-ideal sink for point radiation defects and the features of point radiation defects generation in divisional metals.

References

Modification of Plutonium Dioxide Properties under Exposure to Radiation

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The report shows experimental results of Pu\textsuperscript{239}O\textsubscript{2} electric resistance variations under protracted influence of radiation investigations. Obtained dependence of electric resistance on dose is compared with published results of investigations of Pu\textsuperscript{238}O\textsubscript{2} crystal lattice parameters under influence of radiation with sufficiently higher intensity. Comparison shows that despite significant difference in intensity of radiation influence notable modification of PuO\textsubscript{2} physical properties takes place under dozes \(\lambda \cdot t = 20 \cdot 10^{-5}\).

Investigation of Distribution of Erbium in Uranium – Erbium Oxide Fuel of RBMK by X–ray Analysis

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One of the means, increasing safety and reliability of RBMK Units operation, is adding erbium as a burn-up absorber to the RBMK fuel. A concentration and distribution of erbium in a fuel pellet is an important characteristics of the fuel element fabricated for U–Er fuel usage.

X–ray structure analysis as a ND technique allows to calculate an erbium concentration and determine a manner of erbium distribution in the pellet.

It is shown that the X-ray structure analysis is applicable for a calculation of an erbium content in the fuel. The dependence of a lattice parameter of the fuel on the erbium content is provided. The approximation function of X–ray reflections and the extrapolation function are
matched in the precision method of the determination of a lattice parameter in crystalline materials with a cubic structure.

The methodology to calculate an erbium concentration, a volumetric part of areas with a different erbium content is presented. The methodology was used to investigate samples in seven lots of fuel with a different U-235 enrichment and a different mass portion of erbium.

The condition of the crystal structure of uranium–erbium oxide fuel was investigated after the fuel exposure in the IVV–2M reactor, where the calculation fuel burn up was from 0.67 to 10 MW·day/kgU.

**Comparison of Irradiation Conditions for Fusion Materials in IFMIF and ESS**

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Investigation of the fusion materials behaviour under irradiation conditions close to those expected for future fusion reactor is one of the most critical issue for the design of demonstration power reactor DEMO and fusion reactor prototype ITER. Among other facilities the International Fusion Materials Irradiation Facility (IFMIF) was selected and approved to be suitable for proper simulation of fusion irradiation [1-3]. The design of the European Spallation Source raised a discussion of suitability of this neutron source for fusion materials irradiation. The present study is devoted to comparison of the irradiation conditions for both facilities with respect to their ability to simulate DEMO reactor environment.

Geometry models of these facilities were designed to perform Monte Carlo simulation of neutron spectra at different parts of the installations. Using calculated neutron and primary knocked-on atom spectra various damage characteristics, namely, damage and gaseous atom production rates as well as spallation rates were calculated and compared. In particular it was shown that He/dpa ratio for reflector position (at least in Fe based alloys) is between 5-6, that is about a factor of two lower than expected for DEMO fusion reactor. While the H/dpa ratio at the same position is about 33-36, which is a factor of about 1.5 below DEMO reactor specific ratios.

On the basis of the work the ESS target position initially proposed for allocation of fusion materials irradiation rigs was ruled out as having too hard neutron spectra producing substantial amount of spallation products and resulting in changing phase composition and material properties deterioration of irradiated structural materials. It was also confirmed that although ESS reflector position could be used for obtaining some preliminary results (in case the ESS will be constructed several years earlier than IFMF), the IFMIF facility is necessary for proper simulation of the fusion irradiation conditions and comprehensive investigation of fusion relevant materials behaviour under irradiation.
New Structural Material for Fusion Reactor Based on Ferritic Stainless Steel-Clad V-Ti-Cr Alloy

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As a low neutron activated structure material the vanadium alloys of V-Ti-Cr system are more promising materials for lithium energetic reactors (tokamaks). This is due to favorable combination of their physical and mechanical properties, good compatibility with liquid lithium, high resistance to irradiation-induced damage. V-4Ti-4Cr and V-10Ti-5Cr alloys have good resistance to swelling during irradiation and have not susceptibility to irradiation embrittlement above 400 °C. Mechanical properties and particular elongation of V-10Ti-5Cr alloy are more stable, then V-4Ti-4Cr. Integration and survey of different characteristic data base allow to conclude, that maximum operation temperature of V-10Ti-5Cr alloy could be 750-800 °C in fusion system. However general disadvantage of vanadium based alloys is contamination with interstitial impurities, such as oxygen, carbon, nitrogen, and hydrogen, which cause degradation of mechanical properties and workability. This disadvantage has substantially limited their application as structure materials. To avoid attack of the surroundings the vanadium alloys have been suggested to clad by ferritic stainless steel. Sandwich consisting of V-4Ti-4Cr alloy and ferritic stainless steel plates has been hot and cold deformed. Good adhesion these materials has been achieved. Thus, opportunity of making new low neutron activated, hot temperature strength, corrosion resistant and irradiation resistant structure material for fusion application has been shown.

Study of the Effect of Tritium and Reactor Irradiation on Properties of Materials in the First Wall of Fusion Reactors

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As is known, one of the most important aspects ensuring reliability, radiation and ecological safety of installations in nuclear and thermonuclear power engineering is the effect of trit-
ium, which is accumulated during operation (nuclear and salt liquid reactors) and/or is used as the fuel (fusion reactors), on materials and its penetration to the environment. This problem especially applies to selection of materials and development of the manufacturing technique of such energy-intensive elements of fusion reactors (FR) as the diverter and the first wall.

The present report covers some schedule of work intended to obtain experimental evidence on the effect of tritium, radiogenic helium and the reactor irradiation on the structure and properties of materials and bimetallic elements in the first wall and the diverter of magnetic-confinement fusion reactors. The objects of study will be samples of radiation-resistant steels and a steel/bronze (SS316L/CuA115) bimetallic element of the first wall, which will be supplied by J.F. Stubbins and Celia Elliott (Illinois University, USA).

The fulfillment of the Project work will provide the following information:

- parameters of the penetration process and accumulation of heavy hydrogen isotopes in constructional materials of fusion reactors;
- concentration and localization of hydrogen in the structure of the samples;
- effect of hydrogen on mechanical properties of constructional materials exposed to long-term extension stresses;
- interaction of tritium atoms with radiation-induced defects in the materials studied;
- effect of the surface condition on the interaction between tritium and the materials.

The anticipated results will be very significant from the scientific and practical viewpoints for a reasonable choice of materials and designs of thermonuclear installations ensuring a minimum leakage of tritium to workrooms and the environment.

The experimental basis and the team of researchers, which were formed during fulfillment of the ISTC Project No. 019-94, provide good grounds for implementation of the planned work. The team of researchers not only strives to advance the success achieved under the Project No. 019-94, but also looks for consolidation of the cooperation between the researchers at the Russian Federation Ministry of Power Energy, the Russian Academy of Sciences and the international scientific community.

The penetration processes and the yield kinetics of tritium will be studied using hydrogen permeability and thermal desorption methods. The concentration will be measured by the liquid scintillation spectroscopy method. The microautoradiography method will be used to determine the distribution of hydrogen isotopes in the structure of the materials. The interaction of hydrogen isotopes and radiogenic helium with defects of different origins (dislocations, grain and subgrain boundaries, interfaces, etc.) will be deduced from the variation of mechanical properties, electric resistance, and the residual concentration of tritium. In addition, the methods of X-ray diffraction analysis, positron annihilation, electron microscopy, and nuclear microanalysis will be used at their utmost.

The condition of the interface between the layers in bimetallic structures will be studied on special samples prepared of bimetallic plates. The samples will be tested before and after loading with tritium and radiogenic helium.

The adverse effect of tritium and neutron irradiation on mechanical properties of FR materials exposed to long-term extension stresses presents a special interest from the viewpoint of their service characteristics. Standard short-term tests cannot reveal the real effect of hydrogen on embrittlement of materials under continuous loads (which occur most frequently in service),
while long-term tests allow determining how plastic properties of constructional materials are affected even by a small concentration of gas impurities.

The goals of the proposed study fully comply with the ISTC objectives, since it provides an opportunity of alternative peaceful activities for specialists in nuclear weapons, who will be able to use their experience and knowledge for solution of worldwide problems concerned with generation of thermonuclear power, environmental protection, and safety of fusion reactors. The study will facilitate the integration of Russian scientists into the world scientific community.

Effects of Actual Aging (Self-Irradiation) of Delta-Stabilized Pu Alloy with Gallium on Microstructure, Mechanical Properties, δ–α′–Phase Transformation Macrokinetics in Weak Shock-Waves


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Peculiarities of dynamic behavior and properties of newly cast delta-stabilized plutonium alloys with gallium have been already investigated in the weak shock -waves [1-3].

Accumulation of the radiogenic helium in the crystal lattice of alloy, because of α-decay of plutonium nuclei, at the room temperature during the prolonged real-time self-irradiation, can effect not only on the mechanical properties of the alloy at its quasi-static and high-rate deformations in the initial δ – phase state, but also on the kinetics of the δ – α′ – phase transition in stress waves.

The set-up of explosive experiments and the first investigation results of the high-rate elasto-visco-plastic deformation in the region of the initial δ–phase state as well as macrokinetics of δ – α′ – phase transition in weak shock waves of the actual 16-years aged δ-alloy are presented. Taking into account [4-8], the features of the microstructural changes of the prolonged self-irradiated alloys, which are responsible to the kinetics change of the δ – α′ – phase transformation, are discussed.

References


**Effects of Prolonged Self-Irradiation on Shear Strength of Unalloyed Pu under Explosive Loading**


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Accumulation of the radiogenic helium in the unalloyed Pu crystal lattice and defects, which were induced by prolonged self-irradiation of the material during its storage, can result in lowering of its mechanical properties, particularly in shear strength. In addition to [1-2], the set-up of explosive experiments and the first investigation results with the samples of the prolonged actual aged unalloyed Pu are presented. The effect of self-irradiation during the 1 – 15 years on the kinetics of elastic precursor relaxation and change of Pu shear strength in the weak shock-waves were experimentally estimated.

**References**


Features of Microstructural Changes in Unalloyed Coarse-Grained Anisotropic Depleted Uranium after Shock-Wave Loading in the Range of 20-50 GPa at Loading Impulse Duration of 0.5-1.3 μs

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The set-up of five shock-wave experiments and the first results of microstructural investigations of the loaded and recovered samples are presented.

The high anisotropy and inhomogeneity of intragranular plastic deformation of blocks, differently oriented to the shock wave front, have been revealed. The completeness of this effect in stress waves of different amplitude $\sigma_{xx} = 20…50$ GPa and duration $\tau = 0.5…1.3$ μs is analyzed. The observed changes in microstructure and microhardness of coarse-grained depleted uranium are discussed. A feasibility of proceeding the polymorphous $\gamma$-$\beta$-$\alpha$-transformations in unalloyed uranium after its shock compression during the unloading is analyzed with allowance made for [1,2].

References

Structural Changes in Reactor Alloy of the Hastelloy Type during Long-Time High-Temperature Exposure

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A CrNi89MoNb nickel alloy of the Hastelloy type, which contained (mass %) ~0.05 C, 8 Cr, 11 Mo, and 1.4 Nb, was proposed as a promising constructional material for salt liquid nuclear reactors [1]. This alloy has a relatively large corrosion and embrittlement resistance at high temperatures. The goal of the present study was to determine structural changes, which take place in the alloy of the Hastelloy type during long-time exposure at 650-750 °C. If secondary phases precipitate at boundaries during long-time aging, one may predict proneness of the alloy
to intercrystalline corrosion and corrosion stress cracking. The alloy was studied in two structural states after high-temperature rolling and after quenching from 1200 °C. In the former case, the deformed alloy had a large density of dislocations and high hardness (HRB 110). In the latter case, the alloy contained coarser grains with a small density of dislocations, which was characteristic of the quenched state. The alloy hardness was HRB 82. From the Table it is seen that hardness changed little when the deformed alloy was annealed at 550-650 °C. Hardness decreased to HRB 94.5 during annealing at 750 °C. This was due to recrystallization. Hardness of the quenched alloy remained unchanged when it was held at 550 and 750 °C. Hardness increased from HRB 82 to HRB 89 when the alloy was held for 1000 hours at 650 °C. Aging took place at this temperature. Niobium carbides could only be identified among strengthening phases in the electron diffraction patterns. Some carbides were located as sparse chains at grain boundaries. This could have some adverse effect on corrosion and corrosion-mechanical resistance of the alloy. Dispersed carbides or intermetallics were not detected at boundaries of fine grains in the hot-deformed alloy. Therefore, one may think that the hot-deformed state of the Hastelloy surpasses its quenched state regarding resistance to intercrystalline corrosion and corrosion-mechanical cracking.

Table

<table>
<thead>
<tr>
<th>Aging temperature, °C</th>
<th>Aging time, h</th>
<th>0</th>
<th>20</th>
<th>50</th>
<th>100</th>
<th>500</th>
<th>1000</th>
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<td>550**</td>
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<td>650</td>
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<td>82</td>
<td>83.5</td>
<td>82</td>
</tr>
</tbody>
</table>

*Initial alloy after hot deformation. **Alloy after water quenching from 1200 °C (30 min).

This study was supported by the International Scientific and Technical Center (Project No. 1606).

References

VI. Facilities and Techniques of Experiment
Neutron Diffraction Studies of Internal Stresses in Structural Materials for Nuclear Reactors

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For investigations of internal stresses in structural materials various techniques of non-destructive testing have been used so far: x-ray diffraction, ultrasonic scanning, measurement of magnetic induction and susceptibility, Barkhausen effect etc. Unfortunately, all these tools have serious limitations. High resolution neutron diffraction has been adopted for solution of this problem about 10 years ago and has been widely expanded due to a whole series of important advantages in comparison with traditional methods. The most important of them are: the identification of internal stress deep in the sample volume (up to 2 cm for steel), high spatial resolution (about 1 mm at any direction), simultaneous measurement of average deformation in gauge volume and microstresses in crystal grains, the determination of the deformation anisotropy and stress distribution for each component of multiphase material, the possibility of studying both magnetic and nonmagnetic compounds.

At FLNP in Dubna specialized equipment for internal stress measurements in volume materials and details has been set up and tested. These studies are performed with the high-resolution Fourier neutron diffractometer (HRFD) at the IBR-2 pulsed reactor. Multi-axes goniometer for determination of the stress tensor, loading machine with 60 kN load, mirror furnace up to 2000°C are used for experiments. The minimal gauge volume is about 2×2×2 mm³, typical time needed for measurement of a diffraction pattern is around 2–4 hours.

At HRFD a series of experiments in main directions of this field has been carried out. For instance, the mechanical properties of austenitic steel samples at various applied load, including cyclic one, have been investigated. At different degree of low cycle fatigue the residual stresses and amount of the martensitic phase, which appeared as a result of the plastic deformation of the initial austenitic phase, were measured. Measurements of residual stresses in shape welded steel have been performed. The residual stresses, phase composition and mechanical properties of new prospective materials – Cu/W and MoSi2/SiC gradient compounds, Al/Al2O3, Ni3Al/Al2O3 composites etc. – have been investigated. The results of neutron diffraction experiments are used for optimization of manufacture processes for obtaining materials with needed mechanical properties.

Investigations of Thermomechanical Effect in Metals on Impulse Electron Accelerators EMIR-M and IGUR-3

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Set-up and experimental results of the measurements of the thermomechanical effect in metals carried out on the installations IGUR-3 and EMIR-M [1] are presented. These installa-
tions operated in the regime of electron beam output in the atmosphere. The fluences of electron energy on the spot of sample displacement were 5 - 8 cal/cm². The loading impulse profiles were measured by means of a quartz gauge, and the fluences of energy, by steel calorimeters of full absorption. Pressure amplitudes and values of mechanical impulses in the samples were calculated in accordance with the classical knowledge of generation and spreading of stress mechanical waves in metals [2-5].The experimental results for some materials with different $\rho$ and $Z$ are displayed in the table.

**Table**

Measured shock pressure parameters of metals under electron beam irradiation on EMIR-M and IGUR-3 (fluence of energy 1 cal/cm²)

<table>
<thead>
<tr>
<th>Parameter of stress pulse</th>
<th>Al (AMg6)</th>
<th>Ti (VT-1)</th>
<th>Fe (St.3)</th>
<th>Cu (M1)</th>
<th>Ta</th>
<th>Pb</th>
<th>Sn</th>
</tr>
</thead>
<tbody>
<tr>
<td>$P_{\text{max}} \times 10^6$, Pa</td>
<td>$17^{12}$</td>
<td>$11^{13,15}$</td>
<td>$26^{13}$</td>
<td>$25^{13}$</td>
<td>$23^{13}$</td>
<td>$25^{13}$</td>
<td>$13^{12}$</td>
</tr>
<tr>
<td>$t_{1/2}$, ns</td>
<td>$350^{70}$</td>
<td>$220^{130}$</td>
<td>$120^{130}$</td>
<td>$200^{130}$</td>
<td>$70^{115}$</td>
<td>$250^{130}$</td>
<td>$200^{120}$</td>
</tr>
<tr>
<td>$I$, Pa·s</td>
<td>5.9</td>
<td>2.4</td>
<td>3.1</td>
<td>5.0</td>
<td>1.6</td>
<td>6.2</td>
<td>2.6</td>
</tr>
</tbody>
</table>

It is seen from the table that smaller duration of irradiation pulse and higher average energy of electrons on the accelerator IGUR-3 in comparison with the accelerator EMIR-M resulted in increase both in the stress pulse duration and the magnitude of mechanical impulses.

**References**


**Research Center of Simulation and Modeling of Irradiation Phenomena in State Scientific Center of Russian Federation**

“Institute of Physics and Power Engineering”

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An opportunity of foundation of the Center for investigations by simulation and modeling of irradiation phenomena in the SSC RF IPPE is considered.
The objective of the foundation is to create the experimental and theoretical basis for a substantiation of a choice of irradiation resistant advanced materials for reaching ultrahigh nuclear fuel burn-ups and for providing the technical and economical effectiveness and competitiveness of future nuclear power engineering.

The research base of the Center consists of a set of ion and electron accelerators: EGP-15, EGP-10M, ILU-100, EG-2.5, EG-1, KG-2.5, KG-0.3, pulsing reactor BARS-6, diffractometer DIN-2PI at the reactor IBR-2.

Primary tasks of the Center are the following:

- Express assessment of irradiation resistance of candidate structural materials for various nuclear power facilities including fast reactors, VVERs, fusion reactors and accelerator driven systems (ADS) at design and beyond design irradiation doses.
- Accumulation of experimental data on microstructural changes, precipitation and radiation-induced segregation in structural materials under ion and neutron irradiation that is necessary for the further development of radiation damage theory.
- Modeling of the microstructure evolution and precipitation in alloys under irradiation and calculating energy and diffusion characteristics of point defects and point defect clusters using the methods of molecular dynamics.
- Studies of radiation-induced processes in dielectric materials under conditions of powerful pulsing and continues irradiation.

The main directions of fundamental and applied investigations in the field of radiation damage physics which are planned to carry out in the Center are presented too.

It seems that the organization of the Center in IPPE will compensate the loss of the accelerators of the Kharkov Institute of Physics & Technology (Ukraine) after disintegration of the USSR, and also will lead to an essential acceleration and to reduce the price of development of radiation resistant materials by means of carrying out the simulation of radiation damage using accelerators of charged particles (ions and electrons).

For the present the SSC RF IPPE possesses the necessary experimental base and personnel potential for making such a Center.

Scanning Probe Microscopy of Radiation Damages of Proton Absorbers

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Using of carbon materials in high intensity proton linacs can decrease radioactivity level in 10-100 times. As a result, operation such equipment will become more safety. To avoid degradation and spattering of carbon material it’s useful to cover it by metallic film.

This paper presents results of the study of radiation resistance of carbon materials (small-grained and boron modified graphite) which were covered by nickel film for using in high intensity proton linacs. The testing samples were exposed by proton irradiation with fluencies up to $2 \times 10^{18}$ p/cm$^2$. The angle of irradiation was varied from $0^\circ$ to $90^\circ$ concerning surface of sam-
The irradiated surfaces were investigated by scanning probe microscopy [1]. The examinations stability of carbon plates in conditions of proton beam radiation were carried out. The permissible levels of a fluency of protons for the test carbon materials were estimated. The vacuum testing has shown, that the materials are satisfactory for accelerators.

Radiation examinations showed that such structures «carbon material - nickel» have a small degradation for fluencies of particles loosed during 10 years accelerating process. As a result, the mentioned structures can be successful apply as proton absorbers.

References

Investigation of Behavior of Model Heat Producing Rods for Ignition Pulse Reactor under Pulse Neutron Heating at YAGUAR Reactor

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The report presents results of literature data analysis concerning investigation of behavior of model heat producing rods fabricated from enriched to 36% uranium. Test conditions were heating up to 1000°C during 1.5 ms and cooling with water stream (stream velocity – 5m/s).

Requirements to design of model heat producing rods and experimental measurement device (MD) for reactor investigations were settled.
Elaborated designs for model heat producing rods and MD, experimental parameters checking devices are described.
Possible accidents during carrying out experiments are evaluated. Technical methods of prevention are proposed.
Reactor YAGUAR was used for experiments on heating and cooling of model heat producing rod fuel velocity in real scale of time.

Miniature Scanning Tunneling Microscope for Intrareactor Investigations

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Construction and capabilities of designed and manufactured miniature scanning tunneling microscope for carrying out wide-ranging investigations inside and thermonuclear plants are described.
A head of the microscope has been specially designed for study and monitoring of surface degradation of the elements and units of thermonuclear plants. It is equipped with a transport device and a special drive for transporting in vacuum. The drive is controlled by either computer or manually. The studies are carried out both under shutdown and working conditions i.e. while the thermonuclear fusion is running in plasma. In both cases it is possible to study surfaces of working elements of nuclear and thermonuclear plants (including difficult of access zones) as well as to study the surface degradation of specimens, which are charged with the head of the microscope beforehand.

Controlling of the head and obtaining of the data is carried out by the computer and power sources, which are installed beyond a radiation effect zone. Depending on radiation level in the zone where the designed mini-microscopes operate the microscope and the electric cables can be reusable or used once.

Thus, scanning tunneling microscopy – high-resolution non-contact technique of study of surface – is now applied to surface degradation analysis inside the nuclear and thermonuclear reactors and other similar plants. Before, for the accurate study of surface degradation of the inner components of the thermonuclear plants little pieces (templates) were cut from the components or used the test-specimens.

**Monte Carlo Simulation of Neutron Yield from Be, B, Li and C Targets Irradiated with 10-MeV Proton Beam**

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The Monte Carlo simulation results of neutron yield from $^9$Be, $^6$B, $^6$Li, $^7$Li, $^{13}$C and boron carbide targets irradiated with 10-MeV proton beam are presented. The depth-distribution of energy deposition and neutron yield as well as energy-angle distributions of neutrons emitted by $^{13}$C and boron carbide targets are presented too.

Monte Carlo simulations are done using PRIZMA code developed at RFNC- All Russian Institute of Technical Physics to solve the problems of transport of photons, neutrons and charged particles in matter.

The presented results allow making the proved choice of a target design and material taking into account of thermal loading and energy-angular neutron distribution.

This research was spent in interests of the ISOL project of an intense source of radioactive ions (Isotopes Separator On Line) within the framework of SPES program (Study and Production of Exotic Species) which is being carried out in the Italian Institute of Nuclear Physics (Legnaro National Laboratories).
Ion Track Nanotechnology

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Among the currently developed methods of synthesizing various in morphology and properties nanostructures in solids, a special place is given to the so-called nuclear technology that includes a direction of studying the processes of formation and practical application of ionic tracks generated in solids at their irradiation by high energy ions. The particular structure of track fields allows one to prognosticate wide perspectives of their application, for example, in microtechnologies. The empty or nearly empty track fields can be filled by atoms of practically any sort.

Particular interest has recently appeared to the use of ionic tracks for the formation from various materials of one-dimensional nano- microstructures such as nanowires and microtubes, with the help of the so-called template technology. The replicas from etched tracks by way of a galvanic deposition in them of metal atoms are the first step for the development of such a microtechnology on the basis of track membranes.

In a number of cases of practical application, it is necessary to form a nanowire structure on massive substrates (the so-called metal microbrushes). It is connected with a necessity of considerable (tens - hundreds times) increase of the surface activity of the object, for example, magnification of radiating or immersing, absorptive or desorptive abilities.

Metal microtubes of strictly fixed sizes can find wide application for production of various sensor devices, in systems of differential gear transmission of anionic or cationic molecules, microcanisters for medicines, toxiferous and radioactive substances, etc.

Using the metal nanowires on a massive substrate, for example, as electrodes for electric erosion treatment, allows one to form metal nanoclusters on the surface of solids (metal, semiconducting, polymeric ones, etc.)

This investigation presents the results of the developments of methods for synthesizing metal nanowires and microtubes on the basis of track membranes as well as the formation of metal nanoclusters on the surface of various solids.

Neutrino Method of Fuel Burning-Out and Radiation Creep Rate Monitoring

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In the present work the essentially new method of restoration of each nuclear density component of nuclear fuel isotope structure and accordingly dynamics of their change during work of a reactor is offered. On an example of known experimental data the return task of neutrino diagnostics of a reactor active zone is considered. This return task decision, on the one hand,
allows to restore with high accuracy the current values of nuclear density of each of a compo-
nent of isotope structure of nuclear fuel, and with another - opens a real opportunity of creation
neutrino technologies of diagnostics of time evolution of isotope structure of nuclear fuel and
the reactor power in a mode on-line.

The correlation passive location algorithm structure of inside reactor division neutrino
sources is submitted. The developed signals processing algorithm structure is a basis for practi-
cal realization of neutrino sources passive location system. The description of such system and
its reception aerial is submitted.

The automatic system of the remote continuous control after the degree of fuel burning out
and fuel radiation creep rate is considered in view of fuel arrangement in a reactor active zone,
and consequently, diagnosing of its residual resource/service life with the help of model not ex-
cess of limiting burning out, model not excess loading - durability and models a parameter -
field of the admission. The radiation creep rate estimation will be carried out by a method of
Monte Carlo with the help of the simulation program using as entrance parameters of an estima-
tion of concentration of radiation point defects (vacancies and interstitials), formed by division
nucleus splinters. Estimations of concentration of radiation point defects turn out by the appro-
priate estimations of neutrino source activities. The complex of the simulation programs is de-
veloped, allowing to check up the developed algorithms and to specify a lot of parameters, for
example, such as an interval of digitisation of signals, a base time interval for construction of
discrete cross-correlation function and others.

Mass-Spectrometric Facilities for Isotope and Chemical Analysis of
Multi-component Mixtures

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Basic principles, technical characteristics, design philosophy, and working condition of the
special-purpose mass-spectrometric facilities for multi-component mixture isotope and chemical
analysis are presented.

The base of the developed devices is a time-of-flight mass-spectrometer (mass-reflectron)
built at a linear scheme. Method of recording and mathematical processing of a number of the
impulses of current coming from the detector operates in real time. It makes possible to identify
spectra with up to 1024 partial components. Developed apparatus is intended for solving ana-
lytical problems and for the automated optimized control of the technological processes.

One of the developed facilities is oriented to work with gas containing tritium mixtures. Its
resolution is 600; measured masses range is 1-50 a.e.m.; cycle analysis duration is 0.4 s. The
second facility is proposed to be used in the experiments with transuranium elements and corre-
sponding technologies. Its resolution is 500; measured masses range is 1-600 a.e.m.; isotopic
sensitivity threshold by uranium is 5·10⁻⁶; cycle analysis duration is 0.1-0.3 s.
Developed devices combine:

1. high sensitivity in a wide range of the measured masses
2. measuring rapidity,
3. design simplicity,
4. working flexibility,
5. compactness,
6. compatibility with the external nets,
7. lower cost compared with foreign analogues.

Automated Hydrogen Measuring System at IVG.1M reactor

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System is dedicated for real-time registration of hydrogen (and its isotopes) concentration in two volumes of experimental device during irradiative material testing experiment at IVG1M reactor.

System allows conducting of automated simultaneous real-time sampling and registration of chemical composition of gases in operational volumes of two chambers of experimental installation (in inlet and outlet sides, for example). That is especially important for experiments on study of diffusion parameters (with method of hydrogen permeation, for example).

Measurement part is based on mass-analyzing radio-frequency gouges of omegatron type RMO-13 and MX6407-P (for light masses) with appropriate set of hardware-software tools. Original programmable oscillator of omegatron operational frequency and programmable high-voltage ramp for MX6407-P deflection system were designed with purpose to provide computer control for both mass-analyzers. CAMAC interface is used to link measurement system to IBM PC.

System provides possibility to measure wide mass-spectrum in a chamber under irradiation as well as simultaneous measurement of concentration changes of up to four masses in one chamber and registration of spectrum of light masses (2-6) in second chamber. Also it allows operation in data analysis mode when measurements are finished. Selection of operational mode, set-up of measurement duration, sampling frequency and data analysis are provided by graphic terminal of IBM PC.

System supports data acquisition and processing during four-hour reactor power session with following technical characteristics:

- Number of measure channels – 2 (one-channel operation is possible);
- Input signal – analogous, $10^{-5}$ - 10.0 V voltage;
- Channel sampling frequency – up to 0.1 Hz

Software is functioning in environment of operational systems MS-DOS or Windows (in DOS emulation mode). Post-measurement data processing provides visual analysis and filtration of measured arrays.
Described automated system is used for investigation of hydrogen- and tritium-permeation parameters of F82H and MANET stainless steels and vanadium alloys within the frame of international program of ITER-DEMO Thermonuclear Reactor.

**Calorimeter for Intense Electron Beams Dosimetry**

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One of the ways for loading materials with strain pulse is exposure of them with a high-power electron beam by duration 25-100 ns. To calculate the resulted thermal pressure it is required to measure the energy density value of falling electrons.

In [1] the construction for the unit of total-absorption calorimeters is developed (see Figure), which allows conducting measurements of the value of electrons energy density above 200 J/cm² in the center and at the periphery of the sample without its damage.

To use the developed structure for diagnostic purposes, the thermal characteristics of calorimeters are assessed in the given paper and the influence of electron spectrum, firms and calorimeters sizes on the measurement results is studied. Moreover, the independence of measurements of calorimeters warming-up on sample material is provided with protection against the reflected electrons with shields 7 and 8.

To determine the energy share absorbed in calorimeter material, \( \chi \), with Monte Carlo technique by MCNP4B code the calculations of electrons transport are conducted in the bodies with a form and sizes fit with the calorimeter construction. It is shown that the share of absorbed energy is mainly determined by lateral dimensions of calorimeters and average energy of electrons in a beam, and it is weakly dependent on a form of energy spectrum.

Analysis of thermal processes in the calorimeter construction after it have been heated with electronic beam and then cooled allows determining of the calorimeter average temperature \( <T_0> \), which, in its turn, is associated unambiguously with the value of electrons energy transfer in the beam by a ratio

\[
W = \frac{h \cdot d}{\chi} \left( H(<T_0>) - H(T_{am}) \right),
\]
where \( h \) – calorimeter thickness, \( d \) – steel density, \( H(T) = \int C(T)dT \), \( C(T) \) – iron heat capacity, \( T_{am} \) – ambient temperature.

The proposed calorimeter construction is many a time applied in experiments. Its use enabled to study the spall phenomena in copper and aluminum alloys, to measure the value of Gruneisen coefficient of pyrocarbon [2] and so on. The construction seemed to be serviceable both as for hardness to electron beam and for using it as a separate single node.

References

Implanter for Ions of Hydrogen Isotopes Based on Accelerator of NG-12 Device


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The results of updating of the neutron generator accelerator NG-12I are stated for its use as an implanter for ions of hydrogen isotopes (H\(_1^+\) and D\(_2^+\)) into silicon wafers. Ions energy is 175-210 keV, beam current is up to 10 mA [1]. For updating aims the 90° track for beam transportation is used, with the installed devices as scanning module, the ion pipe, the measurement module and the module for the wafer fastening.

For wafers exposure the system of ion beam scanning in two perpendicular-reciprocal directions is assembled and perfected. Currently the system enables to exposure the wafers of 100 mm diameter; in perspective – up to 200 mm. The uniformity of wafers exposure is perfected with using calorimeters of pyrocarbon. The recording and control system for exposure uniformity by a wafers perfected with using 21 calorimeters. The obtained non-uniformity at beam current of 0,35 mA is approximately ±10%. The approbation of exposure mode of current 1 mA is conducted, but for practical purposes the modes with current above 0,35 mA have not however been perfected yet.

Currently the device enables to exposure the wafers with protons and deuterons within wide temperature range. With that purpose the related calculations are conducted and the special constructions are developed for the silicon wafer to be fastened, that enables to change the temperature of the pad, on which the wafer is placed. Under different circumstances more than 90 silicon wafers were exposed. The trial bonding of the implanted wafers with oxidized ones for getting silicon – on – insulator-plates (SOI-plates) is performed. The obtained transport area of film is ≈100%.

References
Proton Accelerator-Based Intense Source of Radioactive Ions
(project approved by ISTC)


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The proposed project is aimed at the R&D study of a radioactive ion source in the framework of the SPES program at LNL. LNL has proposed a double-acceleration design for production of ISOL-type RIB (Radioactive Ion Beam). A primary proton beam (with the energy up to 100 MeV, average power up to 300 kW, diameter 1 cm) accelerated in the superconducting linac is directed to a special neutron target and produces an intense \(3 \times 10^{14} \text{ cm}^{-2} \text{s}^{-1}\) flux of fast neutrons. The obtained neutron flux is directed to a hot thick target made of \(^{235}\text{U}\) compound. The vapor of radionuclides ionized, extracted from the target with the energy of 20-60 keV, and then separated into isotopes in the experimental zone for low energy experiments or further acceleration up to the energy 1-5 MeV/n.

The problems of source development are discussed and the way of its production is proposed.

Source of High-Energy Resonance \(\gamma\)-Rays Based on Proton Storage Ring with Electron Cooling

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Possibility of resonance \(\gamma\)-rays source with energy 9.172 MeV (nitrogen line) is discussed. \(\gamma\)-rays are produced by excited \(^{14}\text{N}\) nuclei obtained when nuclei of \(^{13}\text{C}\) captures the proton. Design of this source based on proton storage ring is proposed. The reaction takes place on thin inside \(^{13}\text{C}\) target. On the basis of proton beam heating compensation we propose to use electron cooling. The estimations of various source systems parameters are presented. It is shown this design is more effective than design based on direct accelerator.

Elastic-Plastic Crack Propagation under Irradiation and Cyclic Loads Caused by Thermoacoustic Instability of Heat-Carrier in Active Zone

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In [1] conditions at which occurrence thermoacoustic instability of heat-carrier (TAI) in an active zone of some reactors is possible, in particular for water-water reactors (VVER installations of reactor) VVER-1000 they are determined. Occurrence TAI at a reactor normal exploita-
tion (including transition periods) results to that thermal emission element’s (TEE’s) are thus under influence of cyclic dynamic loadings which amplitude on tentative estimations makes 0.5 kg/mm\(^2\) \div 2.0 kg/mm\(^2\), and frequency – 50 Hz \div 100 Hz. In metal uranium TEE’s (both in the uranium core, and in a protective environment) thus there is an elastic - plastic development of cracks. In [2] the generalized diagram of radiation-thermal creep of the metals, showing dependence of creep mechanisms on thermodynamic parameters such, as temperature, loading and neutron flux density is submitted. In [3] it is shown, that in metal uranium at moderate temperatures and high neutron flux densities the radiation point defect concentrations considerably may exceed thermally equilibrium, also it is submitted the theoretical model of the established radiation creep within the framework of the mechanism of dislocation gliding and climbing, based on the conception of a dislocation as nonideal sink for point radiation defects. The similar phenomena is necessary to expect and in a material of a protective environment which because of close contact to uranium also is exposed to intensive operation of division nucleus splinters of uranium. Computer modeling of radiation creep of metal uranium fuel has revealed its temperature nonlinearity [4]. These temperature nonlinearity have served the reason for search of nonlinear effects and modes (in particular, concentration waves of point defects and their influence on radiating creep) in open nonlinear stochastic system which the system metal uranium fuel under an irradiation [5] is. As mechanisms of radiation-thermal plastic deformation in top of a crack, are identical to mechanisms of radiation-thermal creep the changes of mechanisms of radiation-thermal creep received in [2-5] law on thermodynamic parameters may be the basis for development of the appropriate mechanisms of elastic - plastic development of cracks in metal uranium fuel.

In the given work for VVER installations of reactor with perspective metal uranium fuel are determined possible areas of thermodynamic parameters (temperature, loading and neutron flux densities) at which occurrence TAI, causes elastic - plastic development of cracks with possible course of plastic deformation on mechanisms to the similar mechanisms offered for radiation-thermal creep.

References
Reactor Antineutrino Cross-Correlation Spectrometry

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In [1] importance of studying of reaction of return $\beta$-disintegration on a proton is marked. It is caused by necessity of the decision of some the important problems as fundamental (structure of weak interactions, neutrino oscillations etc.), and applied value (the control of a nuclear reactor being the most powerful antineutrino source). In [2] it is shown, that for the neutrino control of reactor fuel isotope structure the measurement of reactor antineutrino power spectra is necessary. In the same place the basic opportunity of a correlation passive location antineutrino sources of division reaction with the help cross-correlation signal processing of two spatially carried antineutrino detectors (an estimation of a delay of useful signals on a maximum of cross-correlation function) for the first time is shown. In [3] it is emphasized, that as the length neutrino oscillations depends on its energy the neutrino energy measurement is necessary at their detecting, otherwise the average on energy transition probabilities are accessible to measurement only.

In the given work algorithms of a cross-correlation method of detecting and measurement of reactor antineutrino energy spectrums are submitted. The method allows to carry out measurement of antineutrino energy spectrums at their detecting. The developed method is actual, as it is necessary for the decision of some the important problems both fundamental, and applied character. In particular it may be harmoniously combined with the developed signal processing algorithms of cross-correlation system of passive location neutrino sources.

References

Development of Measurement and Governing Systems Meant for Study with Hydrogen Isotope Mixtures

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This article gives a review of developments of equipment and systems of measurements and governing of facilities meant for studies conducted with hydrogen isotope mixtures. Requirements and approaches to development of such systems are analyzed.

The paper describes an automated system of control and governing of the complex of preparation of gas mixture of “TRITON” facility meant for carrying out of experimental re-
searches of muonic catalysis of fusion nuclear reactions in the ternary mixtures of hydrogen isotopes H/D/T in the broad range of temperatures and pressures.

The automated system of control, governing and acquisition of data of “Prometey” universal stand meant to study the phenomena of accumulation and transmission of hydrogen isotopes by metals and structural materials is also described.

**Synthesis, Study and Application of Aza- and Diazabicyclononanes as Complexing Agents and Preparations for Radiation Hygiene and Medicine**


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New fundamental methodological principles as well methods for the directed synthesis of skeleton aza- and diazabicyclononanes using diazo-compounds, norbornenes, 1-mono- and 1,3-dinitropropane derivatives have been developed.

The ways to create significant structural units and practically useful compounds have been proposed by means of simplifying the total chemical process owing to the unification of the stages of generating toxic and dangerous intermediate reagents and their transformation to 3-aza- and 3,7-diazabicyclo[3.3.1]nonanes. A convenient method for the one-stage synthesis of 1,5-dinitro-3,7-diazabicyclo[3.3.1]nonanes has been developed using the fourfold condensation of 1,3-dinitropropanes with formaldehyde and primary amines under Mannich’s reaction conditions. It was found, that the nature of amine effects significantly on the direction of the reaction.

\[
\begin{align*}
\text{R}^1 & = \text{H, Ph, 4-BrC}_6\text{H}_4, 2,4-\text{Cl}_2\text{C}_6\text{H}_3; \text{R}^2 = \text{H, Me, PhCH}_2, \text{CH}_2\text{CH}_2\text{OH, cyclo-C}_3\text{H}_5 \\
\text{H} & + \text{CH}_2\text{O} + \text{R}^2\text{NH}_2 \xrightarrow{60-83\%} \text{R}^1\text{N} - \text{N} - \text{R}^2 \\
\end{align*}
\]

Chelate complexes of the given compounds with Cu, Pd and Rh chlorides and acetates have been produced. It was shown, that 3,7-diazabicyclo[3.3.1]nonane fragment of molecule behaves as bidentate ligand forming stable complexes with transition metals.

Might selective extractants for hydrometallurgy and radiochemistry have been proposed using oligomer and polymer materials produced on the base of 3-aza- and 3,7-diazabicyclo[3.3.1]nonanes and their analogies.

In the series of 3-aza- and 3,7-diazabicyclo[3.3.1]nonanes three compounds have been revealed, which exceed antiarrhythmic drugs used at present in antiarrhythmic activity, toxicity and wide therapeutic effect.
VII. Some Aspects of Physics of Radiation Phenomena in Semiconductors and Insulators
Effect of the Nanoparticle Size and Structural Defects on Magnetic Properties of
Antiferromagnetic and Ferromagnetic Oxides

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Magnetic nanoparticles have attracted a lot of attention in recent years. The magnetic properties of bulk materials and nanoparticles may be different. Near surface the orientation of magnetic moments may be altered due to the loss of three-dimensional structural periodicity and broken exchange bonds. The surface and interface effects, such as the spin disorder and superparamagnetism, were found in the cases of magnetic metals and ferromagnetic spinels [1]. The understanding of the intrinsic properties of nanoparticles is of extreme interest not only from the fundamental point of view, but also for the technological application of the nanocrystal materials.

In this work we report study on effects of particle sizes, microdeformations and radiation defects on the magnetic susceptibility \( \chi(T) \) for nanocrystalline ionic compounds CuO and Mn3O4. The nanoparticle sizes were determined by means of scanning tunnelling microscope. CuO is a low-dimensional antiferromagnet, which transforms into 3D state at \( T_N=230 \) K. Near \( T_N \) a maximum of the susceptibility typical for 3D-antiferromagnets is absent and only the slope of the \( \chi(T) \) curve changes [2]. Mn3O4 is a ferrimagnet with \( T_N=41.9 \) K and has the complicated magnetic structure. The triangular spin ordering of Yafet-Kittel type is realized below \( T=33 \) K. The temperature dependence of the reciprocal susceptibility \( 1/\chi(T) \) has a hyperbolic shape in the wide range of temperatures \( T_N < T < 400 \) K and obeys the Cure-Weiss law at \( T > 400 \) K [3].

The single-phase nanocrystalline samples CuO were prepared by two methods. The high-dense nanocermics with average grain size \( d=5-100 \) nm were obtained by the action of spherical shock waves. It has been found that the decrease of a grain size \( d < 70 \) nm leads to the increase of the susceptibility and the appearance of the contribution \( \chi \sim 1/T \) in the low temperature range \( T < 140 \) K [4]. At high temperatures \( T > 230 \) K the values of \( \chi \) are coinciding for all samples. After three years of storage the grain sizes in shocked CuO remain invariable. The disappearance of the contribution \( \chi -1/T \) may be due to a relaxation of microdeformations and transition of grain boundaries into equilibrium state. The loose nanopowders CuO with the particle size of 15, 45 and 60 nm were prepared by gas condensation and oxidation of copper. For nanopowder CuO the susceptibility decreases at increasing \( T \) in the range \( T=80-600 \) K. The correlation between susceptibility and nanoparticle size is observed for both series of nanocrystal samples CuO. Nanoceramic samples Mn3O4 with the minimal grain size \( d=0.06 \) \( \mu \)m prepared by the action of shock waves have a higher values of the susceptibility than polycrystals. At \( T=300 \) K the value of \( \chi \) decreases as the magnetic field increases.

Thus, in oxide nanomaterials the susceptibility is strongly influenced by the particles size, density and microstructure details of both core and surface. The observed magnetic properties of the nanocrystal samples CuO and Mn3O4 are explained by the breaking of exchange bonds for surface ions and the destruction of long-range order.
The work is supported by Russian Foundation for Basic Research, grant No. 01-02-96403 and contract of the Ministry of Science and Technology №40.012.1.1.1153

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Quantitative Model of Irradiated Semiconductor

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Radiation processes complexity hampers creation of irradiated semiconductor quantitative model suitable for use in radiation technologies and in radiation resistance assessments. In spite of the data bulk they don't meet the main point viz. why under irradiation some materials obtain eigen conductivity and in other low-ohmic \(n(n^+)-\) or \(p(p^+)-\)zones form. Therefore at present basic way of researches is data storage on radiation defects (RD) characteristics that requires experiment bulk. In such a way radiation physics of Si has been developing. However in the main part of the compound materials these data are until fragmentary for imaging overall picture of radiation damages. It causes developing prognostic models of irradiated semiconductors.

It displays the model for quantitative analyses of electrical properties and for estimation of stationary (limiting) Fermi level position \((F_{lim})\) in irradiated semiconductors. In the model \(F_{lim}\) value is identified with a deepest defect level \(E_B\) of the crystal. The model allow for the fact that RD are deep states and so only integral characteristics of crystal energy spectra are important for them. It allows to calculate position \(E_B\) as a single energy level in semiconductors with analogous chemical bond type [1]:

\[
2 \sum_{a,b} l/(E_B - E_{ad})^3 = \frac{\partial^2 G_0(E_B)}{\partial E^2} = 0,
\]

where \(E_{ad}\) is energy spectra of the crystal, \(G_0(E_B)\) is Green function of the perfect crystal. It should note that the model of the deep level doesn't connect with some specific defect of the crystal. In the model just a single basic feature of the deep center is considered namely the most powerful localization of its wave function among other defect states of the crystal. This circumstance ensures weak sensitivity of this level both to a character of the defect and crystal matrix of semiconductor.

As is seen from the table values \(E_B\) are neighbours to experimental ones \(F_{lim}\) in irradiated semiconductors and their changes depending on material band-gap width are similar. On the base of research it can conclude that defect states, which levels close to the level \(E_B(=F_{lim})\), are the most steady in the crystal since they have the smallest generation energy. Consequence of this is: (1) generation processes of complexes of dopants and RD with deep levels in doped...
semiconductors that are well known for Si; (2) clusterization of RD during further annealing or high-temperature irradiation; (3) stability of deep defect levels to heat treatment.

The presence of the energetic gap in the band structure of the non-metallic materials (semiconductors, dielectrics) results in their high sensitivity to the various violations in the lattice structure (intrinsic lattice defects, dislocations, etc.). The lattice defects accumulation in these materials upon the high energy radiation results to the tremendous change of theirs electronic properties that is widely used in practical aims but at the same time it results the small radiation hardness of these materials in comparison with metals. The history of radiation effects investigation in such materials are usually based on the data about the electronic and microscopic nature of the defective states are generated in the crystal lattice upon bombardment. It requires a

### Table

Calculated values of minimum band-gap $E_g$, levels $E_B$ and experimental quantities $F_{sa}$ (in relation to valence band ceiling, eV) and equal limiting electrical parameters of irradiated semiconductors

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>1.20</td>
<td>0.47</td>
<td>0.39</td>
<td>$p$-type, $\rho=10^5$ Ohm-cm</td>
</tr>
<tr>
<td>Ge</td>
<td>0.78</td>
<td>0.26</td>
<td>0.13</td>
<td>$p$-type, $\rho=10^{16}$ cm$^{-3}$</td>
</tr>
<tr>
<td>AlSb</td>
<td>1.60</td>
<td>0.63</td>
<td>0.5</td>
<td>$p$-type, $\rho=10^6$ Ohm-cm</td>
</tr>
<tr>
<td>GaP</td>
<td>2.37</td>
<td>1.16</td>
<td>1.0 $\pm$ 0.2</td>
<td>$\rho \geq 2 \times 10^{13}$ Ohm-cm</td>
</tr>
<tr>
<td>GaAs</td>
<td>1.51</td>
<td>0.77</td>
<td>0.6</td>
<td>$\rho \approx 10^{6,3}$ Ohm-cm</td>
</tr>
<tr>
<td>GaSb</td>
<td>0.87</td>
<td>0.24</td>
<td>0.02-0.05</td>
<td>$p^+\text{-type, } p^\approx 3 \times 10^{18}$ cm$^{-3}$</td>
</tr>
<tr>
<td>InP</td>
<td>1.43</td>
<td>1.03</td>
<td>1.0</td>
<td>$n$-type, $n \approx (3-6) \times 10^{12}$ cm$^{-3}$</td>
</tr>
<tr>
<td>InAs</td>
<td>0.39</td>
<td>0.52*</td>
<td>0.52</td>
<td>$n^+\text{-type, } n \approx 3 \times 10^{16}$ cm$^{-3}$</td>
</tr>
<tr>
<td>InSb</td>
<td>0.14</td>
<td>0.09*</td>
<td>0.0</td>
<td>$p^+\text{-type, } p \approx 7 \times 10^{17}$ cm$^{-3}$</td>
</tr>
</tbody>
</table>

Thus under irradiation Fermi level of the crystal is shifted in the most deep state for the crystal that define evolution of semiconductor electrical parameters and its limiting electrical characteristics. It allows to predict processes of radiation modification and estimate semiconductor radiation resistance especially where there isn't reliable experimental data.

The work has been made under financing of ISTC (project #1630).

References

huge volume of the data about radiation-induced defects (RID) properties. Meantime, the nature and origin of these defects unknown in the most of materials. Therefore, main aim of these researches is to develop such approach, which can give a fundamental understanding of radiation-induced effects with scarce information about the nature and parameters of RID.

It is known that a common effect for non-metallic materials is the pinning of the electronic chemical potential (Fermi level) in a final (ultimate) position $F_{\text{ult}}$ upon irradiation. It enables to consider the radiation effects in these materials in the frame of the unified approach that gives the opportunity to predict theirs electronic properties after bombardment without the detailed information about the nature and the behaviors of RID. The present paper focuses on the general approach to the Fermi-level pinning in the great group (more than 20) semiconductors upon irradiation. The correlation between the electronic properties, the $F_{\text{ult}}$-position after irradiation and the peculiarities of the band spectra of the crystals are revealed. These correlation were analyzed using some theoretical models: (1) the model of the radiation "compensation" [1], (2) the charge (local) neutrality models [1,2]; (3) the model of the "deepest" defective state [3,4]. The calculations obtained proved good correspondence with experimental data in the irradiated semiconductors and explains the correlation between different theoretical approaches to the problem of the Fermi-level pinning in these materials. The data are presented here can be used for analysis the general totality of the experimental results in the irradiated semiconductors and for prediction theirs electronic properties after high energy bombardment.

References

$^{11}$B and $^{25}$Mg NMR Estimates of Electron Density-of-States in Superconducting MgB$_2$ Disordered by $n^1$-Irradiation

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The influence of structural disorder on the electron states in conduction band of MgB$_2$ is question of great interest both in physics and technology. As known high-pressure technology in sintering of the compacted MgB$_2$-materials results in stressed crystal structure accompanied by decrease of $T_c$ and broad superconducting transition in comparison with as grown at ambient pressure crystals. Radiation disordering by neutrons is probably the purest method to study the influence of the induced structural disorder on the physical properties regarding to the motion of carriers in conduction band. As shown in [1] superconducting temperature $T_c$ drops below 10 K whereas the initial crystal structure is preserved under $n^1$-irradiation up to the fluence of thermal neutrons $\Phi = 10^{15}\text{cm}^{-2}$. The moderate irradiation leads to anisotropic expansion of the crystal lattice with increase of the c/a ratio resulting in increase of the interlayer distance. The Riet-
veld analysis refinement of X-ray diffraction patterns yields some decrease in the occupation number at Mg-sites.

NMR line shift and nuclear spin-lattice relaxation rate $T_1^{-1}$ of $^{11}$B and $^{25}$Mg were measured in superconducting MgB$_2$ ($T_{c\text{out}}$=38 K) structurally disordered by nuclear reactor neutrons up to the fluence of thermal neutrons $\Phi = 1 \times 10^{19}$ cm$^{-2}$. The temperature of superconducting transition was shifted down to $T_{c\text{irrad}} = 7$ K under irradiation. The change due irradiation in the partial electron density of states (DOS) at the Fermi energy of boron and magnesium were traced by taking into account that $T_1^{-1}$ of $^{11}$B and $^{25}$Mg are determined by hyperfine magnetic interactions with carriers. According the NMR data obtained
- the partial DOS of s-states is not influenced by disorder;
- substantial decrease in DOS of 2p-states occurs under irradiation;

The NMR estimates for DOS using the McMillan formula show that critical temperature decreases in irradiated MgB$_2$ mainly due to reduction in the partial DOS of p states of boron.

References

Long-Lived Radiation Defects on Implanted Silicon

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The existence of radiation defects on implanted silicon surface was discovered by means of recently developed technique, based on measuring of optical constants connected with thermal conductivity change. These defects alter optical constants (complex index coefficient, reflection coefficient) of irradiated silicon surface, and then during the storage of irradiated sample these alterations relax over a long period of time: up to month after implantation.

Investigations were carried out allowing to ascertain that long-term relax does not connect with absorption–desorption processes, and that it is defined by structural alterations of irradiated silicon layer and long-term processes of reconfiguration.

Investigations of observed effect were carried out for ion species, which are most frequently used in silicon implantation: boron, phosphorus, and arsenic, also for ions, neutral by transistance, in particular, argon.

Well-founded conclusions about observed effect nature were stated.
Investigation of Defectiveness of Nanocrystal Ceramics CuO by Positron Annihilation Method


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The unique properties of promising materials such as radiation-modified and nanocrystal ones, are defined by their structure and specified set of defects. The positron annihilation method is the effective tool to investigate structural and electronic properties, in particular, vacancies and their agglomerates, microvoids and changes in the electronic structure associated with defects. In the current paper the high-density nanoceramics and CuO microceramics with the size of crystallites of 15-90 nm and 5-15 \( \mu \)m, correspondingly, have been investigated by the method of angular correlation of annihilation radiation (ACAR). The CuO nanoceramics has been produced by loading of the blank of CuO microceramics by spherically converging shock waves [1-3]. The loading has been provided by detonation of the layer of RDX-based composition of thickness \( h_{\text{ex}} = 8 \text{ mm} \) at the surface of the spherical sealed casing made of 12C18N10T, with the inside CuO sphere with the initial diameter of 49 mm and density of 70% of the theoretical one. The sizes of crystallites of the compressed core material after its meridian cut has been estimated [3] by means of the scanning tunnel microscopy, as well as from the broadening of X-ray diffraction lines.

Based on ACAR spectra produced in the course of the current work, the pulse distribution of CuO valence electrons is shifted to rather high values as compared to these of metallic copper, that is associated with the chemical bond nature, in particular, with Cu 3d – O 2p hybridization. The defectiveness of nanoceramics produced in the process of explosive processing is essentially higher than in the case of initial CuO microceramics produced by static pressing with subsequent sintering. The nature of nanoceramics defectiveness is defined by the high concentration of oxygen vacancies and/or their agglomerates grouped on crystallite boundaries. The large free volumes (microvoids) have not been revealed, that is consistent with the high density of nanoceramics. To estimate the region of the temperature stability for the produced nanoceramics, its isochronous isothermal annealing has been provided at atmospheric pressure. The increase in sizes of crystallites starts at temperatures higher than 400\(^\circ\)N. According to ACAR data the defectiveness of nanoceramics begins to change from about 300\(^\circ\)N up to the beginning of recrystallization and growth of crystallites [4].

The work has received support of RFFI and the Government of Sverdlovsk region, grant 01-02-96403, as well as FCNTP – 40.012.1.1.1153.

References
Neutron Diffraction Studies of Structural State of Mn₃O₄ after 
Shock Wave Loading

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In some of papers a change in the valent state is pointed out as well as the cause of the oxide 
reduction when irradiated by charged particles and/or loaded by shock waves [1]. The shock-wave 
mechanism is invoked to explain the emergency of defects in the process of irradiating the solids by 
ions at depths considerably greater than the proposed ones [2]. In the current work the neutron dif-
fraction studies of Mn₃O₄ ceramics from the spherically explosive-compressed and recovered comp-
act have been carried out to clarify the nature of damages of Mn₃O₄ crystal lattice following the 
shock wave loading. Mn₃O₄ has a spinel structure and represents a ferromagnetic with Neel tempera-
ture of T_N=41.9 K. The shock-wave loading has been provided by means of detonation the PETN-
based high explosive composition layer with thickness h_HE=5 mm at the surface of the spherical 
sealed casing made of 12C18N10T with the inside blank fabricated from statically pressed and sin-
tered Mn₃O₄ microceramics with the initial diameter of 49 mm and density 70% of the theoretical 
one. The loading parameters as to amplitude and length have been somewhat lower than ones real-
ized in [3]. As a result of spherical shock-isentropic compression the single-phase high-density 
(98%) ceramics has been produced with the size of crystallites of 0.06-2 μm. The diffraction lines of 
the loaded Mn₃O₄ are in a tetragonal correspondence to the damaged spinel and have been broad-
ened as compared to the initial powder, that is associated with small sizes of crystallites and emerged 
internal stresses. At small angles on the diffraction patterns the wide anomaly is observed, which is 
associated, possibly, with the short-range magnet order in the compressed material. The parameters 
of the explosive-loaded Mn₃O₄ lattice are close to the parameters of the initial powder and are as 
high as a = 5.7457, c = 9.4318 Å. The processing of neutron-diffraction patterns by using the pro-
gram FULLPROF has shown that in the explosive-loaded Mn₃O₄ the anionic sublattice is the most 
defective. If in the initial Mn₃O₄ the filling of the oxygen sublattice is somewhat greater than the 
stoichiometric value, then in the loaded sample this value is as high as 3.9. This corresponds to some 
reduction of the oxide and to the increase in the concentration of Mn²⁺ ions. As a magnet moment of 
Mn²⁺ is higher than that for Mn³⁺, the magnetic susceptibility can be increased, as it has been ob-
served in paper [5]. The similar partial reduction of CuO oxide in the course of shock-wave loading 
is pointed out in paper [1].

The work has received support of RFFI and the Government of Sverdlovsk Region, grant 01-
02-96403.

References

Development of Compacting Technology and Processing of Non-Metal Materials Using Spherical Explosive

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The goal of this work is the development of the new method for obtaining the high-density nanocrystal (with crystals of nanometric size) ceramics based on oxides, nitrides and others non-metal materials by means of spherical shock-wave loading. These nanoceramics have high mechanical properties (density, strength, plasticity) and also series of the interesting physico-chemical properties being candidates for practical usage.

The spherical explosive systems developed in RFNC-VNIITF [1] to process the materials are the foundation of this method. Shock-wave loading performs by the explosion of HE layer on the surface of the spherical sealed casing with the initial material inside. Initial material is a statically pressed ingot of microceramics with the density of 70% from theoretical. In the process of the shock-wave loading of microceramics it is compacted up to 99% from the theoretical density and as result of compression and shear deformations we obtain the nano-scale material structure, which is regularly changed along the radius of compact being obtained. Integrity of the sealed casing during the explosive loading is saved. By means of this method [1] copper oxide (CuO), manganese oxide (MnO₃) and some manganites (LaMnO₃) nanoceramics were obtained. The density of nanoceramics is up to 99% from theoretical. Sizes of crystals are 0.01-0.2 μm. Microhardness increases considerably. Copper oxides are a base of the high-temperature superconductors and some applications in the area of catalysis and gas sensors. Manganites are candidates for spin electronics usage.

The advantages of the suggested method are: 1) combination of obtaining the nanocrystal structure and the material compaction in one technological process; 2) high purity of nanoceramics; 3) possibility to produce the nanoceramics with average size of crystallites varying along the compact radius, that makes it possible to obtain in one explosive experiment and to choose the material with the optimal combination of its operating properties and with required crystallite sizes. The developing method can be used for promising nanocrystal materials in the field of catalysts, high hardness and high recrystallization temperature materials and also some functional materials.

The work is performed in the framework of the RFFI Project 01-02-96403 “Ural”.

References


112
Effect of Indexed-Spectrum and 14-MeV Neutrons on Arsenide-Gallium Schottky Field-Effect Transistors

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In the present work, the effect of indexed-spectrum and 14-MeV neutrons on evolution of characteristics of power 3P602G-2 Schottky FETs was investigated. Measurements were carried out on the basis of YAGUAR reactor and NG-12I neutron generator.

As measurements have shown, the parameter most sensitive to neutron action is threshold voltage which decreases with fluence increase.

Since the value of threshold voltage $U$ is proportional to concentration of dope $N$,

$$\frac{\Delta U}{U} \approx \frac{\Delta N}{N}. \quad (1)$$

Assuming that the removal of charge carrier is related to formation of defect clusters, and this effect prevails, then:

$$\frac{\Delta N}{N} = \frac{\Delta U}{U} \approx N_k \cdot v_k = a \cdot v_k \cdot \Phi_n,$$

where: $N_k = a \cdot \Phi_n$ – concentration of clusters; $v_k$ - volume of a cluster, that proves to be true in experiment.

Proceeding from the experimental data and data given in [1], we obtain an estimate of the radiation-induced defect cluster radius (~70 nm).

Clusters concentration increases approximately 2.6 times under irradiation with 14-MeV neutrons.

With a view to analyze the results of the experiment, the size and form of collision cascades(clusters) emerging in GaAs under neutron irradiation were calculated. For calculation, the program TRIM based on Monte-Carlo procedure was utilized [2].

As a result of calculations it was found that the defect clusters are 70-150 nm in size, which weakly depends on the energy of neutron irradiation in the range of 105-107 eV. Clusters concentration grows 3-4 times with increase in neutrons energy from 1 to 14 MeV.

References
Effect of Reactor Neutron Irradiation on InP Single Crystal Structure

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The method of precision measurements was used to study the effect of reactor irradiation (full neutron spectrum with $\varphi_t/\varphi_b \approx 2$) on InP single crystal lattice spacing. Initial InP samples were undoped ($n=1.8 \times 10^{16} \text{ cm}^{-3}$) and Te-doped up to $n=3.9 \times 10^{18} \text{ cm}^{-3}$.

Figure 1 shows the dependence of lattice spacing on carrier concentration in the initial material irradiated with thermal neutron fluence of $F_t = 2.1 \times 10^{19} \text{ cm}^{-2}$. It should be noted that the change of lattice spacing is uncommon for the crystals with radiation defects. At small tellurium concentration lattice spacing decreases under irradiation. And just at $n=3.9 \times 10^{18} \text{ cm}^{-3}$ it increases as compared with the initial value ($\Delta a=5.9 \times 10^{-4} \text{ Å}$).

Figure 2 presents the dependencies of lattice spacing on thermal neutron fluence for InP single crystals with Te concentrations: 1 - $n=1.8 \times 10^{16} \text{ cm}^{-3}$; 2 - $n=4.0 \times 10^{17} \text{ cm}^{-3}$. It is seen that lattice spacing decreases with increasing neutron fluence, and this decrease becomes even more intensive with the growth of Te concentration. As regards high Te concentration ($n=3.9 \times 10^{18} \text{ cm}^{-3}$), it is obvious that properties of irradiated material are determined not only by the concentrations of tin atoms introduced by thermal neutron transmutation doping, generated radiation defects and initial Te concentration. It is the interaction character of these components that makes the main contribution. According to the estimates, an increase of lattice spacing will be an order of magnitude lower even if all Te leaves the solution because of solid solution degradation. The same is true about non-stoichiometry defects. It is evident that residual concentration of radiation defect interstitial components increases at high tellurium concentrations in the initial material. At low Te concentrations vacancy-type defects are predominant.

The work has been done in the frame of ISTC Project #1630.
Silicon carbide (SiC) – is a perspective material of extreme electronics capable to maintain serviceability at high temperatures and levels of irradiation. There were explored 6H-SiC p-n structures received in PTI Ioffe by the method of sublimation epitaxy of p' stratum on the basis of n' Lali substrate under vacuum. P-n junctions were grown on SiC face (0001), with Al (p-type) as a dope. Concentration of non-compensated impurity in p' stratum was \( N_a-N_d = 5-7 \cdot 10^{18} \text{ cm}^{-3} \), and in n' substrate, \( N_d-N_a = 3 \cdot 10^{18} \text{ cm}^{-3} \). The thickness of epitaxial p'-type stratams was 1-2 microns, thickness of a n-type substrate, 400 microns. For investigation, fast pulsed reactor (YAGUAR) with \( \tau_u \approx 1 \text{ ms}, \) \( \bar{E}_u = 1.1 \text{ MeV}, \) and slowing-down radiation unit IGUR-3 \( \tau_u = 25 \text{ ns}, \) \( \bar{E}_\gamma = 1 \text{ MeV} \) were used.

It is shown, that contact potential difference under irradiation preserves its initial value \( (U_v \approx 2.6 \text{ V}) \). The angle of slope of characteristic C-V of the sample irradiated with neutrons is greater than that of non-irradiated, which points to increase in the degree of neutralization of the dope, i.e. generation of donor-like small defects by neutron irradiation. On the assumption of abrupt symmetric p-n junction, concentration of these defects was estimated at

\[
\Phi_n = 7 \cdot 10^{15} \frac{n}{\text{cm}^2}; \quad N_j = 3.6 \cdot 10^{17} \frac{1}{\text{cm}^3}.
\]

The dependence of ionization current on dose rate at \( \tau_u = 1 \text{ ms} \) (YAGUAR) is linear. At \( \tau_u = 25 \text{ ns} \) (IGUR-3) there exists a section of superlinear dependence for both the irradiated and non-irradiated samples. To all appearance, these features of the experiment point to existence of biographic and radiation shallow saturation carrier trapping centres with the recharging time of the same order of magnitude as radiation pulse duration \( (\tau_u = 25 \text{ ns}) \). For experiment interpretation, a simple model taking into account carriers entrapment on saturation and non-saturation levels was analyzed. Satisfactory agreement with the experiment at reasonable values of adjustable parameters was obtained.

The basic direction of development of microelectronics is the making a VLSI circuit on the basis of heterostructures. In them the active elements are formed in submicron layer of silicon isolated from a substrate by a layer of oxide.

These structures, having the highest extent of integration, speed and minimum consumed
power, are stable to action of neutrons. However, the presence of heterolayers puts under doubt the charging stability depended by accumulation of carriers in layers of oxide and on a heterolayers border.

In this work the initial data on charging stability of experimental submicron CMOS transistors, obtained in IFS SB RAN on technology Dela-Cut, are submitted.

The examination of influence of radiations on the drain-shutter character of the MOS transistors with different design data and at miscellaneous potentials on a substrate at the moment of an irradiation was represented.

Under irradiation, at the expense of accumulation of positive charge in subshutter oxide, there occurs decrease of threshold ($V_t$) of the n-channel and increase of $V_t$ of the p-channel transistor. Besides at the expense of accumulation of positive charge in the hidden oxide there is a n-type conduction in the channel on back border, that leads to opening of the n channel transistor.

In the work is shown, that the charging stability is higher for transistors with the smaller sizes of heterolayers. Besides, the changes of $V_t$ essentially depends on potential on a shutter and substrate, which are applied at an irradiation. Is shown, that the best version from a point of view of radiation stability, when at an irradiation on a substrate concerning datum is given negative bias.

The negative voltage, applied to a substrate after an irradiation, improves the performances of transistors, trying to return them to initial values (up to an irradiation). This effect is reversible.

Ge/Si Nanostructures with Quantum Dots
Grown by Ion-Beam Assisted Heteroepitaxy

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Recently the effects of low energy (~ 100 eV) ion irradiation on surface islands nucleation and growth were found under Ge molecular beam epitaxy (MBE) on Si(100) and (111) with pulsed self-ion beam action on the growing layer. The irradiation results in reduction of critical thickness of pseudomorphic Ge layer at which a transition from layer-by-layer to three-dimensional growth occurs [1]. Reduction of the average island sizes and their dispersion, and increase in their density are revealed [2]. Here we present the results of crystallinity investigation of Ge quantum dots embedded in Si structures grown under low energy ion irradiation.

The structures investigated were grown by MBE of Ge (of 4-10 monolayers thick) on Si(100), covered with Si cap layer. The three types of Ge layers were grown:

1) conventional Ge MBE on Si;
2) epitaxy under irradiation with continuous Ge+ ion beam;
3) epitaxy under irradiation with pulsed Ge+ ion beam, correlating in time with the completion of the growing monolayers at layer-by-layer growth mode.
The substrate temperature varied in the range of 300-500°C during Ge heteroepitaxy. The cap layer of 150 nm Si was grown at 500°C by the conventional MBE (no irradiation). Structures were analyzed by Rutherford backscattering/channeling technique with 1.2 MeV He⁺ ions. The backscattering yield for perfect crystal of Si(100) is about 3%. The perfect structure of 2.5% backscattering yield was found in a mode of a pulse irradiation for 1-5 monolayers deposited at temperature 350°C. For lower temperature 300°C the yield exceeded 5% in the similar structures. The enlarged yield was found also in the structures formed with continuous beam irradiation at 300-350°C temperatures. Transmission electron microscopy studies indicated defect-free Ge dots and Si layers for the initial stage of heteroepitaxy (≤5 monolayers) in pulsed irradiation growth mode at 350°C. Continuous beam irradiation was found to induce dislocation around Ge dots.

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References

Radiation Processes in Si Single Crystals

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Due to their high purity and perfection Si single crystals are the best material to be used as a model in solid state physics.

Study of generation and behavior of the point defects in Si with the modern techniques has led to the discovery of unexpected high mobility of vacancies and interstitials. Formation of different defect and impurity complexes and the dependence of the defect-impurity reactions on the intimate processes in crystals.

The systematic approach based on the dominating role of the defect-impurity subsystem extremely sensitive to external influences is presented. An attempt is made to explain such "long-term" problems as: diffusion, non-equilibrium, long-range effects, amorphisation.

We believe the presented approach is valid in its main parts for all solid states, naturally taking into account the specific properties of atoms and structure.
Properties of P-Channel Transistors on Silicon Gate Oxide Created Using BF$_2^+$ Ions for Drain/Source Areas

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Investigations of electrical parameters of p-channel MOS transistors gate oxide were carried out. During drain/source regions formation ion BF$_2^+$ was used. Investigations were carried out of implanted fluorine influence on electrical parameters of obtained dielectric layers by means of accumulating diagrams and Veibull distribution tracing for dielectric charge disruption definition. Implantation of ion BF$_2^+$ was carried out using different energies. As a result extrinsic defects in test MOS-structures gate oxide decreased three times irrespective of implantation energy. Energy level allowing breakdown current increase from 1 to 5-6 mA was obtained experimentally, effective charge of gate oxide reduced, wafer yield increased. Threshold voltage and gate oxide effective thickness increase needs additional investigations, which are realizing at present time.

Investigation of Solid-Phase Processes in Insulator and Semiconductor Materials under Influence of Powerful Electron Irradiation

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Results of the investigations of the processes proceeding in inorganic materials with ionic and covalent bonds under the influence of intensive high-energy electron flows heating the samples up to high temperatures are represented. These processes are called radiation-thermal (RT) ones. The investigations were carried out in the following directions:
- RT sintering and property modification of different oxide ceramics types;
- studying diffusion processes in model (alkali halide monocrystals) and real (ferrites) structures in powerful radiation fields;
- studying electroactivation of implanted semiconductors by radiation-thermal method.

The results obtained are following:
1) The effect of considerable sintering activation of various ceramic materials (i.e. Li-Ti ceramics, high-temperature superconducting ceramics, instrumental corundum-zirconium ceramics) has been established at compacts heating with electron beam. Thereby in comparison with simple thermal annealing the decreasing of sintering temperature by about 200 °C was reached. The radiation activation mechanism of compacting powder materials has been suggested. Data on features of structure and functional characteristics formations of the ceramic materials at RT-sintering of oxide powders and at RT-modification of samples sintered ther-
mally have been represented. The RT-influence was shown to promote phase homogenization of complex oxide ceramics, ferrite ceramics production with minimum level of mechanical stress and defects. The ceramics made by the RT-technique was characterized with improved operational properties.

2) As a result of studying cation impurities diffusion in potassium bromide by using SIMS, it was shown that the intensive electron irradiation didn't affect on high-temperature diffusion of isovalent sodium impurity, but intensified heterodiffusion of multivalent $Al^{3+}$ and $Mg^{2+}$ cations. The temperature dependences of diffusion coefficients, which were determined for the cases of thermal and radiation-thermal annealings, have been analyzed. The analysis of possible mechanisms being responsible for intensification of the investigated diffusion process has been done.

The investigations on affecting powerful electron beam on oxygen diffusion in the polycrystalline Li-Ti ferrites have been performed. The characteristics of oxygen diffusion in non-stoichiometric ferrites were determined by using the original method based on measurement of electrical conduction activation energy. This method allows determining coefficients of grain boundary $D_b$ and volume $D_v$ diffusions. It was shown that in comparison with the thermal annealing the RT one led to increase of both the grain boundary and volume diffusion coefficients. Thereby coefficient of the grain boundary diffusion changed most substantially. The nature of the effect has been discussed.

3) A comparative analysis of electrophysical parameters of ion-implanted structures made of various types of semiinsulating gallium arsenide was performed at different kinds of post-implantation annealings. The technique of annealing ion-implanted gallium arsenide structures by powerful electron beam, energy of which was higher than defect formation threshold, has been suggested. This method has more advantages than the other ones.

**Defect Detection in Binary Zinc-Blende Compounds - Experimental (part-2)**


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At the 2001 Ural-4 Seminar, we presented (Part-1) a theoretical analysis of the effects, defects in zinc-blende binary compounds produce in far IR and EXAFS measured spectra, and made a guess estimation as to the level at which such defects would be detectable.

Availing of the DAΦNE-L synchrotron intense far IR (inaugurated Oct. 2002) and x-ray (expected Dec. 2002) beams, we aim at 1)- checking the level of sensitivity of each of the two methods, 2)- comparing the quantitative effects induced with those predicted by the theoretical treatment.

Crystals of CdTe [a,b], HgTe [c], GaAs [b] both perfect [a] (PVT method), as well as with vacancies [a,b], with H-insite loading [a], with induced LE2 antisites [b] (with monitored content) are considered. Results obtained are meant to be presented.

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Authors Index

A
Abramenko S.I.  67, 75, 80
Abromeit C.  3, 23
Abu Kharroub Mitkhal   39
Afanasev V.N.  89, 97
Afanasev S.E.  39
Akhtyamova L.Z.  111
Alehin V.V.  32
Antoshina L.A.  63
Arbuzov V.L.  4, 5, 9, 47, 105, 110
Arbuzova T.I.  105, 111
Arkhipov V. E.  12, 21
Aryutkin V.S.  74
Asiptsov O. I.  77
Avilov M.S.  99
Azov A.Yu.  36

B
Babaev V.P.  11
Balachov I. I.  17
Balagurov A.M.  89
Baranov M.A.  20
Bateev A.B.  74
Bayankin V.Ya.  19, 20, 36, 51
Belozerov S.V.  79
Belyaev S.P.  18, 26
Berger E.F.  34
Binyukova S.Yu.  39
Blanter M.S.  67
Bleikher G.A.  29
Borodin O.V.  40, 45
Borschhevsky A.O.  86
Brudnyi V.N.  106, 107
Bryk V.V.  40, 45
Bryushkova S. V.  27
Bublik V.T.  114
Burattini E.  119
Bychkov V.  89
Bykov P.V.  19

C
Calvani P.  119
Cebulski J.  119
Chakin V.P.  79
Chekanov V.A.  18, 26
Chemerinskaya L.S.  54, 55, 56
Cherkasov V.I.  84, 85
Chernov I.I.  39, 41
Chernov M.V.  5, 10
Chernyavsky A.V.  118
Chizhikray V.V.  39, 42, 48, 96
Chineykin M.T.  92
Chuev V. V.  68
Chukalkin Yu. G.  21, 34
Constantinescu B.  70

D
Danilov S.E.  4
Davydov L.N.  53
Denisenko E.Yu.  118
Devyatko Yu.N.  5, 10, 11
Dmitriev S.N.  94
Dokichev V. A.  102
Dolinsky Yu.N.  47
Drozdov A.Yu.  20
Drozdovskiy A.  91
Druzhkov A.P.  5, 9, 47, 110
Dubinin S. F.  21, 30, 33
Dvurechenskii A.V.  116

E
Evseev A.L.  72
Evtikhin V.A.  82

F
Fedichkin I. L.  95
Fil’chagin S.V.  101
Filippov V.P.  74
Filyakin G.V.  72
Fortuna S.V.  30
Frangulian T.S.  118
Frantsuzov A.A.  98

G
Garas’ Yu.G.  92
Garner F. A.  8, 17, 21, 32, 71
Gerashenko A.P.  108
Gerasimenko N.N.  22, 109, 118
Gerasimenko N.N. (junior)  109
Gilmudtadinov F.Z.  19, 36, 51
Gizhevskii B.A.  105, 110, 111, 112
Glazkov V.P.  67
Gloushkova N. V.  73
Goloborodsky B.Yu.  54, 55, 56, 57
Golosov O.A.  71
Golubov S.I.  6
Goncharenko Yu.D.  72, 79
Goncharov I.N.  7
Gorbachev D.M.  84, 85
Gordienko U.N.  48
Goshchitskii B.N.  25, 34, 82, 108
Gotthardt R.  23
Grachev A.N.  85, 84
Grinyaev S.N.  106, 107
Groetzschel R.  116
Gromov V.T.  113, 115
Grummon D.S.  23
Gubin K.V.  99
Gubkin I.N.  82
Guidi M. Cestelli  119
Gulevich A.V.  90
Gushchina N.V.  54, 55, 56
Gutakovskii A.K.  116
Gyngazov S.A.  118

J
Jenkins M.L.  13

K
Kalachikov V. E.  73
Kalashnikov A.N.  39
Kalin B.A.  39, 41, 45
Kandiev Ya.Z.  93, 99
Karkin A.E.  25, 34, 108
Kawamura Hiroshi  42
Kazakov V.A.  72, 79
Kennhin E.A.  42, 48, 96
Kharlamov V.S.  11
Khmelevskaya V.S.  7, 63
Khmelnitsky D.V.  97, 48
Kinev E. A.  27, 28
Kirk M. A.  13, 24
Kir’yushkin S.V.  98
Kisiel A.  119
Klevtsov V.G.  42
Kobyliansky G.P.  74, 76, 78
Kolbaenkov A.N.  42, 48
Kolin N.G.  106, 107, 114
Kolotov A.A.  19
Konobeev Yu. V.  8, 28, 32, 58, 60, 90
Konopleva R.F.  18, 26
Konovalov L.N.  67
Kordyukov V.N.  75, 80
Korostin O. S.  68
Kosarev V.V.  92
Kositsyna I.I.  86
Kostenko B.F.  7
Kostromina N.V.  110, 111
Kot N.Kh.  99
Kozikov A.N.  98
Kozlov A. V.  27, 28, 77
Kozlov E.A.  86, 105, 110, 111, 112
Kozlov E.V.  30
Kozlovsky A.V.  95
Kozmanov E.A.  71
Kozodaev M.A.  91, 92
Krivobokov V.P.  29
Krushinsky T.N.  115
Kuchin A.G.  34
Kulikov D.V.  11
Kulinich Y.A.  92, 102
Kulish V.G.  40
Kulsartov T.V.  39, 42, 48, 96
Kupriyanov I.B.  79
Kurdyumov A.A.  43
Kuri'chik E.V.  36
Kurochkin A.V.  98
Kuryakin A.V.  101
Kurzina I.A.  30
Kuzina T.L.  71
Kuznetsov A.R.  3, 35
Kuznetsov E.V.  118
Kuznetsov V. R.  80
L
LaGrange T.  23
Latypov R.N.  79
Laushkin A.V.  67
Lazarev N.E.  11
Lebedev A.A.  115
Levakov B.G.  92
Litvinov B.V.  75
Litvinov D.A.  94, 101
Logatchev P.V.  99
Loginov B.A.  92
Lokhtin L.N.  67
Lukin A.V.  98
Lukin G.V.  93
Luzgin A.V.  55
Lyasota A.M.  75, 80
Lyasota I.A.  47
Lysitsin G.Yu.  92
Lyublinskiy I.E.  82
M
Magda E.P.  75, 92, 98, 102
Makeev O.N.  91, 92
Malakhov A.A.  93
Malkov I.L.  42
Maltsev V.V.  68
Malynkin V.G.  7
Malyshkin G.N.  93
Marcelli A.  119
Marchenkov V.V.  12
Markelov N.N.  84, 85
Markovsky S. N.  95
Martyshkin P.V.  99
Mashinin V.A.  99
Medvedeva E.A.  71
Mel'nik I. A.  30
Meldner R.R.  79
Mendeleva Yu.A.  36
Merkurisov D.I.  114
Mikhalev K.N.  108
Modestov D.G.  93
Mokichev G.V.  98
Möller W.  54
Morozov A.N.  40
Morozov S.N.  99
Mösling A.  81
Moyzes B.B.  118
Mukovskii Ya. M.  21
N
Nakamichi Masaru  42
Naumov S.V.  105, 110, 111, 112
Nazarkin I.V.  18, 26
Neklyudov I.M.  3, 40, 45
Neustroev V.S.  75
Nikitin A.V.  36, 51
Nikolaev A.L.  52
Nikolaev Yu.A.  58
Nikulina A.V.  78
Novoselov A.E.  76, 78
Nucara A.  119
O
Obolensky S.V.  113
Obukhov A.V.  76
Ogorodov A. N.  68, 71
Okit T.  21
Orlov N.G.  115
Orlov V.K. 67  
Osmayev O.A. 53, 59  
Ostrofsky Z.E. 72, 75, 76, 78, 79  
Ovchinnikov V.V. 54, 55, 56, 57  

P  
Panchenko V. L. 77  
Pankratov D.G. 84, 85  
Parkhomychuk V.V. 99  
Parkhomenko A.A. 40  
Parkhomenko V. D. 21, 30, 33  
Paschenko O.V. 29  
Pavlov V.A. 4, 5, 9, 47  
Pechenkin V.A. 8, 58, 60, 61, 90  
Pecherkina N.L. 47  
Pechyorin A.M. 72  
Peregud M.M. 76, 78  
Perminov D.A. 5, 9, 110  
Peshev V.V. 118  
Petrov D.D. 67  
Pevtsov D.V. 79  
Plokhov V.V. 93, 99  
Podgornova I.V. 47, 84, 85, 86  
Polit J. 119  
Poon S.J. 25  
Popov V.P. 98, 115  
Porollo S. I. 32  
Portnykh I. A. 27, 28, 77  
Pribis J. 7  
Pritulov A.M. 118  
Prokopova T.S. 30  
Prozorova I. V. 48  
Pugachev R.N. 74  

R  
Rapp O. 25  
Raspopova G.A. 47  
Raybchikov A.I. 30  
Reshetnikov F. G. 68  
Reutov I.V. 44  
Reutov V.F. 44, 94  
Robouch B.V. 119  

Rogozhkin S.V. 5, 10  
Romanov V.A. 90  
Rusov V.D. 79, 94, 99, 101  
Ruzhitsky V.V. 40  
Rybin P.V. 11  

S  
Sagaradze V.V. 35, 47, 82, 86  
Sakov V.S. 84, 85  
Saksagansky G.L. 95  
Samarin S.I. 93, 99  
Saporzhnikov N.V. 115  
Saprykina I.V. 47  
Sato T. 21  
Savkina N.S. 115  
Schäublin R. 23  
Scherbachev K.D. 114  
Sekimura N. 21  
Semerikov V.B. 71  
Semkov A.L. 98  
Semyonkin V.A. 54, 55, 57  
Sergeev V.M. 67  
Shaaban I.Yu. 94  
Shal’nov K.V. 4, 5, 47, 105, 110  
Shamardin V.K. 72, 75, 76  
Shamovsky V.G. 49  
Shapovalov R.V. 59  
Sharkeev Yu.P. 30  
Shcherbakov E. N. 77  
Sheregii E.M. 119  
Shestakov A.E. 67  
Shestakov V.P. 39, 42, 48, 96  
Shikov A.K. 82  
Shilyaev B.A. 40, 45  
Shimansky G.A. 79  
Shishov V.N. 76, 78  
Shiyankov S.V. 99  
Shmakov A.A. 45, 60  
Shmygin V.G. 84, 85  
Shukaylo V.P. 113, 115  
Singh B.N. 6  
Sivin D.O. 30
<table>
<thead>
<tr>
<th>Name</th>
<th>Pages</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zaluzhnyi A.A.</td>
<td>11</td>
</tr>
<tr>
<td>Zaluzhnyi A.G.</td>
<td>11, 46</td>
</tr>
<tr>
<td>Zheldak U.L.</td>
<td>48</td>
</tr>
<tr>
<td>Zhurb V.I.</td>
<td>40</td>
</tr>
<tr>
<td>Zimin A.V.</td>
<td>84, 85</td>
</tr>
<tr>
<td>Zlatoustovski S.V.</td>
<td>42</td>
</tr>
<tr>
<td>Zouev Yu.N.</td>
<td>47, 75, 82, 84, 85, 86</td>
</tr>
<tr>
<td>Zverev V.V.</td>
<td>48</td>
</tr>
</tbody>
</table>