

## SIMULATION TECHNOLOGIES IN MODERN RADIATION MATERIAL SCIENCE

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The results of simulation experiments on different materials are analyzed. Using irradiation with charged particle beams one can reproduce and examine practically all the known radiation effects and investigate physical nature of these effects in more details under well-controlled conditions. Characteristics of some radiation sources used for studies of radiation effects and radiation resistance and experimental procedures are presented. The accelerator systems, high-tech instrumentations and methodologies for the analysis of experimental data provide a comprehensive tool for determination of mechanisms of radiation damage and selection of materials with high radiation resistance.

### 1. INTRODUCTION

Increase of safety of existing nuclear reactors, construction of new generation of economical fast reactors (FR) and fusion reactors needs the development and production of materials that can operate in the severe conditions of the cores of nuclear reactors [1, 2]. Development of structural materials for operating and future nuclear plants represents very complex science-technologic problem. It is very important that materials for reactor building ensure safe and effective operation of existing nuclear plants and met the highest operational characteristics for reactors of next generations.

Under the influence of fast particles and radiation complex structure-phase transformations occur; these transformations cause the considerable changes and, as a rule, the degradation of initial properties. Therefore to determine the general mechanisms of the change of structure and physical-mechanical properties, prediction of serviceability of materials for reactor cores it is necessary to study processes of nucleation and evolution of defect structure of metals and alloys under irradiation. Insufficient understanding of physical processes responsible for radiation damage of solids is one of main factors limiting development of radiation-resistant materials, realization of new ideas and original engineering developments.

The defined science-technical challenge is lengthy time (~ 20 years) necessary for development of materials, their testing, licensing, putting in reactor and obtaining of results. The cost of material testing under neutron irradiation for these advanced nuclear systems is continuously increasing while availability of test reactors is steadily decreasing. Last time irradiation possibilities were strongly decreased also due to the shut down of all spectrums of nuclear facilities. Besides the production of radiation-resistant materials is very difficult problem due to the insufficient understanding of the nature of radiation-induced phenomena and material damage practically in non investigated range of high irradiation doses.

Material development for operation in unique conditions of irradiation and evaluation of their radiation resistance consists in the use of existing

irradiation facilities for determination of mechanisms of radiation damage and selection of materials with high radiation resistance, particular *using methods of simulation irradiation.*

Operating research nuclear reactors, special accelerators of charged particles are now the unique facilities that present wide testing possibility for study of radiation resistance of materials, for development of improved radiation-resistant materials and selection of materials for reactors of next generations.

### 2. MAIN TASKS OF SIMULATION EXPERIMENTS

In 1969 Nelson and Mazey [3] performed research on specimens of M316 steel, which were irradiated by ions of C, O and Fe and showed that structure of ion-irradiated specimens was very similar to structure of neutron-irradiated specimens. Since then, many studies have utilized ion irradiation as a reactor surrogate to study radiation damage.

In 1972 the governmental program of activities on physics of radiation damage and radiation material science was approved; this program ensured the scientific assistance of activities on development of new structural materials and prediction of their behavior in fast reactors and in future fusion reactors. KIPT was appointed the main organization in this problem. Use of accelerators in radiation material science and wide scaled investigation in the field of physics of radiation phenomena in Kharkov institute of physics and technology (KIPT) were started in 1974. During the past four decades a large experience of the use of charged particle accelerators in simulation technologies was accumulated. Ion irradiation technologies have been shown to considerably reduce the time and material resources required for selection and optimization of chemical composition, thermal and thermal-mechanical treatment of structural materials.

Over the years the following general observations and conclusions were reached.

– Main phenomena limiting the use of materials (embrittlement of reactor pressure vessels, low temperature swelling of austenitic steels, growth and forming of zirconium alloys, swelling of claddings and

wrappers of fast reactors) are connected to one another by common physics of phenomena characteristic for irradiated materials but are showed in specific conditions.

– Possibility of the use of materials in nuclear power assemblies is determined by the irradiation conditions – by energetic spectrum of neutrons, by the temperature of irradiation and so on. These conditions determine the behavior of ensembles of point defects and transmutants responsible for degradation mechanisms and dimension instability and may be studied by simulation technologies.

– The presented goal – the reaching of commercially necessary levels of nuclear fuel burn-up – may be realized only on the base of modern scientific ideas about the role of physical mechanisms of microstructure evolution that are associated with variation of initial physical-mechanical properties under irradiation. Creation of radiation resistant materials is very complicated due to: the insufficient knowledge of the nature of the radiation-induced phenomena and material damage in practically no-investigated range of very high irradiation doses.

Development of theoretical models, experimental investigation and accelerator simulation of processes of radiation damage, evolution of defected structure of irradiated materials give the possibility to predict the material behavior in real conditions of their lengthy operation in cores of nuclear reactors.

Behavior of point defects and of their complexes produced under irradiation, the high concentration and high diffusive mobility of defects induce a number of processes (Fig. 1) not observed or weakly demonstrated in conditions without irradiation.

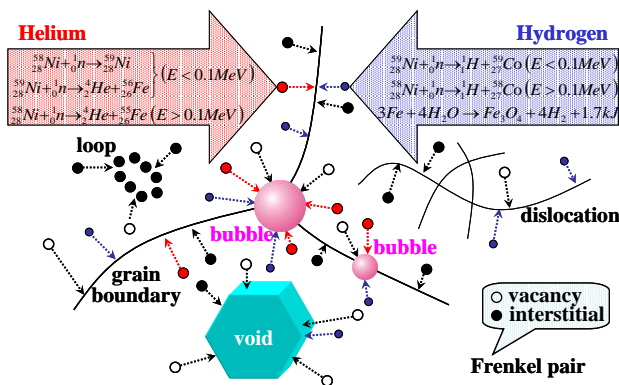


Fig. 1. Main relations in structure and composition of stainless steel under irradiation [1]

Degradation of initial properties and the loss of radiation resistance are caused by radiation-induced evolution of microstructure and micro composition. Irradiation of structural materials at temperatures of reactors operation creates the unprecedented possibility of the changes of microstructure, of mechanical properties and even of external dimensions of structural components at the expense of swelling, growth and creep. Phenomena of radiation damage are defined by the conditions of irradiation in concrete NEP – by energy spectrum of neutrons, by irradiation temperature, transmutants etc.

Main mechanisms of degradation and dimension instability arise from following processes:

- 1) Displacement of atoms from their lattice sites;
- 2) Radiation induced segregation;
- 3) Long-range migration and clustering of defects;
- 4) Preferential interaction with edge dislocations;
- 5) Transmutants formation and their interaction with point defects.

The main tasks, which are needed in using accelerators, are such:

– Investigation of fundamental processes. (Particle collisions; quantification of kinetic properties of radiation defects; simulation of formation & growth of defects; defect characteristics depending on radiation dose (type, size, density, etc.).

– R&D materials for fast reactors (swelling and embrittlement). Observation of radiation-induced microstructure such as segregation and hardening.

– Microstructural predicting for possibilities of life extension for operation reactors; RPV steels (dpa rate), RVI (low temperature embrittlement).

– Gases influence on mechanisms of radiation damage. Synergetic effect of helium and hydrogen in fusion and spallation systems.

Simulation experiments in investigations of radiation damage of materials have the few obvious **advantages** in comparison with reactor tests:

– Precise continuous control of experimental parameters of irradiation (temperature, flux, etc.);

– Possibility of differential and direct investigation of different factors influence on structure-phase evolution under irradiation; ideally suited for optimizing alloying composition;

– Absence of induced radioactivity; specimens can be handled in conventional conditions;

– Relative cheapness of experiments realization.

The use of accelerators of charged particles will allow to:

– Conduct experiments under cyclic and other non-stationary regimes of irradiation according to the planned program;

– Conduct irradiation in pulsed regime or concurrent with continuously implant helium or atoms of other gases in any relation with number of displaced atoms;

– Reach exposure doses not yet achieved in nuclear plants;

– Change the rate of damage in large limits;

– Obtain the extensive information on influence of irradiation conditions: mass and energy of bombarding particles, rate dose, rate of gases implantation, pulsed beam on formation of radiation porosity, dislocation structure evolution and phase state of irradiated material.

Simulation experiments together with advantages have substantial **disadvantages and limitations**:

– difference in recoil spectra and the structure of primary radiation damage;

– phase stability at high dpa rate – and increased temperatures – changing of typical for reactor experiment conditions for nucleation and growth of voids;

– injected interstitial effect leads for typical in simulation experiments decreasing of void size;

– difficulties in simulation of transmutants accumulation (mainly He and H). This problem can be solved only with multibeam accelerators;

– stress induced by irradiation – surface proximity can go to abnormal evolution of radiation-induced structure.

But despite these **disadvantages and limitations** now irradiation on accelerators is one possible choice in the absence of high flux neutron irradiation facility. Many nuclear facilities are now shut down (FFTF, RAPSODIE, DFR, PFR, Superphenix, EBR-II, BR-10, BN-350 etc).

It is possible to minimize the disadvantaged and limitations of simulation experiments by the use of accelerators of new types (dual or triple beams accelerators) and by modern high-technology methods of investigations (PEM (loops, cavities, precipitates); STEM+EDS+EELS (segregation of grain boundaries); TAP (clusters of solutes); AES, XPS (segregation of surface); FIB and nano indentors (technologies of micro specimens necessary for development of new materials and testing of materials for nuclear and fusion facilities); EXAFS, SANS (structure evolution on nano scale); nuclear-physical methods – RBS + channelling (non destructive analysis, nature of point defects and their localization in lattice).

### 3. SELECTION OF IRRADIATION CHARACTERISTICS FOR SIMULATION EXPERIMENTS

During the study of physical base of radiation damage of materials including the radiation swelling it is necessary to use all available irradiation facilities. It will allow to determine the effect of structure of primary radiation damage, of rate dose, of surface influence and of ion implantation on the development of radiation porosity and to determine general and special mechanisms of radiation swelling. By selection of the type, energy and intensity of particle flux in simulation experiments one can remove the side effects and realize conditions similar to reactor irradiation.

The main criterion determining the selection of particle kind that reproduces the primary processes of material damage under reactor irradiation is the structure of primary radiation damage. The preliminary conclusion about the degree of reproducibility of primary event of reactor damage may be presented on comparison the spectra of primary knocked-out atoms or some integrator introduced so that their ratio characterizes the number of defects produced by the same way. The performed calculation [4] shows that irradiation of metals by self ions simulates the process of point defect production. The advantage of irradiation by self-ions is the fact that irradiation of pure metals by self ions doesn't produce alloying of metals. Therefore it was concluded that for irradiation of composite alloys it is preferable to use ions of one the main component of irradiated alloy.

Selection of particle energy is made balancing the surface and ion implantation influences on processes occurring in irradiated material. The degree of reproducibility of primary processes of ion interaction with the matter in the damage peak and in adjacent

layers at ion energy higher 0.5 MeV depends weakly on primary energy of ions [5]. At 800...1000 K and dose rate  $(1...5) \cdot 10^{-3} \text{ dpa} \cdot \text{s}^{-1}$  surface effect in steel and nickel specimens spreads on the depth 200...300 nm. Ion beams of metal with energy 2.5...5 MeV are widely used in practice that allows reaching the accepted limits of effects of surface and of ion implantation on development of porosity in investigated layer (Fig. 2). At higher ion energies the limits are induced by the tendency of intense ion beams to deposit excessive energy, creating heat removal problems and temperature control.

The degree of reproduction of nuclear reaction products is determined from comparison of PNR spectra for compared kinds of irradiation [6, 7]. In experiments with gas ion irradiation and with coincident beams area for investigation is selected where gas atoms are implanted in required ratio with number of displacements per atom.

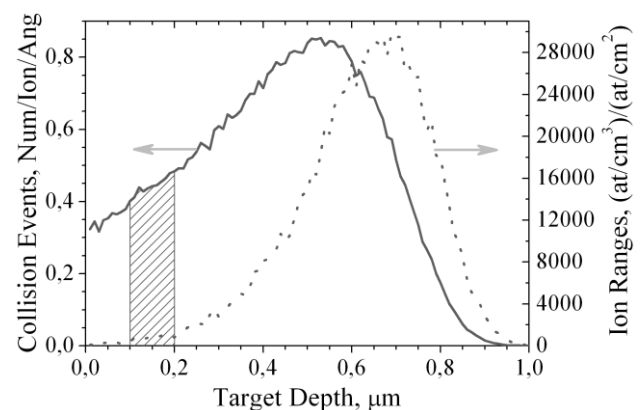


Fig. 2. Determination of the depth layer (shaded) that is optimal for microscopy investigation of void swelling.

Profiles of damage and deposition of chromium ions with energy 1.8 MeV during ion irradiation of steel [8] were calculated using the Kinchin-Pease option of SRIM-2011 for correct comparison with neutron-induced swelling of this steel [9]

The established standard practice of simulation studies [10] prescribes the calculation of the number of atomic displacements per atom (dpa defined [10] as “a unit of radiation exposure giving the mean number of times an atom is displaced from its lattice site”) as an adopted metric of correlation of the radiation damage relevant effects in metals and alloys subjected to different irradiation environments. This allows comparison of the results of accelerator and reactor based irradiations as well as of those of different experimental groups [10].

The quantification of spatially dependent dpa is a complicated radiation transport problem mostly solved by means of the Monte Carlo (MC) modeling software. The SRIM package [11] is a publicly available [9] practically standard [10] user-friendly tool of such kind of calculations applicable to  $\sim 10$  (0...4) keV ion beams irradiation of planar layered targets. The TRIM MC code of the SRIM package simulates depth profiles of irradiation induced vacancy-interstitial Frenkel pairs (FPs) using the binary collisions approximation (BCA) method. Under the assumption that each FP arises in a

single atomic displacement, a common practice is to scale dpa at a given ion fluence  $\Phi$ ,  $\text{cm}^{-2}$ , with the vacancy profile the TRIM code outputs in the VACANCY.TXT file.

TRIM offers two options for the FP distributions simulation. The express “quick damage” (QD) method simulates only the trajectories of primary ions and the production of primary knock-on atoms (PKAs). The total FP production rate is then calculated analytically within the scope of the modified Kinchin-Pease (K-P) [12, 13] model of the secondary displacement function (SDF)  $\nu(T)$ , the number of the secondary knock-on atoms (SKAs) produced by a PKA of energy  $T$  at a user-supplied value of a stable FP production threshold energy  $E_d$ . The alternative “full cascades” (FC) damage MC simulation method simulates the overall collision cascade explicitly down to certain cut-off energy  $E_{\text{fin}} \sim 1$  eV of BCA applicability.

The SRIM package is a well approved and powerful tool suitable for atomistic simulation of ion beam interaction with solid, ion implantation, sputtering, and the production of radiation defects. However, when applied to the reactor materials science relevant computational dosimetry of ion beam driven simulation irradiations, it must be used with caution. The parameters and the settings of the BCA simulation have to be chosen accordingly to the specific goal of computer modeling and irradiation experiment, and should not be omitted in publications of simulation results.

Different regimes of the code operation output physically reasonable but semantically different dosimetric quantities. To compare with the reactor damage dosimetry data, the “quick damage” simulation option is the preferred one since its results are found to be the most closely conformant with the “NRT standard dpa” prescribed by the standard practice of simulation studies (Fig. 3).

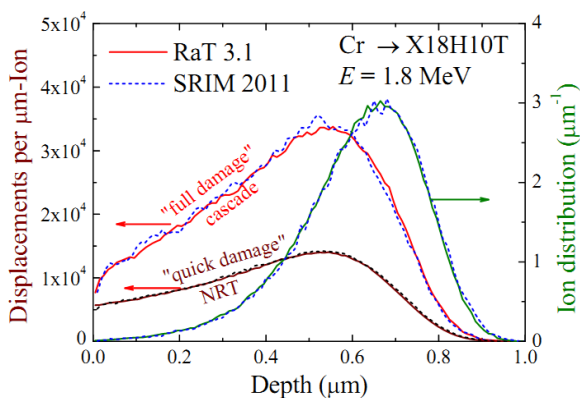


Fig. 3. Doping and damage profiles calculated for the case of the simulation irradiation [14] using the FC and QD options of TRIM (dotted curves) and the appropriate algorithms of the RaT MC code (solid curves) [15]

The application of other options deviates the results from the working standards. One is free to use them for “what-if” and sensitivity analyses. But an uncritical use

of the “full cascade” option outputs can result in overestimation of the expected NRT dpa. This can be especially critical in the case of ultrahigh-dose irradiations when considering the dpa dependent threshold effects (e.g. swelling).

These conclusions are based on current regulations and are subject to change at the expected refinements of the NRT standard. The new generation GEANT4 based multi-purpose Monte Carlo radiation transport code RaT successfully reproduces the results of atomistic SRIM simulations. Due to its flexibility and capability of consistent simulation of coupled neutron-gamma, electron and ion beam relevant irradiations, it is a prospective platform for incorporation of new standards in the computational support of radiation materials science studies.

Increase of radiation damage that had determined the expediency of simulation experiments is always the principal reason of non reproducibility of reactor swelling in simulation experiments. The higher is the difference in damage rates in reactor conditions and in simulation experiments the lower is the probability of that the variations in porosity development caused by increase of damage rate will be completely removed by temperature shift.

On selection of particle flux intensity (rate dose) it is necessary to take into account that changes induced by acceleration of damage must be removed by the temperature shift (Fig. 4). The following relationship between “dose rate ( $\Phi$ ) and temperature ( $T$ )” must hold as  $T_1/T_2 = A \ln(\Phi_2/\Phi_1)$  [16].

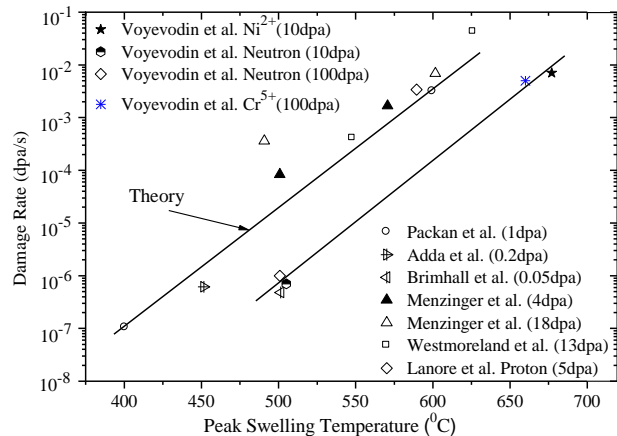


Fig. 4. Summary of all available data on damage rate-induced temperature shift of swelling in nickel [1, 17]

Structure-phase transformations are reproduced in two-stage experiment when for formation of void population and structure-phase transformation characteristic for reactor irradiation in reactor or in accelerator at first with low rate of dose accumulation ( $10^{-7} \dots 10^{-5}$  dpa·s $^{-1}$ ) and when with fast accumulation of high doses. Experience accumulated to date shows that simulation-specific problems discussed earlier have been studied sufficiently to allow understanding of most experimental results and allow use of accelerators to probe the behaviour of metals at very high doses.

The field of accelerator technology is exciting and dynamic. As a result the accelerator community is able to provide brighter light sources, higher collision rates in particle generation, and more precise measurements of physical properties (Tables 1-3).

Higher damage rate as a result of higher cross-section of charged particles interaction with materials in accelerators ( $10^{-2} \dots 10^{-4}$  dpa/s) in comparison with rates of displacements in the different reactors ( $10^{-6} \dots 10^{-8}$  dpa/s) allow achieving necessary doses much faster, for few hours (Fig. 5).

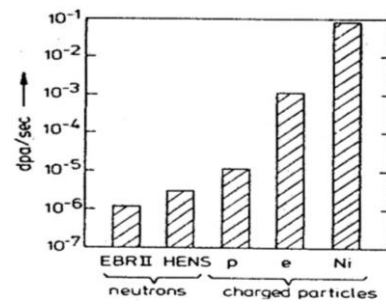


Fig. 5. Damage rate of fast reactor neutron and charged particles [18]

Table 1

Research centers in the world dedicated to irradiation technologies

### Dual & Triple Beam Accelerators

Laboratory	Facilities	Specifications	Ref.
Dual or triple MV ion beams			
MSD, IGCAR Kalpakkam, India	1.7 MV Tandetron 30...150 kV Ion implanter	Heavy and Light Ion Damage rate up to $7 \cdot 10^{-3}$ dpa/s	[18] [19]
TIARA Takasaki, Japan	Cyclotron accelerator AVF K110 3 MV Tandem accelerator 3 MV Single-ended accelerator 400 kV Ion implanter	Heavy Ion Light Ion/Proton Damage rate $10^{-3}$ dpa/s	[20] [21]
HIT Tokyo, Japan	3.75 MV Van de Graaff 1 MV Tandetron	Heavy Ion and He/H injection Damage rate $10^{-4} \dots 10^{-2}$ dpa/s	[19] [21] [22]
DNE Nagoya University, Japan	2 MV Van de Graaff 200 kV Ion implanter		[19]
FZ Rossendorf, Germany	6 MV Tandetron 3 MV Tandetron 500 kV Ion implanter	Heavy Ion and He/H injection	[19]
FSU Iena, Germany	3 MV Tandetron JULIA 400 kV Ion implanter ROMEO 10 kV Ion beam implanter LEILA	Range of ion fluences: Xe ion $3 \cdot 10^{12} \dots 4 \cdot 10^{15}$ ions $\text{cm}^{-2} \cdot \text{s}^{-1}$ ; N ion $8 \cdot 10^{13}$ ions $\text{cm}^{-2} \cdot \text{s}^{-1}$	[19] [23]
DuET Kyoto University, Japan	1.7 MV Tandem accelerator 1 MV Single-ended accelerator	Heavy Ion and He/H injection Damage rate $5 \cdot 10^{-5} \dots 10^{-3}$ dpa/s	[21]
TIARA JAEA Takasaki, Japan	3 MV Tandem accelerator 3 MV Van de Graaff 400 kV Ion implanter	Heavy Ion and He/H Damage rate $\sim 10^{-3}$ dpa/s (Fe ion)	[19] [21]
JANNUS Saclay (CEA), France	Epimethee – 3 MV Pelletron (Multi-charged ions 200 keV < E < 60 MeV. H, D, He beams for Ion Beam Analysis). Yvette – 2.5 MV Van de Graaff (Positive single-charged ions: H, D, $^3\text{He}$ and $^4\text{He}$ (He flux $\sim 10^{13}$ ions/s)). Japet – 2 MV Tandem accelerator (Sputtering source. Almost any element except rare gases. Hydrogen for triple beam)	Heavy Ion and He/H injection	[19] [24]
Lawrence Livermore National Laboratory, CA USA	10 MV Multi-purpose tandem accelerator 1 MV Nuclear Microprobe	Heavy (C, Fe, U) 30...80 MeV He 4...27 MeV H/D 2...18 MeV	[25]
ESUVI & GiV NSC KIPT Kharkov, Ukraine	1.8 MV Van de Graaff Spread source of gas ion (He/H)	Heavy Ion and He/H injection Damage rate $5 \cdot 10^{-5} \dots 10^{-2}$ dpa/s	[26] [27]
ESU-2 NSC KIPT Kharkov, Ukraine	1.6 MV Van de Graaff 50 kV He and H/D ion implanters	Ions (He, Ne, Ar, Kr, Xe) Damage rate $2 \cdot 10^{-3} \dots 2 \cdot 10^{-1}$ dpa/s Ions (He, H, N, O, D) Ion fluxes $10^{13}$ He/ $\text{cm}^2 \cdot \text{s}$ and $10^{14}$ D/ $\text{cm}^2 \cdot \text{s}$	[28]

Table 2

Single &amp; dual beam coupled to transmission electron microscope (TEM)

Laboratory	Facilities	Specifications	Ref.
CARET Multi-Beam HVEM facility, Hokkaido University, Sapporo, Japan	Electron irradiation (1.3 MeV) Dual beam irradiation (1.3 MeV e <sup>-</sup> + 100 keV He <sup>+</sup> ) 10...200 appmHe/dpa	Damage rate: 10 <sup>-5</sup> ...10 <sup>-3</sup> dpa/s Maximum damage: ~ 30 dpa/day H or He injection rate: > 10 appm/dpa	[20] [21] [29]
Cyclotron & Dynamitron, Tohoku University, Japan	AVF Cyclotron, 4.5 MV Dynamitron accelerator (Proton and He ion)	He, C, N, O, Ne, Ar, Kr Ion (He) Damage rate 10 <sup>-3</sup> dpa/s	[21] [29]
IVEM-Tandem Facility, Argonne National Laboratory, USA	Hitachi H-9000NAR (100...300 kV) Ion irradiation (most ions, 40 keV...1 MeV)		[19] [30]
JANNUS Orsay (Paris-Sud University and CNRS), France	Aramis – 2 MV Tandem with external negative ion source + Penning source inside the terminal. (Almost all the elements except rare gases 500 keV < E < 15 MeV). IRMA – 190 kV Ion implanter Bernas-Niersource. (Almost all the elements – a few keV < E < 570 keV) 200 kV TEM	Almost all the elements except rare gases	[19] [24] [31]
MIBL University of Michigan, USA	3 MV Ion Tandetron 1.7 MV protons 400 kV implanter	Ions Damage rate 10 <sup>-5</sup> ...10 <sup>-3</sup> dpa/s	[32] [33] [34]

Table 3

Comparison of irradiation parameters for different particle types [35, 36]

Particle type	Energy, MeV	Dose rate, dpa/s	Dose, dpa	
Electrons	1	2×10 <sup>-3</sup>	28	
Protons	3.4	7×10 <sup>-6</sup>	7	1 dpa ~ 1 day
Ni ions	5	5×10 <sup>-3</sup>	25	10 dpa ~ 1 hr

Heavy ions "generate" the highest defect production rate, but they possess very short penetration depths, as demonstrated in Fig. 2. Therefore, heavy ion accelerators with the beam energies from the hundreds keV to a few MeV are mainly used for producing high levels of defect concentration in thin layers of the irradiated material (Fig. 6).

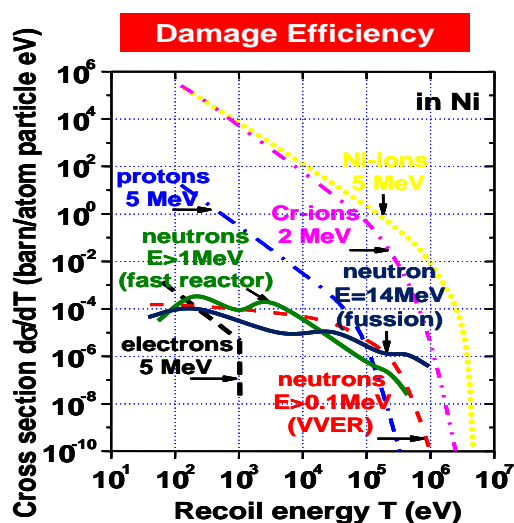


Fig. 6. Damage efficiency of different particles [1, 37]

Fig. 6 shows that damage efficiency of various irradiated particles is quite different showing why the choice of particles for irradiation simulation experiments is very important.

#### 4. ELECTRON ACCELERATORS AND HIGH VOLTAGE ELECTRON MICROSCOPES

Electrons are subject to many large-angle scattering events; hence range straggling is severe. In radiation damage studies, however, the primary concern is with the passage of electrons through relatively thin targets in which the fractional energy loss is small. This loss can be estimated for many purposes using the following general prescription.

Electrons with energy 1...10 MeV induce displacement of atoms and produce in metals defects as isolated Frenkel pairs [38]. Therefore HVEM with electron beams with energy 1 MeV and higher is widely used not only as high-resolution researching facility but also as irradiation accelerator of electrons. Density of electron beam in modern electron microscopes reaches  $2 \cdot 10^{24} \text{ 1/(m}^2 \cdot \text{s)}$  [39], the rate of displacement in metals makes  $10^{-4} \dots 10^{-2} \text{ dpa} \cdot \text{s}^{-1}$  that is 3-4 order higher than the rates of displacement observed in reactor conditions. The advantage of high-voltage electron microscope (HVEM) is the possibility to irradiate relatively thick (to 3 mkm) targets and study the process of developments

of radiation-induced defects of structure in dynamics. This is of great importance for study of mechanisms of nucleation and growth of dislocation loops, voids and precipitates [40, 41] also as for understanding of the effect of gases and dislocation in development of radiation porosity [42, 43] for determination of energetic characteristics of point defects and their agglomerates [44] determination of relation between porosity developments and transformations in other components of defected structure of the material [45, 46] for study of characteristic properties of behavior of voids and dislocation loops near dislocations, twins, grain boundaries [47], for investigation of voids and dislocation loops produced by preliminary neutron or ion irradiation [48, 49].

As was noted early, electrons with energy 1...10 MeV induce displacement of atoms and produce in metals defects as isolated Frenkel pairs. This has been of foremost importance in developing our understanding of radiation damage, as it made studies of defect creation mechanisms.

The processes of point defects annealing in low-alloyed materials Zr-Sc and Zr-Y, after low-temperature irradiation with 2 MeV electrons has been investigated. The temperature intervals of main stages of irradiation are determined [50].

In NSC KIPT the irradiation was performed on electrostatic electron accelerator ELIAS, manufactured by High Voltage Engineering Corporation, model KS/3000. Energy of electrons on specimen surface was equal to 2 MeV, density of beam current – 10 mkA/cm<sup>2</sup>, area of irradiation – 10×25 mm<sup>2</sup>.

Fig. 7 shows the difference curves of spectra of isochronous annealing of Zr and alloys. In fact these are the temperature ranges of formation of complexes of the type radiation defect (interstitial/vacancy)-atom of alloying element. Spikes below the axis of abscissa indicate the intensification of annealing in alloys in comparison with pure Zr and identify the temperature ranges of decay of early formed complexes.

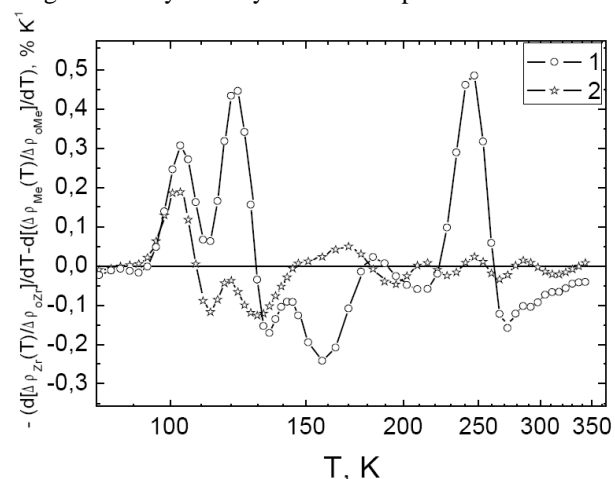


Fig. 7. Dependence of difference of isochronous annealing spectra of Zr and alloys Zr-Y and Zr-Sc on annealing temperature: 1 – difference of spectra of Zr and alloy Zr-Y; 2 – difference of spectra of Zr and alloy Zr-Sc [50]

Using the method of low-temperature (~80 K) irradiation with electron energy of 2 MeV and measurements of electrical resistance, annealing of radiation point defects were investigated in low-doped alloys Zr-Gd, Zr-Dy, and Zr-La.

It was experimentally established that impurity atoms such as La, Dy and Gd interact effectively with point defects in zirconium matrix. Such interaction can result in formation of interstitial-impurity and vacancy-impurity complexes. This influences considerably on the processes of annihilation and redistribution of radiation defects and must be taken into account in development and modification of zirconium-base alloys for the core of nuclear power reactors (Fig. 8) [51].

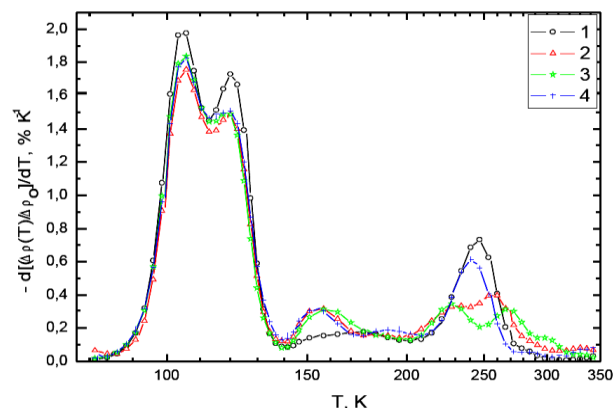


Fig. 8. Spectrums of isochronous annealing of Zr and Zr-Dy, Zr-Gd, and Zr-La alloys irradiated with electrons ( $E = 2 \text{ MeV}$ ,  $T_{irr} = 82 \text{ K}$ ,  $D = 1.4 \cdot 10^{19} \text{ e}^-/\text{cm}^2$ ): 1 – pure Zr; 2 – Zr-Dy alloy; 3 – Zr-Gd alloy; 4 – Zr-La alloy [51]

The irradiation with high-energy electrons ( $E > 8 \text{ MeV}$ ) leads not only to generation of Frenkel pairs but also to formation of complex radiation defects and nuclear reactions products. It allowed to separate the mechanisms of radiation embrittlement. High temperature embrittlement exists under irradiation by electrons with  $\gamma$ -quanta with energy higher than the level of nuclear reactions [52].

The main reason of difference in swelling under irradiation by neutrons, heavy ions and electrons with energy 1 MeV is the difference in primary processes of radiation damage. It was shown that in the case of electron irradiation only formation of isolated Frankel pairs – of interstitial atoms and vacancies – occurs and irradiation by heavy ions and by neutrons causes the formation of cascades that then form vacancy loops producing an excess of interstitial atoms and simultaneous nucleation of vacancy and interstitial loops then proceeds.

The more important difference of void evolution under ion irradiation and under irradiation by electrons with energy 1 MeV is the difference in the rate of their nucleation. Under ion irradiation for saturation of void concentration sufficiently high irradiation doses are necessary (~60 dpa for steel deformed on 30%) and under electron irradiation the void concentration stabilizes under considerably lower doses: usually at 10 dpa and lower and only in special cases – at 30 dpa.

Influence of vacancy loops nucleating in cascades under material irradiation with neutrons or heavy ions on processes of swelling, of radiation creep were discussed in literature repeatedly [53]. Their main role at steady state course of radiation damage processes is the intensification of processes of point defects recombination.

Plotting of swelling characteristic of investigated steels (dose dependence, temperature dependence of

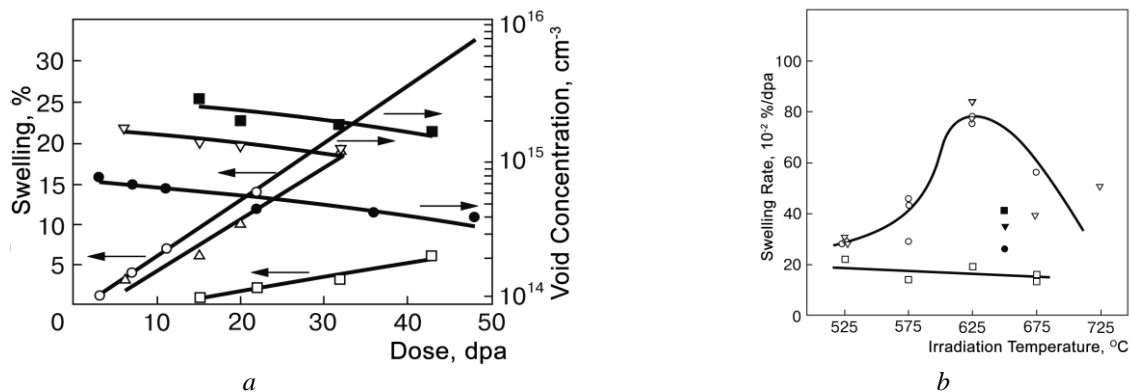


Fig. 9. Dependence: a – of swelling and of void concentration on irradiation dose in steel EI-847 under irradiation in HVEM ( $T_{irr} = 600\text{ }^{\circ}\text{C}$ ,  $\circ$  – solution annealing  $1050\text{ }^{\circ}\text{C}$  30 min;  $\nabla$  – 10%ST;  $\square$  – 30%ST); b – rates of swelling on temperature at electron (1 MeV;  $\circ$ ,  $\nabla$ ,  $\square$  – HVEM) and ion (3 MeV;  $\bullet$ ,  $\blacktriangledown$ ,  $\blacksquare$  – ion accelerator) irradiation of steel EI-847 in different structural states ( $\circ$  – solution annealing  $1050\text{ }^{\circ}\text{C}$  30 min;  $\nabla$  – 10 %ST;  $\square$  – 30%ST) [54]

Therefore, investigation of swelling regime of studied austenitic steels under irradiation by 1 MeV electrons in HVEM allows to monitor the vacancy voids evolution in the process of irradiation and to obtain additional information on physics of studied phenomena [48].

## 5. LIGHT ION ACCELERATORS. CYCLOTRONS

For investigations of radiation effects such as strengthening, embrittlement, creep and growth of materials it is necessary to use charged particle beams with the energy providing zone of homogeneous damage through all the irradiated specimen thickness. For radiation damage physics studies of solids the high-energy beams of protons,  $\alpha$ -particles, ions of carbon or nitrogen, etc., that can produce homogeneous defect structure along all the thickness of irradiated samples are used. The grain sizes in austenitic stainless steels are of 20...30  $\mu\text{m}$ . The maximum thickness of the samples for mechanical tests must be of 100...250  $\mu\text{m}$ . Therefore, for these purposes it is necessary to use charged particle beams with the energy providing a zone of homogeneous damage through all the thickness of the irradiated specimen. The used (e, $\gamma$ )-beams do not exceed the reactor neutron fluxes in displacement production rate but in respect of helium accumulation, the high energy electron and  $\gamma$ -beams are more effective than fast neutrons, by two orders of magnitude approximately. This fact makes it possible to simulate expressly high temperature radiation.

Cyclotron is a very useful instrument for study of different effects of radiation damage by their use of light ions. Investigation of radiation damage of structural materials is one of main direction of the use of

swelling rate (Fig. 9,a and b) certificate that at irradiation temperature of 575  $^{\circ}\text{C}$  the rates of swelling of austenitic steel and of 10% cold worked steel became approximately the same.

Increase of irradiation temperature to 625  $^{\circ}\text{C}$  leads to the situation when swelling rate in deformed specimens with 10% cold worked became higher than in annealed steels.

cyclotron CV-28 in NSC KIPT. Researches of Julich Researching Center have earlier developed methodologies that allow simulate variation of mechanical properties of materials for nuclear reactors under bombardment by light ions that are generated by cyclotron CV-28 [55]. They also have developed methodology of works with miniaturized specimens and have presented recommendations for realization of mechanical testing of miniaturized irradiated specimens [56, 57]. Also is presented the method of simulation of mechanical properties variation spallation targets by introduction of helium by direct implantation. Results of such investigations are very important for realization of safe nuclear power in the future.

Reference [58] presents a study of homogeneously  $\alpha$ -implanted specimens of a 9Cr-1Mo (EM10) martensitic steel at 550  $^{\circ}\text{C}$  to a concentration of 5000 appm. The irradiation apparatus is located at a beam line of the Julich Compact Cyclotron. The initial energy of the  $\alpha$ -beam (27.4 MeV) was then degraded by a rotating wheel consisting of 24 aluminium foils of different thicknesses. In order to obtain a homogeneous implantation, the beam was scanned at sawtooth frequencies of typically 300 Hz in both directions across the specimen. The bubbles are small (average diameter 5...10 nm) and clearly faceted (Fig. 10).

The aim of this paper was to analyses the He bubbles in an  $\alpha$ -implanted EM10 steel and to determine precisely the density and pressure by EELS. The main results are the following:

- The high quality data obtained allowed to establish two linear relationships: one between the energy shift and the estimated He density, and another between the pressure and the inverse bubble radius.



– By applying the equation of state developed by Trinkaus, it has been shown that the bubbles are under-pressured.

Approaches to methodology of applying in RRC KI cyclotron irradiation and procedure of simulating damage up to 100  $\mu\text{m}$  deep to 10...20 dpa those parallel

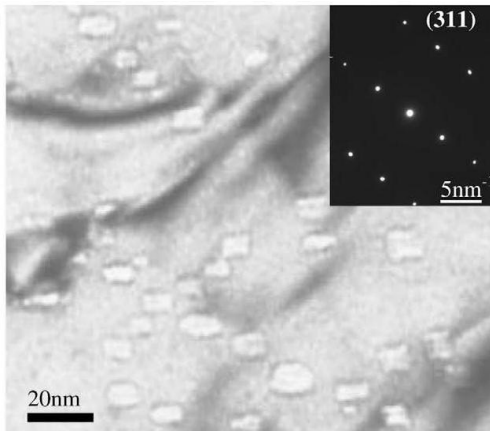
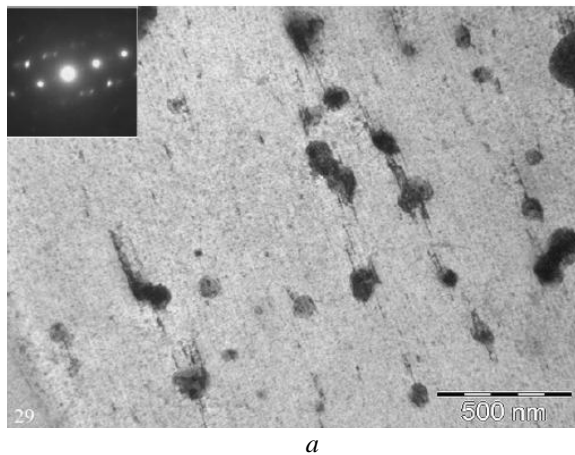


Fig. 10. TEM of micrograph obtained along the [311] zone axis and the corresponding diffraction pattern [58]

Under neutron irradiation zirconium alloys are subject to structure transformations, elements redistribution and acceleration of uniform corrosion, particularly, for intermetallic type Zr-alloys. Correlation between these processes has the interest for investigations of new Zr-materials to operate in-pile for long time. Neutron irradiation in reactors and difficulties with corrosion tests of irradiated samples



typical VVER burners, subsequent corrosion and TEM examinations are shown [59]. The typical results of the numerical calculations of distribution profile of generation rate of point defects under irradiation of Zr alloy by 15 MeV helium ions at irradiation dose  $10^{17} \text{ cm}^{-2}$  are given in Fig. 11.

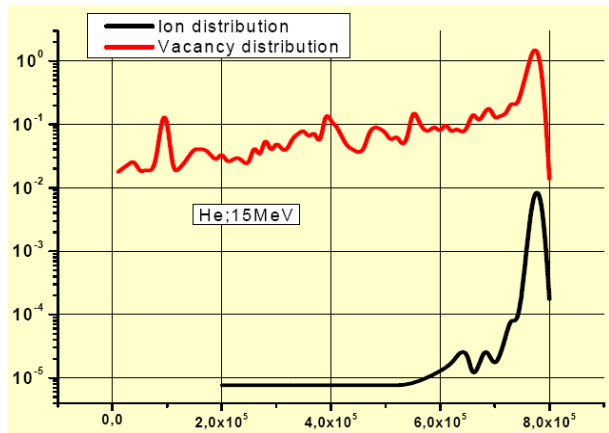


Fig. 11. The distribution profile (depth in  $\text{\AA}$ ) of generation rate of point defects (number per atom) during irradiation of Zr alloy by 15 MeV helium ions to an irradiation dose  $10^{17} \text{ cm}^{-2}$  [59]

raise the using accelerators of charged particles to achieve high damage levels during shorter periods, that makes experiments faster and reduces their cost.

For the comparison in Fig.12 we can see microstructure changes in neutron irradiated Russian Zr alloys E635 up to neutron dose  $F_n = 0.5 \cdot 10^{26} \text{ n/m}^2$  and damage dose 2 dpa.

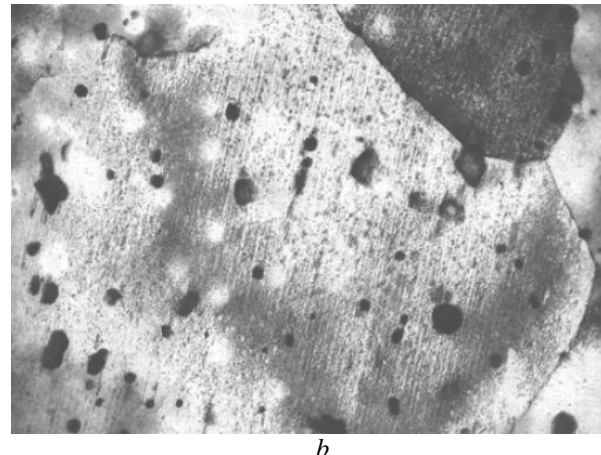


Fig. 12. Microstructure of Zr alloy E635 irradiated on cyclotron by helium ions (a) and neutron up to dose:  $F_n = 0.5 \cdot 10^{26} \text{ n/m}^2$  and damage dose 2 dpa [59]

The obtained results show the distribution of precipitates and ordered dislocation loops ( $\alpha$ -type) with the average diameter of them  $D = 10 \dots 20 \text{ nm}$  and bulk density  $N = 10^{16} \text{ cm}^{-3}$  loops. The same physical picture has been observed after charged particle irradiation on microstructure of Zr alloy E635 irradiated on cyclotron by protons with the energy 4 MeV at temperature  $T = 350 \text{ }^\circ\text{C}$  and irradiation dose 1 dpa ( $2 \cdot 10^{17} \text{ p/cm}^2$ ), where has also been observed the ordered structure of dislocation loops ( $\alpha$ -type). These obtained experimental

results confirm the good correlation between microstructure changes in irradiated Zr alloys after neutron and charged particle irradiations at comparable dpa level (1...2 dpa).

## 6. HEAVY IONS IRRADIATION

Achievement of high burn-up (20...25% of h.a.) in fast reactors demands to solve the swelling problem of cladding and wrapper materials. Up to now, void swelling is the main limiting factor for using structural

materials for the fast reactors and the reactors of future generations [29]. In this field, advantages of accelerators are much more considerable because allow comprehensive studies of the input of different factors, which influence on voids nucleation and growth. Many of these factors, such as the role of structure-phase evolution, influence of crystal lattice, gaseous impurities etc. [1, 60, 61] are studied. The role of different alloying elements in radiation behaviour of cladding for fuel elements has also been investigated [62].

The combination of *rate theory* (to approximate the temperature shift required to compensate for changes in dose rate in order to produce the same microstructure effect at a fixed dose) and the *efficiency* for producing freely migrating defects can account for the effect of particle type and dose rate in particle beam experiments used to simulate neutron irradiation effects in austenitic stainless steels [63].

Standard practice for neutron radiation damage simulation by charged-particle irradiation provides ASTM E 521 [10]. Important excerpts are given below.

#### Choice of Particle.

Since the accelerated particles usually come to rest within the specimen, the possibility of significant alterations in specimen composition exists with concomitant effects on radiation damage. If metallic ions are used, they should be of the major constituents of the specimen. Electron irradiation poses no problems in this regard.

#### Choice of Particle Energy.

Three criteria should be considered in the choice of particle energy:

(1) The range of the particle should be large enough to ensure that the region to be examined possesses a preirradiation microstructure that is unperturbed by its proximity to the surface.

(2) The point defect concentration during irradiation in the observed volume should not differ substantially from that expected of irradiated volumes located far from free surfaces.

(3) The energy deposition gradient parallel to the beam across the volume chosen for observation should be small over a distance that is large compared to typical diffusion distances of defects at the temperature of interest.

The best measure of surface influence is the observation of denuded zones for the microstructural feature of interest. The width of denuded zones for voids can be significantly larger or smaller than those observed for dislocations. The volume of the specimen to be examined should lie well beyond the denuded zone because steep concentration gradients of point defects may exist on the boundary of such zones. Gradients in the deposited energy can be reduced by rocking the specimen (varying the angle between the beam and the specimen surface), but local time dependent flux variations will exist.

#### Free Surfaces.

Free surfaces, acting as sinks for point defects, alter the point defect concentration profiles, the densities and configurations of various microstructural sinks as well as their rate of growth, and the kinetics of various phase transformations. (Grain boundaries may exhibit similar

effects.) Surfaces also serve as sites for solute segregation and may allow entry of various elemental contaminants. The most serious influence of the surface is the depression of point defect concentrations. Since this effect increases as the temperature is increased, it may preclude an unperturbed measurement of the full temperature response of a given phenomenon using charged-particle irradiation.

The presence of surfaces must also be taken into account in neutron experiments where the phenomenon under study is sensitive to the local chemical environment (for example, sodium, fission gas, helium, or hydrogen charging). Since most charged particle studies are conducted in a vacuum, this aspect of the neutron experiment may be impossible to simulate.

#### Self-interstitial.

When a specimen is bombarded with self-ions, the ion is injected as a self-interstitial that is indistinguishable from a displacement-produced interstitial. The latter, however, has a vacancy counterpart while the injected interstitial does not. Theoretical methods have been developed for evaluating the reduction in cavity volume caused by injected interstitials. The effect can be significant and is largest at low irradiation temperatures and for intrinsically low-swelling materials where recombination is the dominant mode of loss of radiation-produced point defects. The effect of injected interstitials can be minimized by extracting data at depths well removed from the projected range of the bombarding ions. When the effect of injected interstitials is large, the use of step-height measurements as a measure of swelling is precluded.

#### Elemental Redistribution.

Irradiation generally leads to a redistribution of alloying elements and impurities caused by differences in the interaction of each species with the vacancy and interstitial fluxes. In addition to these effects, which occur even when material is uniformly irradiated, additional redistribution may be introduced during ion irradiation by the variation in damage rate along the particle path or by defect concentration gradients produced by free surfaces. Gradients in damage rate can be minimized if necessary by rocking the specimen during irradiation or by variably degrading the energy of the ion beam. Free surface effects are avoided by using sufficiently high ion energies.

#### Rastering.

Rastering (periodic scanning) of a focused beam over the specimen will subject the specimen to periodic local flux variations. It is recommended that a rastered beam be avoided for the simulation of a constant neutron flux, although it may be appropriate for the simulation of a pulsed neutron flux. Radiation-induced defect structures that evolve under such pulsed conditions can differ substantially from those that evolve in a constant flux. It should be noted that pulsed operation is an inherent characteristic of many accelerators.

Using the above procedures and recommendations for irradiation with charged particle beams one can examine practically all known types of radiation effects and investigate the physical nature of these effects in detail under well-controlled conditions. Some successful

examples from studies conducted at KIPT are presented below.

Under irradiation not only change of initial structure proceeds but also the change of initial solid solution composition, formation and modification of second phase precipitates is observed as a result of interaction of point defects fluxes and atoms of solid solution. Irradiation modifies the structure-phase state of steels and alloys at the expense of diffusion processes acceleration and as a result of such structure-phase transformations that are impossible under conditions of thermal equilibrium [1].

Investigation of segregation on dislocation segments composing the part of dislocation network had showed that the same tendency revealed at investigation on neutral sinks was preserved: enrichment by Ni and Si and depletion by Cr and Fe was observed.

Measurements of segregation profiles on large defected Frank loop (Fig. 13) have showed the considerable increase of nickel and silicon concentration and also decrease of concentration of Cr and Fe. Factors of enrichment/depletion have changed as follows: Ni→1.5; Cr→0.78; Si→1.8, that is, nickel content was 1,5 times higher and content of silicon was 1.8 times higher after irradiation.

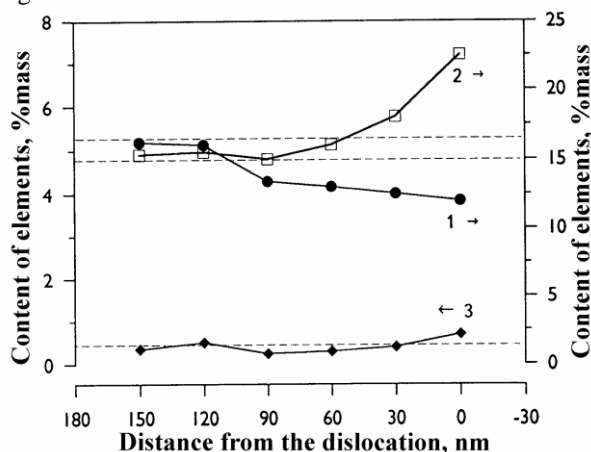


Fig. 13. Distribution of elements in stacking fault plane of Frank loop (dotted lines show the mean content of element in matrix): 1 – Cr; 2 – Ni; 3 – Si (EI-847(A) ( $Cr^{3+}$ ,  $E = 3$  MeV,  $T_{irr} = 600$  °C,  $D = 25$  dpa) [1, 64]

Similar behavior of segregants with higher level of segregation on defected Frank loop was detected in steel PCA irradiated in reactor FFTF to 15 dpa at 520 °C [8]: content of Ni in the plane of stacking fault exceeds the content of Fe and Ni becomes the dominant element of microcomposition of Frank loop, that is dislocation loop is surrounded by the region of nickel “microalloy” with composition that differs considerably from composition of matrix of the base steel. Level of segregation decreases with the moving away from the plane of stacking fault and at a distance greater than 50 nm segregation changes the sign because the enrichment of defected plane by the same elements causes their removal from adjacent matrix. Decrease of loop size decreases considerably the level of segregation of all

elements. About 80% of dislocation loops with diameter higher 80 nm show the detected segregation and loops with size lower 40 nm didn’t demonstrate such segregation.

Between evolution of MC precipitates under irradiation and regime of dose dependence of swelling there is the close relation – with the increase of irradiation dose the concentration of fine dispersed precipitates MC decreases and their size increases. The increase of irradiation dose causes the change of precipitates shape from spherical to globular and platelet. It was observed that the moment of precipitate shape change coincides with the start of the regime of fast swelling (Fig. 14).

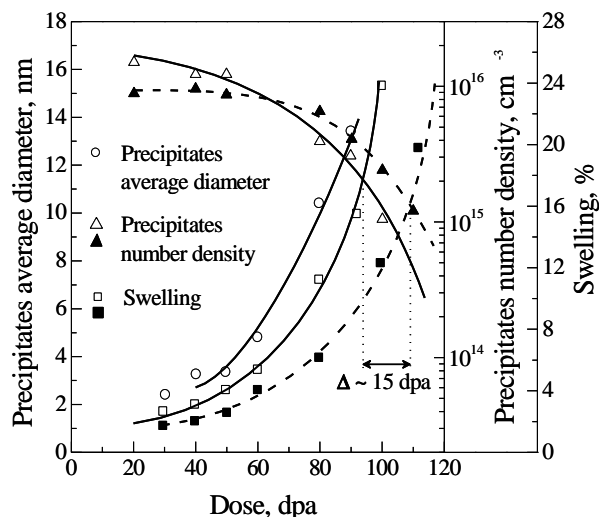


Fig. 14. Relation of MC precipitate evolution with swelling of steel EI-847 ( $Cr^{3+}$ ,  $E = 3$  MeV,  $T_{irr} = 650$  °C) [1, 65]

The described modification of precipitates shape means the loss of coherency by MC precipitates. This initiates the mechanisms of phase evolution discussed earlier that leads to the heavy infiltration and accelerated growth of precipitates at the expense of the flux of solute atoms from matrix.

Irradiation by heavy ions is now the unique instrument to study the swelling behaviour of ferritic-martensitic steels, allowing us to attain super high irradiation doses. In NSC KIPT the possibility of high-dose irradiation is currently being realized with the study of swelling of a number of ferritic-martensitic steels in the temperature range 430...550 °C [8]. An example is shown in Fig. 15 where Russian duplex alloy EP-450 was irradiated with 1.8 MeV  $Cr^{3+}$  ions to doses as high as 300 dpa. It was determined that the maximum of swelling of ferrite grains in EP-450 lies at ~ 480 °C. After a lengthy incubation period of ~ 150 dpa a transition to steady-state swelling proceeding with a rate ~ 0.2%/dpa was observed. It is shown for the first time that swelling of ferritic steel may reach ~ 25% [66]. The tempered martensite grains in EP-450 resisted swelling to much higher doses.

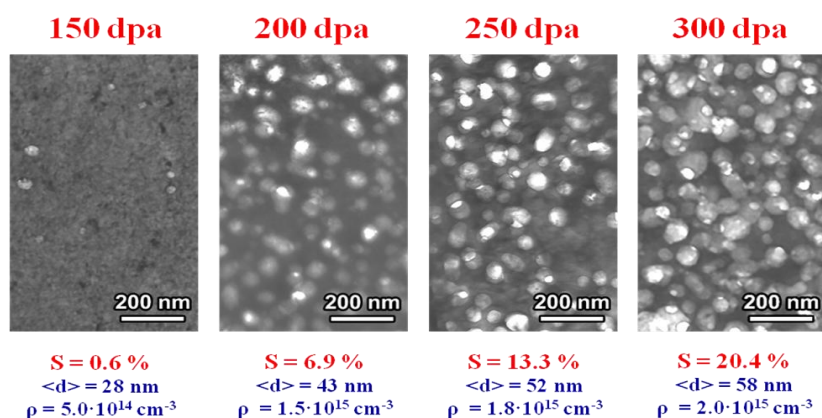
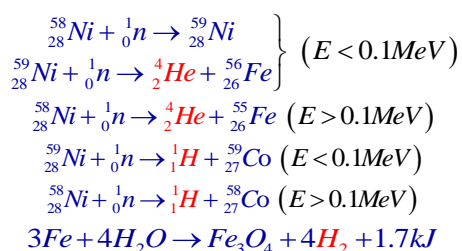


Fig. 15. Void volume fraction observed in ferrite grains of EP-450 after irradiation with 1.8 MeV Cr<sup>+3</sup> ions. After calculation of swelling from void volume fraction, the swelling at 300 dpa is ~ 25%, reached at a steady-state swelling rate of ~ 0.2%/dpa [66]

## 7. MULTI-BEAM TECHNIQUES

Structural materials in nuclear plants under neutron exposure are exposed to products both chemical and nuclear in nature which are produced simultaneously with radiation damage. Primary sources of helium and hydrogen in thermal reactors especially arise from nuclear reactions in nickel isotopes and hydrogen can also arise from corrosion, as shown below.



The topicality of investigation of helium and hydrogen behavior in metals had increased sharply together with investigation of these gases interaction with defects in the crystalline lattice, evolution of defected structure and microstructure investigating the effect of helium and hydrogen on mechanical properties of structural materials. Many results testify to the negative effect of helium and hydrogen upon joint generation or implantation [67].

For instance, radiation effects and tolerance of several steels under ion and electron irradiation on hardening, phase stability, helium embrittlements were studied using He implantation on nano-oxide dispersion strengthened steels [68]. Experimental procedures used were ion beam irradiation in the DuET facility using 6.4 MeV Fe ions at 650 °C and 5 · 10<sup>-4</sup> dpa/s to ~ 60 dpa.

The size distribution of complex oxides before and after ion irradiation was compared, and any significant modification could not be detected. Microstructural evolution did not occur, indicating that the dispersed oxides in the ODS steels are rather stable under the tested conditions. The cavities in a reduced activation ferritic steel formed on dislocations and at grain boundaries, but the cavities in the ODS steel were mainly formed at the interfaces of oxide particles with matrix. Oxide particles act as trapping site for helium suppressing swelling and intergranular embrittlement as

a result of preventing helium from gathering and transporting to grain boundaries. Suppression of irradiation hardening and grain boundary degradation are anticipated for ODS steels up to 1000 appm He at around 550 °C.

Basing on (the availability of) the current STCU accelerator project “Evaluation of the performance of ferritic-martensitic steels under gas conditions relevant to advanced reactor concepts”, development and improvement of a special ion irradiation system will be completed; that will provide a state-of-art irradiation test facility. This facility will be used to determine the full parametric performance of all ferritic-martensitic steels currently being developed in the world community for advanced reactor applications (STSU Project #3663). This facility will allow irradiation under gas generation conditions specific for each reactor concept. Iron ions will be used to generate radiation damage without gas. It will also allow co-implantation of both helium and hydrogen at reactor-specific levels using a new, novel concept of a three-ion single beam rather than the usual three accelerator approach. Analysis had showed that a good alignment of damage profiles and of profiles of hydrogen and helium ions paths is possible under energy of 1.8 MeV of Cr<sup>+3</sup> ions, 20 keV of hydrogen and 40 keV of helium. The corresponding deposition profiles for 18Cr10NiTi steel are presented on Fig. 16.

Series of experiments was performed on austenitic iron-chromium-nickel steel 18Cr10NiTi that is the main materials for pressure vessel internals of WWER reactors. Analysis of the effect of synergistic irradiation of materials by beams of gaseous and heavy ions on defect structure evolution was performed on microscopes JEM-100CX and JEM-2100. Considerable influence of gases on swelling of studied steel was revealed [69-71]. The temperature dependence of swelling under irradiation only by heavy ions of chromium has the maximum at 615 °C (Fig. 17). Maxima of the curves of swelling under irradiation by gases in any configuration were observed at 600°C. Under double and triple irradiations voids are observed already at 450 °C that is not characteristic for irradiation by chromium ions only.

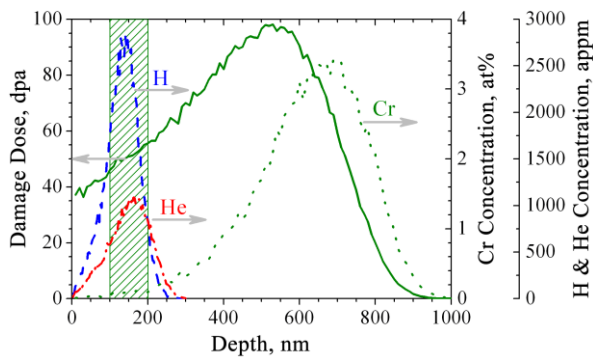


Fig. 16. Profiles of damage (—) and deposition (·····)  $1.8 \text{ MeV Cr}^{3+}$ , Range profiles of  $20 \text{ keV H}$  (- - -) and  $40 \text{ keV He}$  (- · - ·) in steel  $18\text{Cr}10\text{NiTi}$ . ( $n_{\text{Cr}} = 1 \cdot 10^{17} \text{ Cr ions/cm}^2$ ,  $n_{\text{H}} = 2.4 \cdot 10^{15} \text{ H ions/cm}^2$ ,  $n_{\text{He}} = 1.2 \cdot 10^{15} \text{ He ions/cm}^2$ ). The shaded area will be investigated by electron microscopy [69]

While helium alone extended the temperature range of swelling, hydrogen was much more effective to increase swelling over the same range. When irradiated with both H and He the effects are more complex, decreasing swelling somewhat at lower temperatures but increasing it at higher temperatures (Fig. 17).

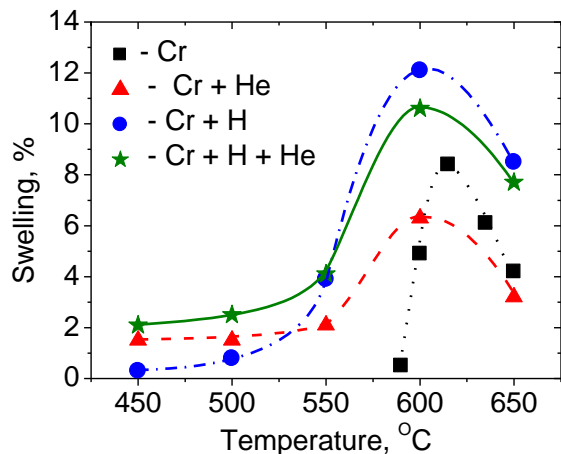


Fig. 17. Temperature dependence of the swelling steel  $18\text{Cr}10\text{NiTi}$  after irradiation: ■ -  $1.8 \text{ MeV Cr}^{3+}$  to a dose  $50 \text{ dpa}$ ; ▲ -  $1.8 \text{ MeV Cr}^{3+}$  to a dose  $50 \text{ dpa}$  and  $60 \text{ keV He}^+$  up to  $1000 \text{ appm}$ ; ● -  $1.8 \text{ MeV Cr}^{3+}$  to a dose  $50 \text{ dpa}$  and  $30 \text{ keV H}_2^+$  up to  $2000 \text{ appm}$ ; ★ -  $1.8 \text{ MeV Cr}^{3+}$  to a dose  $50 \text{ dpa}$ ,  $30 \text{ keV H}_2^+$  up to  $2000 \text{ appm}$  and  $60 \text{ keV He}^+$ ,  $1000 \text{ appm}$  [71]

Under irradiation only by heavy ions the dependence of void concentration on temperature repeats practically the temperature curve of swelling, namely concentration increases in low-temperature range ( $590 \dots 615 \text{ }^\circ\text{C}$ ) and decreases at higher temperatures. Under irradiation by Cr+He and by Cr+He+H one order decrease of voids concentration is observed. For irradiation by Cr+H considerable increase of void concentration is observed (approximately 3 times higher) in rather narrow low-temperature range  $450 \dots 500 \text{ }^\circ\text{C}$  but at higher

temperatures of irradiation the void concentration became 3 times lower.

It was revealed that after co-irradiation by heavy and gaseous ions the efficiency of grain boundaries and of surface influence on development of radiation porosity is low. Areas free from voids along the grain boundaries and specimen surface are absent.

Temperature dependence of microstructure evolution of steels after irradiation with heavy ions of chromium and gaseous ions of helium (Cr+He) and hydrogen (Cr+H) has the characteristic like-bell appearance. Swelling maximum under irradiation with Cr+H is twice higher than swelling under irradiation with Cr+He while under triple irradiation with (Cr+He+H) swelling is insignificantly lower than under irradiation with Cr+H.

Irradiation of specimens with implantation of helium induces the increase of swelling in low-temperature range. It is caused by the fact that atoms of helium stabilize void nuclei decreasing their critical radius. Hydrogen has the similar effect on nucleation of voids but its effect is significantly lower and possibility appears for faster growth of voids because the effective radius of vacancies capture by voids increases. Swelling increase is also observed for triple irradiation.

Implantation of helium also as implantation of hydrogen in studied steel contributes to the process of voids production but slows down their growth. It is revealed that for gas irradiation in the range of investigated temperatures the concentration of voids increases and their size decreases.

The accumulation, migration, distribution and desorption of helium and hydrogen (deuterium) implanted in  $18\text{Cr}10\text{NiTi}$  steel over a wide range of implanted gases were studied [72]. Concentrations characteristic of generation by both fusion reactors and PWRs and VVER-1000 reactors were investigated.

Data on trapping, retention and microstructural evolution of helium in the temperature range  $300$  to  $900 \text{ K}$  and post-implantation annealing at  $300$  to  $1800 \text{ K}$  were obtained. It was experimentally established that the helium distribution profiles in the range of annealing temperatures  $290 \dots 900 \text{ K}$  do not change. A variation in helium distribution profile was observed only at an annealing temperature of  $\sim 1100 \text{ K}$ , resulting in a helium concentration decrease of  $\sim 30\%$ .

In the case where  $18\text{Cr}10\text{NiTi}$  stainless steel specimens were first irradiated by  $\text{Cr}^{3+}$  ions to  $50 \text{ dpa}$ , the retained deuterium concentration is found to increase. Fig. 18 shows the distribution profiles of deuterium implanted at room temperature to  $1 \cdot 10^{16} \text{ cm}^{-2}$  in the initial state compared with the profile of a specimen irradiated by  $\text{Cr}^{3+}$  ions. Comparison of the profiles shows that after irradiation at  $20 \text{ }^\circ\text{C}$  and after annealing, the concentration of deuterium is higher and the half width of the distribution is larger when the specimens have been previously irradiated with Cr ions. The amount of deuterium retained in the Cr-implanted specimen after annealing at temperatures of  $90$ ,  $200$  and  $500 \text{ }^\circ\text{C}$  is  $40$ ,  $20$  and  $2\%$  with respect to the amount of deuterium retained at room temperature.

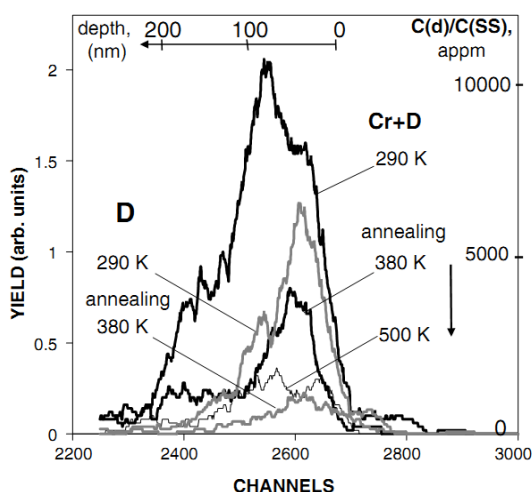


Fig. 18. The distribution profiles of deuterium implanted to  $1 \cdot 10^{16} \text{ cm}^{-2}$  at room temperature and after annealing at 90 and 200 °C, compared to the specimen with similar implantation that had been previously irradiated with  $\text{Cr}^{+3}$  [72]

As one can see from a typical SRIM calculation the level of displacement damage increases almost linearly, increasing about a factor of two from 0 to 4000 Å, but averaging ~ 50 dpa in the gas-implanted region for this calculation.

Deuterium has significant mobility at room temperature. During its migration through the chromium damaged layer it is becoming fixed in traps created by  $\text{Cr}^{3+}$  ions. As a result the deuterium distribution profiles are observed to widen. A decrease in the concentration level always takes place during annealing but the capture and retention of deuterium following  $\text{Cr}^{3+}$  ion irradiation is several times higher compared to the capture by defects created by irradiation by deuterium alone.

These results support the possibility that displacive irradiation of stainless steels in environments producing large amounts of helium and hydrogen will experience enhanced trapping of significant amounts of hydrogen, with possible consequences on the onset and amount of void swelling, development of hardening and possibly on irradiation-assisted stress corrosion cracking.

## 8. CORRELATION OF RESULTS OF REACTOR AND ACCELERATOR EXPERIMENTS ON RADIATION SWELLING OF MATERIALS

During development and conduct of simulation experiments two purposes were pursued: firstly – accelerate the process of understanding of swelling; secondly – realize in practice the results of simulation experiments for prediction of material behavior under reactor irradiation on the base of simulation experiments.

Simulation experiments had largely contributed to understanding of phenomena of radiation damage of materials. Majority of mechanisms of radiation porosity was found during simulation experiments. During irradiation of nickel and steels by ions of inert gases development of two systems of voids was detected [73];

similar behavior was also detected under reactor irradiation.

During simulation experiments the hypothesis about considerable role of dislocation structure and its transformation in development of porosity was confirmed [73-76] influence of gases on porosity development was also studied [73, 74, 77-81] a new phenomenon-formation of void lattice was observed [81, 82].

Additionally simulation experiments have shown promise of the use of some materials with high size stability under irradiation as materials for fuel cladding tube as and wrappers of channels for fast reactors; these experiments have provided the extensive information on the influence of thermal-mechanical treatment, of composition and alloying in material behavior under irradiation [83-86]. Introduction of simulation experiments in reactor material science determined in a short space of time the relationship of radiation swelling with other phenomena under irradiation, allowing the program of further reactor experiments to be adjusted or corrected, also allowing for mechanisms and phenomena revealed in simulation experiments to be identified; allowing preliminary classification of materials according to their tendency to high swelling.

In the first approximation it can be considered that for each kind of particles there is the effective dose ( $D_i = k_{ij}D_j$ ) which determines the fraction of vacancies which were not subjected to annihilation and which were realized as porosity. Theoretical and empirical development of doses relationship under which radiation-induced swelling has the same value (or relations of swelling values under the same dose) is realized.

In reference [87] proton irradiation has undergone considerable refinement as a radiation damage tool. Numerous experiments have been conducted and compared to equivalent neutron irradiation experiments in order to determine whether proton irradiations capture the effects of neutron irradiation on microstructure, microchemistry, and hardening. In some cases, benchmarking exercises were conducted on the same native alloy heat as neutron irradiation in order to eliminate heat-to-heat variations that may obscure comparison of the effects of the two types of irradiating particles. The following examples cover a number of irradiation effects on several alloys in an effort to demonstrate the capability of proton irradiation to capture the critical effects of neutron irradiation. These examples represent a comprehensive collection of comparison data between proton and neutron irradiation and taken together serve as a good example for the capability of charged particles to emulate the effect of neutron irradiation on the alloy microstructure.

Similarly, ion bombardment can be used to study swelling if we are careful to understand its peculiarities and limitations. It is necessary to minimize the surface influence and to avoid the influence of the injected interstitial.

Using heavy-ion irradiation at very high dpa rates ( $10^{-2}$  and  $10^{-3}$  dpa/s) and doses (5...100 dpa) and coupling the results to available neutron data a swelling (Fig. 19) function within the framework of a single

empirical model has been developed that specifically incorporates the effect of dpa rate on void swelling [88-91].

Mathematical relationship for swelling of the 18Cr10NiTi steel is obtained:

$$S = (0.25 - 0.022 \ln k) \cdot \phi(D - 103 + 0.1T - 2.6 \ln k) \times \exp\left\{-\frac{(T - 690 - 15.5 \ln k)^2}{2 \cdot (12.3 - 1.9 \ln k)^2}\right\}$$

where  $S$  is swelling in %,  $D$  is the damaging dose in dpa;  $T$  is the irradiation temperature in °C;  $k$  is the dose rate in dpa/s; and the function  $\phi(x)$  is defined by expression:  $\phi(x) = x \cdot \theta(x)$ , where

$$\theta(x) = \begin{cases} 1, & x > 0 \\ 0, & x \leq 0 \end{cases}$$

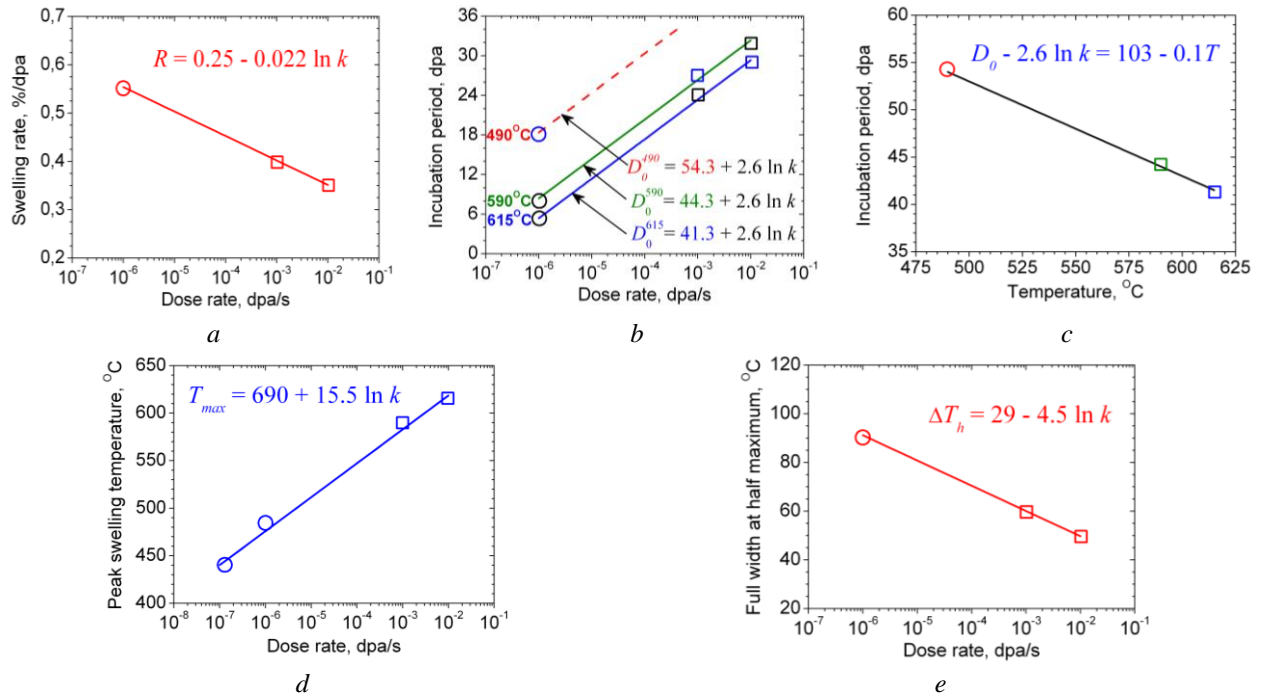


Fig. 19. Peak swelling rate of 18Cr10NiTi steel (a), incubation period (b), peak swelling temperature (d) and temperature distribution (e) versus the dose rate and the coefficient entering into the approximating function versus irradiation temperature (c) in 18Cr10NiTi steel. The symbols  $\circ$  and  $\square$ , respectively, designate the reactor and accelerator data [89]

Swelling maps constructed from this model permit forecasting of the behavior of the steel in WWERs under the required irradiation conditions, not only at already attained exposure doses, but more importantly to higher dose levels that will be reached following plant life extension.

Fig. 20 shows predicted dose-temperature maps of 18Cr10NiTi swelling that were calculated in [89] at different dose rates typical of accelerator irradiation ( $k = 10^{-3}$  dpa/s), fast reactor ( $k = 10^{-6}$  dpa/s) and low-flux thermal reactor ( $k = 10^{-8}$  dpa/s) environments.

The swelling becomes progressively larger at lower dpa rates. The generality of this phenomenon is supported by other studies on a variety of austenitic steels, such as that of Budykin and coworkers [92] and Seran and Dupoyu [93]. In every case the shortened incubation period at lower dpa rates leads to earlier and therefore more swelling at the lower dpa rates. In one exceptional study the 18Cr10NiTi steel when irradiated in the BR-10 fast reactor at a very low dpa rate ( $1.9 \cdot 10^{-9}$  dpa/s) was observed at 350°C to be clearly swelling after accumulating only 0.6 dpa [94].

Fig. 19 shows that with increasing dose rate at a given dose the temperature corresponding to the swelling peak shifts towards higher temperatures. This temperature “shift” is known to be due to the necessity of keeping constant the relationship between the rates of point defect formation and disappearance at sinks so that the vacancy supersaturation level characteristic of charged particle irradiation conditions should be maintained at reactor-relevant levels [95].

Therefore an empirical function incorporating both ion bombardment and fast reactor data of annealed 18Cr10NiTi steel has been developed to provide prediction of void swelling anticipated in the austenitic core internal components of WWER reactors, especially under conditions expected due to plant life extension. This function explicitly contains dependence not only on the dpa level and irradiation temperature but also the dpa rate, an approach not normally taken in earlier studies that produced equations containing no dose rate dependence.

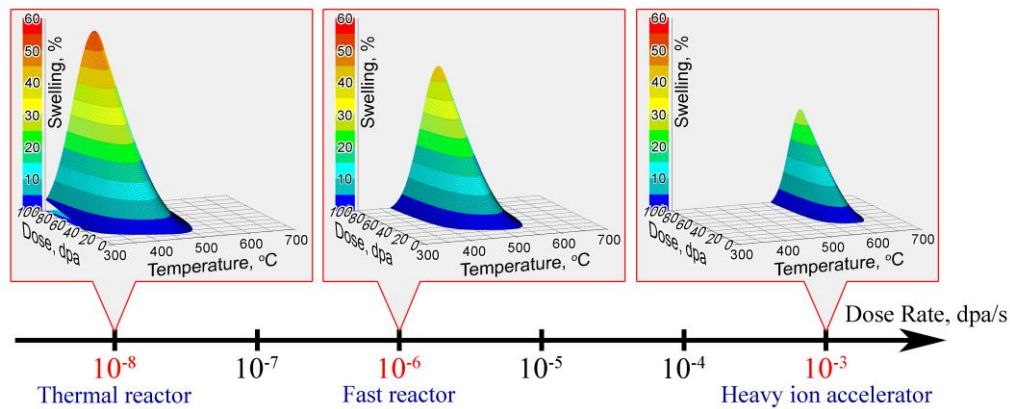


Fig. 20. Temperature-dose maps of  $^{18}\text{Cr}^{10}\text{NiTi}$  steel swelling for different dose rates, calculated by the empirical function [89]

## 9. PHILOSOPHY OF PREDICTION RESULTS FOR PRACTICAL PURPOSES

Results presented here for stainless steels may serve as an example of simulation use for technological purposes. The use of irradiation in accelerators of charged particles in NSC KIPT, involving monitoring of microstructure development, understanding of phenomena involved in processes of swelling has allowed to formulate the scientific presumptions about the industrial levels of swelling resistance of these steels [4]:

- Stability of dislocation structure that serves as controller in distribution of fluxes of point defects and thereby determines the rate of further evolution as result of different absorbing possibilities for point defects and possible segregation;
- Stability of solid solution is determined by possibilities of element segregation and by characteristics of sinks;
- Role of precipitate as predominating mechanisms suppressing swelling, depends in the extreme on alloying elements included into precipitates and capable to change the nature of precipitates;
- Evolution of defected structure in steel under irradiation represents the competition between survived phases (MC,  $\text{Fe}_2\text{P}$ ) and phases evolved as a result of solid solution decay ( $\gamma$ , G and  $\text{M}_6\text{C}$ ). This competition may be extended by optimizing composition and thermal-mechanical treatment;
- The task for future-investigation of synergetic effects of Ti, Nb, V, P, B, Si with possible formation of system of fine stable precipitates under irradiation and possible decrease of radiation-induced segregation;
- It is supposed that the possible way of improvement of radiation characteristics of austenitic materials may be the development of ODS-austenitic steels.

### Philosophy of radiation resistance of stainless steels:

Attainment of acceptably low levels of swelling is directly associated with formation of very stable microstructure under irradiation. Effect of alloying and treatment therefore lies in the following:

- Formation of more stable dislocation structure (preserving of Frank loops with their inherent low

mobility) and increase of level of point defect recombination. This may be achieved via cold deformation or by segregation of alloying elements on dislocation components that decreases their mobility;

- Preserving of fine carbide precipitates (TiC) and phosphides ( $\text{Fe}_2\text{P}$ ) as a main factor of swelling suppression in these steels, shifting the dose range of formation of G-phases and  $\eta$ -carbides into the area of higher doses;
- Delay of formation of G-phase and  $\eta$ -carbides will preserve in solid solution sufficient quantity of such elements as Ni, Si and P which strongly influence the nucleation and growth of voids.

## CONCLUSION

The present necessity of accelerators use is dictated by following main tasks:

- Provide the proper understanding of mechanism of radiation damage in nuclear materials; obtaining of information on the origin of point defects and on interaction between them.
- Determine the correlation between radiation-induced defects, structure-phase evolution and mechanisms of material degradation.
- Study the stability of systems with nano-scaled properties. It is of great importance for development and prediction of radiation behavior of nano-precipitates in ODS steels under high doses that are the more promising materials for reactors of next generation.
- Joint irradiation (reactor + accelerator). Despite the experimental complexities this method may present the best results for prediction of radiation behavior of materials under very high doses. Formation of defected structure characteristic of reactor irradiation may be reached on the stage of subsequent irradiation in accelerator.
- Develop the procedure for prediction of radiation behavior of materials right up to doses characteristic for reactors of next generation.

To achieve the main goal to develop materials for safe and economic operation of existing reactors and developed nuclear plants the close collaboration of theoreticians, researchers and designer is necessary.



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## ИМИТАЦИОННЫЕ ТЕХНОЛОГИИ В СОВРЕМЕННОМ РАДИАЦИОННОМ МАТЕРИАЛОВЕДЕНИИ

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Проанализированы результаты имитационных экспериментов на различных материалах. Используя облучение пучками заряженных частиц, можно воспроизвести и исследовать практически все известные радиационные эффекты, а также изучить физическую природу этих эффектов более детально в хорошо контролируемых условиях. Представлены характеристики некоторых источников облучения, используемых для исследования радиационных эффектов, радиационной стойкости, и экспериментальные процедуры. Ускорительные системы, высокотехнологичные измерительные приборы и методики для анализа экспериментальных данных являются всеобъемлющим инструментом для определения механизмов повреждения и отбора материалов с высокой радиационной стойкостью.

## ІМІТАЦІЙНІ ТЕХНОЛОГІЇ В СУЧАСНОМУ РАДІАЦІЙНОМУ МАТЕРІАЛОЗНАВСТВІ

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Проаналізовано результати імітаційних експериментів на різних матеріалах. Використовуючи опромінення пучками заряджених часток, можна відтворити та дослідити практично всі відомі радіаційні ефекти, а також вивчити фізичну природу цих ефектів більш детально в добре контрольованих умовах. Представлено характеристики деяких джерел опромінення, що використовуються для дослідження радіаційних ефектів, радіаційної стійкості, та експериментальні процедури. Прискорювальні системи, високотехнологічні вимірювальні прилади та методики для аналізу експериментальних даних є всеосяжним інструментом для визначення механізмів пошкодження та відбору матеріалів з високою радіаційною стійкістю.