IRRADIATION EFFECTS IN STRUCTURAL COMPONENTS OF NUCLEAR REACTOR: AN EXPERIMENTAL NANOSCALE POINT OF VIEW

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1. INTRODUCTION

Maintaining, all along the reactor life, a high level of safety and energetic and economic nominal performances are the most important topics for the future of nuclear energy. In this area, the control of materials ageing plays a key role. Predicting the long-term evolution of the mechanical properties for the materials (in service or new) submitted to irradiation during the reactor life remains a major challenge [1-4]. For that, many research activities are concerned with irradiation effects on mechanical properties of real, or model, materials, for in reactor or accelerator irradiations. But, while the multi-scale modelling of materials under irradiation has been well developed these past years [5,6] the experimental studies of the evolution of the microstructures at the atomic level are quite rare. This is mainly due to the lack of analytical capacity at nanometer scale for material irradiated in real conditions (neutron irradiation).

Neutron irradiation deeply modifies the properties of materials [7,8]. Some of the changes come from specificities of neutron irradiation such as the dynamics of the specific defects created by large displacement cascades accompanied by the creation of foreign atoms by nuclear reactions. The nature and the defect concentration depend on many factors like neutron fluence and energy, temperature, material composition, and crystallographic quality. Indeed, the evolution under irradiation of the microstructure of materials results from complex dynamics on an atomic scale from the coupled migrations of defects and impurities [7,8]. These nano-structural modifications are at the origins of the “ageing” of materials and thus of the possible reduction in their durability and/or performance. The nanometric scale observations make possible real progress in the understanding of these phenomena, such as aggregation of defects, elimination of the defects on grain boundaries or on nanometric particles, atomic segregations or precipitations, at the origin of the material degradation [9].
Only the powerful tools recently developed allow these nanoscale studies necessary to understand the microscopic and macroscopic phenomena. These characterizations are relevant to support the theoretical bases of multi-scale modeling from atoms to mechanical properties.

2. BASIC MECHANISM AT THE ORIGIN OF PHASE TRANSFORMATIONS UNDER IRRADIATION IN NUCLEAR MATERIALS

Irradiation in nuclear reactor can lead to large dimensional changes of metals, ceramics and alloys (irradiation creep, swelling and growth) and also significant changes in their mechanical behavior (hardening, loss of ductility, embrittlement, DBTT shift …). The rupture mechanisms can be strongly modified, like the apparition of quasi-cleavage in highly irradiated austenitic steels for example. The first observations of these phenomena were in the late 60’s (1967). These modifications are due to an important evolution of the microstructure induced by irradiation, voids, dislocation loops, segregation, precipitation, amorphization…. These microstructural evolutions, different from those observed during thermal aging, are governed by the continuous formation of point defects, interstitials and vacancies, and by the modification and the enhancement of the diffusion processes. These basic concepts were introduced in the 70’s like the induced segregation in 1974. In non-metallic compounds, the charge states of the defects can also be modified by the electronic excitation.

2.1 Elastic collisions

Elastic collisions between neutrons and atoms of the crystalline lattice result in the production of Frenkel pairs (vacancies and interstitials). The spatial distribution of defects at the time they are produced, strongly depends on the energy transferred to the primary knocked atom ("energy" of the displacement cascades). Indeed high energy primary atoms can displace secondary atoms, which can themselves displace other atoms, and so on. The material suffers an internal irradiation by projectiles which transfer the whole of their energy to the knocked-on atom during a head-on collision. When the energy of primary or secondary atom, or eventually of an ion further down in the cascade, is between 1 keV and 50 keV typically, its mean free path becomes comparable with the interatomic distances. It will therefore set to motion a considerable number of atoms, up to several hundreds, inside a volume ranging over a few tens of angstroms in radius. We have then a displacement cascade. In chemically ordered compounds, disorder is introduced by replacement sequences: strings or rings of atoms moves by one atomic distance in the process of Frenkel pairs formation. Also, it must be keep in mind that the fraction of the projectile energy which is transferred to the lattice in the form of defects or chemical disorder is very small, the larger part is dissipated into heat. The material heats up. The results obtained from numerical simulations greatly contribute to the understanding of the morphology and the time dependent evolution of the displacement cascades.
The debris of a displacement cascade is at the origin of all microstructural evolution that may be observed in the metallic structure. A general description is a vacancy rich core surrounded by an interstitial rich periphery. The vacancy rich cores often collapse into clusters also leaving free vacancies. The interstitials behave in the same way. In other words, point defects clusters (interstitial or vacancy) are formed as well as Frenkel pairs.
Even at very low temperatures, where the point defects are immobile, the concentration of Frenkel pairs will saturate (1%) as irradiation proceeds. At large point defects concentrations the distance between defects is small and athermal recombination or agglomeration will operate. As a consequence, the rate of accumulation of Frenkel pairs under low temperature irradiation is parabolic. At higher temperatures, point defects migrate by thermally activated jumps, and can thus disappear because of a large variety of defect/defect reactions.
In general the point defects concentration is a result of the competition between defects production and elimination. Three major mechanisms contribute to defect elimination: i) vacancy-interstitial recombination, ii) point defect elimination on defect sinks (dislocation, grain boundaries, free surfaces etc…), iii) agglomeration in the form of cavities, stacking fault tetrahedra, dislocation loops.
The point defect agglomeration and elimination at sinks induce the microstructural evolution.

2.2 Microstructural evolution

During irradiation, elements of the microstructure which act as point defects sinks absorb both interstitials and vacancies. If the number of absorbed vacancies differs from that of interstitials, lattice sites are created or destroyed at the sink: i.e. cavities will grow, dislocation loops will grow or decay, dislocation climb will increase the dislocation density. Point defects may also agglomerate. The most commonly observed evolutions, at least in cubic materials are as follow: 1) for small cumulated damage (1 dpa or less), dislocation climb, their density increases. If the dose rate is large enough, interstitial dislocation loops nucleate and grow in size. When the number density is large enough, dislocations interact and a dislocation network builds up; 2) for large doses (5 dpa and above) and under appropriate conditions cavities may nucleate and grow.
Whatever the irradiation conditions, self interstitial atoms always cluster in the form of a disk of atoms inserted between two dense planes: the disk edge is an interstitial dislocation loops. Vacancy clusters, on the contrary, are either two- or three dimensional objects: respectively vacancy loops or cavities and stacking fault tetrahedra (in the FCC structure).
The time evolution of the population of free vacancies and interstitials is described by the solution of the set of ordinary differential equations:

$$\frac{\partial c_v}{\partial t} = G_v - R\ D_i c_i c_v - \sum_j k_{j,v}^2 D_v c_v + G_{v,e}$$

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cv et ci are the vacancies and interstitials concentrations (per unit volume). The first term is the point defects creation rate. The label refers to sink type and characteristics. The term $G_{v,e} = \sum_j k_{j,v}^2 D_v c_{v,j}^e$ correspond to the vacancies emission from sinks. $c_{v,j}^e$ is the equilibrium vacancy concentration at each sink

In the absence of irradiation, a binary alloy may be either in the form of: i) a solid solution (where A and B atoms are distributed either in a random manner or with some degree of local order), ii) of a long range ordered structure (the A and B atoms are arranged in a periodic manner on a single lattice), iii) a two phase alloy, the second phase being either a solute rich solid solution or an ordered compound. If the alloy is prepared by an appropriate high temperature treatment in the form of a solid solution it will achieve on further annealing at a lower temperature one of the above structure.

Under irradiation the evolution may be completely different. Indeed the existence of a point defect supersaturation and the fact that point defect concentration on sinks is the equilibrium one leads to gradient and fluxes of point defect. According to the formalism of the thermodynamics of irreversible processes, the fluxes of species are dependent. As a function of the different concentrations, fluxes of the species **J** in a binary alloy **(**A-B) under irradiation may be written:

$$\begin{cases} -\mathbf{J}_v\Omega = D_{vv}\nabla c_v + D_{vB}\nabla c_B \\ -\mathbf{J}_i\Omega = D_{ii}\nabla c_i + D_{iB}\nabla c_B \\ -\mathbf{J}_B\Omega = D_{Bv}\nabla c_v + D_{Bi}\nabla c_i + (D_{BB}^{v} + D_{BB}^{i})\nabla c_B \end{cases}$$

D_{ij} is the diffusion matrix element. Thus, the evolution of the point defects population in the binary alloy under irradiation as well as the B concentration is given by :

$$\begin{cases} \dfrac{\partial c_i}{\partial t} = G - \mathrm{div}\mathbf{J}_i - R_{iv}D_{ia}c_i c_v \\ \dfrac{\partial c_v}{\partial t} = G - \mathrm{div}\mathbf{J}_v - R_{iv}D_{ia}c_i c_v \\ \dfrac{\partial c_B}{\partial t} = -\mathrm{div}\mathbf{J}_B \end{cases}$$

If the cross diffusion coefficient can be neglected, the only effect of irradiation is to sustain a large defect supersaturation and therefore to increase the solute diffusion coefficient. The precipitation process will be accelerated. The above argument implies that the defect supersaturation does not modify the thermodynamics of the system, which is the case at least in the metallic alloys. This phenomena is called : *irradiation enhanced precipitation*.

In many cases, the cross diffusion terms cannot be ignored and a solute segregation or deplpetion appears at the point defects sinks. If the solubility limit is exceeded, precipitation occurs. Since such a precipitation process only occurs because of irradiation and at predetermined location (defect sinks) it is currently named : *irradiation induced heterogeneous precipitation*. In certain undersaturated solid solutions, *irradiation induced precipitation can proceed in an homogeneous manner*, i.e. not associated to point defects sinks. Here again the cross diffusion terms are at the origin of the clustering of solute. However what triggers the defect flux is not the presence of a defect sink but the fact that the recombination of Frenkel pairs may be enhanced in a solute rich region.

3. EFFECT OF IRRADIATION ON STRUCTURAL MATERIALS OF NUCLEAR REACTORS

In regard to structural materials of current reactors, a better understanding of ageing is essential to justify the lifetime extension of pressurized water reactors (PWR), beyond the 40 years design life. In 2011, 58 PWRs are in operation in France, and 2 units have passed their third 10-year inspection. For 27 of the 900 MWe reactors, the regulatory surveillance program has provided data for irradiation doses close to those expected after 40 years of operation. The correlation used in France up until 2008 to evaluate the Reactor Pressure Vessel (RPV) embrittlement was based on an empirical formula (FIS) [10], developed from test reactor data. Its upper bound character is satisfactorily verified with the most recent surveillance data; however, it underestimates the general trend of embrittlement increase with irradiation dose. For this reason and in view of life extension, the use of an improved correlation is necessary for the embrittlement assessment at higher fluence. The work to determine a new correlation is carried out in several steps: comparison of existing correlations with the surveillance data, constitution of a database, observations of irradiated microstructures, choice of a model form, model fitting and validation. The difficulty today is that very few data exist for high doses and results from accelerated tests are not fully reliable due to the complex effects of dose rates. In addition, high doses may also produce additional hardening features like the so called « late blooming phases [11]. To go beyond the correlations and understand the phenomena occurring at high doses, analyses of the different irradiated microstructures (plate, forging, welding materials) is urgently needed in order to develop models of precipitation and defect accumulation. The long term evolution of internal structure austenitic steels (swelling, irradiation assisted stress corrosion cracking) represents also major stakes, and again, analyses at the nanometer scale are crucial to understand and predict the degradation phenomena [9].

For cladding materials of the current reactors (zirconium alloys), work needs to be carried out on the damage mechanisms (creep/growth under irradiation, corrosion by the primary circuit, stress corrosion cracking enhanced by iodine) that limit the burn up of the fuel assemblies and availability of the reactor [12]. The observation of these phenomena at the atomic scale could for instance help to understand how a few ppm of sulfur can have such an impact on the creep properties of the cladding and in general contribute to the optimization of their chemical composition.

Extensive work is needed on projects for a sustainable nuclear energy to allow technological breakthroughs and innovations for all GenIV reactor types. In particular, structural materials must withstand high fast-neutron fluence at high-temperature as

well as to comply with new reactor coolant [13] which will require the development of new materials: ODS/CDS/NDS (oxide, carbide, nitride dispersed strengthened steels), ceramic materials such as SiC/SiC composite, etc. The R&D activity will also be concerned with the coating or functionally gradient materials. For example, the choice of ODS for SFR cladding needs to be validated, and the choice of a cladding material is one of the principal technological hurdles for the development of GFR [14]. The strength of this material at high temperatures has been demonstrated but its behavior under irradiation still needs to be assessed.
For fusion reactors the stakes relate to the development of structural materials adapted to the operating conditions for the first wall, the divertor and the blankets.
For radioactive waste, optimization of the recycling of long life elements, resulting from the reprocessing of used fuel, is required. The aim is to separate them and to burn them in fast neutron reactors (transmutation) or in dedicated systems ADS (Accelerator Driven System) type. These long-life elements will be incorporated in a transmutation matrix that will resist to very high irradiation doses.

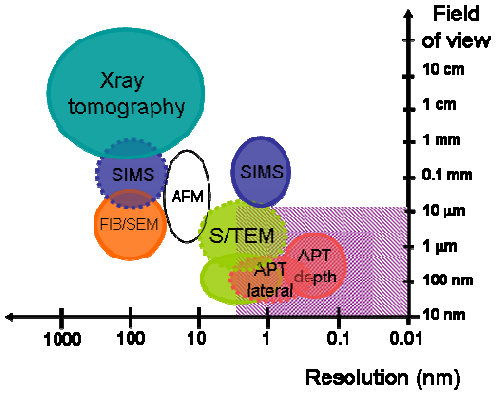
4. TRACKING MATTER AT THE ATOMIC SCALE

4.1 High resolution techniques

The elementary mechanism at the origin of the degradation of materials under irradiation occurs at the atomic scale. Thus tracking matter at the atomic scale is important to improve our understanding of irradiation effects in materials, a fortiori for structural materials for nuclear reactor. Many microstructural studies have been performed to account for the formation of extended defects as voids, precipitates, loops … and to predict the lifetime of materials. Between the 70's and the 90's, the conventional TEM and conventional spectroscopy brought a lot of information to confirm or to improve our understanding of these phenomena. However, the observation of the irradiation effects in matter was not accurate enough and many phenomena were still not understood or just suggested. After the 90's the combination of higher resolution instruments such as Atom Probe Tomography, HRSTEM as well as Small Angle Neutron Scattering gave new insights in this research field. However their application to active materials or material as different as ceramics, glasses, metals and alloys (mixed with oxides nanoparticles) and nanostructured materials were only taking their first faltering steps, the instruments were still in development and dispersed.

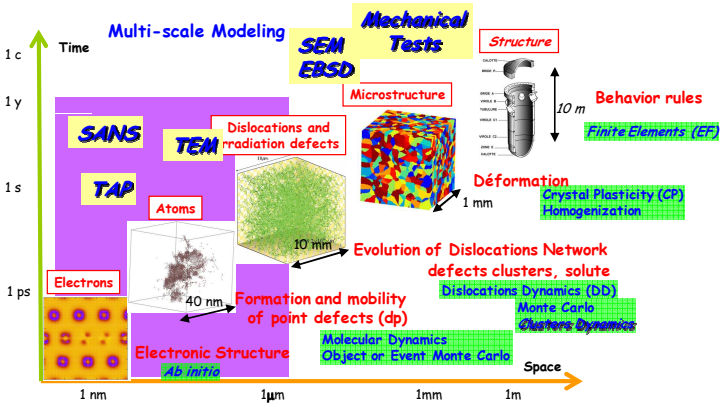
Now, with the progress in scientific instrumentation, high resolution analytical microscopy tools are available and mature: GIF Quantum Filter, Cs corrector for TEM, sample holders for in-situ straining, heating, tomography for high resolution scanning transmissions electron microscope, large angle femtosecond laser pulsed atom probe tomography with various wave lengths in order to quantitatively analyze different kinds of materials in the best and reproducible conditions, Cross Beam Station where the nanomanipulation and nanomachining of samples can be routinely performed. These techniques and their coupling are nowadays the state of the art techniques to go further in the research of the irradiation impact on the ageing and integrity of nuclear structural materials.

The following figure illustrates the high resolution nanoanalytical tools and the continuum scale of observation using these complementary techniques.



The spatial resolution and field of view of Atom Probe Tomography (APT), Scanning Transmission Electron Microscopy (STEM), Scanning Electron Microscopy (SEM) with Focused Ion Beam sectioning and X ray Tomography are compared. Full circles are for depth resolution, dash circles for lateral resolution. It is universally accepted that experimental studies of irradiation effects require techniques falling in the purple square. The potential of Atom Probe Tomography coupled with TEM is evident. For ceramics, a statistical approach by X-ray diffraction is a mandatory complement of these local techniques.

One of the goals of these experiments is to establish a strong link between experiments and the physics-based multi-scale modelling of irradiation effects. Various simulations are used at various scale, as well as their coupling (Ab-initio calculations, Molecular Dynamics, Object or Event Kinetic Monte Carlo, Cluster Dynamics, Dislocation Dynamics…see C. Becquart presentation). This approach has reached a highly predictive power, in particular thanks to ab initio methods, which allow the calculation of the properties of radiation defects (structure and mobility of vacancy and interstitial type defects). Experimental data at the appropriate scale (i.e. atomic scale) are however often lacking to validate these simulations. This strong modelling activity needs experiments in order to validate some of the latest concepts proposed in particular regarding the unexpected properties of some radiation defect clusters and complexes. Conversely, experimental results also stimulate new simulation studies and developments. The scale to scale modeling and experiments are given on the figure below.



These experiments will allow validation of the results obtained from the physical models and their ongoing developments. These experimental validations of physical models describing the first stages of irradiation are very important for their use in predicting of long-term behavior of different materials and the lifetime of components.

4.2 Research still going on

Even if experimental techniques are increasing their capabilities and performences, as well as modelling, numbers of questions in these fields are still open.

In bainitic steels used for the Light Water Reactor (PWR or BWR) vessels for which the irradiation dose is very low (< 0.1 dpa even for 60 years), the irradiation leads to both nucleation of clusters consisting in minor elements of the steel (as seen by APT) and point defect clusters (it is still unknown today if they are small dislocations loops? [16-21]). These clusters are too small to be studied by conventional transmission electron microscopy. A few results are available but limited to low-activity samples. The results need to be completed by a more systematic study of different vessel materials and highly irradiated (representative for 40 years and beyond) to determine chemical effects on clusters and cluster kinetics but also to determine the nature of point defect clusters. This is essential in order to propose new approaches and formula closer to microstructural evolutions and to have a more reliable extrapolation of macroscopic behavior of materials and the lifetime of this component.

For austenitic steels used for internal structures of LWR or for subassemblies of sodium fast neutrons reactors (SFR) for which irradiation is very high (active samples), analysis at atomic scale [22-26], with atom probe tomography for example, is very important to determine the first stages of microstructural evolution, segregation at different sinks (surfaces, grain boundaries, dislocation lines and loops …), precipitation induced by irradiations (γ', G-phases…). This information is necessary to improve our understanding and thus the modeling of swelling resistance that is very dependent on the element in solution (Cr, Ni, Si, Mo, N …), radiation-induced hardening, irradiation creep or stress corrosion cracking (Irradiation Assisted Stress Corrosion Cracking). The knowledge of the local stress state and segregation at surfaces and grain boundaries will help to determine mechanisms at the origin of IASCC. However, techniques described in the previous paragraph only give a partial view of irradiation effects. Indeed, even if APT is the most suitable tool to characterise nanometric chemical heterogeneities, it is not able to detect crystalline defects such as nanovoids, cavities or dislocation loops. Such features largely contribute to irradiation-induced ageing of materials. Transmission Electron Microscopy (TEM) is a complementary technique of APT since it allows observation and characterisation of these small defects. So the combination of these two techniques is an important step toward the understanding of basic mechanisms of material ageing under irradiation. The instrument needed for such experiments should be equipped with spherical aberration (Cs) corrector, which allows Scanning TEM imaging with a high resolution. Energy Dispersive and Electron Energy Loss Spectroscopies (respectively EDS and EELS) detection systems would be implemented, allowing chemical analysis from lightest to heaviest elements, and open to radioactive materials. Thus it will be possible to identify and quantify the structural defects induced by the neutron irradiation but also to estimate local variations of chemistry associated to these defects.

For cladding materials of LWR (Zr alloys [27-33]), atomic scale studies must give additional information on dissolution of Laves phases which are present before irradiation in Zr alloys and also on nucleation of β-phases in Zr-Nb alloys under irradiation and their evolution at high doses.
Materials proposed for the future SFR cladding [34,35] are ferritic-martensitic alloys reinforced by an uniform dispersion of oxides. The presence of nano-oxides (radius 1 – 2 nm) in these alloys provides good creep deformation resistance. Also, these nano-oxides allow trapping of the hydrogen and helium produced by nuclear reactions, mainly in Fusion conditions. The nanoscale of oxides makes very difficult to study by conventional transmission electron microscopy and asks means at this scale as TAP, HRTEM and GIF system. Mechanisms at the origin of the creep resistance are not completely understood and need complementary studies at the scale of the oxides, interaction with dislocations, interfaces with matrix ... Also, the stability of these nano reinforcements under irradiation and their interface with matrix governed the evolution under irradiation and aging of mechanical properties and dimensional stability of this kind of steels.

For ceramic materials [36], same methods must be applied. However, one has to take into account to some specific phenomena which are absent in metallic systems: presence of several sub-lattices which behave differently under irradiation (induced disorder in cationic sub-lattice in spinels for ex.), effect of the electronic excitation either on the damage process or in the irradiation induced annealing (in SiC for ex.), different charge states of the defects which influence their diffusion… Moreover, one must have tools for the characterization of the phase transitions (monoclinic to tetragonal in zirconia for ex.) or of the amorphization kinetics, the grain rotation and the grain fragmentation… All these phenomena can be understand only if they are characterized at the nanometric scale with local approach (analytical HRTEM) for the defects and impurities clustering and for the evolution of the dislocation network, or with a statistic approach (X-ray diffraction) for the evolution of the structure, the texture, the grain size, the residual stress...

Also other large instruments must be considered : Synchrotron SOLEIL with the Multi-Analysis on Radioactive Sample (MARS) beamline and Leon Brillouin Laboratory as well as Pierre Sue Laboratory.

5. CONCLUSION

“It is only a paper reactor until the metallurgist tells us whether it can be built and from what.”

Norman Hilberry, Director, Argonne National Laboratory, 1957 to 1961

This laconic statement made by N. Hilberry in the late fifties is even more appropriate nowadays. Materials are at the forefront of nuclear power. It was true for Generation I reactors in the 1950’s, it is true for present Generation II and III reactors and is even more acute for tomorrow’s Generation IV and fusion reactors: the challenging operating conditions imply that in most cases, the use of conventional nuclear materials is excluded, even after optimization and a new range of materials has to be developed and qualified for nuclear use. Research in physical metallurgy, mechanic of materials and modelling is needed as well as their teaching as offered at this school: *Effets d'environnement sur le comportement mécanique et la dégradation des matériaux*, AUSSOIS (21-25 01/13).

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