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Modelling of long term kinetic evolution: a fruitful relationship between experiment and theoretical development

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Abstract

Recent developments in multi-scale modelling, based on atomic scale calculations, are leading to a growing conviction that modelling will soon be used to design material components for nuclear reactors. In this article we discuss this assumption on the basis of the relationship between experimental studies and theoretical calculations of the microstructural evolution of materials under irradiation. In the first part of the paper, the available numerical models for long term microstructural evolutions are briefly reviewed. The experimental methods are presented in a second part. In the third part, several examples of fruitful relationships between modelling and experiments are discussed. ***To cite this article: A. Barbu, C. R. Physique 9 (2008).***

Résumé

Modélisation des cinétiques d'évolution à long terme : une fructueuse confrontation entre expériences et développements théoriques. Les développements récents de la simulation multi-échelles, fondée à l'échelle atomique ont amené la communauté à penser que la modélisation serait bientôt utilisable pour concevoir les matériaux des composants de structures des réacteurs nucléaires. Dans ce papier, nous discutons cette hypothèse séduisante sur la base de la confrontation entre expériences et développements théoriques. Les modèles numériques existants pour simuler l'évolution microstructurale à long terme des matériaux sont rappelés dans une première partie. Dans la seconde, les méthodes expérimentales utilisables pour valider ces simulations aux différentes échelles sont présentées. Enfin, quelques exemples de confrontations fructueuses entre expériences et développements théoriques sont présentés dans une dernière partie. ***Pour citer cet article : A. Barbu, C. R. Physique 9 (2008).***

Keywords: Multi-scale modelling; Kinetic evolution

Mots-clés : Simulation multi-échelles ; Évolution cinétique

1. Introduction

Modelling of the long term kinetic evolution of microstructures under irradiation makes sense only if validation with experiments is carried out. Indeed, the elementary mechanisms must be known and characterized quantitatively in order to provide parameters for multi-scale modelling. Except perhaps for ab-initio calculations which are very robust but limited to the determination of the properties of very small objects, methods based on empirical potentials

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are not safe and must be validated by experiments. Furthermore, it is always possible to omit an important mechanism. Critical comparison between experiments and theoretical developments is not only useful for validating code, but also cross fertilizes both, as has been the case since the beginning of material science under irradiation.

A short critical review of the various numerical codes available to treat the long term evolution of microstructures is given first. Then, the available experimental methods for validation of the multi-scale modelling at the different scales are discussed. Some typical examples of fruitful relationships between experimental data and results of simulation are presented. Finally perspectives concerning the coupling between multi-scale modelling and experiments are discussed.

2. Modelling of the long term evolution of the microstructures induced by irradiation

There are obviously no numerical codes able to treat the totality of the microstructural evolution under irradiation. Two types of code can be distinguished:

- Atomistic Kinetic Monte Carlo (AKMC) and Mean Field (MF) models, which are particularly well adapted to treat the micro-chemical evolution such as segregation and precipitation;
- Object Kinetics Monte Carlo (OKMC), Event Kinetics Monte Carlo (EKMC) and Cluster Dynamics (CD), which are well adapted to the clustering of point defects or solute atoms in the dilute approximation.

2.1. AKMC and MF codes (Soisson [1], Barbe and Nastar [2])

AKMC considers a rigid crystallographic lattice. Each reticular site is occupied by an atom of type A or B (for a binary alloy) in interaction or by a point defect. Pair interaction energies are usually considered. The whole thermodynamics is consequently taken into account. The atoms move via point defect jumps. A Monte Carlo method based on the time residence algorithm, which considers that the jump probability depends on the pair interactions at equilibrium position and at the saddle point, is used. This model is particularly well adapted to treat segregation and precipitation in concentrated or dilute alloys.

MF models have the same basis as AKMC. However, only the very near neighbours of the jumping defects are treated in detail. Outside, the system is treated as a mean medium. If the space is taken into consideration, this model is also able to treat segregation at grain boundary or surfaces in concentrated alloys. In addition, the Onsager coefficients for matter transport by vacancy or self-interstitial atom (SIA) in crystalline solid solution can be obtained using this method (M. Nastar [3], in this volume).

2.2. OKMC and EKMC modelling (Domain, Becquart, Malerba [4], Dalla Torre et al. [5])

Both models do not consider atoms but objects such as point defects, point defect clusters, solute atoms and solute clusters. Each object is referenced in space by using a discrete coordinate for OKMC or by a continuous coordinate for EKMC. The physical system can be seen as a gas of clusters. Consequently, the concentrations of objects must be necessarily low. This condition is generally satisfied for point defect and point defect clusters. Concerning solute atoms, only dilute alloys can be treated. The location of the objects is only defined by their centre of gravities. Their shapes are not considered explicitly.

In OKMC, the underlying array used to locate the object is usually the actual lattice array of the crystalline structure. The movement of the objects is described by a set of jump frequencies from lattice site to lattice site. The effective occurrence of jumps is treated by a time residence algorithm. Reactions between two objects take place when the distance between them is smaller than a reaction radius depending on the nature of the objects. A cluster can also emit monomers (single SIA, single vacancy, single solute atom). Input data, jump frequencies and binding energies, are in principle those obtained at a higher level of the multi-scale modelling using atomistic calculations based on first principles or empirical potentials (Fu and Willaime [6], in this volume; Fu et al. [7]). The primary radiation damage is obtained from a data base of displacement cascades given by Molecular Dynamics simulations (Bacon [8]). Cascades are introduced at random in the OKMC simulation box according to the irradiation flux and energy spectrum of primary knock-on atoms.

In EKMC, the positions of the objects are continuous and so the detail of the trajectory is not considered: jumps are gathered into trajectories obeying a continuous time diffusion law. It is the event probabilities that are considered

in these methods and not the jump probabilities. The advantage is that the jumps which do not give rise to an event (a reaction between two objects) are not treated explicitly. EKMC is consequently very efficient when the number density of objects is low. The EKMC input parameters are the same that those for OKMC but for diffusion coefficients instead of the set of jump frequencies.

These models are particularly well adapted to simulate the microstructure evolution in terms of point defect clusters such as dislocation loops and voids. They are also able to treat the effect of impurities such as carbon atoms present initially or He atoms generated in nuclear reactions, He bubble formation and evolution and, if the solute content is low, precipitation.

2.3. Cluster dynamics simulation (Hardouin Duparc et al. [9], Christien and Barbu [10])

In OKMC and EKMC modelling, correlations between objects of the microstructure are fully treated. The drawback is that the simulated volume is limited to a cube of size some microns and that microstructural evolution cannot be simulated under actual conditions up to the end of life of the structural elements of nuclear reactors.

This is not the case in cluster dynamics (CD) models. This is at the expense of the loss of spatial correlations. As in OKMC and EKMC models, the system is seen as a gas of clusters with the same consequence that the total volume fraction occupied by the clusters must be small. CD is based on rate theory. Consequently the real system is replaced by an effective medium in which all processes occur continuously in time and space. The evolution of the number density of clusters of each size n and of a certain type, embedded in the effective medium is described by a set of ordinary differential equations that can only be solved numerically. Indeed, to reach the typical size of dislocation loops or voids which are observed experimentally, for instance by transmission electron microscopy, the number of differential equation is very large. Several approximate numerical schemes are used to reduce this number. It is worth noting that the CD codes are particularly efficient in terms of computational cost, allowing an exploration of a large range of experimental conditions (temperature, flux). The inputs are exactly the same as for EKM.

3. Experimental methods for validations

Two kinds of experiments are to be considered. The first allows the validation of the elementary mechanisms. The second consists of experiments for determining the microstructures obtained at large fluences during continuous irradiation at temperatures at which all kinds of point defects are potentially mobile or effectively mobile.

3.1. Experimental irradiations

Various types of irradiations can be used to validate multi-scale modelling: MeV electrons, neutrons, MeV ions and GeV ions.

Irradiations with 1 MeV electrons are interesting as a first step for validation because the primary damage induced by such particles is very simple. It consists mainly in Frenkel pairs (FP): a SIA and a vacancy. Of the particles which produce displacement cascades, the most interesting in principal are neutrons as these are present in actual nuclear plants. Unfortunately, experiments with neutrons are very difficult to perform and very expensive. Furthermore it is not always possible to introduce an appropriate experimental device in the core of the reactor. In addition, samples become radioactive and are difficult to handle. This is the reason why experiments carried out with heavy ions are very interesting. Heavy-ion irradiations do not activate the sample if the ion energy is not too high and are easy to carry out. It is consequently possible to validate the modelling over large ranges of external parameters (temperature, flux, fluence). The main difficulty with this kind of particle is their low penetration in material. The higher the energy of the ion, the larger the penetration. For this reason, GeV ions would seem being the most appropriate. Unfortunately, at such an energy heavy ions are slowed down mainly by electronic excitations, and microstructural modifications can be produced even in metal and metallic alloys by electronic excitations (Barbu et al. [11]). The best compromise is ions of some tenths of MeV (Serruys et al. [12] in this volume, about the JANNUS facility). The penetration of these ions is only some microns in steels. Surface effects can be important if samples are irradiated as thin foils for transmission electron microscopy or tips for 3D atom probe. Although often pointed out as being a great concern, surface effects are not a real problem for validating multi-scale modelling, as free surfaces can be taken into account in the modelling. As shown on Fig. 1, an interesting point is that the thickness of TEM thin foils or the dimensions of

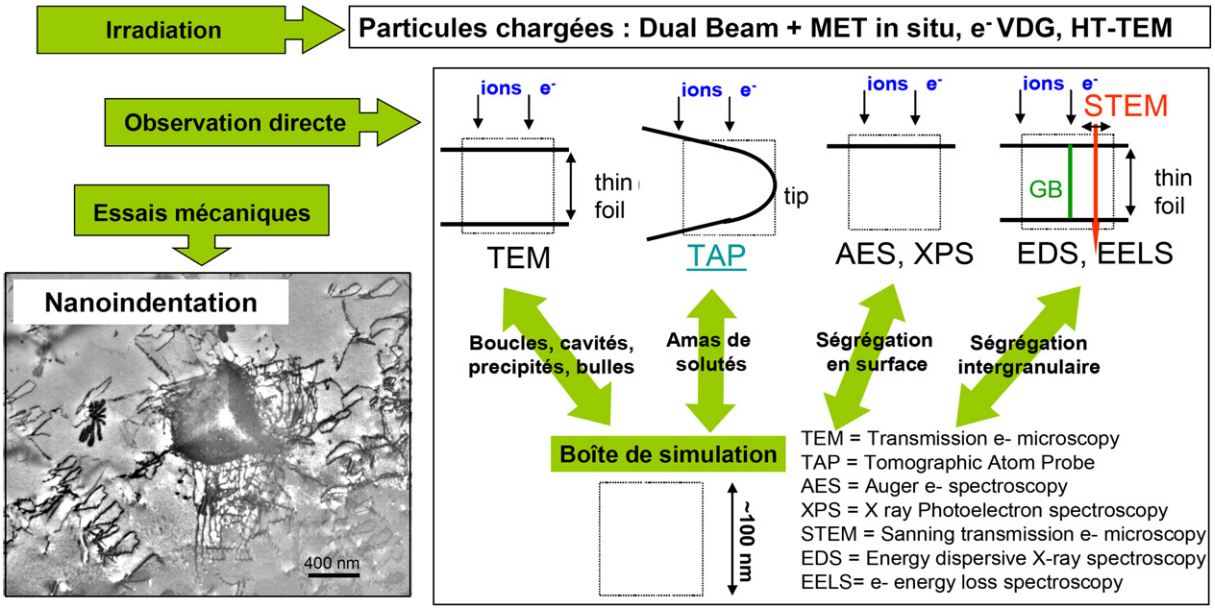

Fig. 1. Relationship between sample geometry for ion irradiation and KMC numerical simulations.

Fig. 1. Comparaison de la géométrie des échantillons pour irradiation aux ions et des cellules de simulation numérique par Monte Carlo cinétique.

the volume investigated by 3D atom probes have the same order of magnitude as the characteristic sizes of the volume simulated by Monte Carlo methods.

It is worth noting that if multi-scale modelling is not able to reproduce the microstructure of electron irradiated materials, it is useless to go further and to try to validate the multi-scale modelling of materials irradiated with particles producing displacement cascades.

3.2. Experimental validations at elementary level

This is mainly a question of validation of the primary damage and of the properties of point defect and small point defects clusters. To be studied experimentally, the defect must be frozen: irradiation must be carried out at temperature at which defects are immobile. As SIA clusters are very mobile if not extremely mobile in metals, liquid helium temperature is generally required.

Experimental determination of the atomic structures of SIAs and SIA clusters is very difficult. As an example, beautiful diffuse X-ray scattering experiments can be mentioned. In these experiments, electron irradiations as well as the acquisition of the X-ray spectra were carried out at 4 K (Ehrhart [13]). To our knowledge, no such a data have been reported for iron probably because of the necessity to have very good single crystal.

Inelastic relaxation experiments can give information about the geometry of the jumps of defects. The method only applicable if the defect possesses a lower symmetry than that of the crystal in which it resides (Nowick [14]). It is consequently not applicable to vacancies (at least single vacancies). In this method the temperature of the experiment must be such that the defect is able to jump but at a not too high frequency. Furthermore, only jumps implying a rotation of the defect can be studied (simple rotation or translation-rotation). More information is obtained if the sample is a single crystal. Such an experiment has been carried out on poly-crystalline iron (Hivert et al. [15]). These authors show peaks that can be reasonably attributed to the translation-rotation jump of $\langle 110 \rangle$ SIAs with an activation energy of 0.32 eV in good agreement with first principles calculations (Willaime et al. [16]).

However, the most extensively used method is based on electrical resistivity. This has the advantage that it is very easy to carry out compared to the methods mentioned above but with the disadvantage of been not selective.

In isochronal resistivity recovery, irradiations are usually carried out with one to two MeV electrons at liquid hydrogen temperature in order to produce immobile FPs at a concentration in the range 1–100 ppm. Isochronal an-

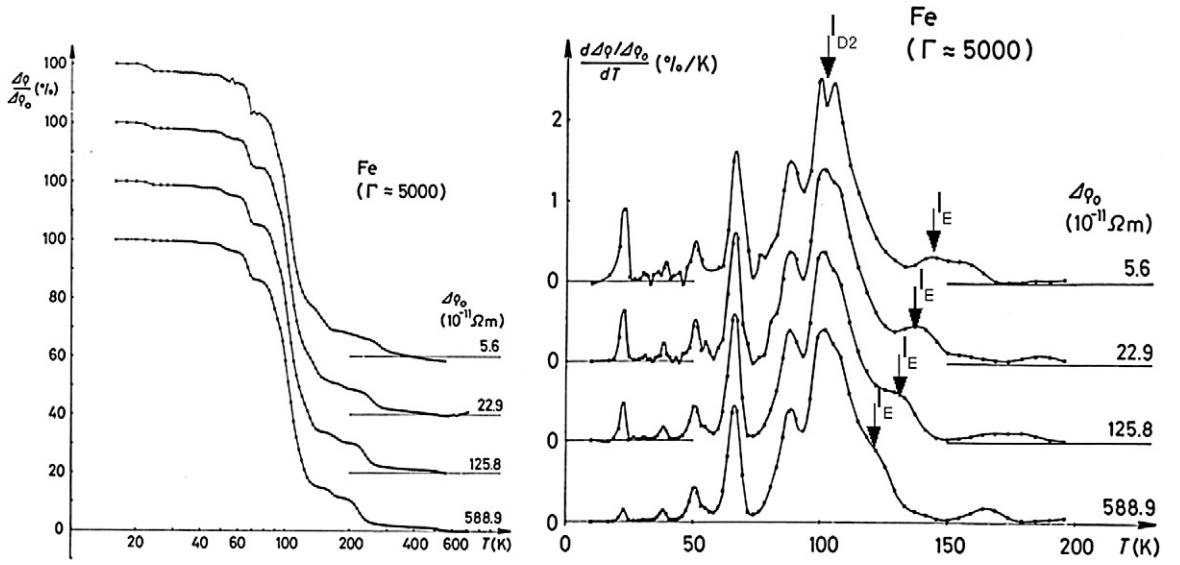

Fig. 2. Isochronal recovery of high purity iron up to 200 K for various initial Frenkel pair concentrations (Takaki et al. [17]).

Fig. 2. Recuit isochrone de fer de haute pureté jusqu'à une température de 200 K pour différentes concentrations initiales de paires de Frenkel (Takaki et al. [17]).

neals of typically 10 minutes are carried out; the temperature is increased after each annealing period. The resistivity measurement is performed at 4 K between each isochronal anneal.

Typical isochronal recovery curves, obtained on ultra pure iron, are shown on the left of Fig. 2 (Takaki et al. [17]). Each drop in resistivity corresponds to a reaction between defects due to the activation of the mobility of at least one defect. The differential curve is usually preferred for obvious reasons (Fig. 2 right). Each peak is usually called a stage.

Being a global quantity, electrical resistivity is unable to determine by itself what physical phenomenon is associated to each peak. Based on other experimental methods or atomistic calculations and intuition, some reaction can be attributed to a given peak. The properties of the peak, such as its change in position with the initial FP concentration, give precious insights into the reactions involved. For instance (Fig. 2), a peak attributed to the correlated recombination between a vacancy and the interstitial of the same FP (I_{D2}) does not move with the initial FP concentration as it is a property of the FP itself. Conversely, the recombination of an interstitial with a vacancy of another FP (I_E) depends on the initial concentration, because the distance between the partners depends on concentration.

The migration energy of a particular species can be obtained if the order of the reaction is known by performing isothermal annealing at two temperatures close to the relevant peak temperature.

Electrical resistivity has also been used extensively to study the primary damage i.e. the damage immediately after the collision between the incoming particle (electron, neutron, ion) and the atoms of the material (Wollenberger [18]). The method consists in following the electrical resistivity during continuous irradiation again at very low temperature. As for isochronal annealing, the great majority of experiments reported in the literature have been carried out using electron irradiations. By performing irradiations at various energies the displacement threshold energy E_d can be obtained. E_d depends on the direction of the target atom impulsion. The E_d distribution can be obtained by using single crystals. From the curve giving the electrical resistivity several elementary quantities such as the cross section of defect creation, the recombination volume, etc., can also be obtained.

The structure of primary damage induced by neutron and ion irradiation is very different from that induced by MeV electrons. Indeed, the transferred energy in neutron-target atom collisions is large enough to create large displacements cascades. Reported studies on samples irradiated with neutrons exist but are rare because of the difficulty to put an experimental device at 4 K in the core of a nuclear reactor. A very interesting result obtained using neutron irradiation is given in Fig. 3, which shows isochronal electrical recovery curves obtained after irradiation with fission and fusion neutrons (Matsui et al. [19]). Both curves are almost the same, showing that the radiation induced primary damage are

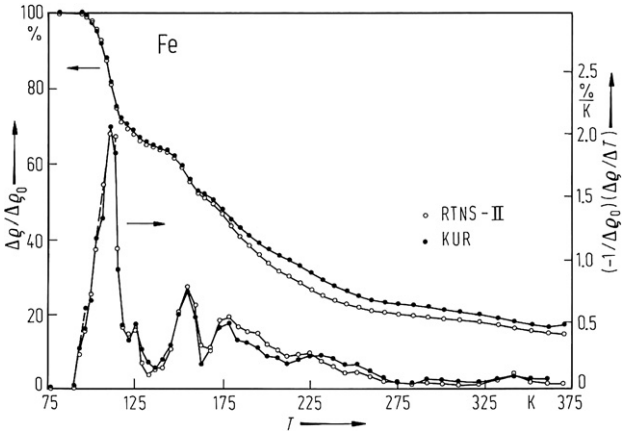

Fig. 3. Isochronal recovery of iron irradiated with fission-neutrons and 14 MeV fusion-neutrons (Matsui et al. [19]).

Fig. 3. Recuit isochrone de fer irradié avec des neutrons de fission et des neutrons de fusion de 14 MeV (Matsui et al. [19]).

practically identical but for the helium production. This can be understood by considering than the 14 MeV neutron cascades split into lower energy sub-cascades identical to those induced by fission neutrons.

Experiments of this type carried under ion irradiations are almost nonexistent. This is due to the small penetration of ions. An evaluation of the possibility to carry out such experiments in the new JANNUS facility is in progress.

3.3. Experimental validation of the long term microstructural evolution

The methods used to study the microstructural evolution during continuous irradiations, at temperatures at which defects are mobile, usually around one third of the melting temperature, are more classical and often more easy to perform. Transmission electron microscopy is used intensively to study point defect clusters if they are large enough. To study micro-chemical evolution such as radiation induced grain boundary segregation or precipitation, analytical transmission electron microscopy or, if a higher resolution is needed, 3D atom probe are commonly used. This latter method is particularly interesting when the size of solute atom clusters is below the resolution limit of TEM which, in diffraction or structure factor contrast, is usually not better than one nanometre.

Point defect clusters which are smaller than about one nanometre in size can play an important role on the mechanical properties of the materials but are difficult to study experimentally, especially SIA clusters. For very small vacancy clusters, positron annihilation techniques can be used if their number density is large enough.

4. Examples of fruitful relationships between experimental data and simulation results

As a first example, we will present the first and as far as we know the only case of a full multi-scale modelling based only on first principles (Fu et al. [7]). It is based on ab initio methods for the defect properties and on a Monte Carlo method for the kinetics. Obviously, it is a very simple case: the isochronal electrical resistivity recovery of electron irradiated ultra pure iron at 4 K mentioned above. As shown on Fig. 4, the recovery curve is very well reproduced. The initial interpretation of Takaki is only partially confirmed. Indeed, stage II must also be attributed to tri-interstitials and stage III must be attributed not only to single vacancies but also to tri and tetra vacancies. Furthermore, the migration of interstitial clusters I_n for $n > 3$ is not required to reproduce the experiments.

Concerning more complicated experimental situations, for example continuous irradiation of non-ultra pure metal, a full multi-scale modelling based on first principals is not yet practicable. Indeed the number of parameters required for such a simulation (such as the formation energies of clusters, the effect of impurities on clusters, the possibility of having mixed point-defect-solute clusters, the mobility of the cluster of all types and sizes) is so high that they cannot be all computed from first principles or even using empirical potentials. The best procedure in this case is certainly to start with the parameters of a simple model material, pure iron for instance for low alloyed ferritic steels. Then one performs a study of the sensitivity of the modelling to these parameters and adjusts the most sensitive ones

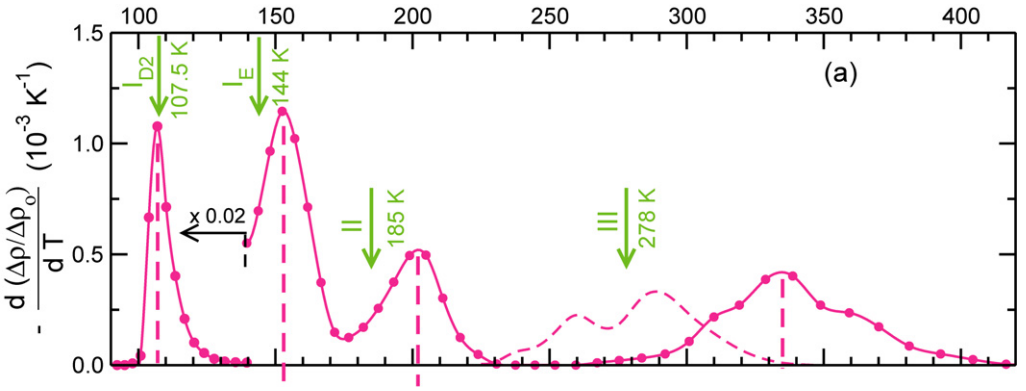

Fig. 4. Multi-scale calculation of the recovery curve of electron irradiated ultra pure Fe (Fu et al. [7]).

Fig. 4. Simulation multi-échelle de la courbe de restauration de la résistivité électrique dans le fer ultra pur (Fu et al. [7]).

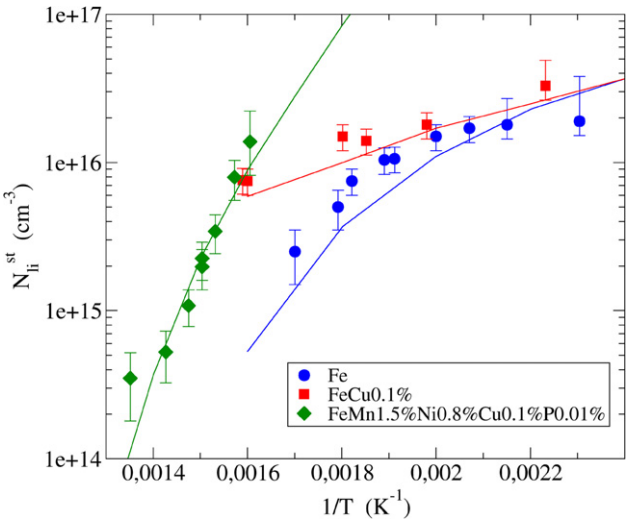

Fig. 5. Stationary number density of interstitial loops as a function of temperature in ferritic model alloys irradiated with 1 MeV electrons in an high voltage electron microscope.

Fig. 5. Densité stationnaire de boucles d'interstitiels en fonction de la température pour des alliages ferritiques modèles irradiés avec des électrons de 1 MeV dans un microscope électronique à haute tension.

within physically reasonable ranges, in order to reproduce the experimental results obtained using different external parameters. The validation consists in testing if the results obtained with some other set of external parameters are in good agreement with experimental results. It is clear that this way to proceed is not totally satisfactory but the possibility to carry out full multi-scale modelling in actual materials under the actual irradiation conditions is certainly not for the near future.

The second example deals with microstructural evolution of ferritic model alloys during continuous irradiation with 1 MeV electrons at large fluences (Hardouin Duparc et al. [9]). Irradiations and observations were carried out at a high damage and between 175 and 400 °C in the CEA Saclay high voltage transmission electron microscope. Fig. 5 gives the temperature dependence of the stationary number density of interstitial loops in a commercial iron, an Fe0.1%Cu alloy and a more complex alloy with composition is close to the matrix composition of RPV steels (symbols). The full lines are the calculated values. For reasons given above, cluster dynamics has been used in this case. To reproduce the experimental data, the vacancy migration energy obtained by calculation performed at the atomic level must be modified. Furthermore, it must be assumed that SIA clusters are immobile. Indeed, the samples being thin foils, any SIA cluster is immediately eliminated at the surface if it is very mobile. The reason that the vacancy migration energy

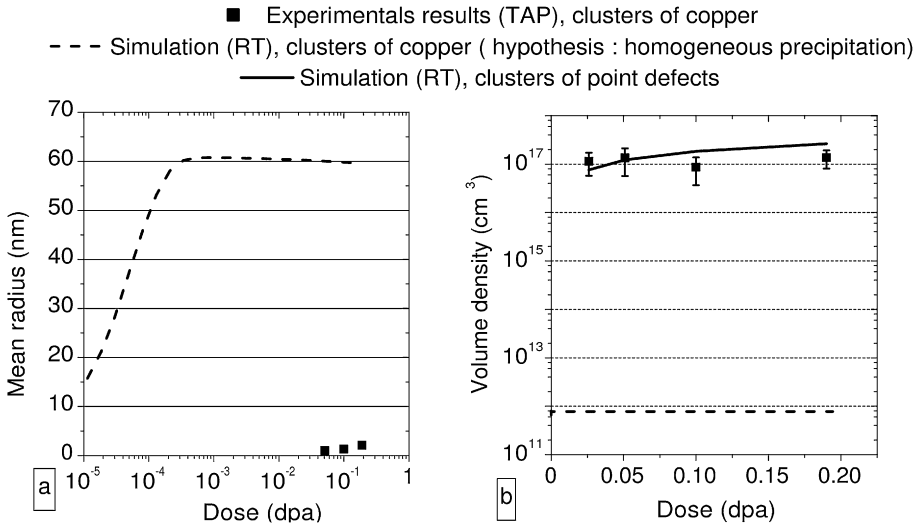

Fig. 6. Comparison between the calculated mean radius (a) and number density (b) of copper precipitates assuming homogeneous precipitation (doted line), the calculated density of point defect clusters (full line) and the experimental number density of copper clusters.

Fig. 6. Comparaison entre la densité calculée de précipités de cuivre en faisant l'hypothèse d'une précipitation homogène (pointillés), la densité calculée d'amas de défauts (ligne continue) et la densité d'amas de cuivre mesurée expérimentalement.

must be increased is clearly due to the trapping effect of carbon (or/and nitrogen) atoms, in agreement with ab initio calculations. The necessity to assume that SIA clusters are immobile or have a very low mobility is not understood (Fu, to be published). The effect of carbon on SIA is not relevant as ab initio calculations have shown repulsion between carbon atoms and SIA clusters (Domain, Becquart, Foct [4]) or more likely only a slightly attraction (Fu, to be published).

The third interesting example of a fruitful relationship between experiments and theoretical developments is the modelling of He desorption of helium implanted iron, which is presented in detail in another chapter of this report (Ortiz, Caturla and Fu [20], in this volume). In this study, numerical calculations were performed using an OKMC code. In good agreement with the result presented in the second example, the migration energy of vacancy is taken to be higher than the values given by first principal calculations and traps must be introduced to considerably slow down interstitial clusters. It is worth noting that this assumption was already introduced in OKMC simulations of the microstructural evolution of ferritic steels irradiated in reactor at low temperatures (Domain et al. [21]).

The fourth example concerns the mechanism of formation of solute rich clusters observed by 3D atom probe in irradiated pressure vessel steels of water pressurized nuclear reactors. It is usually assumed that such clusters are formed by homogeneous precipitation of copper-rich precipitates. The validity of this hypothesis was tested on a low copper content ferritic binary FeCu model alloys (Radiguet et al. [22]). A cluster dynamics code devoted to the formation of point defect cluster and the simultaneous homogeneous precipitation was used for this purpose. The parameters of point defect clusters were adjusted by performing an experimental study of point defect clusters under ion irradiation at a dose rate and fluences such that large interstitial loops were easily observed by TEM. The parameters describing the homogeneous precipitation of copper were acquired using experimental results obtained in irradiated high copper content alloys, typically around 1% Cu. Indeed for such a high concentrations, the mechanism has been shown to be unambiguously homogeneous (Christien and Barbu [10]).

A comparison was made of the calculated number density of copper precipitate in copper alloys containing only 0.1Cu, assuming an homogeneous precipitation, with the number density of the copper clusters observed experimentally by 3D atom probe in samples irradiated with neutrons in the BR2 Belgian experimental reactor. This comparison gave rise to the conclusion that the mechanism of formation cannot be homogeneous. Indeed, as shown in Fig. 6, the number density of precipitates assuming an homogeneous precipitation mechanism is 10^{12} cm^{-3} (dotted line) when the experimental value is 10^{17} cm^{-3} (squares).

The most reasonable assumption to explain the experimental high values is that nucleation of solute clusters is heterogeneous on point defect clusters. These point defect clusters are unfortunately not observed by TEM: either

they do not exist or they are too small to be observed experimentally by TEM. The number density of point defect clusters given by the cluster dynamics simulation shows that they are very likely under the resolution limit of the microscope and that their number density is of the same order of magnitude as the experimentally observed solute clusters (Meslin, to be published).

5. Conclusion

The constant improvements in the capability of computers raises the issue of whether the behaviour of materials under irradiation can be predicted quantitatively by modelling. At this time, it is possible to do this, starting from first principal calculations, only in very simple situations. For industrial materials submitted to actual conditions it is clearly not realistic. The parameters calculated for a simple material, surrogate of the actual material, have to be more or less fitted. Even if this is not totally satisfactory, it is worth noting that the attempt to perform multi-scale modelling has obliged researchers to address many issues that are not considered when only a simple qualitative understanding of the mechanisms is sought.

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