Nuclear materials characterization with Atom Probe Tomography

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Nuclear Materials (reactor vessel, internal structures, fuel rod, glass containment…) undergo degradations under neutron irradiation due to particle/matter interaction. These degradations modify their physical, chemical and mechanical properties and are likely to impact the safety or the operation length of the concerned structures. It is thus crucial to anticipate and quantify these effects in order to ensure an efficient use of present or future nuclear reactors. The phenomena controlling materials behavior under irradiation are on an atomic scale. Understanding the phenomena on this scale is thus essential for the development of a predictive simulation and understanding of the ageing and safety of materials.

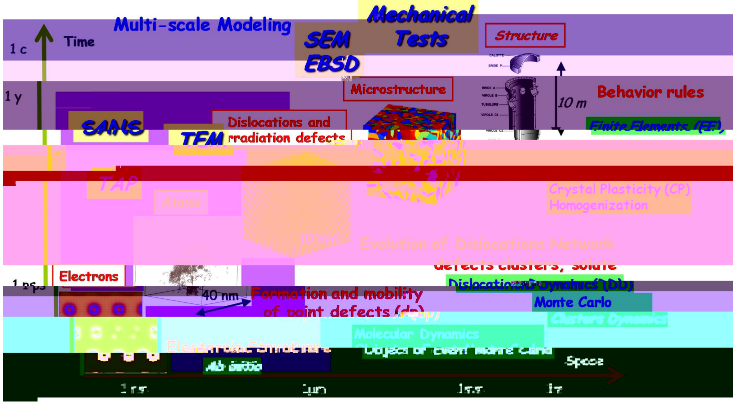

Figure 1: Development of numerical tools and experiments for a multiscale approach

This atomic scale is the first floor of the multi scale approach necessary for modeling and prediction, as illustrated in figure 1. The major technique in this field is the Tomographic Atom Probe, nanoanalytical technique with atomic resolution. Its association with TEM and SEM is of great relevance.

Atom Probe Tomography (APT) is an extension in 3D of the atom-probe field ion microscope (APFIM), an instrument designed in the late sixties by E.W. Müller [1]. APT is the only approach able to map out the 3D distribution of chemical species in a material at the atomic-scale. The principle of APT [2,3] is based on the field evaporation of surface atoms of the specimen (a sharply pointed needle, R ~ 50 nm) and the chemical identification of field evaporated ions by time-of-flight mass spectrometry. The position of atoms at the sample surface is derived from the impact position of ions striking the detector. A major advantage of APT is its quantitativity. The local composition in a small selected region of the analyzed volume is simply derived from the number of atoms of each observed species. This is a big advantage compared to number of other instruments. No calibration is required. The in-depth resolution, independent of technologic details of these instruments, reaches 10 picometers. Atomic planes can therefore be imaged and the chemical order can be both exhibited and characterised. The lateral resolution is close to 0.5 nm. This makes it possible the quantitative analysis of small precipitates in alloys or the study of solute segregation to crystal defects (as illustrated in figure 2 [4]).

The last few years have been the witness of a major breakthrough in the development of APT. Formerly limited to metals or good conductors, the implementation of ultra-fast pulsed laser to the instrument opened APT to semi-conductors or oxides [5,6]. Laser pulses give rise to very rapid thermal pulses that promote the field evaporation of surface atoms.

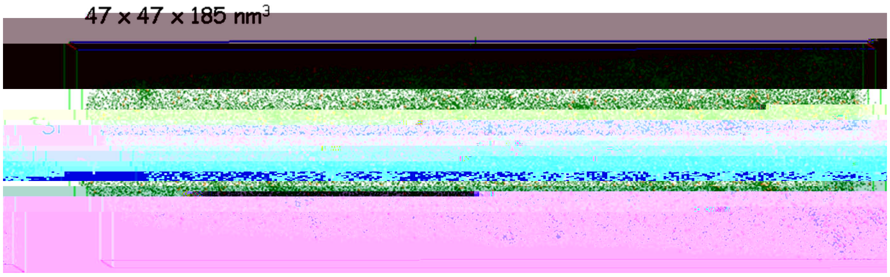

Figure 2 :Tomographic Atom Probe experiment in a RPV steel. Each dot represents an atom (Si or P here). Irradiation induced clusters and segregation on dislocation lines are visible.

In addition, a wider field of view is achieved so that larger area of analysis are obtained (~100x100 nm2) improving therefore statistics together with shortening the number of analyses required to get the relevant information.

This technique brings key information [7,8,9] for the understanding of the evolution of the microstructure of nuclear reactor steels under neutron irradiation, evolution responsible for their hardening and embrittlement.

The research laboratory GPM (Physic Materials Group) is world reknown in the use and development of the Tomographic Atom Probe technic and in the studies of the degradation of nuclear material under severe conditions (irradiation, temperature, environment…). The laboratory is also associated to research programs in collaboration with EDF or CEA in France and is leading a large technical plateform (TAP, SEM and TEM) devoted to the studies and nanocharacterization of radioactive materials.

Professor P. Pareige, Director of the GPM Laboratory and Dr. B. Radiguet, senior researcher in the field of nuclear materials will introduce these different aspects in two join talks.

In a first part, Pr. P. Pareige will describe the GPM Laboratory and its main research topics (Scientific Instrumentation, Physical Metallurgy and Nanosciences), will describe the main progress in the Atom Probe Tomography technique and give a description of the main activities in the field of nuclear materials and the new technical plateform for nanoanalyses of radioactive materials.

In a second part, Dr. B. Radiguet will illustrate these research activities in the framework of the development of materials for GEN IV reactors. It is well known that these reactors are challenging for materials due to their high operating temperatures, high neutron doses and corrosive environments. Different materials are under development in order to fulfil the requirements of Gen IV reactor cores and structures (ceramics, ferritic/martensitic (F/M) steels, Oxide Dispersion Strengthened (ODS) steels…). This second part will focus on ODS steels which are promising candidates since they exhibit a limited swelling under irradiation, characteristic of F/M matrix, and excellent creep and tensile properties at high temperatures, thanks to a dense dispersion Y, Ti, O nanoparticles. Two aspects will be tackle: (i) the APT characterization of these steels, taking into account the bias that can be induced by the field evaporation of the samples and (ii) the stability of the nano-particles under ion irradiation up to high doses.

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