

Materials subjected to fast neutron irradiation/Matériaux soumis à irradiation par neutrons rapides

JANNUS: experimental validation at the scale of atomic modelling

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Abstract

Ion irradiation is well suited to simulate neutron irradiation because primary knock-on atoms (PKA) produced by neutron collisions are self ions of the target. As the main difference, the energy spectrum of ion-produced PKAs is somewhat broader than in the case of fast neutrons. Studies of the combined effects of target damaging, ion implantation effects, helium and hydrogen production, and the occurrence of nuclear reactions should be performed by co-irradiation experiments (dual or triple beam irradiation). The JANNUS project (Joint Accelerators for Nanosciences and Nuclear Simulation) was started in 2002 in the frame of a collaboration between CEA (Commissariat à l'Énergie Atomique) and CNRS–IN2P3 (Centre National de la Recherche Scientifique–Institut National de Physique Nucléaire et de Physique des Particules). Two experimental sites are involved. At Saclay, three electrostatic accelerators are being coupled: a new 3 MV Pelletron™ machine equipped with an ECR multi-charged ion source, a 2.5 MV single ended Van de Graaff and a 2.25 MV General Ionex tandem. At Orsay, the 2 MV tandem ARAMIS and the 190 kV ion implanter IRMA are being coupled with a 200 kV TECNAI™ transmission electron microscope to allow simultaneous co-irradiation and observation. This paper will first discuss both advantages and limitations of the use of ion beam irradiation to simulate neutron irradiation. A technical description of both set-ups is then presented, and some details will be given concerning multi-irradiation facilities running worldwide. The main application fields of JANNUS will be further detailed. *To cite this article: Y. Serruys et al., C. R. Physique 9 (2008).*

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Résumé

JANNUS : la validation expérimentale à l'échelle de la modélisation atomistique. L'irradiation aux ions peut être considérée comme totalement représentative pour simuler les effets de l'irradiation des matériaux par les neutrons dans la mesure où les premiers atomes de recul sont des ions de la cible elle-même. La principale différence qui subsiste réside dans la forme du spectre en énergie des atomes de recul qui est plus étroite dans le cas des neutrons. Ainsi, il est possible d'étudier l'endommagement des matériaux, les effets d'implantation ionique, la production d'hélium et d'hydrogène ainsi que les réactions nucléaires induites en réalisant des expériences de co-irradiation aux ions (double ou triple faisceau). Le projet JANNUS (Jumelage d'Accélérateurs pour les Nanosciences, le NUcléaire et la Simulation) a été lancé en 2002 en collaboration entre le CEA et le CNRS–IN2P3. Deux sites expérimentaux sont concernés. Le premier situé à Saclay regroupe trois accélérateurs d'ions : une machine 3 MV de type

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Pelletron™ équipée d'une source d'ions multi-chargés ECR, un Van de Graaff simple étage de 2,5 MV et un tandem de 2,25 MV de General Ionex. Le second site situé à Orsay voit le couplage d'un accélérateur mixte de 2 MV (ARAMIS) et d'un implanteur d'ions de 190 kV (IRMA) avec un nouveau microscope électronique à transmission de 200 kV TECNAI™ et permettra ainsi une observation in situ pendant l'irradiation. Cet article discute tout d'abord les avantages et les inconvénients d'une simulation des effets d'irradiation des neutrons par des faisceaux d'ions. Une brève description technique des deux sites expérimentaux est ensuite donnée et quelques informations sont rassemblées sur les autres installations de multi-irradiation disponibles dans le monde. Enfin, les principaux domaines d'application de JANNUS sont présentés. *Pour citer cet article : Y. Serruys et al., C. R. Physique 9 (2008).* © 2007 Académie des sciences. Published by Elsevier Masson SAS. All rights reserved.

Keywords: Neutron irradiation; JANNUS; Atomic modelling

Mots-clés : Irradiation par neutrons ; JANNUS ; Modélisation atomistique

0. Introduction

Synergy between experiments and long term modelling is indispensable in order to increase our knowledge of the kinetic evolution of solids under irradiation. More precisely, elementary chemical–physical mechanisms involved in the evolution of the microstructure, the surface reactivity or the physical and mechanical properties of any material under irradiation have to be quantified through careful experiments, so that input parameters for multi-scale modelling can be introduced into specific codes [1].

Furthermore, the development of new modules for atomistic simulation codes can lead to define and pursue specific irradiation experiments in order to validate the predictions, or their approximations and chosen parameters. Thus, the interplay between experiments and theoretical calculations is useful to cross-fertilize both disciplines.

1. The use of ion beams to simulate neutron-induced damage in materials

Either nuclear reactor irradiation testing or ion beam irradiation experiments serve as the basis of experimental validation of the irradiation behaviour of materials submitted to fast neutron bombardment. Both procedures address the same scale as the atomic modelling. According to the pioneering works of Averback [2,3] and Ullmaier [4], ion irradiation is highly relevant to simulate neutron irradiation, as primary knock-on atoms (PKA) produced by neutron collisions can be considered as self-ions of the target. Table 1 demonstrates the advantages and the drawbacks of neutron damage simulation by means of ion irradiation. The main differences that remain between neutron and ion irradiation lie in the much larger dose rate produced by ions and in the shape of the energy spectrum of these PKA, which leads to a steeper increase of the damage productions versus PKA energy in the case of neutrons (Fig. 1). A spectrum of different ion irradiations with different masses is thus needed to account for the effects of neutrons. Further, it is possible to study target damage, ion implantation effects, helium and hydrogen production, and the occurrence of nuclear reactions by performing co-irradiation experiments (dual or triple beam irradiation). It has been shown in the beginning of the 1980s that sequential irradiation leads to significantly different results as compared to co-irradiation [5]. Thus, it is of essential importance to be able to induce simultaneously ion damage and new atoms in the target material, which, in reality, would be generated by nuclear reactions.

Table 1
Advantages and drawbacks of neutron damage effects by means of ion irradiation

Advantages	Drawbacks
High displacement rates (100 dpa in a few hours) for MeV heavy ions	Dose rate and implantation rate much larger than in real conditions
High and variable He or H/dpa ratios	Small range less (than 5 μm) for a few tens of MeV heavy ions
Homogeneous damage in a few tens of μm for MeV light ions	Surface effects (due to chemical reactivity and sputtering)
No induced radioactivity	Displacement rates limited to 1 dpa in a few hours for MeV light ions
Possibility for in situ mechanical testing	Recoil spectrum different from neutron damage
Possibility for in situ characterization	

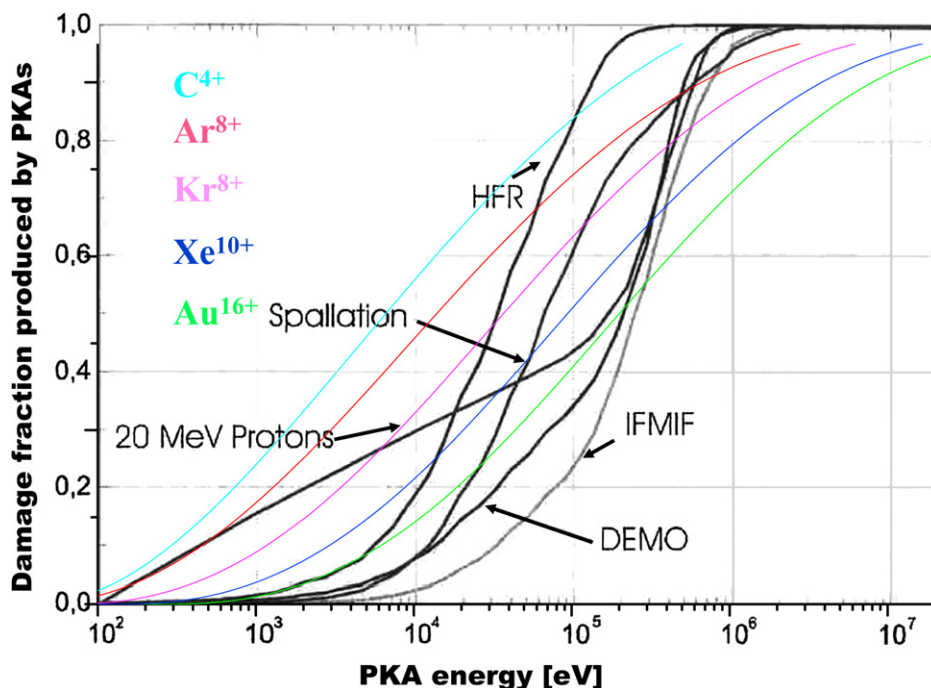


Fig. 1. PKA energy spectra for various neutron sources and for various incident ion beams.

Fig. 1. Spectres énergétiques des Premiers Atomes Frappés (PAF) obtenus par différentes sources de neutrons rapides et faisceaux d'ions.

2. Multi-irradiation facilities available worldwide

Multi-irradiation facilities under operation worldwide can be divided in three categories: those constituted of two or three ion accelerators/implanters, those where a transmission electron microscope (TEM) is connected to one or two ion accelerators/implanters and those where low energy ion guns are coupled with a TEM. Table 2 gives an overview of these facilities; the main application fields explored are listed [6–18]. It is clear that Japanese groups are the world leaders in this field for the study of nuclear materials devoted to fission or fusion applications, as well as for controlled modification of technological materials.

The JANNUS facility with its two experimental sites, one allowing in situ observation during ion beam irradiation and the other one being able to perform triple beam irradiation, is going to offer to the scientific community highly efficient tools, which shall improve the understanding of the behaviour of different kinds of materials under irradiation.

3. Description of the JANNUS multi-irradiation facility

The JANNUS project was started in 2002 in the frame of a collaboration between the Department of Nuclear Materials (DMN), the National Institute for Nuclear Sciences and Technology (INSTN) from CEA Saclay and the Center for Mass Spectrometry and Nuclear Spectrometry (CSNSM) from CNRS-IN2P3 Orsay. This project has received the support of the “Conseil Régional d’Île-de-France” and of the “Département de l’Essonne”. The main goal pursued in the realization and exploitation of the multi-irradiation facility JANNUS is to create a regular and efficient feedback between materials science experimentalists and modellers. The three partners of the project decided to modernize two existing experimental sites: the first one in Saclay that should be mainly dedicated to nuclear applications, and the second in Orsay mostly devoted to nanoscience applications of multi-irradiation.

At Saclay, three electrostatic accelerators will be coupled: a new 3 MV Pelletron™ machine from National Electrostatics Corporation equipped with an ECR multi-charged ion source Nanogan™ from PANTECHNIK company (Fig. 2(a)), a 2.5 MV single ended High Voltage Engineering Van de Graaff used by INSTN for research and teaching purposes and a 2.25 MV General Ionex tandem that will be moved from CSNSM. At Orsay, the two operational machines—a 2 MV tandem Van de Graaff (ARAMIS) equipped with a negative ion source of the Cs sputtering type

Table 2
Multi-irradiation facilities available in the world

Laboratory	Facilities	Application field	Reference
a) dual or triple MeV ion beams			
MSD, IGCAR Kalpakkam, India	1.7 MV Tandetron Ion implanter (30–150 keV)	Irradiation behaviour of nuclear alloys	[6]
HIT Tokyo, Japan	3.75 MV Van de Graaff 1 MV Tandetron	Irradiation behaviour of nuclear alloys and ceramics	[7]
DNE Nagoya University, Japan	2 MV Van de Graaff 200 kV ion implanter	Irradiation behaviour of nuclear alloys and ceramics	[8]
FZ Rossendorf, Germany	3 MV Tandetron 500 kV ion implanter	Synthesis of nanostructured ceramics assisted by irradiation Ion beam modification of materials	[9]
FSU Iena, Germany	3 MV Tandetron JULIA 400 kV ion implanter ROMEO	Synthesis of nanostructured ceramics assisted by irradiation Irradiation behaviour of nuclear alloys	[10]
IAE Kyoto, Japan	1.7 MV Tandetron 1 MV Van de Graaff 1 MV Singletron	Evolution of microstructure under multi-irradiation	[11]
JAERI Takasaki, Japan	3 MV Tandem 3 MV Van de Graaff 400 kV ion implanter	Synthesis of nanostructured ceramics assisted by irradiation Behaviour of alloys and ceramics under irradiation	[12]
DMN Saclay, France (ready to operate at the beginning of 2008)	3 MV Pelletron ÉPIMÉTHÉE 2.5 MV Van de Graaff YVETTE 2.25 MV Tandetron	Irradiation behaviour of nuclear materials Ion beam modification of materials	[13]
b) mono or dual ion beams (> 100 keV) coupled to a TEM			
CARET Sapporo, Japan	1.3 MV HVTEM 400 kV ion implanter 300 kV ion implanter	Synthesis of nano-structured materials assisted by irradiation Behaviour of nuclear materials under irradiation	[14]
Argonne National Laboratory, USA	2 MV Tandem or 650 kV ion implanter 300 kV TEM	Irradiation behaviour of nuclear ceramics	[15]
CSNSM Orsay, France (ready to operate at the beginning of 2008)	2 MV Tandem/Van de Graaff ARAMIS 150 kV ion implanter IRMA 200 kV TEM	Irradiation behaviour of nuclear ceramics and semiconductors Ion beam modification of materials	[13]
c) dual keV ion beams coupled to a TEM			
IMR, University of Salford, UK (under construction)	200 kV TEM Ion implanter (5–100 keV, $A \leq 140$)	Radiation damage on nuclear reactor materials and semiconductors	[16]
JAERI DMD Takasaki, Japan	400 kV TEM 400 kV ion implanter 40 kV ion gun	Radiation effects	[17]
JAERI DMSE Tokai-Mura, Japan	2 × 40 kV ion guns 400 kV TEM	Irradiation behaviour of nuclear alloys and ceramics	[18]

plus a Penning type ion source at the terminal and a 190 kV ion implanter (IRMA)—are going to be coupled together with a 200 kV transmission electron microscope TECNAI-200™ from FEI company (Fig. 2(b)) to allow simultaneous co-irradiation and observation.

The 3 MV Pelletron™ (ÉPIMÉTHÉE) is able to produce almost any ion beam from hydrogen to bismuth, including deuterium ions, in the energy range 300–60 MeV. The 2.5 MV Van de Graaff (YVETTE) accelerates protons, deuterons, helium-3 and helium-4 ions. It is also able to supply carbon and oxygen ions and rare gas ions. The sputtering source of the 2.25 MV tandem (TANDETRON) allows the production of proton and electronegative ion beams (chlorine or iodine for example). The three beams will converge at 15° incidence angle onto the targets in an especially dedicated vacuum chamber. The chamber is implemented with Faraday cups for dose monitoring, energy degraders,



(a)



(b)

Fig. 2. (a) 3 MV Pelletron ÉPIMÉTHÉE at JANNUS Saclay (A. Gonin/CEA); (b) 200 kV TEM at JANNUS Orsay (A. Gonin/CEA).

Fig. 2. (a) Le Pelletron ÉPIMÉTHÉE de 3 MV de l'installation JANNUS de Saclay (A. Gonin/CEA); (b) Microscope Électronique à Transmission (MET) de 200 kV de l'installation JANNUS d'Orsay (A. Gonin/CEA).

and detectors for charged particles, X-rays and gamma rays. The sample holder is able to be cooled down to -150°C or heated up to 800°C .

The IRMA implanter produces ion beams from hydrogen to lead in the energy range 5–570 keV, and ARAMIS is able to produce proton or helium ion beams, and more than forty species of heavier ions ranging from Li to Bi within the energy range 500–15 MeV. The two beam lines will enter the microscope column with a tilt of 22° with respect to the horizontal plane and at 45° from each other in the horizontal plane. The TEM has a spatial resolution better than 0.3 nm. It is equipped with a double tilt sample holder ($\pm 60^{\circ}$) from GATAN company for operation in the temperature range between liquid nitrogen and 800°C . The vacuum can be kept as low as 5×10^{-3} Pa under heating. The ion beam is monitored directly in the microscope column, as close as possible to the sample under investigation.

The JANNUS Saclay multi-irradiation platform is scheduled to become operational at the beginning of the second quarter of 2008 for the dual beam and at the beginning of the fourth quarter of 2008 for the triple beam, while the JANNUS Orsay dual beam/TEM facility is scheduled to start during the first quarter of 2008. In addition to the TEM diagnostics above, both JANNUS facilities will allow in situ characterization of the irradiated samples using ion beam analysis. Specific devices dedicated to electrical or optical measurements are under design. Numerous ex situ characterization methods and devices are available very close to both JANNUS sites, such as X-ray diffraction, scanning electron microscopy (SEM) and atomic force microscopy (AFM), an electron microprobe, a nuclear microprobe, neutron diffraction, atom probe tomography, and a synchrotron X-ray source.

4. Application fields investigated using JANNUS

The main future applications of the JANNUS facilities are listed in Table 3. They include:

- basic irradiation physics;
- fundamental ion beam physics data acquisition;
- irradiation behaviour of nuclear materials dedicated to water cooled fission reactors;
- irradiation behaviour of nuclear materials dedicated to Generation IV reactors;
- irradiation behaviour of nuclear materials dedicated to fusion applications;
- irradiation behaviour of nuclear waste matrices and incineration targets;
- controlled modification of material properties using implantation/irradiation;
- new material synthesis assisted by ion beam irradiation;
- irradiation behaviour of materials dedicated to space applications;

Table 3
Main application fields for the multiple ion beam irradiation facility JANNUS

Tableau 3
Principaux champs d'application de l'installation multi-faisceau JANNUS

Scientific context	Topic
Basic irradiation physics	Combined effects of electronic and ballistic energy losses
Water cooled reactors	Swelling of structural materials under simultaneous ion damage and He accumulation Atomic transport mechanisms (He, rare gases, fission products) under thermal and radiation gradients Ion irradiation induced segregation and precipitation phenomena
Waste matrices and inert fuels	Phase transitions in multicomponent oxide ceramics
Fusion applications	Combined effects of alpha emission and recoil nuclei Microstructural stability (SiC composites)
Generation IV reactors	Combined effects of ion damage, helium and hydrogen accumulation Microstructural stability (ODS steels, carbides, nitrides) Behaviour of new fuels
Ion beam modification of materials	Ion beam assisted synthesis of ceramics Ion beam mixing near interfaces Modification of mechanical, physical or chemical properties for microelectronics, magnetic or optical applications
Teaching	Ion beam physics for analysis or irradiation purposes

- teaching and training in the areas of radiation interactions with matter, irradiation effects and ion beam analysis. Special efforts will be devoted to the study of synergistic effects between damage and helium and/or hydrogen production.

Some works concerning relevant materials for fusion application and carried out using multiple ion beam irradiation have been recently published. All of them clearly demonstrate that the combined effects of displacement damage, hydrogen and helium atoms significantly affect the irradiation behaviour of the investigated materials. First, the group from JAERI Tokai-mura have investigated the synergistic effects of displacement damage and atomic hydrogen and helium on swelling of the ferritic/martensitic steel F82H [19]. The swelling of F82H steel under triple beams with 18 appm He/dpa and 70 appm H/dpa was larger than under dual beams with 18 appm He/dpa. The swelling in F82H under triple beams increased with decreasing irradiation temperature from 0.1% to 3.2%, while swelling under dual beams was between 0.04% and 0.08%. On the other hand, in the case of triple beam irradiation with a high ratio of gas/dpa, the swelling tended to increase with irradiation temperature [19]. The second example deals with vanadium-based materials. Pure vanadium (99.8% purity) was irradiated at 500 and 600 °C either by 5 MeV Ni ions (single beam) or Ni + 260 keV H and 600 keV He simultaneously [20]. When only Ni ions were used, voids formed in the region near the surface up to a depth of ~0.5 µm when irradiated at both temperatures. However, in the region of the damage peak, voids were not observed. Needle-like precipitates, probably a carbide, of about 100 nm length were observed for any specimen covering the full ion penetration depth. Moreover, in the specimen irradiated at 600 °C, the granular precipitates were over a depth range from 1.0 to 1.5 µm. Void formation was observed over the whole ion range when the specimen was subjected to Ni + He simultaneous irradiation; needle-like precipitates were also observed [20]. The last example concerns advanced composite ceramics submitted to triple beam irradiation (4.3 MeV C or 7.84 MeV Si + 1.2 MeV He + 380 keV H) [21]. Nogami and coworkers have shown that the shrinkage of SiC fibers and the apparent shrinkage of the interfacial material, carbon between the matrix and the fibers at the irradiated surface, were mainly attributed to displacement damage caused by irradiation [21].

Before concluding, we briefly address a specific application example of the triple beam irradiation facility at Saclay in the field of nuclear fusion. The experiment will be devoted to compare the evolution of the microstructure of a typical reduced-activation ferritic/martensitic steel submitted to: (i) a pre-implantation with 1.7 MeV $^3\text{He}^+$ and/or 0.53 MeV $^1\text{H}^+$ and subsequent irradiation with a self ion beam (12 MeV $^{58}\text{Ni}^{4+}$ for example); (ii) a simultaneous triple beam experiment. The typical range of the ions is about (2.75 ± 0.25) µm. The Ni-induced damage level expected will be in the range 10–50 dpa, the gas incorporation rate will be 10 appm/dpa for helium and 100 appm/dpa for hydrogen. The irradiated specimens will be characterized using TEM and ion beam analysis methods, mainly nuclear reaction analysis (NRA) and elastic recoil detection analysis (ERDA).

5. Conclusion

As written very recently by Ian Cook: “*In spite of the rapidly increasing potential of modelling, experimental validation is obviously still essential*” [22]. Irradiation using multiple ion beams seems to be one of the most promising routes. The JANNUS facility (Saclay and Orsay) will offer in very near future the use of heavy-ion beams to generate damage cascades, and simultaneous helium and hydrogen implantation. By in situ observation using transmission electron microscopy and post mortem analytical diagnostics including X-ray diffraction, ion beam analysis, tomographic atom probe and mechanical testing, we expect to significantly improve the knowledge on this particular branch of materials physics.

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