The 4th international workshop On Transmission Electron Microscopy With In Situ Irradiation is organized in Orsay and Gif sur Yvette, France, from the 16th March to the 18th of March, 2016.

The workshop focuses on the combination of TEM with in situ ion and electron irradiation. Areas for discussion include advances in electron microscopy and ion irradiation techniques as well as current and future research. All scientific fields utilizing the technique are covered including nuclear materials, radiation effects in semiconductors, nanostructural modification, single and multi ion effects, …

WOTWISI-4 is the fourth workshop organized on this scientific area. Previous workshops were held in:

- England, 18th-20th June 2008, University of Salford, The Use of In-Situ TEM / Ion Accelerator Techniques in the Study of Radiation Damage in Solids, Contact: Jonathan Hinks / Stephen Donnelly
- USA, 6th- 9th June 2011, Albuquerque, The second workshop on the use of in situ TEM / ion accelerator techniques in the study of radiation damage in solids, Contact: Khalid Hattar
- Japan, 11th-12th July 2013, Hokkaido University, The 3rd Workshop On TEM With In Situ Irradiation (WOTWISI-3), Contact: Somei Ohnuki

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P2IO is one of the 100 laboratories of excellence (Labex) approved by the French Government in March 2011 within the framework of future investments funded by the large governmental loan, officially called "Grand Emprunt" of 2010. P2IO defines itself as the network of all laboratories in Paris region involved in physics of the infinitely small to the infinitely large and study of conditions for the appearance of life. P2IO acts like as such networks, in a direct way, by funding a number of actions, and more indirectly, by promoting better cooperation between its members and providing a contact point for external partnerships, including those from the Saclay’s Plateau.

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http://www.sciences.u-psud.fr/en/the_faculty.html
The Faculty of Sciences of the Université Paris-Sud, located on the Orsay campus, welcomes over 13,000 students and 2,100 PhD students, and has a staff of 1,700 professors and researchers and 1,800 administrative and technical workers. Its numerous academic buildings and 41 research laboratories are located on an exceptional 200-acre woodland site containing many rare species of flora. Education and research programs cover the fields of biology, chemistry, electronics, computer sciences, mechanics, mathematics, physics, earth sciences and physical education.

National Institute of Nuclear and Particle Physics, CNRS
Founded in 1971, the aim of the National institute of nuclear and particle physics (IN2P3) of the CNRS is to promote and unify research activities in the fields of nuclear physics, particle and astroparticle physics. It coordinates programmes within these fields on behalf of the CNRS and universities, in partnership with CEA.
The goal of this research is to explore the physics of elementary particles, their fundamental interactions and the manner in which they assemble into atomic nuclei, to study the properties of these nuclei and to explore the connections between the infinitely small and the infinitely large. Whilst these main themes represent the core of the discipline, IN2P3 also has several additional vocations: enabling other scientific domains to benefit from its competencies and solving certain problems posed by society (nuclear energy, healthcare), and accompanying universities in contributing to youngsters’ training.
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Venue

The workshop will be held at the I2BC auditorium (Building 21, former IMAGIF) on the CNRS campus in Gif-sur-Yvette, conveniently located 25 kms south of Paris centre (see map next page). It is also very close to the campus of Université Paris-Sud in Orsay, where is located the JANNuS-Orsay facility of the CSNSM, joint research unit of CNRS/IN2P3 and Université Paris-Sud. A visit is scheduled there on the Friday afternoon.

Dinner

A banquet is organized in Paris at the Eiffel Tower on Thursday 17th March evening, including transportation from Gif sur Yvette to Paris and return. Detailed information on transportation will be given during the workshop.

Useful information

Wireless Internet will be available at the I2BC auditorium in CNRS campus, Gif sur Yvette. You will need to login in order to access this service. The access details will be given with your name badge upon your arrival.

Lunches will be served in the building opposite to the venue. Various restaurants for diner are located in Gif sur Yvette city centre, less than 10 minutes walk from the venue. A list is given in your bag.
Location of I2BC auditorium at the CNRS campus, Gif sur Yvette

## Programme (tentative version, 24th Feb.)

**Wednesday, 16th March 2016**

<table>
<thead>
<tr>
<th>Time</th>
<th>Session</th>
</tr>
</thead>
<tbody>
<tr>
<td>9:30-10:00</td>
<td>Welcome</td>
</tr>
</tbody>
</table>
| 10:00-10:20 | A. Gentils, CSNSM, Univ Paris-Sud and CNRS-IN2P3, Université Paris-Saclay, France  
*The in situ dual ion beam TEM at CSNSM: history and overview of the JANNUS-Orsay facility* |
| 10:25-10:45 | B. Muntifering, Northwestern University and Sandia National Laboratories, USA  
*Insight from in-situ analysis of grain boundary character, radiation sequence, and thermal conditions on defect structure evolution in nickel* |
| 10:50-11:10 | J. Hinks, University of Huddersfield, UK  
*Ion-Beam Modification of Silicon Nanowires* |
| 11:15-11:45 | Break                                                                 |
| 11:45-12:05 | C. Jacquelin, CEA, SRMP, France  
*In-situ electron irradiation within a TEM: a way to study the stability and evolution of cavities in pure aluminium* |
| 12:10-12:30 | M. Li, presented by M. Kirk, Argonne National Laboratory, USA  
*TEM with In Situ Ion Irradiation of Nuclear Materials: The IVEM - Tandem User Facility* |
| 12:35-12:50 | Dominique Delille, FEI (Sponsor)  
*The in-situ challenge of better understanding Structure-Properties relationship in nanomaterials : Possible solutions and illustrations* |
| 12:55-14:15 | Lunch                                                                  |
| 14:15-14:35 | T. Nagase, Osaka University, Japan  
*In situ TEM observation of fast electron irradiation induced structural change in Al_{0.5}TiZrPdCuNi High Entropy Alloy (HEA) and High Entropy Glass (HEG)* |
| 14:40-15:00 | M. Kirk, Argonne National Laboratory, USA  
*TEM with in situ ion irradiation and modeling to predict neutron irradiation damage* |
| 15:05-15:25 | S. Peugel, CEA, DEN, DTCD, SECM, France  
*Helium incorporation in nuclear glass studied by in-situ TEM analysis* |
| 15:30-15:50 | S. Donnelly, University of Huddersfield, UK  
*A New Microscope and Ion Accelerator for Materials Investigations (MIAMI-2) Facility at the University of Huddersfield* |
| 15:55-16:30 | Break                                                                  |
| 16:30-16:50 | C. Flament, CEA, SRMA, France  
*In-situ study of nano-precipitates evolution in the 6061-T6 Aluminum alloy under ion and electron irradiation* |
| 16:55-17:15 | C. Sabathyier, CEA, Difen, DEC, France  
*The fuel behavior under ion irradiation investigated using JANNUS-Orsay facility* |
| 17:20-17:45 | M.-F. Barthe, CEMHTI, CNRS, France  
*TEM in situ study of damage induced in W by self ion irradiation* |
<table>
<thead>
<tr>
<th>Time</th>
<th>Session</th>
</tr>
</thead>
</table>
| 10:00-10:20 | C.H. Chen, presented by W.J. Weber, University of Tennessee, USA  
**In-situ TEM characterization of Helium Bubble evolution in Nano-Engineered SiC under Kr²⁺ irradiation** |
| 10:25-10:45 | A. Duchateau, PSL Research University, Chimie ParisTech and CEA, SRMP, France  
**In situ TEM annealing in bcc metals after He implantation: a way to study point defects clusters** |
| 10:50-11:10 | K. Hattar, Sandia National Laboratories, USA  
**Recent Advancements in Sandia’s In-situ Ion Irradiation Transmission Electron Microscope** |
| 11:15-11:45 | Break                                                                                      |
| 11:45-12:05 | D. Kaoumi, The University of South Carolina, USA  
**Effects of ion irradiation on dislocation dynamics in a Ni-based superalloy studied through the use of in-situ TEM** |
| 12:10-12:30 | R.W. Harrison, University of Huddersfield, UK  
**The Effect of He/DPA Ratio on the Micro-Structure of Tungsten Under in-situ Ion Irradiation** |
| 12:35-12:50 | S. Aguy, EDEN Instruments (Sponsor)  
**Protochips In-Operando, in situ electron microscopy solutions: capabilities and applications** |
| 12:55-14:15 | Lunch                                                                                      |
| 14:15-14:35 | K. Arakawa, Shimane University, Japan  
**Structure & Dynamics of Radiation-Produced Defects in Metals: Comparison between Electron and Self-ion Irradiations** |
| 14:40-15:00 | O. Toader, University of Michigan, USA  
**The New Setup at the Michigan Ion Beam Laboratory to Connect Two Beamlines to a TEM** |
| 15:05-15:25 | N. Hashimoto, Hokkaido University, Japan  
**Effect of Heat Load on Microstructural Development in Irradiated Steels** |
| 15:30-15:50 | L. Fave, Paul Scherrer Institut, Switzerland  
**Characterization of the sp²/sp³ ratio of ion irradiated PyC interphases from silicon carbide based ceramic matrix composites** |
| 15:55-16:55 | Posters (see details next page) & break                                                      |
| 16:55-17:15 | J. Ribis, CEA, DEN, DMN, SRMA, France  
**Interface stability under irradiation in Oxide Dispersion Strengthened steel** |
| 17:20-17:40 | M. Gaumé, CEA, DEN, SRMA, France  
**In-situ study of dislocation climb in zirconium alloys under charged particles irradiation** |
| 17:45-18:05 | R. Schaublin, ETH Zurich, Switzerland  
**Impact of H & He in irradiated UHP Fe(Cr) by TEM in situ irradiation** |
| 19:00-23:45 | Bus to Paris (and back to Gif sur Yvette, Hotel Le Village)  
**Dinner at the Eiffel Tower** |
Poster session (Thursday, afternoon break):

- A. Amroussia et al., Michigan State University, USA
  
  *Swift heavy ion irradiation damage in Ti-6Al-4V: Characterization of the microstructure and mechanical properties*

- C. Baumier et al., CSNSM, Univ Paris-Sud, CNRS/IN2P3, Université Paris-Saclay, France
  
  *JANNuS-Orsay facility at CSNSM*

- O. Emelyanova et al., National Research Nuclear University, Russia
  
  *Cavity growth in ODS steels under high dose helium implantation*

- Y. Haddad et al., CSNSM, Univ Paris-Sud, CNRS/IN2P3, Université Paris-Saclay, France
  
  *In-Situ TEM experiment to simulate rim structure formation*

- S. Jublot-Leclerc et al., CSNSM, Univ Paris-Sud, CNRS/IN2P3, Université Paris-Saclay, France
  
  *Influence of ion irradiation and He implantation on the swelling microstructure of austenitic stainless steels*

- M. Loyer-Prost et al., CEA, DEN, SRMP, Laboratoire JANNUS, France
  
  *Ion irradiation and radiation effect characterization at the JANNUS-Saclay triple beam facility*

- G. Victor et al., Université de Lyon, UCBL, Institut de Physique Nucléaire de Lyon, France
  
  *TEM in-situ observations of irradiation damage in boron carbide*

- C. Zheng et al., CSNSM, Univ Paris-Sud, CNRS/IN2P3, Université Paris-Saclay, France
  
  *Nano-size metallic oxide particle synthesis in Fe-Cr alloys by ion implantation*
### Friday, 18th March 2016

<table>
<thead>
<tr>
<th>Time</th>
<th>Speaker &amp; Affiliation</th>
<th>Title</th>
</tr>
</thead>
<tbody>
<tr>
<td>10:00-10:20</td>
<td>T. Prozorov, US DOE Ames Laboratory, USA</td>
<td><em>Magnetosome magnetite biomineralization by magnetotactic bacteria in-situ</em></td>
</tr>
<tr>
<td>10:25-10:45</td>
<td>B. Décamps, CSNSM, CNRS-IN2P3, Univ.Paris-Sud, France</td>
<td><em>In-Situ TEM irradiation of pure iron: Role of He on heterogeneous bubble formation on dislocation loops</em></td>
</tr>
<tr>
<td>10:50-11:10</td>
<td>I. Monnet, CIMAP, CEA-CNRS-ENSICAEN-UCN, France</td>
<td><em>Using in-situ TEM observations during ions/electrons irradiation: a powerful tool for a better understanding of the microstructural mechanisms inducing oxide dissolution in Oxide Dispersion Strengthened ferritic steel</em></td>
</tr>
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<td>11:15-11:45</td>
<td>Break</td>
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<tr>
<td>11:45-12:05</td>
<td>F. Onimus, CEA, SRMA, France</td>
<td><em>A direct comparison between in-situ TEM observations and Dislocation Dynamics simulations of interaction between dislocation and irradiation loop in recrystallized Zircaloy-4</em></td>
</tr>
<tr>
<td>12:10-12:30</td>
<td>D. Gorse-Pomonti, LSI, Ecole Polytechnique, CNRS, CEA, Université Paris-Saclay, France</td>
<td><em>A TEM study of electron radiation damages in two calcium silicates and a magnesium rich silicate</em></td>
</tr>
<tr>
<td>12:35-14:00</td>
<td>Lunch</td>
<td></td>
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<tr>
<td>14:00-14:20</td>
<td>B. Doyle, Sandia National Laboratories, USA</td>
<td><em>Calculating and Mapping Unintentional Ion Channeling in Polycrystalline Materials</em></td>
</tr>
<tr>
<td>14:25-14:45</td>
<td>L. Belkacemi, CEA, DEN, SRMP, France</td>
<td><em>Radiation effect on ion-irradiated under-saturated Fe1%at.Mn and Fe1%at.Ni alloys</em></td>
</tr>
<tr>
<td>14:50-15:00</td>
<td>Closing session</td>
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</tr>
<tr>
<td>15:00-15:30</td>
<td>Transfert Gif-Orsay</td>
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<tr>
<td>15:30-17:30</td>
<td>Visit of the JANNuS-Orsay facility, CSNSM lab</td>
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</table>
Abstracts

(listed in alphabetical order)
EDEN Instruments is a High-tech company specializing in the distribution of Electron Microscopy Equipments. We deliver a wide range of accessories dedicated to the samples study in SEM microanalysis EDS, EBSD, WDS, for correlative microscopy, specimen preparation and for In-Situ TEM electron microscopy.

In this talk, we will present the Protochips In-Operando, in situ electron microscopy solutions: capabilities and applications.

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In this presentation we will explore the most recent results using the Atmosphere Gas Environmental Cell and the Poseidon flowing liquid and electrochemistry systems and provide updates on new technology in our Aduro heating and electrical biasing system. The Atmosphere 200 Gas Environmental Cell combines our patented MEMS technology and holder-based closed cell design with innovative software and gas handling hardware, allowing for atomic-scale resolution at gas pressures up to 1 atm and sustained temperatures up to 1000°C and is compatible with analysis tools including EDS and EELS. The Poseidon liquid cell surrounds samples in a self-contained and fully hydrated flowing and mixing chamber directly within the TEM. Samples and processes that previously required freezing or could not be imaged without resin embedment or desiccation can now be studied and observed in liquid and at high resolution. With the Poseidon Electrochemistry system, real-time electrical analysis can also be achieved. Both systems are fully EDS compatible.

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Contact Details:
EDEN Instruments
Mr Stéphane AGUY
Tel: 00 33 6 08 06 70 81
Email: stephane.aguy@eden-instruments.com
Site web: www.eden-instruments.com
Swift heavy ion irradiation damage in Ti-6Al-4V:
Characterization of the microstructure and mechanical properties
Aida Amroussia (1), Carl J. Boehlert (1), Florent Durantel (2), Clara Grygiel (2), Wolfgang Mittig (3),(4), Isabelle Monet (2), Frederique Pellemoine (3)

(1) Department of Chemical Engineering and Materials Science, Michigan State University, East Lansing, Michigan 48824-4437
(2) CIMAP-CIRIL, BP 5133, 14070 CAEN CEDEX 5, France
(3) Facility for Rare Isotope Beams FRIB, Michigan State University, East Lansing MI 48824-1321, USA
(4) National Superconducting Cyclotron Lab, Michigan State University, East Lansing MI 48824-1321, United States

ABSTRACT

Due to their low activation, corrosion resistance, good mechanical properties, and their commercial availability, Ti-alloys, especially the α+β alloy Ti-6Al-4V (wt%), are considered for different applications in nuclear industry. Ti-6Al-4V is also being considered as a structural material for the beam dump for the Facility for Rare Isotope Beams (FRIB) at Michigan State University: a new generation accelerator with high power heavy ion beams. FRIB will provide primary beams from O to U with an energy of 200 MeV/u for heavy ion beams, and higher energies for lighter beams. A rotating water-cooled thin-shell Ti-alloy drum was chosen as the basic concept for the beam dump. In this study, samples of Ti-6Al-4V were irradiated at the CIMAP-GANIL Facility in France using different ion beams at 1 MeV/u to investigate the changes in microstructure and mechanical properties due to swift heavy ion irradiation damage. A dual dose and temperature dependence in the hardness results for irradiated Ti-6Al-4V was observed: Samples irradiated at higher temperature exhibited higher hardening for similar dose than the one irradiated at room temperature. Similar behavior was observed during irradiations with neutrons [1], protons [2] and swift heavy ions [3]. In these studies however, only the post irradiation examination of the final irradiated microstructure at a certain damage level was performed which leaves gaps in our understanding of the damage mechanisms. Successful prediction of this material’s performance overtime and under irradiations requires an understanding of the basic formation mechanisms of radiation-induced defects at initial damage stages (lower doses) and its accumulation at higher dose levels which results in the complex features in the microstructure [4]. For these reasons, further investigations using in-situ transmission electron microscopy (TEM) are planned. These in-situ TEM irradiations offer the unique capability to investigate the evolution of the damage through continual imaging and observation and allow for quantitative and qualitative microstructural studies.

References:
Structure & Dynamics of Radiation-Produced Defects in Metals: Comparison between Electron and Self-ion Irradiations

Kazuto Arakawa¹, Ryota Nagasawa¹, Cédric Baumier², Brigitte Décamps², Estelle Meslin³, François Willaime³, Hidehiro Yasuda⁴, Hirotaro Mori⁴, Shigeo Arai⁵, Nobuo Tanaka⁵, Takafumi Amino⁶, Shiori Ishino⁷

¹Department of Materials Science, Shimane University, 1060 Nishikawatsu, Matsue 690-8504, Japan
²CSNSM, Orsay Campus Bat.108, 91400 Orsay, France
³CEA, DEN, Service de Recherches de M’etallurgie Physique, F-91191 Gif-sur-Yvette, France
⁴Research Center for UHVEM, Osaka University, 7-1 Mihogaoka, Ibaraki 567-0047, Japan
⁵Ecotopia Science Institute, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8601, Japan
⁶Nippon Steel & Sumitomo Metal Corporation, 1-8 Fuso-Cho, Amagasaki, Hyogo 660-0891, Japan
⁷Faculty of Engineering, the University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656, Japan
arakawa@riko.shimane-u.ac.jp

Abstract

Nuclear-fission and fusion materials are degraded primarily due to the accumulation of lattice defects in response to energetic neutron irradiation, where point-defect clusters are directly produced in addition to point defects (self-interstitial atoms (SIAs) and vacancies) in the primary damage process, which is called “collision cascade.” To precisely predict the life time of these materials, accurate understanding of the structures and dynamic properties of radiation-produced defects is required.

We have directly revealed dynamic properties of individual nanoscale SIA clusters in metals, using in-situ TEM ([1-3]). Recently, we have extracted the dynamic properties of single SIAs, from the formation process of TEM-visible SIA clusters under high-energy electron irradiation, where primary defects generated via knock-on displacement are only point defects unlike neutron and ion irradiation, with a high-voltage electron microscope (HVEM) ([4, 5]).

In this talk, we will primarily focus on the structures and dynamics of radiation-produced point defects and point-defect clusters in high-purity tungsten. Firstly we will show some of our results on dynamic properties of SIAs and SIA clusters, which have been mainly obtained with HVEMs in Osaka University and Nagoya University in Japan. And, we will provide our results on the formation processes of point-defect clusters under self-ion irradiation, which have been obtained with an ion-accelerators combined TEM in the JANNuS-Orsay facility in France. Through the comparison between these results, we will try to extract the effects of collision cascades in the formation processes of point-defect clusters.

References

TEM in situ study of damage induced in W by self ion irradiation.

E. Autissier\(^1\), M-F. Barthe\(^1\), M. Sidibe\(^1\), P. Desgardin\(^1\), C. Genevois\(^1\), B. Décamps\(^2\), R. Schäublin\(^3\)

\(^1\)CEMHTI, CNRS/Orléans University, 3A Rue de la Férollerie, 45071 Orléans, France
\(^2\)CSNSM - Centre de Sciences Nucléaire et de Sciences de la Matière, Bâtiment 108, 91405 Orsay
\(^3\)Metal Physics and Technology Department of Materials, ETH Zürich, HCI G515, 8093 Zürich

emmanuel.autissier@cnrs-orleans.fr

Abstract

Tungsten (and W alloys) has been chosen to cover part of the divertor in fusion reactor ITER, and envisaged for first walls in DEMO, because of its low sputtering yield, low hydrogen solubility and high-melting temperature [1, 2]. In these tokamak, W materials will be submitted to He /H fluxes from the plasma, radiation damage due to the impinging 14 MeV fusion neutrons and high temperature. It is of greatest importance to foresee what will be the effect of these extreme conditions on the W material. In this work we focus on the characterization of the microstructure evolution under irradiation in tungsten in the objective of bringing new experimental data for the modeling of radiation embrittlement of W based materials. We use 2 different complementary techniques to observe the damage: Positron annihilation spectroscopy (PAS) and transmission electron microscopy (TEM).

In order to simulate the damage induced by recoil atoms created by neutron exposure that the tungsten based components will be facing in a tokamak, irradiations are performed in well characterized pure W samples with W ions. In this study we combine W irradiations performed at low energy (1.2 MeV) in Orsay in thin lamellae allowing for in situ TEM observation and at higher energy (20 MeV) in Saclay allowing for bulk irradiation that is closer to the fusion irradiation condition.

We already showed by using Positron Annihilation Spectroscopy [1] that at high energy (20 MeV) vacancy clusters are created and that their size depends on the irradiation temperature and post-irradiation annealing temperature.

In this presentation we will focus on the study of the in situ irradiations performed in thin lamellae at two temperatures (RT and 973K) and at a fluence of \(2 \times 10^{12} \text{ cm}^{-2}\) corresponding to a damage dose of 0.01 dpa (using quick calculation with SRIM software). In situ under irradiation and ex situ observations have been performed. The film recorded during in situ irradiation shows the fast appearance of defects that are essentially immobile at RT (except for some escape to the surface) but move when irradiation is performed at 973K. These defects accumulate and can grow at high temperature (see figure 1).

The ex situ g.b=0 study of these defects shows that in sample irradiated at RT, the majority of them are \(1/2<111>\) loops. Most of them are isolated. Some \(b=<100>\) loops are also observed but in lower number, as already observed by X. Yi and co-authors in W irradiated with 150 keV W ions [3].

In sample irradiated at 973K, the majority of defects are \(1/2<111>\) loops. The \([111]\) and/or \([11-1]\) type agglomerate into rafts. The others, of the type \([-111]\) and/or \([1-11]\), are isolated. \(<100>\) loops are also observed. The fraction of \(<100>\) loops is larger at RT than in 973K irradiated sample.

Ex situ observations in over and under focused condition show the presence of cavities (vacancy clusters) whatever the irradiation temperature is. Their size is at the limit of TEM observation with an objective aperture, which means a diameter of about 0.6±0.1 nm in RT irradiated sample (see figure 2) and a diameter of about 1.4±0.2 nm with some larger cavities of 3 nm in diameter when irradiation is performed at 973K. The number of observable cavities is lower in RT irradiated sample than at 973K.
To complete this work the RT irradiated sample has been annealed at 573 and 773K during 1 hour under vacuum, the same condition as the one used in the previous PAS studies. It is observed that the size of cavities increases with temperature (as already observed in PAS studies) to reach an average diameter of 1.3±0.2 nm. In addition, the number of observable ones strongly increases.

It is interesting to note that these results are in good agreement with the PAS studies already performed, for a higher damage dose of 1 dpa. The increase of the vacancy clusters size has been observed for annealing at temperature higher than 473K and for irradiation temperature of 873K [4].

These results are in good agreement with the one obtained by A Hasegawa and co-workers who have shown that dislocation loops and voids are induced in pure W by irradiations with neutrons [5].

Figure 1: defects observed after 10 sec of irradiations in W irradiated with 1.2 MeV W ions at 0.01 dpa at different temperatures: a/ RT, b/ 973 K. Images extracted from films acquired under irradiations with the same enlargement.

Figure 2: Over focused (left) and under focused (right) images in W irradiated with 1.2 MeV W ions at 0.017 dpa at RT.

Acknowledgements: Experiments done at JANNuS (Joint Accelerators for Nanoscience and Nuclear Simulation) Orsay at CSNSM (Orsay, France) and Saclay at CEA (France) which are part of the EMIR French accelerators network.

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement number 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

References
We present a unique in-situ Transmission Electron Microscope coupled with two ion accelerators (ARAMIS and IRMA) enabling in-situ observation of the material microstructure modifications induced by ion irradiation/implantation. The facility is operating in three modes: i) TEM + IRMA, ii) TEM + ARAMIS and iii) TEM in dual beam (IRMA + ARAMIS).

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Radiation effect on ion-irradiated under-saturated Fe1%at.Mn and Fe1%at.Ni alloys

L. Belkacemi¹, E. Meslin¹, B. Radiguet², B. Décamps³, J. Henry⁴ et A. Lopez⁵.

¹CEA, DEN, Service de Recherches de Métallurgie Physique, F-91191 Gif-sur-Yvette –France
²Groupe de Physique des Matériaux UMR CNRS 6634, Université et INSA de Rouen - France
³CSNSM, CNRS-IN2P3-Univ.Paris-Sud 11, UMR 8609, Bât. 108, 91405 Orsay, France
⁴CEA, DEN, Service de Recherches de Métallurgie Appliquée, F-91191 Gif-sur-Yvette –France
⁵CEA, DEN, Service d’Etude des Matériaux Irradiés, F-91191 Gif-sur-Yvette –France

lisa.belkacemi@cea.fr

Abstract

Reactor pressure vessel (RPV) steels embrittlement under neutron irradiation is the main lifetime limiting factor of nuclear reactors. The RPV embrittlement is primary due to the impeding of dislocation glide caused by their interaction with nanometric clusters composed of point defects and/or solute atoms which form under irradiation. Point defect clustering can lead to the formation of crystallographic defects such as dislocation loops or voids (Figure 1). It is necessary to better understand the diffusion mechanisms leading to the formation of these irradiation-induced defects in view of improving the predictive models for RPV embrittlement.

In this study, the effect of Mn and Ni in under-saturated ferritic model alloys Fe1%at.Ni and Fe1%at.Mn was experimentally investigated at the atomic scale by Transmission Electron Microscopy (TEM) and Atom Probe Tomography (APT) on ion-irradiated samples. The irradiation with 10 MeV Fe⁵⁺ ions was performed at 400°C at a nominal damage rate of 10⁻⁴ dpa.s⁻¹ to a nominal displacement damage of 2.5 dpa.

Figure 2 shows TEM micrographs performed on FIB slices taken out of the three bulk specimens after irradiation. The detailed analysis shows that the microstructure totally depends on the composition. In pure Fe, an homogeneous distribution and a significant number density of both dislocation loops and voids were revealed. However, in Fe1%at.Ni, voids number density was still high, whereas dislocation loops number density was very low. In Fe1%at.Mn, a high number density of dislocation loops was
observed, in opposition to voids. Radiation-Induced Segregation (RIS) of Mn was recorded by STEM/EDX in the vicinity of dislocation loops and grain boundaries, according to observations made on dislocation lines [1]. An additional analysis realized by APT supported these observations by showing Mn depletion within the matrix. The results obtained indicate that Mn would significantly contribute to swelling reduction, while Ni would limit the formation and/or growth of dislocation loops. Diffusion mechanisms of solute atoms leading to the preferential formation of dislocation loops or voids are still not well understood under irradiation. By creating Mn-Vacancy complexes, Mn would reduce vacancies clustering phenomenon, which would partly explain the low density of voids in Fe1%at.Mn [2, 3]. Furthermore, reduction of the mobility of interstitial and vacancy clusters by Ni would probably promote recombination process in Fe1%at.Ni [4].

To go further, we plan to carry out in-situ irradiation of Fe1at.%Mn and Fe1at.%Ni using charged particles such as ions or electrons. Setting imaging and irradiation conditions using ions would enable us to better understand how irradiation defects form while taking into account the cascade effect. On the other hand, using 1 MeV electrons as irradiating particles would remove the cascade contribution to damage defect formation and simplify the modelisation of the RIS phenomenon.

Acknowledgements: This project has received funding from the Euratom research and training program 2014-2018 under grant agreement No 661913 and was supported by the French Network EMIR. The authors acknowledge the JANNuS Saclay team for the irradiation performed in this study.

References
In-situ TEM characterization of Helium Bubble evolution in Nano-Engineered SiC under Kr$^{2+}$ irradiation


$^1$Materials Science & Engineering Dpt., University of Tennessee, Knoxville, TN 37996, USA.
$^2$Materials Science & Technology Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831, USA.
$^3$Center for Integrated Nanotechnologies, Materials Physics & Applications Division, Los Alamos National Laboratory, Los Alamos, NM 87545, USA.
$^4$Division of Nuclear Engineering, Argonne National Laboratory, Argonne, Illinois 60439, USA
$^5$Nuclear Engineering Dept., North Carolina State University, Raleigh, NC 27695, USA.
cchen30@vols.utk.edu

Abstract

Understanding the evolution of helium bubbles under irradiation in SiC is important for applications in next generation nuclear power plants. The real time evolution of microstructure and bubbles in nano-engineered (NE) 3C-SiC under ion irradiation has been studied. The evolution of individual He bubbles in SiC foils during 1 MeV Kr$^{2+}$ irradiation at 350 to 800 °C has been followed with in situ transmission electron microscopy (TEM). Significant bubble coarsening is not observed under TEM with increasing dose up to 20 dpa. On the contrary, a decrease in average bubble size is observed as the damage dose exceeded 10 dpa. Above a dose of 10 dpa, a majority of helium bubbles decreased in size while keeping a spherical shape. As illustrated in Fig. 1, the bubble size distribution contracts and the average bubble diameter decreases with increasing damage level. Besides the shrinkage in size, some of the larger bubbles split into smaller bubbles, which contributed to the overall decrease in average bubble diameter. During irradiation at 800 °C, a few bubbles exhibited a small but discernable growth, as shown in Fig. 2. Compared to literature results for bulk SiC under irradiation [1], helium redistribution and release is observed in the TEM sample under irradiation at 800 °C. Defect migration associated with stacking fault (SFs) confinement has also revealed that nano-layered SFs structure is radiation tolerant below the dose of 40 dpa at 800 °C.
Fig. 1. Normalized bubble size distribution in NE SiC after irradiation at 800 °C, the average diameter of helium bubble decreases as damage increases.

Fig. 2. Micrographs of helium bubble evolution before/after irradiation. Bubble shrinkage, breakup and growth are observed and indicated with an arrow line, dash arrow line and circle, respectively.

References

In-Situ TEM irradiation of pure iron: Role of He on heterogeneous bubble formation on dislocation loops

Brigitte Décamps 1, Daniel Brimbal 2,*, Sandra Moll 3,*, Estelle Meslin 3, Jean Henry 2, Hélène Lefaix-Jeuland 3, Alain Barbu 3

1 CSNSM, CNRS-IN2P3-Univ. Paris-Sud 11, UMR 8609, Bât. 108, 91405 Orsay, France
2 LA2M, CEA/Saclay, DEN/DMN/SRMA/LA2M, 91191 Gif-sur-Yvette Cedex, France
3 SRMP, CEA/Saclay, DEN/DMN/SRMP, 91191 Gif-sur-Yvette Cedex, France

* no more in CEA
brigitte.decamps@csnsm.in2p3.fr

Abstract

High-purity iron and Fe–Cr model alloys are of particular interest, as ferritic–martensitic steels are candidate materials for use as structural materials in future fusion reactors. Large quantities of helium will be produced by transmutation reactions in future fusion reactors, helium having an effect on the mechanical properties and swelling of metals [1–3].

In order to study the effect of helium, the microstructural evolution under irradiation of model materials (pure Fe and Fe-Cr alloys) using the JANNuS (Joint Accelerators for Nano-science and Nuclear Simulation) platform has been studied. Such platform is designed to supply a large range of ion irradiation and implantation conditions, allowing notably in-situ Transmission Electron Microscopy (TEM). For this purpose, high purity iron (fabricated at the “Ecole des Mines de Saint-Etienne”) has been first irradiated in-situ (JANNuS Orsay platform) in a TEM with 1MeV Fe+ ions while simultaneously implanting 15keV He+ ions, at 500°C. Results [4] showed that, at 0.8dpa/540 appm, dislocation loops and helium bubbles are present with a heterogeneous bubble formation on dislocation loops: a majority of them are observed inside large dislocation loops.

To better understand this result obtained after a dual-beam irradiation (Fe/He), pure Fe has been irradiated in-situ in the TEM with only He (15 keV) at 500°C. The analysis of the samples after single-beam irradiation reveals similar heterogeneous bubble formation within loops as shown in Figure 1.

Figure 1: Bright field TEM images: Bubbles and dislocation loops in a-Fe irradiated at 500°C to 1100 appm He: Left: g=[110] (s>>0), defocus=-6µm; Right: g=[110] (s>>0), defocus=+6.74µm.
If the quantity of He is increased (3100 appm), bubbles are formed in the vicinity of the dislocation loop cores probably blocking their extension as well as in grain boundaries (GBs).

To better understand the evolution of the microstructure under He irradiation, a special in-situ irradiation is performed in the TEM as no observation can be performed in the TEM during irradiation with He. A first He irradiation is performed at a dose of 1100 appm, a microstructural characterization of different zones exhibiting loops is performed and then a second He irradiation is made at a dose of 2000 appm He. Again TEM observation is done in order to follow the evolution of loops as shown in Figure 2.

![Figure 2: Bright field TEM images: Bubbles and dislocation loops in α-Fe irradiated at 500°C to 1100 appm He (Left image: g=[110] (s>>0), positive defocus) and later on (right image) at 2000 appm g=[110] (s>>0), positive defocus.](image)

The present work is related to the TEM study of the microstructure of pure iron irradiated in-situ in the TEM with only He (15 keV) at 500°C. The detailed analysis of the evolution of the material under irradiation at different fluences, notably using sequential irradiation, will be presented and discussed with respect to the precedent dual-beam work. Thus, a mechanism explaining the heterogeneous nucleation of helium bubbles within dislocation loops will be proposed and discussed.

**Acknowledgments**

Experiments done at JANNuS (Joint Accelerators for Nanoscience and Nuclear Simulation), Orsay, France, and supported by the French Network EMIR, European Fusion Development Agreement (EFDA) and Needs (Osmose).

The authors acknowledge the outstanding work of the JANNuS Orsay team.

**References**

The *in-situ* challenge of better understanding Structure-Properties relationship in nanomaterials: Possible solutions and illustrations

*Dominique Delille*

Dominique.Delille@fei.com  +33 686 570 723

Abstract:

The Electron Microscopy of tomorrow appears more and more to be highly dynamic, with the growing development of many different ways to look at real-time in-situ experiments. Being one of the world leading suppliers of scientific instruments in the field of Transmission Electron Microscopy, FEI pursues its efforts in developing in-situ microscopy at ultra-high resolution by not only better integrating existing third party portable in-situ solutions and sample holders, but also keeping its leadership on the very demanding market of Environmental Transmission Electron Microscopes (ETEM). This presentation will highlight the latest FEI developments in both directions, showing how much improvement has been made in ETEM through presentation of several unrivaled results, as well as how much the versatility of the most recent FEI TEMs family, including Talos and Themis, can accommodate the use of various in-situ sample holders, either from FEI or from other suppliers, with the fruitful help of the new FEI CMOS Ceta 16M camera. Here as well, some very realistic examples will show how much a ‘standard’ TEM, Cs corrected or not, can associate with the most advanced in-situ solutions to become a surprising laboratory at the sub-nanometer scale…

**Key words:** TEM, STEM, ETEM, in-situ, 3D EDS Tomography, CMOS camera.
A New Microscope and Ion Accelerator for Materials Investigations (MIAMI-2)

Facility at the University of Huddersfield

S.E. Donnelly, G. Greaves and J.A. Hinks

School of Computing and Engineering, University of Huddersfield, Huddersfield, HD1 3DH,

United Kingdom

The original Microscope and Ion Accelerator for Materials Investigations (MIAMI-1) instrument began operating in 2010 at the University of Salford before moving to the University of Huddersfield in 2011. It is now being joined by a brand new state-of-the-art MIAMI-2 facility which is being constructed with funding from the United Kingdom’s Engineering and Physical Science Research Council (EPSRC). Based on an H-9500 300 kV transmission electron microscope custom-designed and built by Hitachi for this project, the new system will incorporate dual high- and low-energy ion beams, advanced analytical capabilities and complementary in situ experimental techniques. MIAMI-1 and MIAMI-2 will be housed in a new suite of laboratories creating a centre of excellence in the field of TEM with in situ ion irradiation. The specifications, capabilities and experimental possibilities of the new MIAMI-2 facility will be presented.
Calculating and Mapping Unintentional Ion Channeling in Polycrystalline Materials

Barney L. Doyle, Khalid M. Hattar, Daniel C. Bufford and Brittany R. Muntifering

Sandia National Laboratories PO Box 5800 Albuquerque, N.M. 87185 U.S.A.

Abstract

It is important to include the effect of ion channeling when considering the equivalence of using ions accelerated to high energies to simulate the displacement damage produced by energetic neutrons because ions that channel are expected to make significantly less displacement damage than that calculated. The primary knock-on atoms (PKAs) recoiled by neutrons are omnidirectional and of such low energies that they are unlikely to channel [1], but the monodirectional high energy ions implanted as PKAs will certainly channel if individual crystallites in the material are oriented, albeit randomly, with their crystallographic axes or planes aligning with the direction of the beam. This presentation will, we think for the first time, ameliorate this one uncertainty in ion-neutron equivalence by describing an approach to quantitatively determine the probability that accelerated ions will channel in textured polycrystalline materials and even map the position on the sample where such ion channeling occurs.

The first step in this process is the determination of “half-angles” $\psi_{1/2}$ for axial and planar channeling of the ion used in the exposure for the crystal lattice of the sample. An Excel program was developed for this purpose [2] and at http://www.sandia.gov/pcnsc/departments/iba/ibatable.html. The program can calculate the $\psi_{1/2}$ for all ions at any energy for all crystal axes and planes up to hkl=555 for mono-atomic cubic crystals, i.e. bcc, hcp and diamond lattices.

The second step is to produce an “equal-angle” projection (not to be confused with a stereographic projection) that maps ion channeling conditions onto the unit sphere. Only one octant of this sphere is really needed however due to the symmetry of all the cubic lattices. This is also done using Excel, and a description of this process can be found in Ref. [3]. Such a plot is shown in Figs. 1a and 2a for 2 MeV Si on fcc Au. Here the white dots represent axes where their radii correspond to axial $\psi_{1/2}$’s and the planes are plotted as lines with width of 2 times the planar $\psi_{1/2}$’s.

The third step is to determine the crystalline texture of the sample. This was done on the I$^{\text{III}}$TEM at Sandia [4] using Precession TEM. The typical Euler orientation angles ($\varphi_1$, $\Phi$, $\varphi_2$) of each crystallite in the <111> textured polycrystalline Au TEM sample were then determined as the electron beam scanned in x and y across the sample. This set of Euler angles are then converted by an Excel program to a discrete Inverse Pole Figure (IPF) which can be plotted with the same “equal-angle” projection as the channeling map. This is done if Fig. 1a for a normal incident Au beam, and in Fig. 2a for the sample tilted 60° which is the orientation used during I$^{\text{III}}$TEM experiments. The color of the discrete IPF points follows a square-root frequency scale from light blue to yellow. If an IPF point lies in a white region of the channeling map, channeling will occur, and visa versa for points in the black regions. The probability of channeling for these two sample tilts of 0° and 60° is 44% and 29%.

Finally, maps of where channeling occurs on the sample are made. Each of the IPF points corresponds to an xy position scanned by the TEM beam, if an IPF point overlaps a channeling region the point is plotted gray, if not it is black. Maps for 0° and 60° tilts are in Figs. 1b and 2b. We plan to use such maps to differentiate regions on samples where we predict ion channeling and following ion exposure to see if the defect concentration differs from neighboring regions that don’t channel.

The aspect of this theory with greatest utility is quantifying the probability of channeling, as that can be used to scale up the fluence of ions required to reach a desired dpa level, e.g. in the 60° tilt case above, the fluence of ions would need to be increased by 1/(1-0.29) or 41% from TRIM calculations.
Figure 1. a: Equal Angle Channeling map and Discrete Inverse Pole Figure for normal incident 2 MeV Si on <111> textured polycrystalline fcc Au. b: Spatial map of where channeling will occur (gray regions) on the poly Au sample for normal incidence.

Figure 2. a: Same as above for 60° incidence. b: Same as above for 60° incidence.

References

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In situ TEM annealing in bcc metals after He implantation: a way to study point defects clusters

A. Duchateau1,2, F. Prima1, Rebecca Alexander2, E. Meslin2, T. Jourdan2, M.-C. Marinica2, H. Lefaix-Jeuland2

1 PSL Research University, Chimie ParisTech - CNRS, Institut de Recherche de Chimie Paris, 75005, Paris, France
2 CEA, DEN, Service de Recherches de Métallurgie Physique, F-91191 Gif-sur-Yvette, France
anneduchateau0@gmail.com

Abstract

Within nuclear reactor cores, structural components are subjected to high neutron fluxes whose main effect is to create a large amount of point defects such as vacancies and self-interstitial atoms which can aggregate into point defect clusters. The resulting nanometric clusters subsequently have a drastic influence on the macroscopic properties of materials and can lead for example to hardening, through the pinning of dislocations on clusters, or swelling, as a consequence of the formation of cavities [1,2,3]. Several bcc metals are envisaged for the next generation fission reactors and fusion reactors. Our study is focused on α-iron, vanadium and tantalum.

It is commonly accepted that in most metals, interstitial clusters can only grow as two dimensional dislocation loops. However, recently a three dimensional periodic structure for self-interstitial clusters in body-centered-cubic (bcc) metals was proposed [4] (Figure 1). The underlying structure corresponds to C15 Laves phase. Such C15 clusters were observed in molecular dynamics simulations of displacement cascades [5]. Using atomistic calculations, it was demonstrated that in α-iron these C15 aggregates are highly stable and immobile, and that they are strong pinning centers for dislocations [6]. It is thus necessary to better apprehend C15 cluster formation as well as their elementary properties. However experimental evidence for such 3D SIA clusters is still lacking. The main reason being their size, which is most likely below the resolvable limit of conventional TEM.

Very recently a new method was used to make interstitial dislocation loops grow during in-situ isochronal and isothermal annealing [7]. Interstitial loop coarsening by Ostwald ripening can provide insight into single point defects but is very difficult to observe in α-iron and many other metals where nanoscale vacancy clusters dissociate and annihilate loops. By implanting helium in the samples at room temperature at a carefully chosen energy, we observed Ostwald ripening of loops by
transmission electron microscopy during in situ isochronal annealing. This coarsening of loops results in a sharp increase of the mean loop radius at around 850 K in α-iron (Figure 2). Cluster dynamics simulations demonstrated that loops evolve due to vacancy emission and that such experiments give a robust estimation of the sum of the formation and migration free energies of vacancies which will lead us to the entropic contribution in pure α-iron, vanadium and tantalum.

![Figure 2: Dislocation loops radius evolution during in situ isochronal annealing in α-iron](image)

The calculations predict that, at small sizes, in α-iron and tantalum the C15 clusters are more stable than the loops in variance with vanadium for which the loops are the most stable configuration of the self-interstitial clusters. To probe the C15 clusters experimentally, we will try to make them grow by vacancy emission using this in situ annealing method.

Another way to study C15 formation would be TEM in situ irradiation with self-ions to generate cascades which would allow us to see C15 formation, growth and their possible destabilization and dissociation into 2D interstitial clusters.

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**References**

Cavity growth in ODS steels under high dose helium implantation

O. Emelyanova\(^1\), K. Prikhodko\(^2\), M. Ganchenkova\(^1\), V. Borodin\(^1,2\), P. Dzhumaev \(^1\),

P. Vladimirov\(^3\), A. Moeslang\(^3\)

\(^1\) National Research Nuclear University (MEPhI), 115409, Kashirskoe highway 31, Moscow, Russia

\(^2\) National research centre “Kurchatov institute”, 1, Akademika Kurchatova pl., Moscow, 123182, Russia

\(^3\) Karlsruhe Institute of Technology, Institute for Applied Materials – Applied Materials Physics, Hermann-von-Helmholtz-Platz 1, 76344 Eggenstein-Leopoldshafen, Germany

eolga@bk.ru

Abstract

Oxide dispersion strengthened (ODS) steels have a great potential to be employed as high performance structural materials for fusion and fission applications [1,2]. Since a rather high concentration of secondary gas impurities will be generated in the fusion environment, helium effects on the microstructure of ODS steels will be critical for radiation stability of reactor structural materials during operation [3,4]. Both steels were implanted by 40 keV He\(^+\) ions up to a fluence of \(5 \times 10^{20}\) m\(^{-2}\) in the temperature range 293K-923 K. Helium effects on ODS-EUROFER and EP-450-ODS were investigated in a transmission electron microscope (TEM) equipped with energy loss spectroscopy (EELS).

Two ion implantation regimes were used. In one case samples were irradiated at room temperature and then were subject to in situ annealing. Alternative, samples were immediately implanted at 923 K. In this report we present and compare TEM examination results for samples implanted in both regimes. Statistical analysis of bubbles density and size distribution was carried out. Swelling rates have been calculated from the statistical data and compared with the data from non-ODS counterpart of investigated steels. Measurements of the helium density inside individual bubbles and cavities were performed by PEELS in STEM mode.

It was shown that high dose helium implantation at high temperature results in the formation of a notable amount of large helium bubbles with different shapes: either regular cubic, or platelets oriented in \(<100>\) direction. Helium bubbles were found nonuniformly distributed within the subgrains of both steels. Microstructural features like grain boundaries and dislocations seem to have significant effect on He bubbles formation in contrast to yttrium oxide particles, which do not affect the distribution of bubbles at these irradiation conditions (fig. 1). The obtained swelling levels indicate rather high resistance of ODS-steels to helium swelling in comparison with a base material; cf. 0, 93 % swelling for EP-450-ODS against 6,5% for base EP-450.
Fig. 1 Bright field image of EP-450-ODS after He implantation at 923 K

References

Characterization of the sp²/sp³ ratio of ion irradiated PyC interphases from silicon carbide based ceramic matrix composites

Loïc Fave¹, Cécile Hébert², Manuel A. Pouchon¹

¹ Laboratory for Nuclear Materials, Paul Scherrer Institut, OHLD/016, Switzerland
² CIME, École Polytechnique Fédérale de Lausanne, Switzerland

loic.fave@psi.ch

Abstract

Silicon carbide based composite claddings (SiC/SiC) have been receiving a lot of attention in the frame of accident tolerant fuel (ATF) research. Thanks to its outstanding mechanical stability at high temperature, high thermal conductivity and overall good resistance against radiation damage, silicon carbide is considered for both GenIV and LWR reactor types.

In the frame of a research project focused on the thermal conductivity of the aforementioned composite material, a strong emphasis is put on the pyrolytic carbon (PyC) interphase linking matrix and fibers within the material. Electron microscopy techniques are used to study the material at the micro- and nano-scale. The radiation induced changes of this interphase are characterized by acquiring EELS spectra and energy filtered transmission electron microscopy (EFTEM) maps at fiber/matrix intersections. With this technique, data cubes in the x-y-ΔE space are acquired and used to quantify the speciation of the carbon atoms found in the PyC interphase. This technique has been previously used by Yan et al.[1], [2] in a study of the mechanical properties of non-irradiated PyC layers.

Based on the spectra taken at different locations, the ratio between carbon atoms hybridized in sp² and sp³ states is used to determine the extent of the damage caused by the exposure to ions and neutrons. The method used to obtain said ratio is well documented in a paper by Zhang et al. [3].

Figure 1: PCA reconstructed EFTEM series showing the interface between two fibers (bottom right and first third of the micrograph on the left). Data stems from pristine SiC/SiC, showing a graphite-like PyC interphase.

Three days of beamtime at JANNuS-Orsay of the CSNSM, Orsay, France which is part of the EMIR French accelerators network have been granted to us. During these, TEM lamellae have been exposed
to a self-ion irradiation (i.e. using Si and C) ions in order to study the dynamics of the damaging mechanisms occurring within the PyC interlayer.

The results from this irradiation campaign will be presented and put into perspective by comparing them to results obtained on electron irradiated nuclear graphite published by Voronov et. al [4], [5] as well as our own data acquired on neutron irradiated SiC/SiC claddings.

These observations have been carried out on lamellae extracted from cladding tubes obtained from industry partners through the CARAT (DE-NE0000566) and MatISSE (EU grant agreement n°604862) research programs. This project is funded through the ccem.ch MeAWat project.

References
In-situ study of nano-precipitates evolution in the 6061-T6 Aluminum alloy under ion and electron irradiation

Camille Flament\(^1\)*, Joël Ribis\(^1\), Jérôme Garnier\(^1\), Jean Henry\(^1\), T. Vandenberghhe\(^1\), A. Gentils\(^2\), C. Baumier\(^2\), O. Kaïtasov\(^2\), Alexis Deschamps\(^3\)

\(^1\)CEA Saclay, DEN, DMN, SRMA, CEA Saclay, F-91191 Gif sur Yvette, France
\(^2\)CSNSM, Univ Paris-Sud, CNRS/IN2P3, 91405 Orsay, France
\(^3\)SIMAP - Grenoble INP Phelma, BP75, 38402 Saint Martin d'Hères Cedex, France

camille.flament@cea.fr

Abstract

The 6061-T6 Aluminum alloy, whose microstructure contains Al(Fe,Mn,Cr)Si dispersoids and hardening needle-shaped β'' precipitates (Mg, Si), has been chosen as the structural material for the core vessel of the Material Testing Jules Horowitz Nuclear Reactor [1]. Because it will be submitted to high neutron flux at a temperature around 50°C, it is necessary to study microstructural evolutions induced by irradiation and especially the stability of the particles which have an impact on the mechanical properties of the alloy. Thus, to study the behavior of dispersoids and β'' nano-phases under irradiation, in-situ electron and ion irradiations have been performed associated to characterization techniques like Energy Filtered Transmission Electron Microscopy (EFTEM), High Resolution and Atom Probe Tomography.

The precipitate characterization by EFTEM demonstrates that Al(Cr,Fe,Mn)Si dispersoids display a core/shell organization with a (Fe,Mn) enriched core surrounded by a Cr rich shell. The in-situ 1MeV electron irradiation of this organization in a High Voltage Electron Microscope (CEA Saclay, SRMA) shows an enhancement of the core/shell structure by irradiation. This result is discussed in terms of Radiation-Enhanced Diffusion (RED). Under electron irradiation atomic mobility is more likely to result from point defects diffusion rather than from ballistic displacements. It is then proposed that radiation-enhanced diffusion favors the effect of unmixing forces and Cr interface segregation, allowing the system to relax toward the equilibrium core/shell structure [2].

The stability of the β'' needle-shape nano-phases has been investigated under ion irradiations. Dark Field microscopy in the [100] orientation reveals that β'' completely dissolve after the high dose ex-situ W\(^{3+}\) (2MeV) irradiation performed at the Jannus Saclay facility. However a new precipitation of (Mg,Si,Cu) rich clusters, which have a common direction with [112], occurs. An in-situ Au\(^{2+}\) (4MeV) irradiation at the Jannus Orsay facility highlights that both clusters and β'' coexist. In order to understand the mechanism responsible for such evolutions, an in-situ Au\(^{2+}\) ion irradiation of an untempered 6061 alloy (without β'' initially) has been performed. Dark Field observation in the [112] orientation during irradiation shows that clusters precipitate from the solid solution. It is proposed that vacancy drag (like Si-V) may play a key role in their precipitation.

References


*Currently working at CEA Saclay, DEN, DMN, SRMP.*
In-situ study of dislocation climb in zirconium alloys under charged particles irradiation

M. Gaumé1,2, F. Onimus1, T. Vandenberghe1, L. Dupuy1, F. Mompiou2

1 CEA, DEN, Section for Applied Metallurgy Research, 91191 Gif-sur-Yvette, Cedex, France
2 CEMES CNRS, 29 rue Jeanne Marvig, 31055 Toulouse Cedex 4, France

Abstract

In Pressurized Water Reactor, fuel cladding tubes, made of zirconium alloys, are subjected to mechanical loadings under neutron irradiation leading to in-reactor deformation. Although the macroscopic behavior of zirconium alloys under irradiation is well known, the microscopic deformation mechanisms still need to be better understood. The aim of this work is to study the microscopic deformation mechanisms occurring under charged particles irradiation and especially the climb of dislocations due to irradiation.

In situ electron irradiation experiments have been conducted on recrystallized Zy-4 at temperatures ranging from 400 to 500°C using a 1 MeV High Voltage Electron Microscope (HVEM). During these in situ experiments <a> loop growth has been observed and characterized depending on irradiation temperature and dose. The dislocation loops nucleate after few minutes of irradiation which correspond to 0.6 to 1 dpa, the damage rate being of 13 dpa/h. Their diameter and density increase rapidly up to a diameter of about 30 to 50 nm after 7 dpa (Hellio, de Novion, Boulanger, 1988). The majority of these loops are of interstitial nature, as proved by the use of the inner/outer contrast method (Griffiths, 1994; Kelly, Blake, 1973). The helical climb of the <a> dislocations has also been observed under electron irradiation. This phenomenon is known to be responsible for the irradiation creep deformation (Figure 1).

Zr ion irradiations of recrystallized Zy-4 have been performed under the same conditions using an ion accelerator (Jannus-Orsay facility) and the results have been compared with the observation of electron irradiation.

References

The *in situ* dual ion beam TEM at CSNSM: history and overview of the JANNuS-Orsay facility

JANNuS-Orsay technical staff:
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JANNuS-Orsay local contacts:
Cédric Baumier, Brigitte Décamps, Franck Fortuna, Aurélie Gentils, Stéphanie Jublot-Leclerc

*CSNSM, Univ Paris-Sud, CNRS/IN2P3, Université Paris-Saclay, 91405 Orsay, France
Presenter’s e-mail address: aurelie.gentils@csnsm.in2p3.fr*

**Abstract**

*In situ* Transmission Electron Microscopy (TEM) studies of materials submitted to ion beams have been running since the 1980’s in the CSNSM lab in Orsay, France, under the scientific leading of Marie-Odile Ruault, now retired. The first TEM, a Philips EM400 microscope operating at 100 kV, was connected in 1980 to the 190 kV ion implantor IRMA [1], with a perpendicular ion beam with respect to the microscope. The second microscope installed in 1994 was a Philips CM12 operating at 120 keV with a 0.37 nm resolution. It was equipped with EDX and a double tilt holder working from liquid nitrogen temperature up to 700°C. The connection to IRMA was still perpendicular to the microscope’s column. Several dynamical studies concerning crucial features of materials evolution submitted to ion beams were undertaken, and most of them are reviewed in [2].

Launched in 2009, JANNuS, ‘Joint Accelerators for Nano-science and Nuclear Simulation’, is an open facility for the scientific community in the fields of ion implantation/irradiation effects, and nanosciences and nuclear materials using ion beams [3]. The JANNuS facility is a member of the EMIR French accelerator network [4], located in Saclay and in Orsay. In JANNuS-Saclay facility, three ion accelerators are coupled together allowing triple ion beam irradiation of materials [5]. In JANNuS-Orsay facility, by combining two ion beams in a transmission electron microscope, it allows well-controlled modelling-oriented experiments where you can tune the simultaneous production of nuclear recoil damage versus implantation for a large array of ions. Located at 20 km from Paris in the CSNSM[6], the JANNuS-Orsay facility [6] is built around a 200 kV Tecnai G² 20 Twin FEI microscope coupled with two ion accelerators (IRMA in the energy range 10-570 keV [1] and ARAMIS, a home-made 2 MV Van de Graaff-Tandem accelerator, delivering ions in the range 0.5-15 MeV [7]). This setup, with a microscope resolution of 0.27 nm, allows *in situ* observation of the material microstructure modifications induced by single/dual ion irradiation/implantation. Due to the complementary analytical equipments (EDX, STEM and GIF) associated with a large range of specimen holders (nitrogen cooling, heating up to 1300°C…), it offers a large potential to simultaneously study structural and/or chemical modifications and defects induced by implantation and/or irradiation using ion beams.

The history and overview of this existing *in situ* TEM facility will be shown in this presentation in the light of scientific results obtained throughout the years.

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1 Centre of Nuclear Science and Material Science, joint research unit of Université of Paris-Sud and CNRS/IN2P3, member of Université Paris-Saclay
General view of the JANNuS-Orsay in situ TEM facility of the CSNSM, showing the TEM connected to IRMA and ARAMIS ion accelerators lines

References

A TEM study of electron radiation damages in two calcium silicates and a magnesium rich silicate

Marie-Noëlle de Noirfontaine¹, Mireille Courtial¹,², Frédéric Dunstetter¹, Marcel Signes-Frehel¹, Guillaume Wang³, Pierre-Eugène Coulon¹ and Dominique Gorse-Pomonti¹

¹Laboratoire des Solides Irradiés, Ecole Polytechnique, CNRS, CEA, Université Paris-Saclay, 91128 PALAISEAU cedex, France
²Université d’Artois, 1230 rue de l’Université, CS20819, 62408, Béthune, France
³Laboratoire Matériaux & Phénomènes Quantiques, CNRS, Université Paris-Diderot, 75205, France

Dominique.gorse-pomonti@polytechnique.edu

Abstract

We present a contribution to the general study of radiation damages in complex ceramic oxides. The structural evolution under electron irradiation of three orthosilicates, the structure of which is characterized by isolated \([\text{SiO}_4]^4\) tetrahedra, is followed in the electron microscope. The flux values vary over a wide range, typically between $2.7 \times 10^{17}$ and $2.2 \times 10^{22}$ e·cm$^{-2}$·s$^{-1}$, and the highest doses approach $10^{25}$ e·cm$^{-2}$.

For the two calcium silicates, with increasing electron flux, the following damages are observed: i) quasi instantaneous decomposition of the silicates particles into CaO nanocrystals separated by SiO$_2$ rich amorphous areas at low flux; ii) once reached a threshold flux, formation of an amorphous crater fully calcium depleted in tricalcium silicate but not in dicalcium silicate; iii) structural evolution of the crater areas that recrystallize post mortem in the dicalcium silicate but not in the tricalcium silicate; iv) hole drilling in tricalcium silicate once reached a threshold flux of $7.9 \times 10^{21}$ e·cm$^{-2}$·s$^{-1}$ and a dose of $2.3 \times 10^{24}$ e·cm$^{-2}$ (see figure below) whereas dicalcium silicate still amorphizes under the electron beam up to a flux value of $2.2 \times 10^{22}$ e·cm$^{-2}$·s$^{-1}$.

For the magnesium rich silicate, the available results suggest a similar sequence of radiation damage effects with increasing flux, only shifted to lower values of flux by at least two orders of magnitude.
This last point should be related to the structural and compositional characteristics of this magnesium rich silicate, by comparison with the above studied calcium silicates.

Our focus will be on the chemical analysis of the damaged areas (decomposition areas at “low” flux, amorphous crater or compound closing the hole during the healing process at “high” flux). In particular the question of their precise composition will be specifically addressed. This information is strongly required in order to be able to propose a convincing damage mechanism for each orthosilicate studied.

References
In-Situ TEM experiment to simulate rim structure formation

Yara Haddad*, Frédérico Garrido, Aurélie Gentils
CSNSM, Univ Paris-Sud, CNRS, Université Paris-Saclay, Orsay Campus, France
Yara.Haddad@csnsm.in2p3.fr

High burnup structure (or rim structure) is observed at the peripheral region of the nuclear fuel pellet with thickness of 100-200 µm where several observations and characterizations are notable in that zone [1-5]:

1- Pu content and burnup increase.

2- Development of fission gas pores (leading to a porous region) with a typical diameter 1-2 µm and the maximum porosity between 10% and 22%. The fission gas pore density increases with increasing the local burnup.

3- A large reduction in crystallite size, where the original grain, having a typical size of around 10 µm, subdivides into sub-micron grains with a size of about 0.2 to 0.3 µm. In these fine grains, a lower overall dislocation density and a much lower density of intergranular fission gas bubbles and precipitates are found.

4- A decrease in the content of Xenon fission product within the UO$_2$ grains (athermal release of Xe from the UO$_2$ grains).

The electron transmission microscopy (TEM) technique is one of non-destructive methods that can be applied to observe the rim structure and to identify the mechanisms which are responsible to the appearance of such structure.

Therefore, in-situ TEM experiment was performed at JANNuS-Orsay to reproduce the specific microstructure of the high burnup structure of the irradiated nuclear fuel. This is experimentally simulated by using a very simplified model system – namely uranium dioxide single crystals – and 260-keV Xe ion beam at 500 °C for generating radiation damage and for doping the material with xenon fission product. Preliminary results will be presented, showing the importance of the various relevant parameters involved in the formation of high burnup structure, and helping in clarifying the synergies between them.

References:

The Effect of He/DPA Ratio on the Micro-Structure of Tungsten Under \textit{in-situ} Ion Irradiation

R.W. Harrison, H. Amari, G. Greaves, J.A. Hinks and S.E. Donnelly

School of Computing and Engineering, University of Huddersfield, Huddersfield, HD1 3DH

Abstract

Tungsten (W) has been chosen as the material for use in the divertor of the International Thermonuclear Experimental Reactor (ITER) due to its high melting temperature, excellent thermal conductivity and low sputter yield. As a plasma-facing material, W will be exposed to high-heat flux, high-energy neutrons (>14 MeV) and He production from \((n,\alpha)\) reactions as well as injection from the plasma. He has a low solubility in these materials and generally agglomerates in areas of high defects (e.g. vacancies and grain boundaries). Thus, He production from \((n,\alpha)\) transmutation reactions in nuclear reactors will adversely affect the properties of structural materials. The ratio of He concentration to atomic displacements (He/DPA ratio), will vary greatly in the divertor from \(~0.2\) appm He/DPA in the bulk to \(1000s\) at the surface. Divertor local temperature will generally operate at above 1000°C and may reach as high as 3000°C. Reports on the effects of He/DPA ratio on the microstructure of W in the literature are often difficult to extract and compare due to variations in the experimental conditions between studies. For instance, it is not straightforward to compare the results of neutron irradiation with those of dual-beam ion irradiation (self-ions and He), differences in total fluence, irradiation temperature and starting material composition. Using the Microscope and Ion Accelerator for Materials Investigation (MIAMI) facility, the effect of varying the He/DPA ratio on the damage microstructure of tungsten has been studied in the temperature range 500–1250°C at damage levels up to 100 DPA. Simply by changing the energy of the He ions, irradiating a 50 nm TEM foil, over the range 40–100 keV the He/DPA ratio can be varied from tens to hundreds of He-appm per DPA. The resulting damage microstructure (dislocation loops and He bubbles) has been characterised by TEM as a function of He/DPA ratio, temperature and damage level, thus generating a detailed complete data-set of defect microstructure evolution in identical tungsten samples as a function of these variables.
Effect of Heat Load on Microstructural Development in Irradiated Steels
N. Hashimoto, E. Suzuki
Hokkaido University, Sapporo, Hokkaido, Japan

Thermal neutron induces cascade damage and defect clusters would form in structure materials. When the severe accident or some crucial troubles happens and results in the stop of coolant system, it is predicted that temperature would rise up in a short time and then cooled down rapidly or slowly. In such heat load might affect the mechanical property of structural materials. In order to investigate the influence of heat load on mechanical properties, this study is focused on the change of microstructure such as the size and the density of Frank loops, line dislocations and voids in electron- and ion-irradiated 316L stainless steel. The heat treatment at several heating and cooling rates after irradiation to 0.1 dpa indicated less change in the size and the number density of black dots. This suggests that the influence of heat load on microstructure is small at early stage of irradiation. On the other hand, heating up to 700 °C for 20 min. and then cooling down to 290 °C for 800 min. resulted in the growth and unfaulting of Frank loops. While, the sample heated up for 5 min. and cooled down for 5 min. indicated the growth of voids. From these results, it would be suggested that a low rate cooling down results in the growth of Frank loops, and also a high rate cooling down after a high rate heating up does the growth of voids.
Recent Advancements in Sandia’s In-situ Ion Irradiation Transmission Electron Microscope

Khalid Hattar¹, and Barney L. Doyle¹, and Daniel L. Buller¹

¹Sandia National Laboratories PO Box 5800 Albuquerque, N.M. 87185 U.S.A.
khattar@sandia.gov

Abstract

This presentation will highlight the recent development of the In-situ Ion Irradiation Transmission Electron Microscopy (I³TEM) facility and some of associated results that have been achieved to date. This facility is located at the Ion Beam Lab at Sandia National Laboratories in Albuquerque, New Mexico, U.S.A. The move of the 6 MV Tandem and the instillation of the JEOL 2100 was started in late 2010 with the first beam in the TEM achieved in April 19, 2011. From that date to the present, the I³TEM facility has been under nearly continuous development [1]. The historical development can be clearly seen in the complexity of the beam line and TEM control units added, Figure 1 A-C.

The facility currently combines a JEOL 2100 LaB₆ TEM, a 6 MV HVE EN Tandem Van de Graaff–Pelletron accelerator, and a 10 kV Colutron G-1 ion accelerator. The two ion beams enter the TEM through the same port nearly orthogonal to the electron beam. Through detailed ion optics design and multiple beam line developments it is now possible to ion irradiate and implant from both the Tandem and Colutron concurrently. In addition, the Colutron was outfitted to permit two gas sources to be operated simultaneously, which permits a limited triple beam capability when a high-energy heavy-ion from the Tandem is concurrently irradiated with a mixture of He⁺ and D₂⁺ from the Colutron. Two optical ports have been added to the TEM permitting real time photoluminescence, cathodoluminescence, and ion beam induced luminescence, as well as rapid alignment of the ion beam optics. To mitigate many of the concerns regarding in-situ TEM experiments, a large chamber capable of handling coupon size samples has been incorporated just upstream of the TEM. In addition to the ion beam capabilities, the TEM has also been outfitted with an ASTAR/Nanomesas precession electron diffraction system, two tomography stages, two mechanical testing stages, two environmental stages, a heating stage, and a custom electrical biasing stage. This combination of in-situ capabilities permits direct juxtaposing of extreme conditions that materials might encounter in real world environments. A few examples will be highlighted to demonstrate the capabilities of the system to study the effect of various ion species [2], various ion energies [3], in-situ TEM observation [4], as well as order and synergistic ion beam exposure [5].

In addition to the current capabilities and associated results, the I³TEM is still very much a facility under development. Work is currently underway to:

1) Add an additional 1 MV Tandem accelerator to the facility completing the current energy gap between the ion beams run from the Colutron (up to 20 keV) and directly from the SNICS source (46 keV) and those accelerated by the Tandem (minimum of 800 keV).

2) Convert a 90˚ bending magnet to a 45˚ bending magnet in order to reach the maximum energy of our Tandem accelerator (greater than 88 MeV).

3) Add adequate spectrometry to both the TEM and Colutron to better characterize the ion beams

4) Upgrade the electron optics of the TEM to improve the time resolution [6].

These current and planned capabilities should provide a rich variety of extreme environments that can be observed and analyzed all with the resolution of the TEM.
Acknowledgements

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Figure

Figure 1: The evolution of the In situ Ion Irradiation TEM (I^3TEM) facility at Sandia National Laboratories. A) JEOL 2100 TEM installed. B) TEM connected to a 6 MV EN Tandem Accelerator. C) 10 kV Colutron added to I^3TEM. D) Current status of the I^3TEM with planned addition of electron optics for nanosecond time resolution, 1 MV accelerator to complete the energy range, and a new bending magnet to permit greater than 88 MeV ions.

References

Ion-Beam Modification of Silicon Nanowires

I. Hanif, G. Greaves, S.E. Donnelly and J.A. Hinks

School of Computing and Engineering, University of Huddersfield, Huddersfield, HD1 3DH,

United Kingdom

Semiconductor nanowires are important candidates for incorporation into the next generation of nanotechnologies as resonators, diodes, transistors, generators and sensors etc. As well as their nanoscale dimensions, their behaviour and properties are also different from their bulk equivalents because of quantum effects, their increased surface-to-volume ratios and degrees of freedom which can enable novel options for modification and assembly. Previous work by various groups have demonstrated how ion beams can be used to induce plastic deformation in semiconductor nanowires. However, these published results demonstrate some inconsistencies and various models have been put forward for the mechanism(s) driving the changes. Ion irradiation experiments have been performed to induce bending in silicon nanowires whilst under observation via transmission electron microscopy using the Microscope and Ion Accelerator for Materials Investigations (MIAMI) facility. These new results will be presented in this talk and discussed in the context of existing literature and proposed models.
In-situ electron irradiation within a TEM: a way to study the stability and evolution of cavities in pure aluminium

C. Jacquelin\textsuperscript{1}, M. Loyer-Prost\textsuperscript{2}, T. Schuler\textsuperscript{1}, E. Meslin\textsuperscript{1} and M. Nastar\textsuperscript{1}

\textsuperscript{1}: CEA, SRMP (Service de Recherches de Métallurgie Physique), 91191 Gif-sur-Yvette Cedex, France

\textsuperscript{2}: CEA, laboratoire, JANNUS , SRMP (Service de Recherches de Métallurgie Physique), 91191 Gif-sur-Yvette Cedex, France

camille.jacquelin@cea.fr

Abstract

Al-based alloys are foreseen for the cladding of the future material testing reactor Jules Horowitz. Under irradiation, a large amount of point defects (interstitials and vacancies) are created. They are mobile and may cluster to form extended defects such as dislocation loops in 2D or cavities in 3D. This work is focused on cavities, which may induce swelling and embrittlement. Because of its low Z number, it is possible to create defects in this metal with the typical low-electron energy available within a Transmission Electron Microscope (TEM), as recently obtained at the atomic scale in a Mg metal [1]. Facetted cavities are indeed observed within in-situ experiments performed with 200 and 300 keV electrons. The surface free energy ratio of low-index planes \{100\} to \{111\} in pure Al (99.999\%) are estimated by means of the Wulff construction and measurement of the relative proportion of the corresponding facets (figure 1). This ratio is then compared to recently published \textit{ab initio} values. Note that an experimental direct characterization of the pure Al free surfaces would be almost impossible due to the formation of highly stable Al$_2$O$_3$ oxide. The present results are compared to the facetted cavities observed in pure iron after ex-situ ion irradiation.

Figure 1: Left: Facetted cavities in Al (99.999\%), under focused TEM image, 0.63.10\(^{-4}\) dpa/s (Ed = 16 eV), RT, \(z=\{110\},\) FEI Titan ECP; Right: Corresponding Wulff construction, \(\gamma(100)/\gamma(111) = 1.07\)
The kinetic properties of cavities are also investigated. In situ observations show that the latter appear after the formation of interstitial loops.

Facetted cavities evolution with time (Figure 2) reveals a succession of positive and negative growing rates, which may be interpreted as oscillations between magic number-sized cavities.

Figure 2: Cavities evolution in pure Al (99.999%) under irradiation, (6.3 \(10^5\) dpa/s (Ed = 16 eV), [5.7-8.0 \(10^2\) dpa], RT), z=[001], g=[200], under focused TEM images, FEI Titan, ECP.

To confirm those results, electron irradiation under lower electron intensity and lower electron energy within the TEM are on-going so that more data per unit time will be collected. The effect of irradiation flux on the cavity formation will be studied.

References

Influence of ion irradiation and He implantation on the swelling microstructure of austenitic stainless steels

Stéphanie Jublot-Leclerc, Marie-Laure Lescoat, Franck Fortuna, Laurent Legras, Xiaoqiang Li, Aurélie Gentils

1 CSNSM, Univ Paris-Sud, CNRS/IN2P3, Université Paris-Saclay, 91405 Orsay, France
2 EDF R&D, Groupe Métallurgie, Les Renardières, 77818 Moret sur Loing, France
3 National Key Laboratory of Thermostructure Composite Materials, Material Science and Engineering Department, Northwestern Polytechnical University, Xi’An, Shaanxi 710072, China

Stephanie.jublot-leclerc@csnsm.in2p3.fr

Extending nuclear power plants lifetime requires a very good knowledge and a good control of the mechanisms of ageing of all their components. This study deals with nuclear reactors material components of Pressurized-Water Reactors (PWR), such as Reactor Pressure Vessel Internals, made of austenitic stainless steels, which are subjected to intense irradiation at elevated temperature. These extreme conditions alter significantly their properties, and a phenomenon of swelling due to irradiation may be possible under PWR conditions. The sensitivity of materials to swelling when irradiated has been studied mainly in the context of work on fast breeder reactors (FBR) above 400°C. It is not therefore possible to extrapolate the FBR data to the operating conditions in PWRs without considering additional factors, and in particular, the effect of He gas produced by the “thermal” neutrons in the PWR spectrum. The main objective of our study is to define, at the nanoscale, the mechanisms and kinetics of swelling occurring in austenitic stainless steels under long-term neutron irradiation, while taking into account synergistic effects of irradiation and helium gas. In this study, experiments were carried out on 316LSH1 industrial austenitic stainless steels at the JANNuS-Orsay facility using single ion irradiation [1] and helium ion implantation [2] as well as simultaneous ion irradiation and helium implantation in the transmission electron microscope. Ion irradiations and implantations were performed at elevated temperatures (200, 450 and 550°C) to doses obtained after several years in reactors. Detailed and quantitative results on the microstructure of the material will be shown and in particular, the influence of temperature, dose and helium on the formation of cavities will be discussed.

Cavities observed by transmission electron microscopy in 316L austenitic stainless steel after coupled Au ion irradiation and He ion implantation at 550°C (fluence of Au and He: 2x10^{15} cm^{-2}).
We acknowledge financial supports from the French National Research Agency through project ANR-11-BS09-006, and from the French EMIR network.

References
Effects of ion irradiation on dislocation dynamics in a Ni-based superalloy studied through the use of in-situ TEM

Djamel Kaoumi, The University of South Carolina, SC, USA,
Mark Kirk, Argonne National Laboratory, IL, USA

Foils of Inconel 617 were deformed in situ in a TEM at 298K without and after ion irradiation with the goal of evidencing the deformation mechanisms in this Ni-based superalloy and how irradiation-induced defects affect these mechanisms. Outside of irradiation, slip bands develop during the straining experiments. The movement of the dislocations in the slip bands, at the interfaces, and about the precipitates is observed in-situ. The arrangement of moving dislocations, especially the propagation of pile-ups of bowed dislocations, is observed in detail. Particularly, the process of discontinuous glide of thousands of dislocations is captured in-situ. The dislocations moving in the slip bands are not preexisting dislocations but originate from grain boundaries and other stress concentrators as strain is induced in the foil. After ion irradiation to a fluence of 1x10^{14} ions/cm^2 with 1 MeV Kr ions, similar straining experiments were done and the impact of the radiation damage on the dislocation behavior was assessed. In contrast with the case outside of irradiation, dislocation motion through the irradiated matrix is observed to be jerky and discontinuous, as the dislocations now progress through a medium with irregularly spaced irradiation-induced defects. These defects form a collective barrier to dislocation motion, strong enough to slow down or even stop dislocation pile-ups inside grains. The progression of the pile-ups proceeds in bursts of multiple dislocations. Sources of dislocations are still active and captured in-situ. Within the slip channels, interactions with irradiation defects can lead to congested channels and heavily jogged dislocations, resulting in a higher driving force for dislocation cross-slip from the channels to the outside matrix, a phenomenon also captured in-situ. Overall the talk will emphasize how the use of in-situ TEM coupled with ion irradiation can help evidence how the mechanisms of dislocation dynamics in such fcc alloys are affected by radiation damage.
TEM with *in situ* ion irradiation and modeling to predict neutron irradiation damage

Marquis Kirk and Meimei Li

IVEM-Tandem Facility, Nuclear Engineering Division, Argonne National Laboratory, Lemont, IL 60439 USA

kirk@anl.gov

**Abstract**

We will review our TEM experiments with *in situ* ion irradiation of thin foil Mo to establish simulation model parameters that can predict irradiation microstructure in both thin foils with strong surface effects and in neutron irradiation of bulk metals. To illustrate one of the unique applications of the IVEM Facility with *in situ* ion irradiation, we summarize recent experiments to measure interstitial defect formation and clustering in thin film molybdenum at 80 C during 1 MeV Kr ion irradiation. Using this *in situ* ion irradiation data we describe the construction of a model for interstitial cluster formation and dynamics that can then be applied to predict results for neutron irradiation of bulk Mo. The latter model prediction is compared to experimental results for neutron irradiation of the same pure Mo material at similar temperature. Careful attention is given to the physics of both ion and neutron damage mechanisms. The loss of mobile interstitial defects to free surfaces is uniquely measured in 3D by electron diffraction tomography and well modeled.
TEM with *In Situ* Ion Irradiation of Nuclear Materials: The IVEM-Tandem User Facility

Meimei Li, Mark Kirk, Pete Baldo, Ed Ryan

*Argonne National Laboratory, Lemont, IL, USA*

*mli@anl.gov*

**Abstract**

Transmission electron microscopy (TEM) with *in situ* ion beam irradiation is an essential tool for understanding fundamental radiation effects, critical to the development of advanced new materials and predictive models of nuclear materials and fuels. The IVEM-Tandem Facility at the Argonne National Laboratory is the leading user facility for radiation damage research. It offers a unique opportunity to test the microstructural response of materials *in situ* to extreme conditions of high-dose irradiation, temperature, and stress, making important contribution to the understanding of physical phenomena and providing invaluable data for validating multiscale materials models.

The IVEM-Tandem has been a user facility for 20 years. It is currently serving more than 30 user projects from universities, national laboratories, nuclear industry and users from abroad. This presentation will provide an overview of the facility capability and highlight user research on advanced nuclear materials and fuels, storage materials for spent fuels, and validation and verification of computer modeling and simulations.
Ion irradiation and radiation effect characterization at the JANNUS-Saclay triple beam facility

M. Loyer-Prost¹, G. Gutierrez¹, F. Leprêtre¹, Y. Serruys¹, E. Bordas¹, H. Martin¹, S. Pellegrino¹,², C. Cabet¹

¹ CEA, DEN, Service de Recherches de Météllurgie Physique, Laboratoire JANNUS, 91191 Gif-sur-Yvette, France
² CEA, INSTN, UEPTN, Laboratoire JANNUS, F-91191 Gif-sur-Yvette, France

Abstract

In complement to in-situ TEM analysis performed on the JANNuS-Orsay facility, the JANNUS-Saclay irradiation facility provides a triple ion beam for well-controlled modelling-oriented experiments. The system consists of high vacuum chambers and beamlines, which are connected with electrostatic accelerators. The accelerators can be operated concurrently to deliver up to three simultaneous ion beams on one target sample to study the effects of displacement damage and/or implantation of a large selection of ions on microstructural changes of materials. Samples can be irradiated in the wide temperature range from liquid nitrogen to 850K. Along with experimental developments in irradiation/implantation chambers, continuous efforts have been made to implement ex situ and in situ characterization techniques. This paper gives an overview of JANNUS-Saclay current status with a focus on on-line instrumentation, in situ Raman spectroscopy and ex situ characterization by ion beam surface analysis (NRA, ERDA, PIXE). Access to the facility for national and international users is provided thru the French network EMIR http://emir.in2p3.fr/).
Using in-situ TEM observations during ions/electrons irradiation: a powerful tool for a better understanding of the microstructural mechanisms inducing oxide dissolution in Oxide Dispersion Strengthened ferritic steel

I. Monnet¹, O. Kaitasov², P. Dubuisson³

¹CIMAP, CEA-CNRS-ENSICAEN-UCN, BP 5133 F-14070 Caen cedex 5, France
²CSNSM, CNRS-IN2P3-Université Paris-Sud, Bât. 108, F-91405 Orsay, France
³CEA DEN/DPC, CEA Saclay F-91191 Gif sur Yvette cedex, France

monnet@ganil.fr

Abstract

In the scope of Generation IV forum, future nuclear reactors would operate in extreme conditions, meaning high temperature (350-700°C) and high radiation damage (more than 150 dpa). Oxide dispersion strengthened (ODS) steels are potential candidates as structural components for such future reactors. These ODS ferritic/martensitic steels exhibit low swelling under irradiation and offer improvements of the out-of-pile strength characteristics above 550°C due to the fine dispersion of oxides. However, the stability of the reinforcing oxides under irradiation has to be assessed in order to ensure sustainable mechanical properties in service.

An experimental irradiation in Phenix of a ferritic ODS type alloy indicates that yttrium oxide encoring dissolution under neutron irradiation [1]. For this type of materials, several kind of phenomena can be responsible for such a dissolution (thermal dissolution, recoil dissolution, dissolution induced by displacement cascade, dissolution due to inelastic interaction, ...). Irradiation with neutrons shows the influence of dose and, in a smaller part, of temperature. But this kind of irradiation does not allow us to determine the influence of inelastic interaction, Frenkel pairs or displacement cascades formation because all this type of phenomena are present. In order to improve the understanding of the mechanisms of dissolution, DY and four other ferritic steel (Fe-9Cr) reinforced respectively by Al₂O₃, MgO, MgAl₂O₄ and Y₂O₃ were irradiated with different charged particles and observed in a transmission electron microscope during the irradiation with ion or electron.

- Irradiation with 1 MeV Helium does not induce any modification, neither in the chemical modification of the particles nor in their spatial and size distribution. Since most of the energy of helium ions is lost by inelastic interaction, this result proves that this kind of interaction does not induce oxide dissolution. Nevertheless using very high electronic stopping power induces amorphous tracks inside the oxide [2].
- Irradiation with electrons in a high voltage microscope (THT, CEA-Saclay, France) leads to a significant dissolution. By using in-situ observations, we evidence that the radius decrease is proportional to the dose [3].
- The comparison between irradiation with ions (300 keV Ar+, CSNSM-orsay, France) (displacements cascades) and electrons (Frenkel pairs only) shows the importance of free point defects in the dissolution phenomena [4].

Insight from in-situ analysis of grain boundary character, radiation sequence, and thermal conditions on defect structure evolution in nickel

Brittany Muntifering¹,², Khalid Hattar²

¹Northwestern University, 2145 Sheridan Road, Evanston, IL 60208.
²Sandia National Laboratories, PO Box 5800 Albuquerque, N.M. 87185 U.S.A.

Abstract

The structural materials of future nuclear reactors will be exposed to high energy neutrons that cause atomic displacement cascades and transmutation reactions, which produce significant quantities of helium and hydrogen. A combination of heavy-ion irradiation, causing displacement cascades, and gaseous implantation, simulating transmutation products, is a technique used to rapidly emulate neutron damage as well as elucidate fundamental physical radiation mechanisms related to the damage process [1]. In-situ TEM irradiation experiments are critical in characterizing active mechanisms in radiation damage and developing an understanding of the role helium and hydrogen play in materials degradation.

This talk focuses on recent experimental results on nickel involving in-situ TEM self-ion irradiation and gaseous implantation. In-situ ion irradiation TEM experiments were conducted at the In-Situ Ion Irradiation TEM (I³TEM) facility at Sandia National Laboratories [2]. Nickel was chosen as a model face centered cubic system to gain insight into defect evolution for more complicated reactor materials such as austentic steels. Coarse grained and nanograined nickel were tested to elucidate the role that grain boundaries play in radiation-induced defect formation and evolution. Self-ion irradiation at 3 MeV and gaseous implantations of He or D₂ at 10 keV were performed sequentially, in both orders, as well as concurrently. This was done in order to probe the role of irradiation sequence on the microstructural evolution in overlapping radiation environments. Post irradiation annealing was performed to study the thermal aging of these defects.

In certain cases, the sequence of the heavy ion irradiation and gaseous implantation had a significant impact on the distribution of the resulting defect structure. Figure 1 demonstrates the defect structure after a nanocrystalline nickel sample was first self-ion irradiated, figure 1a, and then implanted with helium, figure 1b. In this case, an even distribution of cavities developed through the grain structure. The reverse order of irradiation, where the films were first implanted with helium, figure 1c, then self-ion irradiated, figure 1d, showed an increased density of cavities at the grain boundaries. In contrast, self-ion irradiation and deuterium implantation did not result in any visible cavities, and the sequence of the irradiation had little or no effect on the final loop structures.

Post-irradiation annealing was performed to study the thermal evolution of irradiation-induced defects. In the case of helium implanted and self-ion irradiated nanocrystalline films, annealing resulted in cavities nearly as large as the film thickness, demonstrated in figure 2a. Orientation mapping, figure 2b, demonstrated that some cavities were larger than the grains and crossed multiple grain boundaries, suggesting that the boundaries have negligible ability to limit the growth of cavities with temperature. An unexpected electron beam effect was also observed during annealing of a self-ion irradiated nanocrystalline sample, where voids were formed only in the area illuminated by the electron beam during annealing. Based on diffraction patterns analyses, it is hypothesized that the electron beam enhanced the growth of a NiO layer resulting in a decrease of vacancy mobility during annealing. The electron beam used to investigate self-ion irradiation ultimately significantly affected the type of defects formed and the final defect microstructure [3].
Figure 1: a) Nanocrystalline nickel after self-ion irradiation b) the same film after additional helium implantation. Nanometer size voids are evenly distributed throughout the grains. c) Nanocrystalline nickel implanted with helium d) the same film after additional self-ion irradiation. Nanometer sized voids were present throughout the film, but with a higher concentration along grain boundaries.

Figure 2: a) Cavities in helium implanted then self-ion irradiated nanocrystalline nickel after annealing b) Orientation map obtained by Precession Electron Diffraction of the same region as (a)

Acknowledgments
The authors thank Prof. Jianmin Qu, Drs. Dan Bufford and Remi Dingreville, and Mr. Aaron Dunn, Michael Marshall and Dan Buller for insightful conversation and collaboration, as well as Drs. Samantha Lawrence, Brian Somerday, and Doug Medlin for materials. This work is primarily supported by the US Department of Energy’s Nuclear Energy University Program (DE-NE0000678). Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy’s National Nuclear Security Administration under contract DE-AC04-94AL85000.

References
In situ TEM observation of fast electron irradiation induced structural change in Al_{0.5}TiZrPdCuNi High Entropy Alloy (HEA) and High Entropy Glass (HEG)

Takeshi Nagase\(^1,2\), Akira Takeuchi\(^3\), Kenji Amiya\(^3\)

\(^1\) Research Center for Ultra-High Voltage Electron Microscopy, Osaka University, 7-1, Mihogaoka, Ibaraki, Osaka 567-0047, Japan

\(^2\) Division of Materials and Manufacturing Science, Graduate School of Engineering, Osaka University, 2-1, Yamada-Oka, Suita, Osaka 565-0871, Japan

\(^3\) Institute for Materials Research, Tohoku University, 2-1-1 Katahira, Aoba-ku, Sendai 980-8577, Japan

Abstract

High-Entropy Alloys (HEAs) and High-Entropy Glasses (HEGs) consist of multiple metallic elements with a nearly equimolar composition to maximize the mixing entropy, which in turn stabilizes the solid-solution phase in HEAs and an amorphous phase in HEGs. HEAs and HEGs have the potential to be used as highly refractory materials resistant to irradiation damage, and thus preliminary works on the irradiation damage in HEAs [1-4] and HEGs [5] were investigated by High Voltage Electron Microscopy (HVEM). In the present study, the structural change in bcc solid solution phase and an amorphous phase in Al\(_{0.5}\)TiZrPdCuNi alloy [6] (See Table 1) stimulated by fast electron irradiation was investigated by in situ TEM observation using the ultra-high voltage electron microscope Hitachi H-3000 installed at Osaka University, Japan.

A part of work was supported by JSPS KAKENHI Grant Number 15K06484, and scientific research grant from Kansai Genshiryoku Kondankai.

Table 1 Nominal Composition of Al\(_{0.5}\)TiZrPdCuNi High Entropy Alloy

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Ti</th>
<th>Zr</th>
<th>Pd</th>
<th>Cu</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>Atomic ratio</td>
<td>0.5</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>at.%</td>
<td>9.09</td>
<td>18.18</td>
<td>18.18</td>
<td>18.18</td>
<td>18.18</td>
<td>18.18</td>
</tr>
<tr>
<td>wt.%</td>
<td>3.54</td>
<td>12.56</td>
<td>23.93</td>
<td>27.91</td>
<td>16.67</td>
<td>16.39</td>
</tr>
</tbody>
</table>

References

A direct comparison between in-situ TEM observations and Dislocation Dynamics simulations of interaction between dislocation and irradiation loop in recrystallized Zircaloy-4

F. Onimus\textsuperscript{1}, J. Drouet\textsuperscript{1,2}, L. Dupuy\textsuperscript{1}, M. Gaume\textsuperscript{1,2}, F. Mompiou\textsuperscript{2}

\textsuperscript{1}Service de Recherches M\textsuperscript{e}tallurgiques Appliqu\textsuperscript{e}es, CEA-Saclay, 91191 Gif-sur-Yvette, France

\textsuperscript{2}Centre d’Elaboration de Mat\textsuperscript{e}riaux et d’\textit{d}tudes Structurales, CNRS UPR 8011, 29 rue J. Marvig, 31055 Toulouse, France

\texttt{fabien.onimus@cea.fr}

\textbf{Abstract}

A better understanding of the effect of irradiation on the mechanical properties of zirconium alloy is important in nuclear applications. While dislocations usually glide in the prismatic plane before irradiation, basal slip is observed after irradiation. Clear bands containing a low loop density are also reported (Onimus et al., 2004). Although these phenomena are a consequence of interactions between dislocations and irradiation loops, they are not yet clearly understood.

To tackle this problem, elementary interactions need to be well identified. In this talk, we report observations of interactions both during in-situ straining experiments in a transmission electron microscopy (TEM) on recrystallized Zircaloy-4 samples irradiated with Zr ions, and during dislocation dynamics (DD) simulations using the NUMODIS code. While in-situ TEM observations provide valuable information on the occurrence of such interactions and their dynamics in the real material, they fail in retrieving the details of the interactions at the loop scale (<20 nm) (Onimus et al., 2012). On the contrary, DD simulations are able to describe in detail the interactions at such scale but need inputs such as the dislocation mobility law and appropriate stress level (Drouet et al., 2014).

In order to bridge simulations and experiments, we have compared quantitatively the interaction of a dislocation gliding in a pyramidal plane at 500°C with a loop, with a DD simulation performed in the same geometrical and stress conditions. This comparison was made possible in particular by adjusting the mobility law with experimental data.

\begin{figure}[h]
\centering
\includegraphics[width=0.5\textwidth]{figure1.png}
\caption{Interaction between a gliding dislocation and a loop. a) The dislocation is very close to the loop, b) the loop is incorporated into the dislocation as a helicoidal turn.}
\end{figure}
We show that the observed interaction is in agreement with the interaction between a vacancy loop, forming an helicoidal turn expanding along the dislocation before being blocked below the sample surface. This work opens the way to massive DD simulations, provided that the identification of interaction mechanisms and the dislocation mobility laws in the different gliding plane and at different temperatures are retrieved from experiments.

References
Helium incorporation in nuclear glass studied by in-situ TEM analysis

Sylvain Peuget¹, Gaëlle Gutierrez¹, Jonathan Hinks², Graeme Greaves², Stephen Donnelly², Stéphanie Jublot-Leclerc³, Cédric Baumier³, Erwan Oliviero³, Christophe Jégou¹

¹ CEA, DEN, DTCD,SECM, Laboratoire d’Étude des Matériaux et Procédés Actif, 30207 Bagnols-sur-Cèze, France.
² Electron Microscopy and Materials Analysis Group, School of Computing and Engineering, University of Huddersfield, Huddersfield HD1 3DH, UK.
³ Centre de Spectrométrie Nucléaire et Spectrométrie de Masse, CNRS-IN2P3, Université Paris-Sud, Orsay Campus, France.

Sylvain.peuget@cea.fr

Abstract

High-level nuclear wastes resulting from the reprocessing of the spent nuclear fuel from PWR reactors are immobilized in a borosilicate matrix by vitrification. Research is carried out to predict the long-term behavior of the glass under disposal conditions. Helium will be generated from the α-decay of the minor actinides and at the same time the glass will be subjected to atomic displacements induced by the recoil nuclei. In order to predict the helium behavior under geological disposal conditions, the effects of helium build-up were investigated by examining SON68 glass specimens by TEM with in-situ He implantation experiments. The effect of temperature and glass damage was investigated by changing the implantation temperature, performing post implantation annealing and by using double beam irradiation experiments. Observations of implanted glass at 143 K indicate that a helium concentration of around 3at.% is required to nucleate a significant density of nanosized bubbles (Fig 1.). He bubble growth is observed for concentration higher than the number of host sites (>4at.%). These results highlight the large capacity of the glassy network for incorporating helium atoms. The effects of temperature and glass damage will also be discussed.

![Figure 1: TEM micrographs of SON68 glass implanted with 6 keV He ions at 143 K with fluences of: (a) 9; (b) 14; (c) 18 and (d) 23 × 10¹⁶ He.cm⁻². The arrows highlight bubble coalescence with increase of helium fluence. The dotted ellipses indicate the nucleation and growth of bubbles.](image-url)
Interface stability under irradiation in Oxide Dispersion Strengthened steel

J. Ribis¹, A. Gentils², M. Kirk³, Y. Serruys⁴, Y. de Carlan¹

¹ CEA, DEN, DMN, SRMA, F-91191 Gif sur Yvette
² CSNSM, Univ Paris-Sud & CNRS/IN2P3, Bâtiments 104-108, 91405 Orsay Campus, France
³ Materials Science Division, Argonne National Laboratory, Argonne, IL 60439, USA
⁴CEA, DEN, Service de Recherches de Métallurgie Physique, Laboratoire JANNUS 91191 Gif sur Yvette, France

Oxide Dispersion Strengthened (ODS) ferritic steels are considered as promising candidates for nuclear application as cladding tubes for GEN IV reactors. In such reactors, the irradiation damage can reach more than 150 dpa at temperatures ranging between 400°C and 650°C. Thus, the stability of nano-particles has to be guaranteed in order to ensure the materials’ excellent properties.

Under irradiation, where a forced dynamics produced by the external forcing is competing with a thermally activated dynamics, interfaces could be sustained far from equilibrium. In ODS steels, the nano-particles act as sinks for point defects, therefore the stability of their interface under irradiation is of great interest.

In situ ion irradiation was performed at CSNSM-JANNU Orsay to assess the interface stability. First, the orientation relationships between particles and matrix were identified out of irradiation using HRTEM. Cube-on-cube and cube-on-edge orientations are reported. These coherent misfitting interfacial configurations conduct the interfaces to grow under elastic stress effect at the origin of their smooth and flat morphology. After irradiation, same interfaces appear as destabilized since they display morphological changes or diffusenesses that are discussed in both terms of nonequilibrium roughening and faceting. From a practical and applied point of view, the modification of precipitate shapes under irradiation may alter their growth and even conduct to a coarsening saturation, able to modify the mechanical response of the irradiated alloy. Further, interface instability is also susceptible to influence the interaction between nano-particles and point defects. Using the IVEM facility (Argonne National Laboratory), the behaviour of the point defects in the close vicinity of incoherent, semicoherent and coherent interfaces is inferred by in situ observation of the Kr-stabilized cavities attributed to the condensation of vacancies in excess concentration.
The fuel behavior under ion irradiation investigated using JANNuS Orsay facility.

C. Sabathier¹, C. Onofri¹, C. Baumier², G. Martin¹, S. Maillard¹, C. Bachelet², H. Palancher¹, G. Carlot¹, M. Legros³

¹ CEA, DEN, DEC, F-13108 Saint Paul Lez Durance Cedex
² CSNSM, Univ Paris-Sud, CNRS/IN2P3, Université Paris-Saclay, F-91400 Orsay
³ CEMES-CNRS, Université de Toulouse, F-31055 Toulouse

catherine.sabathier@cea.fr

Abstract

Nuclear fission is the main energy generating process in the fuel of a nuclear power plant. During in-pile irradiation, the main modification in the fuel is generated by the very large quantity of fission products and by the defects created by the energy loss of fragment fissions along their paths within the fuel. In order to understand and predict the behaviour of nuclear fuels under irradiation, it is essential therefore to identify and to quantify the mechanisms that underpin the mobility, release and precipitation of gaseous or volatile fission products. To do this, separate effect studies are carried out using ion beams as surrogates for neutron irradiation.

This oral presentation focuses on the experimental observations of the nucleation mechanisms and the influence on the evolution of defects in the uranium oxide of various parameters such as fluence and irradiation temperature. Transmission electron microscopy (TEM) observations of UO₂ polycrystals irradiated in-situ with 4 MeV Au ions were performed in a large range of temperatures from liquid nitrogen temperature to 1000°C to better understand the mechanisms of nucleation. Experiments were carried out at the JANNuS Orsay facility that allows in-situ ion irradiations inside the microscope. The majority of 4 MeV gold ions were transmitted through the thin foil so that radiation defects were mainly investigated by TEM. Observations showed that nano-voids are formed (figure 1) occurs in the irradiation temperature range between the liquid nitrogen temperature until 1000°C in UO₂ thin foils irradiated with energetic heavy ions under an essentially nuclear energy loss regime. The diameter and density of nano-objects were measured as a function of the gold irradiation dose at different irradiation temperature [1]. Moreover, a similar nano-object population has previously been revealed after a Xe implantation performed at 390 keV at 600°C [2, 3]. The nano-object density induced by gold irradiations was compared with Cluster Dynamics simulations [4]. Experimental results were also compared with results obtained through Classical Molecular Dynamics (CMD) simulations [5]. The experimental and modelling results are in good agreement, which suggests a mechanism of heterogeneous nucleation induced by 10 keV cascade overlaps [5]. These results put in evidence mechanism controlled by radiation damage for the formation of nano-voids. These are then likely to be filled in UO₂ with insoluble fission products during reactor operation.

In addition, TEM examinations of the distribution (size and density) of dislocation loops and lines were carried out using a 4 MeV gold irradiation performed at three irradiation temperatures: liquid nitrogen, room temperature and 600°C as a function of fluence. For the irradiation performed at the liquid nitrogen temperature, the TEM observations coupled with the CMD simulations highlight a stage of nucleation of small dislocation loops which probably grow by displacement cascade overlaps up to a critical size (between 3 and 10 nm). The low temperature prevents the migration of dislocation loops and point defects, so the loop density increases and the loop size reaches a constant value with increasing fluence. In the same fluence range, for the irradiation performed at 600°C, the loop density saturates and the loop size increases until 20 nm. The loop size-distribution moves towards the larger loops with increasing fluence. The medium-sized loops disappear at the expense of the larger loops. This is typical of a coarsening mechanism (coalescence and/or Oswald ripening). Furthermore, the
temperature of 600°C and the irradiation enhanced diffusion allow defects and extended defects to move. A continuous nucleation of loops explains the saturation of the loop density and of the proportion of the smallest loops. At a given fluence the loop density is higher for the irradiation performed at 600°C than for the irradiation performed at the liquid nitrogen temperature, which is consistent with CMD simulations [6], which predict that the fraction of clustered interstitials increases with temperature.

For the irradiation performed at liquid nitrogen temperature, at a given fluence, the microstructure exhibits dislocation lines. They are likely formed by the interaction of closed dislocation loops; hence when the loop density is very high. This transformation seems to be due when the small dislocation loops overlap in the volume of the thin foil. The calculated fluence is in good agreement with TEM observations. For the irradiation performed at 600°C, at a 3 or 4 times lower fluence than for the irradiation performed at liquid nitrogen, the TEM observations reveal the transformation of the largest loops into lines. At 600°C, this transformation takes place using the overlap mechanism of dislocation loops. Finally, it seems that with increasing fluence, the final microstructure exhibits only dislocation lines and small dislocation loops (diameter around 5 nm) whatever the temperature of the irradiation. In this case, the temperature irradiation has only an influence on the kinetic of the defect development.

A part of this study was supported by AREVA and EDF in the framework of the ICOMB project.

References

Figure 1: Bright field images of polycrystalline UO2 thin foil irradiated with 4 MeV Au at 600°C at a) $3 \times 10^{14}$ Au/cm$^2$ in over-focused electron beam conditions and b) at $3 \times 10^{19}$ Au/cm$^2$ with a g= 220 reflection with the presence of dislocation loops.
Impact of H & He in irradiated UHP Fe(Cr) by TEM in situ irradiation

R. Schäublin¹, B. Décamps², M. J. Aliaga³, M. J. Caturla³, J. Löffler¹

¹Laboratory of Metal Physics and Technology, Department of Materials, ETH Zurich, 8093 Zurich, Switzerland
²CSNSM, Univ. Paris-Sud, CNRS/IN2P3, Université Paris-Saclay, 91405 Orsay, France
³Dept. Física Aplicada, Facultad de Ciencias, Fase II, Universidad de Alicante, Alicante E-03690, Spain

robin.schaeublin@mat.ethz.ch

Abstract

Reduced activation ferritic/martensitic steels are amongst the most promising candidates for the future fusion reactor structural components that will suffer high heat loads and 14 MeV neutron irradiation doses. In order to reach a thorough understanding of their response to these harsh operation conditions, ultrahigh purity Fe(Cr) alloys are considered for testing. Although many investigations exist published results are somewhat controversial, e.g. on the <100> / ½<111> dislocation loop ratio [1, 2], due to the fact that these are usually obtained by TEM performed on thin foil and it is known that the presence of the free surfaces impact the microstructure due to the so-called ‘image forces’ [3, 4].

Moreover, the impact of helium and hydrogen is considered, for in the fusion irradiation condition large quantities of these gases are expected, namely 10 appm/dpa for He and 40 appm/dpa for H. Until now only the impact of He has been considered, because hydrogen as a fast diffusing atomic species [5] is considered to escape easily to free surface or internal interfaces. Therefore, as mentioned in [6]: ‘Hydrogen embrittlement is most probably negligible at high temperatures because of the high diffusivities of hydrogen in iron- and nickel-based alloys. From the few reported results at low temperatures, the importance of hydrogen effects on the mechanical properties under irradiation or synergistic effects of hydrogen and helium and/or irradiation damage cannot yet be judged decisively’. We have thus undertaken the study of the impact of He and H in UHP Fe(Cr) alloys, using mainly TEM in situ irradiation experiments performed at Orsay, and compared to previous experiments performed ex situ at Saclay [7].

TEM in situ experiments were performed in situ on the JANNuS platform in Orsay with the following irradiation conditions. The displacement damage was performed using 500 keV Fe⁺ ions to a dose of 1 dpa, the irradiation temperature was room temperature, without or without He or H, implanted as 10 keV He⁺ ions to a dose of 1000 appm·dpa⁻¹ or 10 keV H⁺ to a dose of 1000 appm·dpa⁻¹ and 4000 appm·dpa⁻¹. The irradiated material was UHP Fe and UHP Fe(5Cr). The highest concentration of H at 4000 appm·dpa⁻¹, 4 times higher than the content of He, allows to reach the same ratio of 4:1 expected in the fusion reactor first wall. Fe ions were implanted with ARAMIS accelerator while He and H were implanted with IRMA accelerator.

Figure 1 shows that H implanted to a dose of 4000 appm·dpa⁻¹ in UHP Fe has a large impact on the dislocation loop population. In effect, the dislocation loops’ size is visible much larger (Fig. 1a) than the one observed in UHP Fe irradiated without H (Fig. 1b). The number density seems also larger but one should be cautious and consider that in bright field TEM spatial resolution might not be sufficient to reveal the smaller dislocation loops.
Figure 1: TEM bright field image showing irradiation induced dislocation loops in UHP Fe irradiated at RT to 1 dpa with 500 keV Fe⁺ (a) without He or H and (b) together with 4000 appm·dpa⁻¹ H implanted as 10 keV H⁺.

The statistical g·b analysis method [2] was applied in all cases to determine the number densities and the Burgers vector of the dislocation loops. Results will be presented here. These results will serve modelling approaches by providing clean test cases that can be used for validation.

Acknowledgements: experiments done at JANNuS (Joint Accelerators for Nanoscience and Nuclear Simulation) Orsay at CSNSM (Orsay, France), which are part of the EMIR French accelerators network. The authors acknowledge the outstanding work of the JANNuS Orsay team. Work supported by EUROfusion.

References
The New Setup at the Michigan Ion Beam Laboratory to Connect Two Beamlines to a TEM

O. Toader, L. Wang, F. Naab, and T. Kubley

University of Michigan, Ann Arbor MI USA

Michigan Ion Beam Laboratory (MIBL) at the University of Michigan in Ann Arbor Michigan, USA, has recently added the capability to simultaneously run multiple ion beams. The laboratory, equipped with a 3 MV Tandem, a 400 kV Ion Implanter and a 1.7 MV Tandem has also increased the number of beamlines from three to six with two more in the planning stages. Multiple simultaneous ion beam experiments are already in progress and scientists conduct state of the art experiments involving protons and heavy ions. In parallel, we are in planning stages to connect two of the accelerators with a new TEM that will be installed in the spring of 2016. The talk will focus on the details of this installation.
TEM in-situ observations of irradiation damage in boron carbide

G. Victor¹, Y. Pidon¹, N. Moncoffre¹, N. Bérerd¹, C. Esnouf², T. Douillard², A. Gentils³

¹ Université de Lyon, UCBL, Institut de Physique Nucléaire de Lyon, Villeurbanne, France
² Université of Lyon, INSA de Lyon, MATEIS UMR CNRS 5510, Villeurbanne, France
³ CSNSM, Université Paris-Sud, CNRS/IN2P3, Université Paris-Saclay, Orsay, France

Since 2000, and from a project launched by the United States, the Generation IV International Forum (GIF) started researches on new concepts of nuclear reactors. These concepts have to fill new features, especially to (i) enhance the safety, (ii) to increase the fuel exploitability and (iii) to raise the competitiveness of nuclear reactors. Six different reactor concepts were chosen. Among them, the Sodium Fast Reactor (SFR) is one of the most promising. In France, the experience on this technology is already important, since three SFR reactors were built and commissioned in the past decades: RAPSODIE (1967-1983), PHENIX (1973-2009) and SUPERPHENIX (1985–1997). Therefore, a prototype called ASTRID, for Advanced Sodium Technological Reactor for Industrial Demonstration, could be constructed in the next years. This prototype will have to take account of all the criteria mandatory for a fourth generation reactor. For that, researches on new core, cladding and absorber materials presenting high temperature and irradiation resistance properties are necessary.

The main objective of this study is to understand the irradiation behavior of boron carbide (B₄C). Boron carbide is a well-known material used as neutron absorber in several reactors (Pressurized Water Reactor and Sodium Fast Reactor) and one of the most promising for the SFR reactors. However, despite the fact that boron carbide is widely used, its behavior under irradiation is still not fully understood, due to its complex crystallographic structure (a rhombohedral unit cell that contains one 12-atoms icosahedral unit and one linear 3-atoms chain).

This work focuses specially on the effects of ballistic damage on the structural modifications of B₄C. In fact, in reactor, the neutrons mainly interact with the material atoms by elastic collisions, responsible of atomic displacement cascades in the structure. To simulate these collisions, we chose to use 1 MeV Au⁺ ions, corresponding to a penetration depth of approximately 190 nm, and to irradiate at room temperature until a maximum fluence of 4x10¹⁵ at.cm⁻² (~6 dpa). The irradiations were made with the ARAMIS accelerator at the JANNuS-Orsay facility at CSNSM. They were coupled to TEM in-situ observations.

At first, boron carbide samples were sintered at the SPCTS Limoges with the collaboration of A. Maitre, N. Pradeilles and O. Rapaud. Then, thin lamella of boron carbide were prepared at the CLYM¹ (INSA Lyon) using the Focus ion beam (FIB) technique. Four lamella were obtained with different thicknesses (t₁₁ ≈ 210 nm; t₁₃ ≈ 130 nm; t₁₄ ≈ 80 nm; t₁₅ ≈ 190 nm). Moreover, an Electron Backscattered Diffraction (EBSD) analysis was made to determine the grain orientations in the different samples. The figure 1 shows a boron carbide thin lamella observed by SEM (left) and by EBSD (right). The different colors correspond to different grain orientations. We chose to observe one or several grain by samples, with different orientations. Notice that the Au irradiation covers the whole FIB lamella.

¹ Centre Lyonnais de Microscopie (FED 4092 CNRS)
At the beginning of the irradiations at room temperature, we observed a spreading of the diffraction fringes (bending contrast) showing a local curvature of the sample due to an important stress induced in the material. It stabilizes rapidly before the appearance of “black dots” in the lamella at a fluence of approximately $1.2 \times 10^{14}$ at.cm$^{-2}$ (~0.2 dpa). These “black dots” tend to grow with the irradiation until we observe larger stress field contrasts (~20 nm in size) in the material (observe at a fluence of $1 \times 10^{15}$ at.cm$^{-2}$, or 1.6 dpa). The last stage observed is the beginning of the amorphisation of the material around a fluence of $1.3 \times 10^{15}$ at.cm$^{-2}$ (2 dpa). We also observed that crystallographic orientations have no effect on the defect formation or amorphisation of the material whatever is the fluence. Finally, a very interesting result is that, depending on the lamella thickness and/or the electron illumination, a different amorphisation threshold is observed (figure 2).

The authors wish to thanks the NEEDS project for financial support and D. Gosset for his collaboration in this project. The JANNuS-Orsay technical staff is also acknowledged for his assistance during ion irradiation.
Nano-size metallic oxide particle synthesis in Fe-Cr alloys by ion implantation

Ce Zheng¹*, Aurélie Gentils¹, Joël Ribis², Vladimir A. Borodin³,⁴

¹ CSNSM, Univ Paris-Sud, CNRS/IN2P3, Université Paris-Saclay, Orsay, France
² CEA, DEN, DMN, SRMA, Saclay, France
³ NRC Kurchatov Institute, Moscow, Russia
⁴ NRNU MEPhI, Moscow, Russia

* Now working at University of South Carolina, USA (contact: steven524@126.com)

Abstract

ODS (Oxide Dispersed Strengthened) steels reinforced with metal dispersions of nano-oxides (based on Y, Ti and O elements) are promising structural materials for future nuclear reactors. The detailed understanding of the mechanisms involved in the precipitation of these nano-oxides would improve manufacturing and mechanical properties of these ODS steels, with a strong economic impact for their industrialization. A perfect tool to experimentally study these mechanisms is ion implantation, where the various parameters of precipitate synthesis are under control. This report demonstrates the feasibility and advantages of the ion beam synthesis approach as applied to aluminium oxide particle synthesis in Fe-Cr model alloys [1].

High purity Fe-10Cr alloys were implanted with Al and O ions at room temperature and demonstrated quite a number of interesting and unexpected results. For example, transmission electron microscopy observations demonstrated that the nano-oxides had appeared in the Fe-10Cr matrix already upon ion implantation at room temperature. This is very unusual because the standard ion beam synthesis technique requires an additional subsequent high-temperature annealing in order to grow the precipitates from implanted impurity atoms. The nucleation of these nanoparticles, of a few nm in diameter, indicates the mobility of implanted elements already at room temperature, which is evidently promoted by point defects created during ion implantation. The observed nanoparticles were composed of aluminium and oxygen, but additionally included one of the matrix elements (chromium). This indicates that the major steel constituents can play very different roles in the nanooxide synthesis. Another unusual feature revealed by high-resolution TEM experiments [2] is a non-equilibrium crystallographic structure of aluminium oxide compound (cubic γ-Al₂O₃ type rather than more common corundum). Some other observations strongly contributing to the understanding of the underlying mechanisms of nanoparticle nucleation and growth will be discussed.

A mechanism involving the precipitation of nano-oxide dispersed in ODS alloys is proposed in this presentation based on the obtained experimental results and the existing literature data. The results on model alloys are fully applicable to industrial materials. Indeed, ion implantation nicely reproduces many key aspects of the microstructural processes involved in conventional powder metallurgy based technique of ODS steel production.

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