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Plasma–Wall Interaction for Irradiated Tungsten and Tungsten Alloys in Fusion Devices

Summary Report of the Second Research Coordination Meeting

Seoul National University, Seoul, Republic of Korea

8–11 September 2015

Report prepared by

H.-K. Chung and B. J. Braams

November 2016

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ABSTRACT

The Second Research Coordination Meeting of the IAEA Coordinated Research Project on Plasma–Wall Interaction for Irradiated Tungsten and Tungsten Alloys in Fusion Devices was held in Seoul, Republic of Korea, hosted by the Nuclear Engineering Department of Seoul National University. Eighteen projects participating in the CRP were represented together with one guest project and IAEA staff. The participants reviewed their work over the first two years of the CRP and made plans for coordinated activities, especially in the areas of damage characterization and thermal desorption spectroscopy. The proceedings of the meeting are summarized in this report.

November 2016

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INTRODUCTION

Coordinated Research Projects (CRP) are one of the main mechanisms by which the IAEA supports applied research including development of atomic and nuclear data. A CRP on Plasma-Wall Interaction with Irradiated Tungsten and Tungsten Alloys in Fusion Devices was established to improve the knowledge base and databases on properties of irradiated tungsten. The most important topic in this CRP is to understand how tritium retention, tritium migration and ways to extract trapped tritium are affected by radiation damage. Neutron irradiation would be of most interest, but experiments are usually done with surrogate irradiation by charged particle beams (H, D, T, He, Fe, W). This leads to the need to characterize the effects upon microstructure due to different kinds of irradiation and to characterize how changes in microstructure influence the tritium retention and tritium transport properties.

The Second Research Coordination Meeting (RCM) of this CRP was held on the days Tuesday–Friday 8-11 September 2015 at Seoul National University (SNU), hosted by the Republic of Korea through the SNU Nuclear Engineering Department. The local scientific hosts were Professor Gon-Ho Kim and Professor Takuji Oda of the Nuclear Engineering Department. SNU provided excellent meeting facilities and generous support for a social programme.

The meeting featured progress reports from 18 of the 19 projects in the CRP and one guest project. (The CRP project at Kurchatov Institute contributed a written summary but was not represented, while the Department of Nuclear and Quantum Engineering at KAIST presented as a guest.) In addition the meeting featured extended topical discussions on damage and characterization of microstructure, fundamental modelling, benchmarks and common protocol for temperature programmed desorption (thermal desorption spectroscopy; TPD/TDS) experiments, and a code comparison project for TPD/TDS.

The proceedings of the meeting are summarized in Section 2 and the discussions are summarized in Section 3. Work plans of each group are summarized in Section 4. The list of participants is in Appendix 1 and the meeting agenda is given in Appendix 2. Summaries of presentations are presented in Appendix 3.

PROCEEDINGS

The second research coordination meeting of the CRP Plasma-Wall Interaction for Irradiated Tungsten and Tungsten Alloys in Fusion Devices started with the welcome address from Professor Yong-Seok Hwang, the director of CARFRE (Center for Advance Research in Fusion Reactor Engineering), Seoul National University.

It was followed by introduction of participants and a presentation on meeting objectives of the scientific secretary Bastiaan Braams. The agenda was adopted. The meeting proceeded with presentations by participants on their group research activities.

Mizuki Sakamoto from Plasma Research Center, University of Tsukuba reported on Hydrogen isotope retention in tungsten irradiated by heavy ions and helium plasma. Wolfgang Jacob, IPP Garching reported on the influence of radiation damage on deuterium retention in tungsten done. Guang-Nan Luo, ASIPP, Hefei reviewed preliminary results of commissioning of EAST full tungsten upper divertor and deuterium retention and permeation of W irradiated by heavy ions. Heun Tae Lee, Osaka University presented current status of activities in Japan on hydrogen isotope retention studies for neutron-irradiated tungsten materials. Sabina Markelj, Josef Stefan Institute, Ljubljana reported on the deuterium retention and isotope exchange studies in self-ion damaged tungsten exposed to neutral atoms. Yury Gasparyan, National Research Nuclear University "MEPhI" presented the studies of determination of binding energies for hydrogen with radiation defects in tungsten by means of TDS: theory and experimental data. Christian Grisolia, CEA Cadarache reviewed the study of tritium implantation in tungsten based fusion materials.

The second day proceeded with more presentations: Changsong Liu, ISSP-CAS, Hefei reported on retention and diffusion of hydrogen in tungsten: effects of temperature, alloying elements and vacancy clusters. Sergei Dudarev, CCFE, Abingdon presented on the modelling radiation effects in tungsten: from micro- to meso-scales. Takuji Oda, Seoul National University: Assessment and modeling of trap effects on hydrogen diffusivity in tungsten. Hong-Bo Zhou, Beihang University also reported on the modeling and simulation of helium and hydrogen behaviours in tungsten. Shishir Deshpande, IPR, Gandhinagar presented computer simulation of ion-irradiation of tungsten: preliminary results. Christian Grisola presented work of Charlotte Becquart, University of Lille-1, and Marie-France Barthe, CNRS on the topic of modelling of trapping/detrapping of hydrogen isotopes in W material. Davide Curreli presented work of Brian D. Wirth, University of Tennessee, preliminary results from a multiscale approach to modelling plasma-surface interactions involving tungsten. Masashi Shimada, Idaho National Laboratory presented status update on MERCOR-TMAP integration, Tritium Plasma Experiment Modification, and PHENIX program for PMI in irradiated tungsten.

On the third day, P. M. Raole, IPR, Gandhinagar, reported on tadiation damage of heavy ions and H irradiated tungsten - some experimental results. Jochen Linke, Forschungszentrum Juelich reviewed radiation induced degradation of tungsten under thermal and plasma exposure and development of advanced tungsten materials. Davide Curelli presented the work of UIUC (headed by Jean Paul Allain and Anton Neff) on plasma-material interactions under high heat flux conditions and synergistic D/He effects on low Z covered tungsten surfaces. Ho Jin RYU, Nuclear and Quantum Engineering, KAIST: presented work on tungsten-based high entropy alloys and Sergei Dudarev, CCFE, Abingdon reviewed DPA definition and estimates. Summaries of presentations are presented in the Appendix 3.

After all presentations, reviews of experiences with shared materials, discussions about common protol for TDS experiments and discussion about fundamental modelling continued. On the last day,

participants discussed the benchmark experiments and code comparisons for TDS experiments. Discussions are summarized in the Section 3.

DISCUSSIONS

DPA definition and estimates

The concept of "displacements per atom" (DPA) as a characterization of radiation damage came up many times throughout the meeting and on Thursday S. Dudarev briefly reviewed some issues. (The presentation of Dr Dudarev is available on the meeting web page with all other presentations.) The standard DPA measure goes back to a 1975 paper by M. J. Norgett, M. T. Robinson and I. M. Torrens, hence the acronym NRT-DPA. Using an extremely simple analytical model the NRT-DPA characterizes the expected number of defects produced by an incident neutron or ion as a function of the energy available for damage production, i.e. the total energy of the incident particle minus the energy lost due to electronic stopping. The model uses a single free parameter that depends on the target material, the threshold displacement energy.

Detailed MD simulations show that the NRT-DPA overestimates damage production, typically by a factor of about 3. Therefore, proper use of NRT-DPA is as a conventional linear measure of radiation exposure, not as an estimate of number of Frenkel pairs.

More pointed criticism of NRT-DPA is that its calculation is often obscure. It may be obtained as an output of a SRIM simulation of radiation exposure, but the source code for SRIM is not publicly available and the DPA measure that is produced may depend on which version of SRIM is being used.

Benchmark and common protocol for TPD/TDS experiments

Thermal desorption spectroscopy (TDS) or temperature programmed desorption (TPD) are widely used to characterize binding energies of hydrogen or helium in materials. The experiments are relatively simple to implement, but the interpretation of the data is not straightforward due to free parameters and due to several assumptions used in fitting or modeling. On Thursday afternoon the CRP participants discussed ideas about intercomparison of TPD/TDS data and their interpretations among various laboratories. There is agreement among the participants to define some reference experiments and carry out a comparison of the TPD/TDS analysis in the various labs with their various TPD/TDS devices. The experiments should use common samples; samples prepared and exposed to deuterium at one location under controlled conditions and distributed from there to the participating laboratories. The discussions concerned the practicalities of such a comparison exercise and a summary was prepared by H.-T. Lee during the CRP meeting.

Participants in the exercise will perform benchmark experiment in various TPD/TDS devices using samples that were prepared in one location under controlled conditions. The exercise is meant to build confidence and trust in the reproducibility of TPD/TDS results across different laboratories, to compare and evaluate each lab's methods, to identify any systematic errors, to compare and evaluate the performance of different systems, and to create a better basis for common discussion of past and future results. The exercise should start soon and it would be preferable if it lasts no longer than 6 months. (Note in November 2016: samples have now been distributed and the exercise is in progress.)

The following labs from within the CRP intend to participate.

- IPP, Garching, Germany (W. Jacob)

- INL, USA (M. Shimada)
- MEPhI, Russia (Yu. Gasparyan)
- IPP-CAS, China (G.-N. Luo)
- US SciDAC team (D. Curreli)
- Tsukuba University, Japan (M. Sakamoto)
- Aix-Marseille University, France (C. Grisolia)
- Beihang University, China (G.-H. Lu)
- FZ Jülich, Germany (J. Linke)
- Seoul National University, South Korea (T. Oda)
- Toyama University, Japan (Y. Hatano)
- Osaka University, Japan (H. T. Lee)

Also Toronto University, Canada (J. W. Davis) may participate and there can be others.

There should be a single source for the samples (one lab to prepare the standard tungsten material and the standard deuterium exposure) and we wish to have two or three samples for each participating lab. Therefore 24~36 samples need to be prepared. Possible W sources are Plansee (EU), Allied Material (Japan) or recrystallized tungsten. Deuterium loading can be done using a low temperature ECR plasma as shown in the figure below. Participants should use common agreed parameters:

- Ramping rate (1 K/s?)
- Measurement of m/q = 3,4 (2, 18, 19, 20?)
- Delay time between implantation and start of TPD/TDS (<1 month?)
- Sample storage upon reception (dessicator?)
- No surface treatment

Other than the above, each participant's lab should perform TPD/TDS according to their standard procedures and data analysis, e.g. Qmass settings, calibration leaks, etc.. The experimental parameters are to be discussed still: Sample dimensions (10×10×0.8 mm?), min/max ramping rates (0.1-10 K/s), and maximum temperature (~1000 C enough).

Follow-up to the TDS/TPD comparison discussions

A lunch-time meeting was organized by H.T. Lee, W. Jacob and H.-K. Chung on 2 June 2016 at the 22nd International Conference on Plasma Surface Interactions in Controlled Fusion Devices (PSI 2016) in Rome, Italy, with participants from 14 groups represented in the CRP and one additional group (University of Toronto). It was agreed there that the samples would be prepared at IPP Garching and the sample parameters and exposure conditions were specified. The TDS/TPD experimental conditions were discussed and the final specifications will be agreed over email coordinated by H.-T. Lee.It is agreed that this exercise should be concluded before the third meeting of the CRP. A summary report of this discussion at PSI 2016 in Rome is available on the CRP web page.

TDS/TPD simulations comparison exercise

Related to the discussion about a comparison of TDS/TPD experiments the CRP participants discussed a comparison exercise focussed on simulation results from different codes of one experimental thermal desorption case. The most widely used code for this purpose is probably the TMAP code in one of its variations, but other codes are used too. The test case for comparison could involve a foreward or a backward calculation. In either case the experimental protocol is specified. For the foreward calculation one specifies the material conditions and one is asked to simulate the TDS spectrum, while for the backward calculation the TDS spectrum is given and one is asked to interpret it. The options were discussed and M. Shimada provided a summary.

Possible participant groups and their contacts:

- INL (POC: M.Shimada, Codes: TMAP4, TMAP7)
- MEPhI (POC: Yuri Gasparyan)
- CEA and CNRS (POC: Christian Grisolia)
- US SciDAC team (POC: Davide Curreli)
- IPP Garching (POC: Wolfgang Jacob)
- SNU (POC: Takuji Oda)
- ISSP-CAS (POC: Changsong Liu)

This code comparison would be done after the experimental benchmark comparison is completed. The objective of the exercise is to understand differences between different simulations. It is not meant to evaluate codes or to recommend a best code. The anticipated procedure is as follows.

1) One experimental TDS spectrum dataset (e.g. time[s], temp.[K], HD [mole/s], D2[mole/s]) from the experimental comparison exercise will be selected.

2) Modeling geometry (thickness, 1D), deuterium mass transport properties (diffusivity, solubility, recombination coefficient, dissociation coefficient), boundary condition (e.g. c(x=0) equal to 0 or something else), pre-exponential factor of de-trapping and the number of traps to use will be selected. Also the experimental measured deuterium depth profile (via NRA) will be provided by Wolfgang Jacob (IPP-Garching) later.

3) Participants will model the provided experimental TDS dataset with their code(s), hopefully with one fitting parameter for the de-trapping energy.

4) The modeling results will be shared to all the participants, and the differences/similarities will be discussed.

We note relevant earlier related work; the ITPA Div/SOL expert group performed similar benchmarking modeling in 2008-2010 involving just two codes. Fluence dependence on T retention and T permeation were compared as calculated using the TMAP/DIFFUSE (INL/SNL) and WW (MIT) codes independently. The results of this comparison are in an MIT report: [B. Lipshultz et al., "An Assessment of the Current Data Affecting Tritium Retention and its Use to Project Towards T Retention in ITER", Report PSFC/RR-10-4, MIT, April 2010].

Fundamental Modeling

The discussions on Thursday concluded with a review of problems with fundamental modelling, i.e. the electronics structure calculations and development of interaction potentials and rate coefficients based on electronic structure. These discussions were reviewed by T. Oda on Friday morning.

T. Oda reminded us of a fundamental experimental work: [K.-D. Rasch, R. W. Siegel and H. Schultz: "Quenching and recovery investigation of vacancies in tungsten," Philosophical Magazine A, 1980, 41, 91-117]. These kind of data serve as a challenge for theory.

As a guide to the required scale of calculations, participants in the discussions more or less agreed that DFT calculation with around 1000 atoms would be adequate to evaluate hydrogen trap energies (as well as rate constant of trap and detrap processes) of most basic defects including vacancy cluster and simple dislocations with an accuracy required to explain TDE experiments. Calculations on this scale will be available soon, and probably sooner than the time needed to develop a potential model that can give sufficiently accurate results.

For evolution of much larger defects including surface morphology change by plasma/radiation, however, molecular dynamics should be needed. For this, a quality potential model for W-He-H system is vital, which is not currently available. In CRP participants, there are 3 ongoing work for potential model development.

- BOP potential model by Dr. Wirth (just started).
- EAM potential model by Drs. Lu and Zhou (would be completed in this year; better performance than existing BOP models.)
- GAP potential model by Drs. Gabor Csanyi, Duc Nguyen, Oda. (Trying to complete in the beginning of 2016.)

It is also desired to predict defect structures formed by 14 MeV neutron irradiation by computer simulation. For this, we need to appropriately consider non-equilibrium evolution of materials and defects.

Not only hydrogen inventory, but also hydrogen effects on mechanical property and morphology of tungsten needs to be more studied.

There are still gaps between methods: b/w MC-KMC, KMC-MD, MC-DFT. To achieve a comprehensive idea on what will be going on fusion reactors, more research are needed to reduce the gap: for example, an improved binary-collision MC code that can give not only DPA but also the number and space distributions of defect clusters.

Verification and validation were discussed.

Verification: to check whether a physical concept is appropriately reflected in the code. For complicated cases, we have no analytical result. What we usually do is to use more accurate method; for example, verify rate-equation model with MD or with KMC.

Validation: to check whether the code can simulate something practically important, e.g. TDS spectrum, tritium inventory, etc. For validation the first step should be with a simple case. For example, “TDE spectrum with a mono vacancy”, “TDE with (predominantly) grain boundaries”, “TDE with dislocations”, etc.. Then, with simple cases, we determine model parameters (e.g. activation energy and pre-exponential factor). With fixed parameters, we apply the code to complicated cases.

Damage and characterization

On Friday there was a discussion about ways to produce and (especially) characterize damage. Damage can be produced by neutron sources or by other means. The most desirable way to produce damage is to use a 14-MeV neutron source and such sources will be at INEST-HF-CAS, which will have an ion beam in 2016 and neutrons in 2017, and a JAEA source that is moving from Tokai to Rokkasho. The most important fission neutron source for our studies is in the US-JP Titan/Phoenix project. A fission neutron source in under planning in China possibly as a national project. In the EU beryllium studies have priority now, but there should soon be a Eurofusion DEMO project with interest in fission-n/spallation-n studies of damage in tungsten. Other ways to mimic neutron irradiation include an annealing quench; this generates large amount of vacancies near melting and they are preserved during quench and form clusters. (It needs to be clarified how to achieve an annealing quench for W, which has a melting point over 3000^0C.) Electron irradiation generates mainly vacancies. Irradiation by a proton beam or W self-implantation can produce copious damage and is used for that purpose; of course, the damage is not the same as with neutrons. Finally damage can be produced by mechanical deformation.

In order to characterize damage the important tools are Positron Annihilation Spectroscopy (PAS), Thermal Desorption Spectroscopy (TDS), Transmission Electron Microscopy (TEM), X-ray diffraction (XRD), among others. XRD can provide a stress measurement and identify a phase transformation; its use needs further work. Finally it is a major research programme to inventorize and to characterize in a systematic way the damages generated by different ways.

Appendix 1

List of Participants

Yong-Seok Hwang, CAFRE, Seoul National University, Seoul, Republic of Korea

Gon-Ho Kim, Seoul National University, Seoul, Republic of Korea

Takuji Oda, Seoul National University, Seoul, Republic of Korea

Mizuki Sakamoto, Plasma Research Center, University of Tsukuba, Japan

Wolfgang Jacob, Max-Planck-Institut für Plasmaphysik Garching, Germany

Guang-Nan Luo, Institute of Plasma Physics, Chinese Academy of Sciences, Hefei, People's Republic of China

Heun Tae Lee, Osaka University, Osaka, Japan

Sabina Markelj, Josef Stefan Institute, Ljubljana, Slovenia

Yury Gasparyan, National Research Nuclear University "MEPhI", Moscow, Russian Federation

Christian Grisolia, Commissariat a l'Énergie Atomique, Cadarache, France

Changsong Liu, Institute of Solid State Physics, Chinese Academy of Sciences, Hefei, People's Republic of China

Sergei Dudarev, Culham Centre for Fusion Energy, Abingdon, United Kingdom

Hong-Bo Zhou, Beihang University, Beijing, People's Republic of China

Shishir Deshpande, Institute for Plasma Research, Gandhinagar, India

Davide Curreli, University of Illinois at Urbana–Champaign, Urbana IL, United States of America

Masashi Shimada, Fusion Safety Program, Idaho National Laboratory, Idaho Falls, ID, United States of America

Prakash M. Raole, Institute of Plasma Research, Gandhinagar, India

Jochen Linke, Forschungszentrum Jülich, Germany

Ho Jin Ryu, Nuclear and Quantum Engineering, Korea Advanced Institute of Science and Technology (KAIST), Republic of Korea

Bastiaan J. Braams, International Atomic Energy Agency, Vienna, Austria

Hyun-Kyung Chung, International Atomic Energy Agency, Vienna, Austria

Appendix 2

Agenda

Seoul National University, Seoul, Republic of Korea, 8-11 September 2015

Global Convention Plaza, Building 38 - Room 515 (5th floor)

Tuesday, 08 September

09:15–09:45 **Prof Yong-Seok Hwang, Prof Gon-Ho Kim, Prof Takuji Oda, Seoul National University**: Welcome. **Bas Braams, IAEA**: Introduction of participants, review of meeting objectives and meeting agenda.

09:45–10:30 **Mizuki Sakamoto, Plasma Research Center, University of Tsukuba**: Hydrogen isotope retention in tungsten irradiated by heavy ions and helium plasma.

10:30–11:00 **Break**.

11:00–11:45 **Wolfgang Jacob, IPP Garching**: The influence of radiation damage on deuterium retention in tungsten: IPP activities.

11:45–12:30 **Guang-Nan Luo, ASIPP, Hefei**: Preliminary results of commissioning of EAST full tungsten upper divertor and deuterium retention and permeation of W irradiated by heavy ions.

12:30–14:00 **Lunch**

14:00–14:45 **Yuji Hatano, University of Toyama (presented by Heun Tae Lee, Osaka University)**: Present status of activities in Japan on hydrogen isotope retention studies for neutron-irradiated tungsten materials.

14:45–15:30 **Sabina Markelj, Josef Stefan Institute, Ljubljana**: Deuterium retention and isotope exchange studies in self-ion damaged tungsten exposed to neutral atoms.

15:30–16:00 **Break**

16:00–16:45 **Yury Gasparyan, National Research Nuclear University "MEPhI", Moscow**: Determination of binding energies for hydrogen with radiation defects in tungsten by means of TDS: theory and experimental data.

16:45–17:30 **Christian Grisolia, CEA Cadarache**: Study of tritium implantation in tungsten based fusion materials.

19:00 **Social Dinner in the Hoam Faculty House**

Wednesday, 09 September

09:00–09:45 **Changsong Liu, ISSP-CAS, Hefei**: Retention and diffusion of hydrogen in tungsten: effects of temperature, alloying elements and vacancy clusters.

09:45–10:30 **Sergei Dudarev, CCFE, Abingdon**: Modelling radiation effects in tungsten: from micro- to meso-scale.

10:30–10:45 **Break**.

10:45–11:30 **Takuji Oda, Seoul National University**: Assessment and modeling of trap effects on hydrogen diffusivity in tungsten.

11:30–12:15 **Hong-Bo Zhou, Beihang University, Beijing**: Modeling and simulation of helium and hydrogen behaviours in tungsten.

12:15–12:45 **Shishir Deshpande, IPR, Gandhinagar**: Computer simulation of ion-irradiation of tungsten: preliminary results.

12:45–14:00 **Lunch**.

14:00–14:45 **Christian Grisolia, CEA Cadarache (for Charlotte Becquart and Marie-France Barthe)**: Modelling of trapping/detrapping of hydrogen isotopes in W material.

14:45–15:30 **Davide Curreli, UIUC (for Brian Wirth)**: Preliminary results from a multiscale approach to modelling plasma-surface interactions involving tungsten.

15:30–16:15 **Masashi Shimada, Idaho National Laboratory**: Status update on MERCOR-TMAP integration, Tritium Plasma Experiment Modification, and PHENIX program for PMI in irradiated tungsten.

17:30 **Departure from Hoam for Dinner Banquet**

Thursday, 10 September

09:00–09:45 **P. M. Raole, IPR, Gandhinagar**: Radiation damage of heavy ions and H-irradiated tungsten - some experimental results.

09:45–10:30 **Jochen Linke, Forschungszentrum Jülich**: Radiation induced degradation of tungsten under thermal and plasma exposure and development of advanced tungsten materials.

10:30–11:00 **Break**

11:00–11:45 **Davide Curreli, UIUC (for Jean Paul Allain and Anton Neff)**: Plasma-material interactions under high heat flux conditions and synergistic D/He effects on low Z covered tungsten surfaces.

11:45–12:30 **Ho Jin Ryu, Nuclear and Quantum Engineering, KAIST**: Tungsten-based high entropy alloys and composites for plasma facing materials.

12:30–13:00 **Sergei Dudarev**: Review of DPA definition and estimates.

13:00–14:15 **Lunch**.

14:15–15:30 **Discussion**: Review of experience with shared materials; discussion about (need for, content of) common protocol for TDS experiments.

15:30–16:00 **Break**.

16:00–17:30 **Discussion**: Fundamental modelling; status and needs.

Friday, 11 September

09:00–10:45 **Discussion**: Review of TDS experiments and code comparison effort; review of theory discussion.

10:45–11:00 **Break**.

11:00–12:45 **Discussion**: Production and characterization of damage; status and work needs.

12:45–13:30 **Any remaining business**. Plans for meeting report and for next meeting.

13:30 **Close of meeting**.

Appendix 3

Presentations

Hydrogen isotope retention in W irradiated by heavy ions and helium plasma

M. Sakamoto[1], H. Tanaka[1], S. Ino[1], H. Watanabe[2], M. Tokitani[3], R. Ohyama[4], A. Rusinov[4] and N. Yoshida[2]

[1] Plasma Research Center, University of Tsukuba, Tsukuba, Japan

[2] RIAM, Kyushu University, Kasuga, Fukuoka, Japan

[3] National Institute for Fusion Science, Toki, Gifu, Japan

[4] IGSES, Kyushu University (already graduated), Kasuga, Fukuoka, Japan

Properties of deuterium (D) retention in a tungsten (W) sample irradiated by Cu^{2+} ion beam with the energy of 2.4 MeV and a W sample with helium (He) bubbles have been investigated to study effects of the damage on hydrogen isotope retention in W. The damaged W samples were exposed to low energy and high flux D plasma to evaluate D retention through thermal desorption spectroscopy (TDS). The microstructure of the damaged samples were observed with TEM.

Retention property of W irradiated by 2.4 MeV Cu^{2+} ion beam

The TEM observation of W irradiated by 2.4 MeV Cu^{2+} ion beam revealed that most of the dislocation loops (ILs) were nucleated by cascade collisions and each IL could not grow larger individually but aligned ILs grow by coalescing. Nano-voids (< 1nm) were observed in 1 dpa case and they formed densely. The D retention increases with the damage level but it saturates around 0.4 dpa, suggesting that newly introduced defects may be cancelled by already existing vacancies and voids with high density. The desorption peak around 850 K appeared in the TDS spectra in the case of D plasma irradiation, while it did not appear in the case of 2 keV D_2^+ ion beam irradiation. From comparison between TDS spectra of the D_2^+ ion beam irradiation and the D plasma irradiation, the trapping around 850 K in plasma irradiation is considered to be attributed to the defects (i.e. voids) around the damage peak, since the range of the D ion beam could not reach to the damage peak (~600 nm).

Retention property of W with He bubbles

Low energy and high flux He plasma was exposed to the W surface with the surface temperature range of 1700 K to 1900 K. Remarkable fine irregularities with the size of smaller than a few □m were formed on the surface and many bubbles with the size of ~10nm to ~200 nm were observed beneath the surface. Three TDS measurements were made to check He desorption before D plasma exposure. Helium was desorbed in the first TDS, but no He desorption was observed below ~900 K after the second TDS. And then we evaluated fluence dependence of D retention in W with He bubbles using the same sample. The D retention was one order of magnitude higher than that of the bulk W without surface modification in the low fluence case (i.e. 2 x 10^{24} D/m^2) but it decreased gradually with fluence and became almost constant, suggesting that the helium bubbles trap considerable D atoms, but simultaneously they work as a diffusion barrier against deeper penetration of D and generate a diffusion path for desorption of the mobile D atoms.

The influence of radiation damage on D retention in W: IPP activities

W. Jacob, M. Mayer, T. Schwarz-Selinger, J. Bauer

Max-Planck-Institut für Plasmaphysik, 85748 Garching, Germany;

L. Ciupinski, J. Grzonka

Warsaw University of Technology, Warsaw, Poland

Dr. W. Jacob reviewed IPP activities regarding hydrogen isotope retention in damaged tungsten (W). Additional damaging (e.g. neutron damage or high energy ion implantation) strongly enhances H retention in bulk W. In IPP n damage is simulated in laboratory experiments by self-ion implantation, i.e., implantation of 20 MeV W ions. This has the advantage that no chemical effects have to be taken into account because the implanted ion is chemically identical to the sample atoms. The damage profile created by this W ion implantation has a damage peak at a depth of about 1.3 µm and extends to about 2.3 µm.

Damaged W is loaded with deuterium (D) in a well-quantified, low-temperature ECR plasma source [1] with a D flux in the range 0.5 to 1.1×10^{20} D $m^{-2}s^{-1}$. Typically used D fluences are 1 to 10×10^{24} D m^{-2}. The ion energy can be varied between 15 and 600 eV by application of a substrate bias. Samples are exposed to plasma on a temperature-controlled sample holder (T range 230 to 800 K). D loading with floating substrate holder (i.e., ion energy of about 15 eV) is used to decorate the traps produced by the self-ion irradiation. This gentle loading assures that no additional damage is created during loading. In this sense, the amount of retained D is assumed to be a measure of the damage produced by high energy W irradiation. NRA depth profiling shows that D is retained predominantly with in the damaged region of the sample. At low damage (i.e. low dpa values, calculated with SRIM assuming 90 eV displacement energy) D retention increases with increasing damage. It saturates at damage values of about 0.5 dpa[§] [2]. Using D decoration (at the loading temperature of 450 K) as a measure of remaining damage the annealing of damage was investigated. The remaining damage decreases monotonically with increasing annealing temperature. At T = 1170 K the amount of retained D has decreased to about 30% of the value for annealing at 450 K [3]. Annealing also lead to visible changes in TEM images [4]. TEM observations performed by the group of Lukasz Ciupinski show the damage zone which depth is 2.3 µm in good agreement with the range calculations. With increasing annealing temperature the growth of dislocation loops was observed [4].The collaboration between IPP and Warsaw University of Technology is ongoing.

D implantation into W with energies in the keV range also leads to substantial damage creation. For example, D implantation with 3 keV/D at a sample temperature of 134 K leads to huge D concentrations of about 30 at% within the full implantation range of about 70 to 80 nm [5]. This is accompanied by strong blistering of the sample surface.

First experiments were conducted to investigate the damage rate dependence. A variation of the damage rate over three orders of magnitude (using 20 MeV W ions) shows no significant effect for experiments conducted at room temperature [6].

[1] A. Manhard, T. Schwarz-Selinger, W. Jacob, Plas. Sour. Sci. Technol. 20, 015010 (2011)

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Preliminary results of commissioning of EAST full W upper divertor and deuterium retention of W irradiated by heavy ions

Presented by G.-N. Luo

1. Fabrication and commissioning of EAST full W upper divertor

EAST upgraded its upper divertor into full W/Cu-PFCs. The upper tungsten divertor has modular structure with 80 modules in total. Each module supported by inner/outer rail and middle support. The ITER-like monoblock W/Cu plasma-facing units (PFUs) were manufactured by a double Hot Isostatic Pressing (HIP) technology. All the components passed nondestructive testing (NDT) check for dual bonding between W/OFC/CuCrZr tube. High heat flux (HHF) testing was performed with a heat flux of 10 MW/m2 in 1000 cycles.

The full W upper divertor was installed into EAST tokamak from the 2014 campaign. During the commissioning in 2014, leaks related to e-beam welding and damages of the tungsten tiles due to assembly issue were found. After repairing and optimization, commissioning in 2015 was successful to the end of the spring campaign. The divertor survived from 200ºC baking and plasma discharges. Plasma-tungsten interactions (PWI) have been studied in 2014-5 campaigns via monitoring the upper divertor by means of spectroscopies and Langmuir probes.

2. Hydrogen isotopes retention/permeation of irradiated W

To understand the D permeation behavior through bulk damaged W, W foils were irradiated with 122 MeV 20Ne ions and then exposed to D2 gas at 773 K. A quasi-homogeneous distribution of atomic displacement damage to 0.3 dpa within a depth of 50 μm was produced in W using an energy degrader in the irradiation chamber of the Sector-focused Cyclotron at HIRFL (Heavy ion Research Facility in Lanzhou). After irradiation, positron annihilation lifetime spectroscopy (PALS) measurements were carried out using a fast-fast coincidence positron lifetime spectrometer to characterize the irradiation defects in W. For the thermal desorption spectroscopy (TDS), samples were heated in an infrared furnace up to 1273 K with a heating rate of 1 K/s.

Results of PLAS showed a long positron lifetime component of ~400 ps appeared after irradiation, indicating the formation of vacancy clusters with up to 12 vacancies in W. TDS showed a broad D desorption temperature range (730-1173 K) with a high release peak at ~1010 K for the irradiated W specimens. The amounts of D retained in the irradiated W were significantly larger than the annealed ones, which could be attributed to the trapping effects of the vacancy clusters formed by cascade collisions and the clustering of mono-vacancies.

Present status of activities in Japan on hydrogen isotope retention studies for neutron-irradiated tungsten materials

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This work reported on the present status of Japan-US Joint Research Project PHENIX where neutron irradiation of W and W alloys is performed in a fission reactor. The project attempts to simulate fusion conditions of high irradiation temperature, and production of Re, Os transmutation products in a fission environment. To achieve this goal, the progress made on the design and construction of the irradiation capsule with a Gadolinium thermal neutron shield was reported. Experiments are planned in the temperature range of 500-1200°C.

Second, updates on the activities performed under a collaboration program with the International Research Center for Nuclear Materials Science, Institute for Materials Research (IMR-Oarai), Tohoku University was presented. In terms of experimental facilities that are able to handle neutron-irradiated materials, a new compact linear plasma device and long-term gas exposure system for hydrogen loading experiments have been constructed and are now operational. Lower-dose neutron irradiation of W samples (0.02–0.04 dpa) in BR2 reactor (and consequently low Re and Os) at 290 °C has been completed and post irradiation experiments with such samples are planned to begin at the end of this year. To characterize hydrogen isotope trapping in vacancy-type defects first experiments using positron annihilation spectroscopy (lifetime measurements and coincidence Doppler broadening) was performed with W samples irradiated with an 8.5 MeV e-beam (10^{-3} dpa at ~100 °C). The difference in positron lifetime following vacuum annealing, or D_2 gas exposure of e-irradiated specimens was qualitatively discussed. The long-life component of the positron lifetime following D_2 gas exposure was shorter than that after vacuum annealing. This difference may possibly indicate slower vacancy diffusion under the presence of D, but further experiments are required along with complementary TEM analysis.

Third, results of D retention experiments that examined the effects of Re impurity was presented. W-5%Re alloy was irradiated with 6.4 MeV Fe ions in DuET, Kyoto U. at different temperatures, and analyzed using nuclear reaction analysis (NRA), thermal desorption spectroscopy (TDS) and positron annihilation. TDS measurements showed a large decrease in D retention in Re containing samples without a shift in the peak release temperature. In parallel, no significant increase in positron lifetime was observed for W-5%Re alloy. The two observations suggest that the reduction in D retention in W-5%Re samples may be due to a reduction in vacancy-type trap densities.

Finally, experiments examining the displacement energies in W using the ultra high voltage electron microscope (3 MeV) in Osaka U were presented. The results at 300 K for (100) orientation indicates the displacement energy lies in the range 25 eV < E_d < 70 eV. Future experiments at liquid He temperatures will examine the displacement energy dependence on crystal orientation.

Deuterium retention and isotope exchange studies in self-ion damaged tungsten exposed to neutral atoms

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Fuel retention in wall materials of ITER and future fusion reactors is an important issue due to safety limitation of the total tritium amount retained in a device. Recent results from the ITER-like Wall Project at JET [1] have shown, as expected, much lower retention in metallic (W/Be) plasma facing components (PFCs) than in C-based PFCs. For the prediction of tritium retention in future fusion reactors (ITER and DEMO) influence of irradiation by neutrons produced by the fusion D-T reaction has to be taken into account. Radiation-induced damage due to fast neutrons could lead to less favourable results having significant implications for the operation of future reactors. In that respect, successful neutron resistant materials need to be developed. Present indications show that tungsten is the most suitable 'baseline' material for plasma facing component armour (from the EFDA-roadmap). Since neutron irradiated samples are activated and therefore need special handling, high energy ions are used as neutron surrogates to produce neutron-like damaged material needed for experiments dedicated to predict of the above effect. It was shown that fuel retention in damaged tungsten is strongly increased as compared to undamaged tungsten material and for this reason influence of exposure temperature and damage annealing is being extensively studied [2, 3, 4].

In order to study the deuterium retention in damaged tungsten, the samples were damaged for our experiment by 20 MeV tungsten ions at room temperature at Max-Planck Institute fur Plasmaphysik (IPP). Such irradiation results in 2.4 μm deep damaged layer, with defects similar to those created by neutrons[5]. The purpose of our work presented within this CRP is to study the influence of neutron-like damage in a tungsten material on hydrogen retention subjected to gentle loading by deuterium atoms (0.3 eV energy). The goal of this work is to contribute to better understanding of retention that is important for tokamak operation. The atoms are trapped at the produced defects that act as additional strong binding sites for deuterium atoms in the bulk, without inducing any additional damage. We have performed two kinds of experiments with different exposure and sample annealing procedure. In the first case the so called self-ion damaged tungsten samples were exposed to D atoms, flux of 3.5×10^{19} D/m^{2}s, at sample temperatures from 500 K to 900 K to fluence that was sufficient to saturate the damaged layer by deuterium atoms [6]. In the second case the samples were first annealed for one hour at different temperatures (600 K –1200 K) and then exposed to D atoms at 500 K to a fluence of 1.3×10^{25} D/m^2[7]. By analysing the deuterium depth profile obtained by Nuclear Reaction Analysis (NRA), (d(^{3}He,p)α nuclear reaction) in such treated samples we have obtained information on the effect of sample exposure temperature and damage annealing temperature on deuterium retention. It was found that both exposure and annealing temperature have strong influence on the maximum deuterium concentration and on the integrated amount of deuterium in the sample. In both cases the concentration is decreasing with increase of the temperature, however in the case of only defect annealing the deuterium retention is reduced for 60 % at 1200 K as compared to the un-annealed sample. On the other hand the retention decreased for 98% when comparing exposure at 500 K to exposure at 900 K. For the deuterium exposure at elevated temperatures two processes are simultaneously present, defect annealing and deuterium de-trapping from traps produced by

damaging. From the observed results it was shown that the last one is responsible for the more reduced retention at elevated temperatures.

The INSIBA experimental set up at Tandetron accelerator at Jožef Stefan Institute (JSI), Ljubljana enables us to do *in situ* Nuclear Reaction Analysis (NRA) with broad ^{3}He beam (1-4 mm diameter). The dynamics of isotope exchange in damaged W, exposed to atomic deuterium beam, were investigated there at 600 K [8]. Both variations of isotope exchange of H by D and of D by H were measured. The deuterium isothermal desorption was also studied and evaluated in order to be able to resolve the self-desorption from the isotope exchange at 600 K. The bulk isotope exchange was compared also to surface isotope exchange studied at 480 K and 380 K on the surface of damaged W studied by Elastic Recoil Detection Analysis (ERDA). The exchange mechanism was effective both on the surface and in the bulk of damaged tungsten. A simple model was introduced to describe the exchange efficiency on the surface and in the bulk obtaining the exchange cross sections on the surface and in bulk. In both cases an isotope effect was observed, where the exchange of H atoms by D atoms was more efficient than the exchange of D atoms by H.

There is another aspect that was not considered in the study presented above (exposure temperature versus damage annealing), namely, in the reactor both implantation of D ions and neutrals and neutron irradiation will take place at the same time. It was observed in some materials that impurities such as hydrogen, change the behaviour of defect creation and recovery, e.g. on vacancy migration during the recovery stage [9]. For this reason we have started an experiment in our laboratory at the INSIBA set up where both irradiation by 10.5 MeV W^{6+} W ions and D atom loading take place simultaneously. The simultaneous irradiation and loading was performed in temperature range between 450 K and 800 K and results are under evaluation.

Further work will be dedicated to study the synergistic effects of simultaneous multiple-beam irradiation on the deuterium retention. These experiments include i) further work on in-situ experiments with simultaneous heavy ion (tungsten) irradiation and exposure to atomic or low energy deuterium ion beam and ii) in-situ experiments with simultaneous and sequential ^{4}He irradiation and exposure to atomic deuterium beam. In both cases, NRA analysis will be applied for in vacuum deuterium depth profiling.

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Determination of binding energies for hydrogen with radiation defects in tungsten by means of tds: theory and experimental data

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Hydrogen accumulation in the bulk of W is very sensitive to presence of defects. Therefore knowledge of trapping sites parameters is essential for predicting H isotope transport and retention in W plasma-facing components.

In this work, we analyzed applicability of thermal desorption spectroscopy for binding energy determination. It was shown that it is possible to derive the detrapping energy from the set of experiments with different heating rates in a wide range of parameters.

A series of experiments have been done to determine the detrapping energy from various types of defects. Two different types have been identified with Edt = 1,56±0,06 eV [1] and Edt = 2,10±0,02 eV [2]. These two types are, very likely, vacancies and vacancy clusters.

TDS spectra for W samples damaged by 20 MeV W ions has been also analyzed. The detrapping energy for strongest trapping sites was estimated as Edt = 1.7-2.0 eV, which is similar to keV ion irradiation experiments.

The dependence of the steady state D concentration in traps on the incident flux has been demonstrated from comparison of various experimental data in the range of 1019-1024 D/m2s. This dependence is very strong for elevated temperatures [3].

Since DFT calculations predict occupation of various defects with several hydrogen atoms. A new code has been developed in MEPhI which can simulate hydrogen transport in the presence of multiple trapping sites. Using of this approach allows to achieve a better agreement with experimental data.

Investigation of D interaction with the damage produced by 3.5 MeV electron beam are started in collaboration with IPP (Garching). At the moment, several samples were irradiated and pre-characterized by PALS technique. These samples will be exposed to low energy D plasma and analyzed by TDS and NRA technique.

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Retention and diffusion of hydrogen in tungsten: effects of temperature, alloying elements and vacancy clusters

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The solubility and diffusivity of hydrogen in tungsten are fundamental and essential factors to study hydrogen retention in tungsten, but data are scarce and largely scattered. We perform a series of first-principles calculations to predict the dissolution and diffusion properties of interstitial hydrogen in tungsten and the influence of temperature and the defect trapping effect.[1] Our results reveal that both solution and activation energies are strongly temperature dependent. The predicted solubility and diffusivity show good agreement with the experimental data above 1500 K, but present a large difference below 1500 K, which can be bridged by the trapping effects of vacancies and natural trap sites. The present study reveals a dramatic effect of temperature and defect trapping on the hydrogen solution and diffusion properties in tungsten, and provides a sound explanation for the large scatter in the reported values of hydrogen diffusivity in tungsten.

Solute atoms, whether they are voluntary added to tailor the properties of a metal, left from the elaboration process, or introduced during the component lifetime as a result of radiation damage, can affect the properties of hydrogen retention in materials. One of the reasons is that many of them interact strongly with the point defects and hydrogen and change their mobility. We performed a series of first-principles calculations to quantify the intrinsic properties of transition metal solutes and their interactions with point-defect, including vacancies and <111>-crowdions, and hydrogen in tungsten.[2,3] The solute-point-defect and solute-hydrogen interactions are mostly attractive with a few exceptions, and can be well understood in terms of the competition between the chemical and elastic interactions. Based on the calculations, we found that: i) the solute atom can trap multiple hydrogen atoms in their neighbor shells to form many small hydrogen clusters and decrease the hydrogen effective diffusivity, which could, to some extent, prevent the occurrence of large bubbles, but unfortunately would significantly increase the hydrogen retention in tungsten. ii) Solute atoms can narrow the gap in the mobility bias between the vacancy and self-interstitial in tungsten, thereby increasing the recombination rate of the vacancy with the self-interstitial. As a result, these solutes should be expected to exert a restraining effect on the growth of the radiation-induced defects and decrease the concentration of defects, particularly vacancies and vacancy clusters. In this regard, the introduction of these solute atoms should decrease the retention of hydrogen in tungsten. These findings may provide a good explanation for recent experimental results [4-8].

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Assessment and modeling of trap effects on hydrogen diffusivity in tungsten

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Diffusion, solution and trap behaviors of tritium determine tritium inventory in tungsten. Hence, in order to achieve an accurate estimate of the tritium inventory, it is important to construct kinetic models to describe these processes. For this aim, we have been performing multi-scale modeling and experiments to validate constructed models. In this report, we summarize our research progresses on the following 4 subjects, which was presented in the 2nd CRP meeting.

(1) We studied hydrogen diffusivity in tungsten, which is usually described using the equation given by Frauenfelder [1]: $D = 4.1 \times 10^{-7}\exp(-0.39\ eV/kT)$ $m^2\ s^{-1}$ (Eq. 1). This formula was determined by degassing experiment of pre-loaded H_2 gas in 1100-2400 K [1]. However, the activation energy (0.39 eV) disagrees with values determined by first-principles calculation based on the density functional theory (DFT), which is around 0.20 eV [2]. Heinola and Ahlgren [3] examined this disagreement, and showed that the diffusion coefficients obtained with DFT calculation are comparable with those of Frauenfelder's experiment, if experimental data at low temperatures (<1500 K) are excluded. The exclusion decreases the activation energy for hydrogen diffusion in Frauenfelder's experiment from 0.39 eV to 0.25 eV, as given in $D = 1.58 \times 10^{-7}\exp(-0.25\ eV/kT)$ $m^2\ s^{-1}$ (Eq. 2). This finding indicates significant influence of traps even at high temperatures like 1100-1500 K. In order to quantitatively confirm this finding, we evaluated the influence of traps in hydrogen diffusivity in tungsten by kinetic Monte Carlo (KMC) simulation [4]. As a typical trap, mono-vacancy was considered in the simulation. The hydrogen-vacancy interaction energies were taken from DFT calculation results [5]. In results, hydrogen diffusion coefficients reported by Frauenfelder [2] were nicely reproduced if hydrogen and trap concentrations expected in the experiment were employed in the simulation. Therefore, we recommend Eq. 2 as an equation to express the hydrogen diffusivity in tungsten, which was obtained by fitting only to experimental data at 1500-2400 K [3].

(2) We examined the accuracy of DFT calculation because DFT calculation is often employed as the most accurate method in multi-scale modeling and its errors can be propagated to larger-scale models. One intrinsic source of uncertainty/error in DFT calculation is exchange-correlation functionals. For tungsten, we systematically calculated defect formation energies and migration energies with various

functionals. It was confirmed that these energies can be different by up to around 10%, depending on a sort of the functionals and a kind of property to be calculated. As a practical method, comparison of DFT calculation results with several different functionals could give an estimate of possible uncertainty/error in DFT calculation.

(3) Trap effects of vacancy are usually evaluated using the binding energies of the most stable VH_n complex configuration. However, when multiple hydrogen atoms are trapped, because less stable complexes should be also formed in dynamics, the evaluated trap effects would overestimate the true trap effects. Thus using transition state theory and equilibrium theory, we constructed a model to appropriately consider the contribution of less stable defects on vacancy trap effects [6]. Bcc-Fe was selected as the target system because a good potential model to describe V-H interactions was reported [7] and hydrogen behaviors in bcc-W and bcc-Fe are quantitatively similar to each other. As a result, we demonstrated that the overestimation certainly occurs and the constructed model can nicely describe the hydrogen trap effects of vacancy in bcc-Fe at both equilibrium conditions and non-equilibrium conditions [6].

(4) By using TMAP4 code, a scaling law to estimate the tritium retention amount with respect to the ion fluence is obtained. The ion-induced defect fraction based on the Olga's model [8] was calculated by assuming that the defect generation rate is same with the growth rate of the oversaturated depth which was measured by the SIMS depth profile. The calculated scaling law (retention=a×fluence^b, a ~10^3-10^6, b=0.6-0.8) is well agreed to experimental data previously reported.

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Modeling and Simulation of Helium and Hydrogen Behaviours in Tungsten

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Tungsten (W) and W alloys are the most promising candidates for plasma facing materials (PFMs) in Tokamak because of their good thermal properties and low sputtering erosion. However, as a PFM, W will be exposed to extremely high fluxes of helium (He) and hydrogen (H) isotope ions from the

plasma. The bombardment of He and H will give rise to blistering at the W surface, which becomes a key obstacle to develop W as a PFM.

The self-trapping of He should be responsible for the effects of He on the properties of metals. However, the physical intrinsic mechanism for He self-trapping is still unclear. We have investigated the He-He interaction in metals using a first-principles method. We demonstrate that there exists a form of electrophobic interaction between He atoms, which governs their dissolution behaviour in a metal. We predict by first-principles calculations that such an electrophobic interaction drives He to form a close-packed cluster with a clustering energy that follows a universal power-law scaling with the number of atoms (N) dissolved in a free electron gas, as well as W lattice, scaling as $E_c \propto (N^{2/3} - N)$. Validating this concept, we present direct experimental evidence of He bubble formation in an oxide dispersion strengthened alloy, proposed as a principle cladding material for containment vessels in next generation reactor environments. Unifying the explanation for a series of reported experimental observations of close-packed inert-gas bubble formation in metals, this new concept significantly advances our fundamental understanding and capacity to predict the solute behaviour of impurities with closed electron shell structure in metals, a useful contribution to be considered in future material design of metals for nuclear, metallurgical, and energy applications.

Recent experimental studies indicate that the strain in W is directly associated with the growth of H bubbles. In order to understand the effect of strain on H in W, we have investigated the behaviour of H in the strained W using a first-principles method. Surprisingly, we discovered that the H solubility can always be enhanced by anisotropic strain in W, independent of the sign of strain. In a wide range of strain values, the H solubility is enhanced in the strained W. This anomalous behaviour is found to be caused by an unusual H motion induced by anisotropic strain. The common expectation of a monotonic dependence of H solution energy on strain has a prerequisite condition that the H stays at the same location without motion under strain. However, it breaks down for H at the tetrahedral interstitial site, when an anisotropic strain is applied. Our finding suggests a cascading effect of H bubble formation in W: the H solution leads to H bubble formation inducing anisotropic strain that in turn enhances H solubility and more bubble formation.

Status update on MERCOR-TMAP integration, Tritium Plasma Experiment Modification, and PHENIX program for PMI in irradiated tungsten.

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M. Shimada (INL, USA) reported the status of MERCOR-TMAP integration, Tritium Plasma Experiment (TPE) Modification, and PHENIX program for PMI in irradiated tungsten. US-Japan PHENIX program will start HFIR neutron-irradiation to W from Feb.2016 to April 2017. This irradiation will provide large number (> 100) of one-of-a-kind neutron-irradiated tungsten specimen. Tritium Migration Analysis Program (TMAP) is being imported into this MELCOR code in order to produce a more self-consistent safety accident analysis capability (also an update for the TMAP code, making it more advanced than user requested TMAP7).

TPE will be restarted with electrical system in October 2015, and this modification will enable safer plasma operation and achieve much higher ion flux density (> 10^23 m-2s-1), and increase the availability of the TPE for new (> 100) neutron-irradiated tungsten specimens.

Radiation damage of Heavy ions and H irradiated Tungsten – Some Experimental Results

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The objective of this study is (i) to study the structural, microstructural and morphological changes in tungsten after low (0.1 MeV), medium (7.5 MeV) and high (80 MeV) energy heavy ion irradiations and (ii) hydrogen trapped/retained in un-irradiated and pre-irradiated tungsten as a part of the research project entitled "Radiation damage and H/D retention studies on ion-irradiated Tungsten and its alloys - Experiments and Modeling", under the IAEA agreement. The work has been carried out in collaboration with scientists at many Institutes in India as mentioned above.

EBSD technique has been used to understand the grain structure, size and orientation in the samples in which heavy ion (such as W and Au) irradiation has been carried out. EBSD results show that Tungsten (Linke-Plansee) samples have equiaxed grains on the surface. However, in the cross section columnar grains are seen. In the Plansee samples, it is observed that there are larger grains and lattice is strained. The samples are annealed at 900 C before irradiation to relieve stress in the samples. The stress and strain (macro and Micro) have been measured before and after Au irradiation by X-ray Diffraction and it has been observed that the pristine and 900 C annealed samples are tensile strained with respect to standard relaxed tungsten matrix d values. It is progressively relieved with 100 keV Au irradiation and 80 MeV Au irradiation. All irradiated samples show that irradiation tends to increase preferred orientation in (200) and (211) direction and the effect must be extended in substantial thickness of 10s of microns. Increase in the crystallite size is observed in 80 MeV irradiated gold. Positron Annihilation Lifetime Spectroscopy (PALS) results show the effect of annealing in terms of enhanced mono-vacancies at the cost of vacancy clusters. The effect of irradiation is seen in the reduction of mono vacancies.

In order to study the irradiation effects of W ions on retention of hydrogen in tungsten, samples of tungsten were subjected to the irradiation of 7.5 MeV W ions with the fluence of 4.5×10^{15} and 4.5×10^{14} (in two set of samples) followed by 50 KeV hydrogen (H2) implantation with the fluence of 1×10^{18} ions/ cm^2. Elastic Recoil Detection Analysis (ERDA) results show that with the increase in W irradiation dpa value (ion fluence) there is a small increase in the hydrogen trapping. Nano-structuring on the surface of pristine tungsten samples is observed after 80 MeV Au irradiation. However, such nano-structuring is not seen in the 80 MeV Au irradiated annealed tungsten samples. More investigation in this regard is underway.

Radiation induced degradation of tungsten under thermal and plasma exposure and development of advanced tungsten materials

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Within the first working period of the CRP on irradiated tungsten a number of research activities have been performed successfully. The major achievements during this period have been reported during the 2nd RCM Meeting at SNU in Seoul in September 2015:

- Systematic electron beam and laser beam exposure experiments on tungsten with and without ion beam pre-exposure to investigate synergistic effects of particle and transient heat loads on thermal shock performance of W grades
- Detailed analyses on the modification of the microstructure under simultaneous heat and particle loads and impact on W erosion and fuel retention in W
- Quantifications of the fuel retention in pre-damaged W. In these analyses the impact of different impurities (He, N, Ne, Ar) on fuel retention has been determined.
- Analyses on the impact of W surface contamination with oxygen on hydrogen retention and W erosion
- Development of new advanced tungsten materials with improved mechanical properties and oxidation resistance in close collaboration with IPP Garching. W-fiber re-enforced tungsten grades based on Hot Isostatic Pressing (HIP) or Chemical Vapor Deposition (CVD) processes have demonstrated enhanced mechanical strength and fracture toughness. Alloying of tungsten with Cr and Y has shown a 10^{-4} fold suppression of the tungsten oxidation during accidental conditions with coolant loss or air ingress in a fusion power plant.
- A new program has been launched recently on the characterization of commercially available tungsten grades. This will also improve the data base for those W-grades which will be used in the frame of this CRP. Test coupons made from a uniaxial deformed tungsten grade (Plansee AG) have been distributed to those laboratories which had shown interest to perform tests on this material.

High-flux plasma effect on tungsten damaged by high-energy ions

V. Koidan, B. Khripunov, A. Ryazanov

The following work was envisaged for the first two years of the Project:

- Use 3-4 MeV He ions from the Kurchatov Cyclotron to create damaged tungsten samples.
- Calculate primary radiation defect profiles. Expose the irradiated samples to deuterium plasma on LENTA linear plasma simulator and study erosion and deuterium retention using nuclear reaction analysis.
- Carry out erosion and retention experiments and analysis for tungsten damaged by high energy C ions and compare with He irradiation. Use simulations to compare damage by surrogate irradiation and neutron damage.

Tungsten is studied as plasma facing material proposed for application in fusion reactor. Effect of displacement damage on material performance under plasma attack appears to be a serious concern for high-dose irradiations [1-4]. Experimental work has been conducted for the last years at Kurchatov Institute to develop the radiation damage production method and to study the damaged material under

high flux plasma impact. The influence of the radiation damage occurring in tungsten on the result of the plasma effect is investigated. To date, damaged tungsten samples in the interval from 0.1 to 100 dpa have been obtained thus covering the range of interest for ITER and beyond. Irradiations of the material with ions at MeV-energies are performed to obtain damage, and then the irradiated samples are exposed to steady-state high-flux plasma. While carbon based materials were studied in the first stage the present work is focused on tungsten and the method has been developed by employing different high-energy ions (He, C). The results obtained recently on tungsten at high level of radiation damage under plasma impact are reported.

Tungsten at high-level of radiation damage

Polycrystalline tungsten W 99.95 wt.% (Russian grade) is under study. First, $^{4}He^{2+}$-ion irradiations of samples have been performed on accelerator (cyclotron) to doses from 1017 cm^{-2} to 10^{19} cm^{-2} at 3.5-4 MeV (corresponding to the alphas generated in DT fusion reaction). By this, defects are produced to depth of $^{4}He^{2+}$ range in tungsten of about 6 μm. At high helium fluencies given above, the damaged layer acquires a particular porous structure shown in Fig. 1, the pores and cavities supposed to be filled with helium. The results of the experiments with He-irradiated tungsten (swelling effect, erosion in deuterium plasma, surface structure modification, deuterium retention) have been reported elsewhere [5-8]. The structure of the damaged material and the presence of helium have a major influence on the performance of the material under plasma exposure. For example, experimental profile of the implanted helium was also much larger (2-3 μm) than that expected from calculation (0.5-1 μm) [9]. This result is readily explained by strong changes of the material structure in the damaged layer. Swelling effect was detected by profilometry at 2-3 % for the damaged tungsten [9].

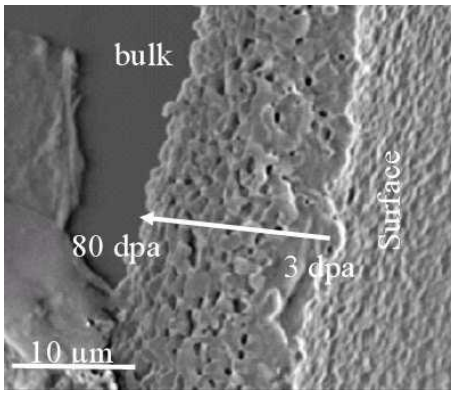


Fig. 1. Surface layer of tungsten damaged by He-ions (3 × 1018 cm-2, 3.5-4 MeV): from 3 dpa at the surface to 80 dpa at 6-micron depth.

Taking into account these particular properties of helium present in tungsten (see also [10]) the work was continued with another ion species. Irradiations of tungsten were made with carbon ions $^{12}C^{+3}$ accelerated to 10 MeV. In this case, the total ion fluency reached 2×10^{17} cm^{-2}. The resulting levels of damage expressed in displacement per atom (dpa) were equivalent to those obtained in He-ion case. Analysis of defect production was based on calculations by SRIM program. Shown in Fig. 2 is distribution of primary defects in tungsten produced by $^{12}C^{+3}$ (Fig. 2). The concentration of the implanted ions is also presented.

The obtained results on tungsten irradiated with helium ions (swelling, erosion in deuterium plasma, surface structure modification, deuterium retention) have been reported earlier, see [5-8]. Swelling in

tungsten after irradiation with $^{4}He^{2+}$ has been measured by changes in linear dimensions of irradiated material and it reached 2-3% (profilometry).

Swelling effect was found also on C-irradiated tungsten. Fig. 3 shows the surface of tungsten sample (SEM) after irradiation with C-ions (10 MeV) to $\Phi = 2\times10^{17}$ cm^{-2}. The photo is taken in the region of the irradiated area boundary visible as a vertical shadowed line in the central part. The surface profile around the boundary is also given in the figure. It exhibits a step corresponding to elevation of the irradiated part over the unirradiated one.

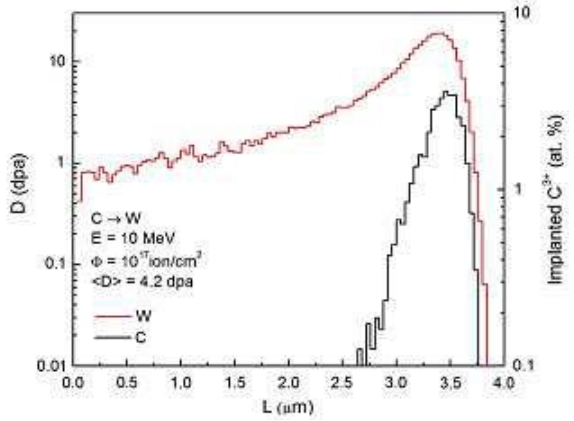


Fig. 2. Primary defect distribution in tungsten irradiated by $^{12}C^{+3}$ ions at 10 MeV to fluence of 10^{17} ion/cm^2. <D> = 12.5 dpa average over damaged layer 3.5 μm in depth.

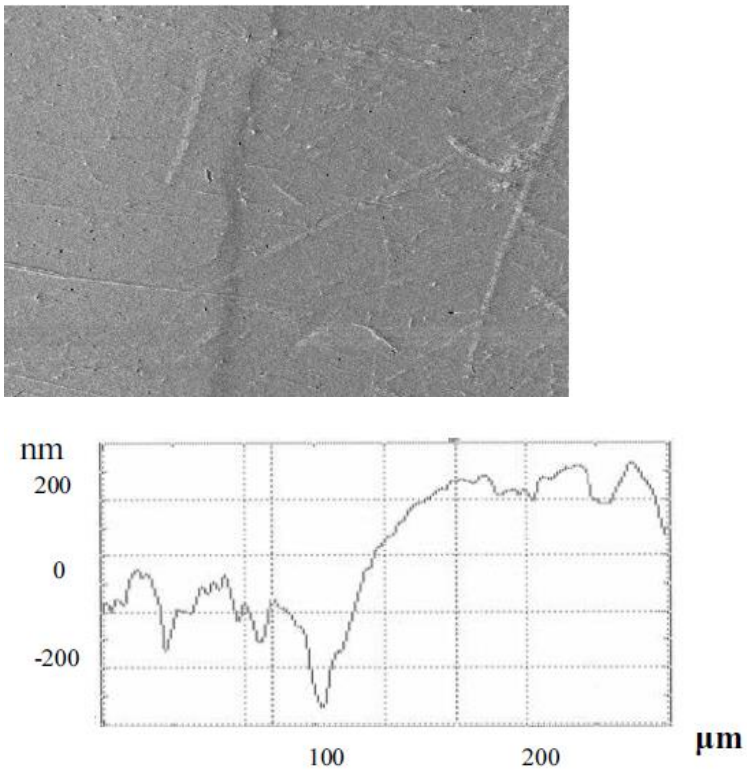


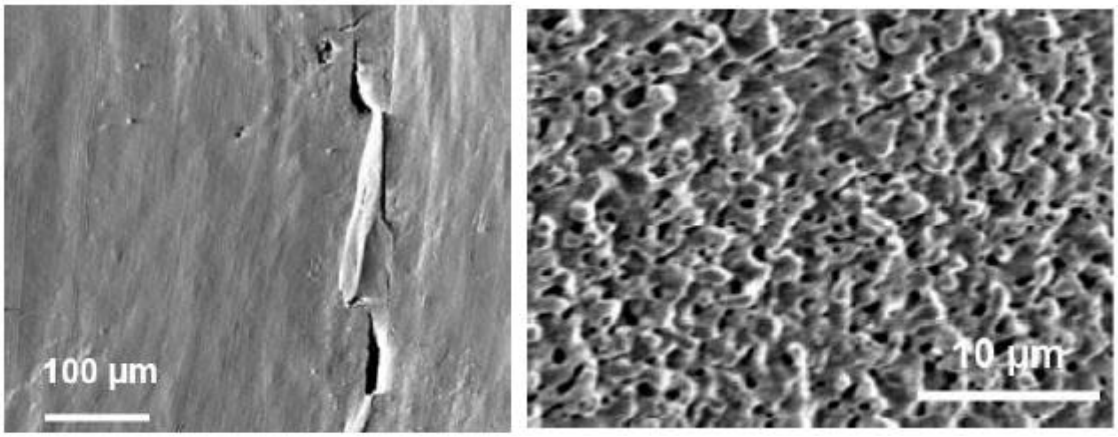
Fig. 3. Boundary of C-ion irradiated area on tungsten sample: the irradiated area is to the right; the left part was masked from irradiation. Surface profile shows elevation of the irradiated part.

Erosion of damaged tungsten in deuterium plasma

Distribution of the defect concentration in damaged layer is highly nonuniform (Fig. 2). Sequential exposures have been carried out to investigate changes in the damaged layer under durable plasma action. Plasma parameters were taken so that erosion condition was realized. Erosion of tungsten at each exposure step was 0.5-1.5 μm, therefore the whole damaged layer could be taken off by erosion in several step sequences [5-8]. By this, relation of erosion rate and surface microstructure with the level of damage at the corresponding layer could be examined.

The results of plasma series with He-irradiated tungsten sample are presented here below for dose $3 \cdot 10^{18}$ He/cm^2 (3,5 MeV, D_{max} = 80 dpa, $D_{min,surface}$ = 3 dpa, <D> = 12 dpa). Fig. 4 shows the sample surface at different stages of the experiment. Fig 4a gives the surface after irradiation (SEM). Significant changes such as blistering related to the splitting of a certain layer from the bulk, cracks of this layer appeared on the surface; the thickness of the damaged layer was about 5 μm. Those features are explained by the character of damage distribution in the surface layer and by important helium accumulation having strong influence on erosion and on the development of the surface microstructure. The studied sample was subjected to a series of 6 sequential deuterium plasma exposures on the LENTA linear plasma simulator:deuterium ion flux (2-3) × 10^{17} ion/cm^{2}s; plasma density 2 × 10^{12} cm^{-3}, electron temperature 6-8 eV, ion energy 250 eV (bias). The sample was actively cooled during exposure. Erosion rate was evaluated by weight loss method. The performance of this series is presented in Table 1.

Important erosion rate from 0.4 to 0.8 mg/cm2 h was measured. Analysis of the surface at each exposure step reflected the damaged layer erosion dynamics in the plasma. Fig. 4b shows the surface after the 4th exposure. One finds here a cellular structure formed during erosion to 3.5 μm (see Table 1) with helium escaping from the pores. Fig. 4c, 4d show the structure of the surface after 6-fold exposure when the total erosion depth was about 5 μm, and the surface facing the plasma displayed the layer of maximal defect concentration. Evaluation of the erosion rate may indicate an increase in this layer (see Table 1).



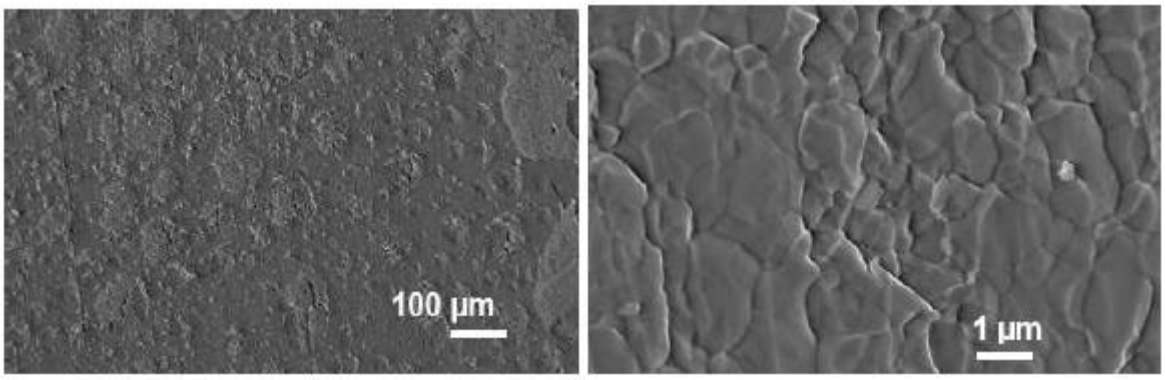


Fig. 4. He-irradiated tungsten (3×10^{18}cm^{-2}, 4 MeV): a – surface after irradiation; b –after 4-fold deuterium plasma exposure, 3.6 µm eroded; c, d – after 6-fold plasma, 5.4 µm eroded.

Exp. Nr	D-fluence, 10^{21} D/cm^2	Erosion depth, µm	Y, at/ion	D, dpa
1	0,9	0,13	$1 \cdot 10^{-3}$	6
2	1,8	0,63	$2.3 \cdot 10^{-3}$	7.5
3	2,7	1,7	$3 \cdot 10^{-3}$	10.5
4	2,4	1,1	$2.4 \cdot 10^{-3}$	15
5	1,6	0,8	$2.8 \cdot 10^{-3}$	19.5
6	1,0	1,0	$5.9 \cdot 10^{-3}$	54
Σ	10,4	5,4		

Table 1. Parameters of deuterium plasma exposures of tungsten sample pre-irradiated with fast He ions ($\Phi = 3 \cdot 10^{18}$ He/cm^2 , 3,5 MeV).

Erosion of C-irradiated samples has been also studied in deuterium plasma at the levels of 10-100 dpa. Both low temperature case (below 100C) and elevated temperature case (500-800 C) of tungsten were under study. Irradiation conditions corresponded to the following data:C ions at 10 MeV, 2×10^{17} ion/cm^2, Dmax = 120 dpa (at 3,5 µm); Dmin surf = 5,5 dpa, <D> = 25 dpa. Plasma exposures of these samples were conducted in conditions close to those for He irradiated samples: D-plasma at 250 eV, 2×10^{21} D/cm^2. Two-fold exposure showed the measured values of erosion yield of about 3×10^{-3} at/ion at ~1 µm depth and about 5×10^{-3} at/ion deeper by 0.5 µm. The effect is illustrated in Fig. 5 where a cross section example is presented as taken after erosion to 1 µm. The structure of the damaged layer is definitely different from that of the bulk material, and appearance of cavities may be supposed.

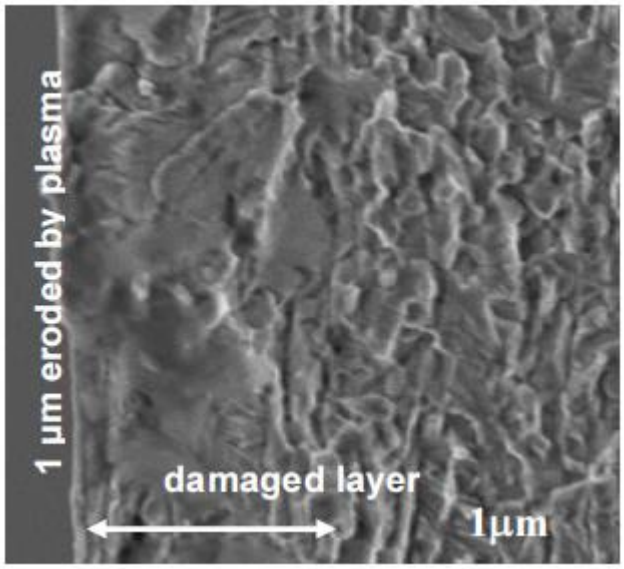


Fig. 5. C-irradiated tungsten cross section (10 MeV, $2 \times 10^{17} cm^{-2}$) after deuterium plasma (1 μm eroded).

The samples irradiated by carbon ions (C^{3+}, 10 MeV, 1.5×10^{17} cm^{-2}) at T_{irrad} = 500 C were also exposed to deuterium plasma. The temperature of the material was also controlled at 500 C during plasma operation till the plasma fluence $(1\text{-}1.2) \times 10^{21}$ D/cm^{-2} was reached. Erosion depth in this case was 0.8 micron, and erosion yield evaluation gave 4.3×10^{-3} at/ion. The obtained value does not differ much from the earlier results for other irradiation and plasma exposure conditions. The surface structure of the sample processed at 500 C after erosion does not show much difference compared to the low temperature case.

Deuterium retention in damaged tungsten

Deuterium retention in damaged tungsten after plasma exposure has been analysed by nuclear reactions methods. The retained deuterium profiles have been registered by ERD analysis. The results obtained for two kinds of irradiations (He, C) provide data for direct comparison of the retention observed in these cases. The data given below was taken for very close plasma exposure conditions (see above). The results are summarized in Fig. 6: two curves represent D distributions for He-irradiated samples (W-03A and W-4 irradiated with He ions at 3-4 MeV, 3×10^{18} cm^{-2} and 3.2 MeV, 3.2×10^{18} cm^{-2} correspondingly), and two others represent distributions for C-irradiated tungsten (W-5 and W-6 in the figure, 10 MeV, $\Phi = 2 \times 10^{17}$ cm^{-2} on both). It appears that all distributions are very similar: deuterium is concentrated mainly near the surface in the layer about 70-80 nanometers deep. The total amount of the retained deuterium is also very close from 1.65×10^{16} cm^{-2} to 1.85×10^{16} cm^{-2} in all cases (see Fig. 6).

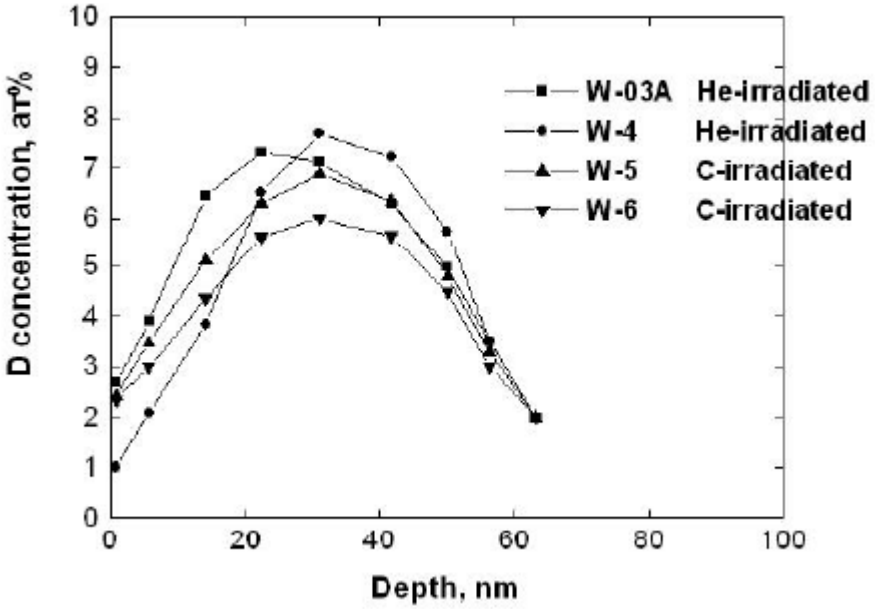


Fig 6. Deuterium concentrations (ERDA) in damaged plasma exposed tungsten: W-03A and W-4 – He-irradiated samples, 2-3 dpa at the surface; W-5 and W-6 – C-irradiated samples 2 dpa at the surface.

Erosion in plasma of W-03A and W-4 was 1.2 μm (by 2.9×10^{21} D/cm^{-2}) and 0.7 μm (by 1.0×10^{21} D/cm^{-2}) correspondingly. The damage level in these layers is assumed to be of about 2 dpa. The C-irradiated samples eroded in plasma by 0.5 and 0.9 μm correspondingly. The damage level in the considered depth is assumed to be about 2-3 dpa. From this, we may conclude that the distributions presented here display very close quantities of the retained deuterium for similar plasma conditions and for close values of the damage level. Similar are deuterium penetration depth and maximal concentrations 6-8% at. at depth of 20-30 nm.

Summary

Tungsten was studied at high level of radiation damage under steady-state deuterium plasma. The damage was produced by high energy ions to simulate fusion neutron effect on the material. The damage of the tungsten samples under study ranged from several dpa to more than 80 dpa and was relevant to the values expected in fusion reactors (ITER, DEMO). Exposure of the irradiated samples was performed in deuterium steady-state plasma on linear plasma simulator in conditions simulating tokamak diverter. Two methods of damage production were tried: MeV-range helium and carbon ions. This study confirmed the observations obtained in the previous work concerning a strong influence of helium on modification of the damaged layer microstructure: great amounts of the accumulated helium led to formation of He-filled bubbles and cavities in the layer (about 6 μm deep) that gave a particular surface structure under plasma bombardment, and by this it could influence the erosion rate of the material. Though no significant change in erosion yield has been measured till now in different series with He-irradiated tungsten, a certain rise of erosion rate was shown at the layer of maximal damage (~80 dpa).

Carbon ion irradiations were made to obtain the damage close to that of He irradiations. The structure of the damaged layer in this case has shown difference from the bulk material with no particular

influence on the measured erosion parameters. The most spectacular effect was found in the comparison of deuterium retention in the studied tungsten for two ions species used for damage production at similar plasma exposure parameters corresponding to erosion dynamic condition. The ERDA analysis has shown very similar depth distributions of deuterium concentration at the surface facing the plasma at close values of damage (2-3 dpa). Close maximal values of 6-8 % at. (at 20-30 microns) and total quantities of the retained deuterium (from 1.65 × 1016 cm-2 to 1.85× 1016 cm-2) were found to occur.

Acknowledgments

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