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### **Plasma-wall Interaction with Reduced-activation Steel Surfaces in Fusion Devices**

#### **Summary Report of the First Research Coordination Meeting**

IAEA Headquarters, Vienna, Austria

9–11 December 2015

Prepared by

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

August 2016

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

### **ABSTRACT**

Seven experts in the field of plasma-wall interaction on steel surfaces together with IAEA staff met at IAEA Headquarters 9–11 December 2015 for the First Research Coordination Meeting of an IAEA Coordinated Research Project on Plasma-wall Interaction With Reduced-activation Steel Surfaces in Fusion Devices. They described their on-going research, reviewed the main data needs and made plans for coordinated research during the remaining years of the project. The proceedings of the meeting are summarized in this report.

August 2016



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## 1. Introduction

Various kinds of reduced-activation steel are being considered as wall material for a fusion reactor, but not enough is known about plasma interaction, erosion and tritium retention in such steels. Erosion brings impurities into the plasma and limits the lifetime of the wall. Hydrogen penetration and retention in the surface removes tritium from the plasma, making it unavailable for fusion.

The mission of the Nuclear Data Section in the area of atomic and molecular data is to enhance the competencies of Member States in their research into nuclear fusion through the provision of internationally recommended atomic, molecular, plasma-material interaction and material properties databases. The Subcommittee on Atomic and Molecular Data of the International Fusion Research Council makes recommendations to the IAEA Nuclear Data Section as to its programme in support of this mission. In its biennial meeting in April 2014 the subcommittee assessed priorities for possible CRPs in the area of plasma-material interaction in fusion devices. The subcommittee recommended a CRP on plasma interaction with reduced activation steel surfaces as its highest priority in that area. This reflects an assessment that (despite consideration given to liquid metals, certain ceramics and once again graphite) really tungsten and steel are the leading candidates for the plasma-facing surfaces in a reactor. The Unit has started a CRP F43021 on “Plasma-wall interaction for irradiated tungsten and tungsten alloys in fusion devices” (2013-2018) and the proposed CRP on steel surfaces therefore focusses on the other main candidate material for the next generation of fusion devices

A Consultancy Meeting was held on 20 August 2014 to prioritise the experimental and theoretical contributions to the proposed CRP. The discussion led to a CRP proposal with a strong experimental component and a theoretical component focussed on simulations in direct support of experiments. The CRP proposal was reviewed and approved by the Committee on Coordinated Research Activities later in 2014, and then the participants were identified.

The new CRP on Plasma-wall Interaction with Reduced-activation Steel Surfaces in Fusion Devices (CRP on Steel Surfaces for short) is devoted to surface processes and thereby it complements a larger body of work that is concerned with properties of steel as a structural material in fusion reactors and with the effect of high intensity neutron irradiation on structural properties of steels. These topics are not in the domain of the Steel Surfaces CRP, but there is overlap. Work on radiation damage in structural steels is relevant to radiation damage in plasma-facing steel. The new CRP will certainly draw upon the developing knowledge about damage processes in structural steel, but the work within the CRP will be devoted to erosion and tritium retention properties and the way in which these are affected by damage.

The first research coordination meeting (RCM) of the new CRP on Steel Surfaces was held 9-11 December 2015 at IAEA in Vienna and the present report is the output of that meeting. At the time of the first RCM there are 7 groups represented in the CRP. They are the plasma-wall interaction group at IPP Garching, Germany, the PISCES team at UCSD, USA, the CAS Lanzhou Institute of Chemical Physics, China, the National Institute for Fusion Science (NIFS) in Toki-city, Japan, the Fusion Reactor Department of NRC Kurchatov Institute, Moscow, Russian Federation, the Institute for Plasma Physics in Kharkov, Ukraine, and the Nuclear Research Centre SCK-CEN in Mol, Belgium.

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.

## 2. Proceedings

The first research coordination meeting of the CRP on Plasma-wall Interaction with Reduced-activation Steel Surfaces in Fusion Devices started with a welcome by the director of division of physical and chemical sciences, M. Venkatesh and the new section head of the nuclear data section A.

Koning. It was followed by a presentation on meeting objectives by the scientific secretary B. Braams. The agenda was adopted and participants introduced themselves.

The meeting proceeded with presentations by participants on their group research activities: P. Wang of Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou, China, on the study of the interaction of RAFM steel with plasma in laboratory devices and under EAST plasma conditions, W. Jacob of Max Planck Institute for Plasma Physics, Garching, Germany on laboratory measurements of RAFM steel erosion and H retention in RAFM steel, Y. Hirooka of National Institute for Fusion Science (NIFS), Toki, Japan on studies of bi-directional hydrogen isotopes permeation through the first wall of a magnetic fusion power reactor, A. V. Golubeva of NRC “Kurchatov Institute”, Moscow, Russia on the retention and permeation of hydrogen isotopes through RAFMs, R. Doerner of University of California at San Diego, La Jolla, USA on the investigation of sputtering of RAFM steel in the PISCES linear device, V. Makhlai of Institute of Plasma Physics, National Science Center “Kharkov Institute of Physics and Technology” (NSC KIPT), Kharkiv, Ukraine on the modification of steel surfaces exposed by hydrogen/helium plasmas streams simulating fusion reactor conditions, and D. Terentyev of SCK-CEN Belgium Nuclear Research Centre, Mol, Belgium on the interaction of high flux plasma with RAFM steels: experimental and computational assessment. Summaries of presentations are presented in the Appendix 3.

After the presentations participants discussed the topics of erosion and material evolution and hydrogen isotope retention and transport, which are summarized in the Section 3.

On the last day, participants reviewed the work plans during the period of CRP, which are summarized in the Section 4.

### **3. Discussion and Conclusions**

Several issues related to the erosion and material evolution and hydrogen isotope retention and transport in reduced-activation steel used for fusion devices were discussed. The following notes are a bit unpolished, but perhaps useful nonetheless for those working in the field.

#### **Possibilities to measure surface composition**

The principal concern is to measure the tungsten concentration (tungsten enrichment) and for this purpose one needs a very sensitive diagnostic with precise depth resolution. It is difficult to extract the information and probably not the highest priority.

Depth resolution of normal Rutherford Back Scattering (RBS) is insufficient.

New method of Medium Energy Ion Scattering (MEIS) was developed by Petter Stroem (?) within Eurofusion; this uses H or He ions in the 100 keV to 400 keV range of energy and it may have better depth resolution.

In-vacuo analysis by Auger Electron Spectroscopy (AES) or X-ray Photoelectron Spectroscopy (XPS) is very surface sensitive, but it is in principle not depth resolved. Depth profiling is possible by sputtering, but then the preferential erosion during sputtering also has to be taken into account.

Glow Discharge Optical Emission Spectroscopy (GDOES) is proposed, but the achievable depth resolution is poor or not well known. It has been used on PSI-2 in Jülich to study preferential sputtering of an Fe/W layer.

Energy Dispersive X-ray (EDX) spectroscopy of cross sections is also possible.

#### **Measurements of differential erosion**

Source term spectroscopy is in principle the most direct measurement. One needs atomic data for Fe and the other constituent and we need to check the status of relevant data. It allows real-time measurements of species-resolved sputtering rates. Source term spectroscopy is being developed for this application to steel surfaces in the PISCES project at UCSD.



One can get better measurements using ion beam analysis for mkm deposited layers, e.g. W-Fe layer.

### **What is the critical upper temperature**

Critical temperature below which we get the self-passivating property. (Presentations here showed about 850K.)

Can we understand it from modelling? We think not, steel is too complicated. Can we study it better?

Can we understand it phenomenologically? There is the issue of Fe diffusion within the steel above a critical temperature. There is the confounding issue of changes in morphology at another critical temperature.

### **Morphology**

Scanning electron microscopy (SEM) provides information on the scale of 100 nm down to about 20–30 nm. Transmission electron microscopy (TEM) provides finer resolution, but it is more difficult.

Laser confocal microscopy?

Depends on temperature at exposure; depends on flux.

We see a reduction of total erosion as more morphology develops; this is not understood.

Depends on angle of incidence; ion beam experiments.

### **Measurement of total erosion rate**

Weight loss measurements.

Measurement of erosion thickness; covering part of samples during sputtering and measuring step height of erosion crater; profile measurement.

### **Shared material samples**

Obvious, or must we identify suitable sources? Eurofer is not commercially available; D. Terentyev can distribute newer samples of Eurofer. Rusfer can also be distributed?

We should think of some joint campaign of specific studies, for example flux and fluence dependence. Then we need to use identical material.

Flanges could be arranged by NIFS? SCK-CEN could provide standard steel samples?

Maybe it is useful to choose the steel that has the highest concentration of W already?

Need discussion about preannealing and other pre-treatments.

### **Modes of exposure for erosion**

Erosion by H/D/T; He; light impurities C, N, O; heavy impurities. In practice for the steel surfaces we primarily anticipate erosion by CX neutral H/D/T. The impurities are always present in plasma experiments, and we must be aware of that. In present experiments we also have the issue of material redeposition; this too is not an issue for the CX exposure in the reactor.

PISCES experiments of erosion are basically done at normal incidence due to the bias voltage. In the fusion device one will have a real angular distribution. This can be studied at much lower fluence in ion beam experiments, and of course it can be simulated in SDTrimSP.

## **Coordinated experiments on erosion**

Try to define some set of experiments using same material, but exposure at whatever range of flux and fluence conditions is appropriate at each site. This would involve at least UCSD, Garching, Kurchatov, SCK-CEN, Lanzhou, Juelich. Need some email exchange about the conditions.

The standard material could be any of the standard RAFM steels (Eurofer, Rusfer, CLAM). It could also be commercial F82H, but we prefer to use one of the RAFMs.

Key question concerns the flux dependence of erosion. Use a high fluence so that weight loss is available.

Plasma experiments can run much quicker than beam experiments. Russ Doerner suggests to run several different samples on PISCES under identical exposure conditions.

## **Data on diffusion of Fe, W within the steel**

Maybe best studied in binary systems, deposited layers.

## **Measurements of H/D (T, He) in the surface**

What are the prospects for in-situ measurements; first few nm, first few mkm? Not a high priority.

Hydrogen retention and depth profile measurement after exposure is more or less standard. (NRA, TDS, sputtering.)

SIMS not really useful for H measurements.

In the areas where there is plasma contact we have bombardment by hydrogen, helium and impurities. We think that steel will only be used in regions where there is no direct plasma contact (regions shielded by a limiter). In that case the dominant source of damage, by far, is CX neutrals of H/D/T. Helium is produced by neutron reactions, but that is not a priority item for us.

## **Interpretation of TDS data; simulations**

More or less the same issues as for TDS for H in W

## **Measurements of hydrogen mobility, permeability**

Transport parameters such as diffusivity and solubility in reduced activation ferritic steel alloys are important to estimate the permeability of hydrogen isotopes through the first wall of a fusion power reactor. Also, the surface recombination coefficient is also important to estimate “dynamic” hydrogen retention.

## **Relation of H-retention to steel composition, preparation**

IPP Garching and Lanzhou LICP both emphasize study of the role of Cr for H retention. The model system would be Fe-Cr-W-H.

## **4. Work Plans**

### **IPP Garching**

**Year 1:** Preparation and characterisation of magnetron sputtered Fe/W thin films. Exposure of thin Fe/W films to D ion beams at low energies (100 - 1000 eV) at 300 K at different ion fluences ( $10^{21}$ - $10^{24}$  D/m<sup>2</sup>) in HSQ device. Quantification of sputter yields (partial and total yields by IBA and weight loss measurements). Supporting investigations of the surface morphology after ion bombardment by SEM. First SDTrim.SP simulations (simulation of the W enrichment as a function of ion fluence at different ion energies). (Remarks: This work is mostly done and will be published soon. Some SEM

imaging is still pending. Modelling work using SDTrimSP is ongoing. IPPs ion-beam experiment, HSQ, is presently being refurbished. The plan is to be back in operation in the middle of 2016.)

**Year 2:** Exposure of thin Fe/W films to D ion beams at low energies (100 - 1000 eV) at 500 and 600 K at different ion fluences ( $10^{21}$ - $10^{24}$  D/m<sup>2</sup>) in HSQ device. Erosion of EUROFER by medium to high energy (100eV/D-1keV/D) at 300, 500, 600K for fluences between  $10^{21}$ - $10^{24}$  D/m<sup>2</sup> in HSQ device. Quantification of sputter yields (partial and total yields by IBA and weight loss measurements). Supporting investigations of the surface morphology after ion bombardment by SEM. Investigation of XPS depth profiles for selected samples. Further SDTrimSP simulations. (Remarks: A few of these measurements have already been done, also in collaboration with R. Doerner, PISCES-A. Further experiments will be started if HSQ is back in operation. With respect to recent experiments regarding the temperature dependence the highest temperature for these experiments should be about 800K. Participation in the joint experiment investigating the flux dependence of sputtering, with detailed conditions still to be defined, should be added to the work plan.)

**Year 3:** Additional selected exposures of thin Fe/W films to D ion beams in HSQ device in if necessary. Additional selected exposures of EUROFER by to D ion beams in HSQ device in if necessary. Sputtering of EUROFER and thin Fe/W films by He. Quantification of sputter yields (partial and total yields by IBA and weight loss measurements). Investigation of XPS depth profiles for selected samples. Investigations of the surface morphology after ion bombardment by SEM. Comparison of experimental data with simulation results. Publication of results.

## LICP Lanzhou

**Year 1:** 1) Characterization the microstructure and composition of as-received Chinese CLAM/CLF-1 steels (XRD, XPS, SEM and TEM); 2) Determination of the recommended heat and polishing standard for Chinese CLAM/CLF-1 steels (XRD, XPS, SEM and TEM)

**Year 2:** 1) Characterization the evolution of surface morphology after erosion, determine the erosion depth of steel after lab plasma exposure using coating mark (ECR-plasma, SEM&FIB and TEM); 2) Investigation the D retention and releasing behaviours of CLAM/CLF-1 steel after lab plasma exposure (temperature and fluence dependence) (ECR-plasma, NRA and TDS) 3) Preparation co-deposition layer and measuring the deuterium retention and releasing (TDS) 4) Determination the gas-driven permeation behaviour using CLAM/CLF-1 steel (TDS&GDP)

**Year 3:** 1) Investigation the Deuterium inventory and releasing behaviours of CLAM/CLF-1 steel exposed to EAST Tokamak environments (MAPES, NRA and TDS); 2) Simulation the D permeation and retention behaviour in steel/iron; 3) Comparing the erosion, fuel retention and releasing results obtained from laboratory and EAST Tokamak environments; 4) Comparing the erosion, fuel retention and permeation results obtained from Chinese RAFM steel with EUROFER, RUSFER and F82H

## NNRC Kurchatov Institute

**Year 1:** 1) Investigation of influence of a dense low-energy plasma irradiation on RAFMs morphology. Plasma irradiation will be performed at LENTA facility in Kurchatov institute, Moscow; 2) Investigation of influence of a pulse heat loads on RAFMs morphology (in collaboration with TRINITY, Russia). Pulse heat loading will be performed at QSPA (Quasistationary plasma accelerator) facility, TRINITI, Russia.

**Year 2:** 1) Preparation of RAFMs samples with introduced radiogenic helium up to 80 appm for further investigations (in collaboration with VNIIEF, Sarov, Russian Federation); 2) Preparation of RAFMs samples damaged with high-energy electrons; 3) Preparation of RAFMs samples damaged with high-energy heavy ions (in collaboration with IPP Garching).

**Year 3:** 1) Investigation of the influence of introduced radiogenic helium on deuterium retention in RAFM; 2) Investigation of the influence of preliminary high-energy electron irradiation on deuterium retention in RAFMs; 3) Investigation of the influence of preliminary high-energy heavy ions

irradiation on deuterium retention in RAFMs. Retention will be investigated by TDS method in collaboration with MEPhI, Moscow, Russian Federation.

## **UCSD PISCES**

**Year 1:** 1) Under controlled plasma conditions, measure the S/XB for Fe I, vary  $n_e$  and Te; 2) Under controlled plasma conditions, measure the S/XB for Cr I, vary  $n_e$  and Te; 3) Investigate the temperature dependence of W surface segregation

**Year 2:** 1) Investigate the surface loss rates of each of the components of steel in real time using visible spectroscopy (quantify these losses with the S/XB data mentioned above), vary ion energy, surface temperatures; 2) Investigate the impact of mixed D/He plasma on steel and surface segregation, vary plasma composition, surface temperature; 3) Compare fluence dependent erosion of a variety of RAFM steels in PISCES plasma

**Year 3:** 1) Investigate the possible use of LIBS (under development in PISCES) to investigate real-time surface composition as a function of plasma fluence; 2) Investigate changes to the surface composition after exposure to transients heat pulses using existing laser systems and the aforementioned real-time diagnostic techniques

## **NIFS**

**Year 1:** Bi-directional permeation experiments will be conducted, using hydrogen, deuterium and their mixture, through a ferritic steel alloy: F82H in a laboratory scale steady state plasma facility: VEHICLE-1. Also, W-coated F82H will be used for these permeation experiments

**Year 2:** Experimental data analysis, using a one-dimensional diffusion code: DIFFUSE and its applications to some of the reactor-relevant conditions. Also, experiments to check the surface composition effects on PDP will be conducted because the surface recombination coefficient might change along with preferential sputtering.

**Year 3:** Extension of bi-directional permeation experiments under some of the reactor relevant conditions, using hydrogen, deuterium and their mixture will be done to be compared with the predictions by the DIFFUSE code.

## **SCK-CEN**

**Year 1:** Prepare a set of materials and characterize their microstructure using scanning/transmission electron microscopy (SEM/TEM), nano-indentation testing (NIT), electron back scattering diffraction (EBSD) imaging, destructive mechanical testing and positron annihilation spectroscopy (PAS). Initiate atomistic simulations to assess hydrogen trapping and migration in the samples taking into account the primary microstructural features.

**Year 2:** Perform high flux plasma exposures at Pilot PSI at DIFFER. Construct a kinetic rate theory model on the basis of the earlier atomistic simulations. Perform and analyse thermal desorption spectroscopy (TDS) experiments to clarify the relation between steel microstructure and hydrogen retention.

**Year 3:** Perform dedicated exposures to validate the kinetic rate theory tool and explore the range of its applicability. Characterize the samples post-irradiation by SEM/TEM, EBSD, NIT and PAS techniques. Finalize the research results and prepare input for the CRP final document.

## **IPP Kharkov**

The operational regimes of the QSPA Kh-50, PPA and MPC devices will be adjusted to achieve adequate variation of energy and particles loads to the exposed steel materials. Plasma streams generated by QSPA, PPA, MPC and plasma layers in front of exposed targets will be characterized by spectroscopy (Ne, Te, N impurity), laser interferometry, analysis of ion masses and energies, high-

speed photography, magnetic and electric probes, Rogowski coils, calorimeters, piezo- and pyro-detectors, and other tools.

Experiments on surface modification of different steels will be initiated using multiples irradiation of material surfaces by quasi-steady and pulsed plasma streams with varied plasma loads and for different initial surface temperatures. Plasma-surface interaction will be studied by high speed CCD camera CMOS PCO AG. Surface diagnostics will include optical (MMR-4) microscopy and scanning electron microscopy (JEOL with X-ray analyser LINK), cross-sectional metallography, X-ray diffraction analysis, elemental analysis using laser mass analyzer and profilometry. Microhardness, roughness and weight loss measurements will also be performed.

Cross-links will be established with other CRP members on joint diagnostics of exposed steel samples, material characterization, and preparation of Round Robin tests of reduced activation steel samples.

## **List of Participants**

**Peng Wang**, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, Lanzhou, CHINA

**Wolfgang Jacob**, Max Planck Institute for Plasma Physics (IPP), Garching, GERMANY

**Yoshihiko Hirooka**, National Institute for Fusion Science (NIFS), Toki, JAPAN

**Anna Golubeva**, Fusion Reactor Department, Division of Tokamak Physics, NRC Kurchatov Institute, Moscow, RUSSIAN FEDERATION

**Russel Doerner**, University of California at San Diego, La Jolla, California, USA

**Vadim Maklai**, Institute of Plasma Physics, National Science Center "Kharkov Institute of Physics and Technology" (NSC KIPT), Kharkiv, UKRAINE

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## **Agenda**

### **Wednesday, 9 December**

**Meeting Room: M0E23**

- 09:30 - 10:00 Welcome, introduction of the participants, adoption of the agenda.
- 10:00 - 10:40 Bas BRAAMS, IAEA: CRP and meeting objectives.
- 10:40 - 11:00 *Break.*
- 11:00 - 12:00 Peng WANG, Study of the interaction of RAFM steel with laboratory and EAST plasma conditions.
- 12:00 - 13:30 *Lunch.*
- 13:30 - 14:30 Wolfgang JACOB, Laboratory measurements of RAFM steel erosion and H retention in RAFM steel.
- 14:30 - 15:30 Yoshihiko HIROOKA, Bi-directional hydrogen isotopes permeation through the first wall of a magnetic fusion power reactor.
- 15:30 - 16:00 *Break.*
- 16:00 - 17:00 Anna GOLUBEVA, Retention and permeation of hydrogen isotopes through RAFMs.
- 19:00 *Social dinner.*

### **Thursday, 10 December**

**Meeting Room: M0E23**

- 09:00 - 10:00 Russel DOERNER, Study of plasma interaction with steel surfaces in PISCES.
- 10:00 - 11:00 Vadym MAKHLAI, Modification of steel surfaces exposed by hydrogen/helium plasmas streams simulating fusion reactor conditions.
- 11:00 - 12:00 Dmitry TERYTYEV, Interaction of high flux plasma with reduced activation ferritic steels: experimental and computational assessment in baseline RAFM Eurofer 97 and its advanced grades improved by thermomechanical treatment.
- 12:00 - 13:30 *Lunch.*
- 13:30 - 15:00 All: Initial review on erosion and material evolution.
- 15:00 - 15:30 *Break.*
- 15:30 - 17:00 All: Initial review on hydrogen isotope retention and transport.

### **Friday, 11 December**

**Meeting Room: M0E23**

- 09:00 - 10:30 All: Discussion and meeting report I: Erosion and material evolution.
- 10:30 - 11:00 *Break.*
- 11:00 - 12:30 All: Discussion and meeting report II: Hydrogen isotope retention and transport.
- 12:30 - 14:00 *Lunch.*
- 14:00 - 15:30 All: Work plan and conclusions.
- 15:30 *Close of meeting.*

## **Presentations**

### **Laboratory measurements of RAFM steel erosion and H retention in RAFM steel**

W. Jacob, K. Sugiyama, M. Balden, M. Oberkofler, T. Schwarz-Selinger, U. von Toussaint

Max-Planck-Institute for Plasma Physics (IPP), Boltzmannstr. 2, 85748 Garching, Germany

Dr. Jacob presented the activities of IPP Garching in this CRP. He first summarized the motivation and scientific rationale for considering the use of RAFM steels as a plasma-facing material at the first wall of a future fusion reactor. A practical outcome of this CRP should be to provide a sound data basis to be able to answer the question whether RAFM steel can be used in recessed areas of the first wall. The sputtering yield of pure iron is way too high, but steel is a complicated mixture of different elements and different phases. During sputtering different elements will be sputtered at different rates such that heavier elements enrich at the surface. This phenomenon is known as preferential sputtering (of the lighter atoms) and it leads to a reduction of the total sputtering yield with increasing ion fluence. Since RAFM steels contain small amounts of tungsten (W) a significant reduction of the sputtering yield due to this mechanism is anticipated.

In a first step, the enrichment of W in an iron (Fe) matrix was investigated. Such Fe/W mixed films can be considered a model system allowing to study effects that are anticipated for steel. Thin (200 to 500 nm) Fe/W mixed films were deposited by magnetron sputtering. The composition and the film thickness were determined by ion beam analysis. Layers with W concentrations of 0.7, 1.5, and 4.2 at% were produced and investigated. After bombardment with monoenergetic deuterium (D) ion beams the species-resolved sputtered amounts were again determined from ion beam analysis. The anticipated W enrichment was clearly confirmed in these experiments. The W enrichment was investigated as a function of initial W concentration, ion energy and ion fluence. As anticipated the measured sputtering yield decreases with increasing particle fluence. It seems that the steady state is not yet reached for total D fluences up to  $10^{24}$  D/m<sup>2</sup>. W enrichment is particularly strong at intermediate ion energies (100 to 200 eV). At higher energies (500 and 1000 eV were investigated) the W enrichment and the reduction of total sputtering yield are less pronounced. A higher initial W concentration leads to a stronger reduction of the sputtering yield. First results from simulations with SDTrimSP were shown. The general agreement between simulations and experimental results for the model layers is of the order of 30%.

Some first experiments were carried out at elevated temperatures (partially in collaboration with R. Doerner at UCSD, PISCES-A) to investigate the effect of temperature. At an ion energy of 140 eV/D and a fluence of about  $1.3 \times 10^{24}$  D/m<sup>2</sup> no clear trend was observed for sample temperatures up to 750 K, but for exposure at 870 K a significant increase of the sputtering yield occurred.

The sputtering yield of EUROFER-97 samples was measured for 100, 200, 500 and 1000 eV/D each for fluences up to  $2 \times 10^{24}$  D/m<sup>2</sup> and for 100 and 200 eV to even higher fluences (up to  $8 \times 10^{24}$  D/m<sup>2</sup> for 200 eV). For 1000 eV/D no significant decrease of the sputtering yield with increasing fluence is observed. For 500 eV/D the yield decreases by a factor of 3. It is not clear whether or not the steady state was reached for 500 eV/D. For 100 and 200 eV/D it appears that a steady state of the sputter yield is reached for fluences of about 3 to  $4 \times 10^{24}$  D/m<sup>2</sup>. For 100 eV/D the yield decreases by about a factor of 4 and for 200 eV/D by about a factor of 8. These data are in good agreement with the trends seen in earlier experiments at PISCES-A performed at 140 eV/D.

Sputtering of the model layers as well as of EUROFER-97 steel leads to pronounced modifications of the surface morphology. The observed morphology varies with applied ion energy, particle fluences and sample temperature. It has to be taken into account that these surface morphologies may strongly influence the measured sputtering yields. The influence of surface morphology on measured sputtering yields is a topic that has to be investigated with more detail in future experiments.



## Investigation of sputtering of RAFM steel in PISCES

R. P. Doerner and D. Nishijima

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While reduced activation ferritic martensitic (RAFM) steel such as EUROFER and F82H is a promising candidate for structural material in future fusion reactors, its use as a first wall material is also considered an attractive option. Thus, investigations of RAFM steel as a plasma-facing material have been recently initiated. In previous studies [1, 2], sputtering properties of RAFM steel were explored, where EUROFER and Fe-W layers as a model system for RAFM steel were exposed to D plasma, and sputtering yields were measured from mass loss. It was found that sputtering yields decrease with an increase in D<sup>+</sup> fluence because of W enrichment on the surface due probably to preferential sputtering of low-/mid-Z elements. Since the sputtering yields were determined only from mass loss, details such as time evolution of sputtered elements remain unclear.

In this presentation, the sputtering behavior of F82H (F82H-IEA (Heat#9741) Plate ID 2-20) RAFM steel [3] is investigated using spectroscopy to obtain element-resolved time evolution of sputtering yields. The main elements of F82H are Fe, Cr (8 wt%), and W (2 wt%). An F82H sample (diameter: 25 mm, thickness: 1.5 mm) was exposed to He plasma for 1600 s in PISCES-B with the following parameters: ion flux  $\sim 3 \times 10^{22} \text{ m}^{-2} \text{ s}^{-1}$ , ion energy  $\sim 80 \text{ eV}$ , and sample temperature  $< 673 \text{ K}$ . During the exposure, spectroscopic measurements were carried out. First of all, no W I lines were detected, meaning that W is not sputtered. On the other hand, Fe I and Cr I lines were observed, but the temporal behavior of Fe I and Cr I emission lines was different. Initially, the Cr I line intensity decreased rapidly, while the Fe I line intensity increased in a similar fashion. After this initial behavior, as the fluence of the exposure increased, both lines showed a slow decrease until eventually reaching a stable value for the remainder of the discharge.

To quantify the sputtered flux, the ionization events per photon,  $S/XB$ , values are necessary for the main elements. Both calculated and measured  $S/XB$  values are available for W I lines [4]. For Cr I, only calculated  $S/XB$  values are available in the ADAS database, and thus we experimentally determined values for a multiple-averaged Cr I 427.07 nm line (425.4, 427.4, 428.9 nm) for verification using the same technique described in [4]. Our value,  $S/XB \sim 1$ , at electron temperature  $\sim 10 \text{ eV}$  is in good agreement with ADAS. Since both calculated and experimental values for Fe I lines are currently not available, we will experimentally measure it. This will allow us to perform quantitative and real-time measurements of element-resolved sputtering yields of RAFM steel.

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[2] K. Sugiyama et al., J. Nucl. Mater. 463 (2015) 272

[3] K. Shiba et al., JAERI-Tech 97-038 (1997)

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## Retention and Permeation of Hydrogen Isotopes through RAFMs

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During operation of Fusion Neutron Source (FNS) and Fusion Power Plant materials (both plasma-facing and construction) will be exposed to high heat fluxes and neutron irradiation. Hydrogen isotopes and He will arise in a bulk. The plasma-facing materials will be damaged by plasma irradiation especially intensive at ELMs and disruptions. The damaging may strongly change the microstructure, ability of a material to retain hydrogen and permeability.

Previously hydrogen retention in Rusfer (Ek-181) RAFM steel from gas and gas-driven permeation though Rusfer in transition permeating regime were investigated. Also the influence of damaging by pulse heat loads (QSPA facility, TRINITY), 20 MeV W implantation up to 0.96 dpa (IPP Garching) and plasma irradiation at LENTA facility (NRC “Kurchatov institute”) on deuterium retention from a gas phase was investigated. It was found that at a depth range of 0.2-2  $\mu\text{m}$  W ions implantation causes 50% increment of retention afterwards. Irradiation at QSPA and LENTA facilities (the last one at 600K) caused 2 times decrease of retention at the same depth range.

In frames of the Project it is planned to focus efforts on the investigation of the influence of different damaging on hydrogen retention in RAFM steels. In particular it is planned to investigate the influence of MeV electrons irradiation and of radiogenic He production on deuterium interaction with RAFM steel.

The MeV electrons irradiation is a way to create point defects or small defect clusters. In experiments with tungsten 10 MeV electrons irradiation was performed, followed by deuterium implantation. TDS of damaged W had higher second peak. This allows associating this peak with Frenkel pairs.

For radiogenic He introducing into the samples so-called “tritium trick” will be used. Flat Rusfer and Eurofer samples with dimensions 12x15x1 mm for retention measurements and Rusfer tubes with length of 250 mm and wall thickness of 0.5 mm will be exposed in tritium. Two expositions at 500 and 250°C for 10 hours at 20-50 atmospheres are planned. Then for several months samples stay in a vacuum container until tritium partially decays to  $^3\text{He}$ . After that the samples should be annealed and if necessary afterwards exposed to hydrogen for replacement of tritium as a result of an isotope exchange. The residual radioactivity of one RAFM steel sample should be below 9 Bq (the limit for handling without special defense). The amount of remaining T is proposed to control by radioluminescence (which is a destructive method), the amount of  $^3\text{He}$  – by mass-spectrometry at melting.

Damaged samples is planned to expose in  $\text{D}_2$  gas at a pressure  $10^4$  Pa in a temperature range of RT-700K and then perform TDS measurements of retention to find out the influence of defects introduced into RAFM steel on D retention. Also gas-driven permeation measurements though samples containing radiogenic He are planned.

## **A study on bi-directional hydrogen isotopes permeation through the first wall of a magnetic fusion power reactor**

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The first wall of a magnetic fusion device is generally defined as the enclosure surface that is nearest to the confinement plasma. Up to ITER, the first wall is essentially the vacuum vessel wall, separating the plasma and the environment. As opposed to this, the first wall of a fusion power reactor is the plasma-facing surface of the breeding blanket structure, which is required to be operated at elevated temperatures for efficient heat exchange. To minimize the thermo-mechanical stress, the first wall is often designed to be  $\sim 5\text{mm}$  or even less in most of the recent power reactor studies.

During the steady state operation of a fusion power reactor, the first wall is exposed to  $\text{D}^+/\text{T}^+$  in the edge plasma on one side and to bred  $\text{T}_2$  in the blanket on the other side when a self-cooled breeder such as FLiBe is employed. As a result, these hydrogen isotopes will penetrate the “thin” wall simultaneously in the two counter directions: in one direction by plasma-driven permeation (PDP) and in the other direction by gas-driven permeation (GDP). The D/T flow into the blanket will necessitate an isotope separation system in the fuel loop and also the T flow into the edge plasma will result in an unwanted increase in edge plasma density, namely, particle recycling  $> 100\%$ . Despite its critical

importance, these technical issues associated with the “bi-directional” hydrogen isotopes permeation through the first wall has not been clearly addressed in the fusion community yet.

In the present work, bi-directional hydrogen permeation through a reduced activation ferritic steel alloy: F82H has been studied in the temperature range from 200 to 600°C, using a laboratory-scale steady state plasma facility: VEHICLE-1 [1]. For PDP experiments, the hydrogen plasma density is of the order of  $10^{10}$ /cm<sup>3</sup> and the electron temperature is around 5 eV, and for GDP experiments the gas pressure is varied from 100 to 700Torr, all relevant to the first wall conditions in a magnetic fusion power reactor. Experimental data have been analysed using the DIFFUSE-code [2].

Under these conditions, hydrogen permeation either by PDP or GDP is essentially diffusion-limited. For the first time bi-directional hydrogen permeation through F82H has experimentally been demonstrated in VEHICLE-1. Hydrogen transport parameters including diffusivity, solubility and also surface recombination coefficient have been evaluated from the PDP and DGP experimental data [3]. Future plans include: PDP and GDP experiments using deuterium, and also all these permeation experiments with tungsten-coated F82H.

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[2] M. I. BASKES, “DIFFUSE83” Sandia Rep. SAND83-8231.

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## **Modification of steel surfaces exposed by hydrogen/helium plasmas streams simulating fusion reactor conditions**

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The project is aimed at experimental simulation of plasma–surface interaction of plasma-facing materials relevant to fusion reactor. Particular attention will be paid to the steels of different grades that can be damaging by extreme particle fluxes and energy loads from the repetitive powerful plasma impacts. Experiments are going to be performed in a quasi-steady-state plasma accelerator QSPA Kh-50 (largest and most powerful device of this kind), short-pulsed plasma gun and magnetoplasma compressor. Heat load will be varied in range of (0.01- 2.4 MJ/m<sup>2</sup>).

Plasma stream duration will be changed from 0.005 ms till 0.25 ms. Steady-state plasma exposures will be carried out also in combination with powerful plasma impacts. This proposal focuses on 3 most critical issues relevant to plasma-facing materials of fusion reactor: (1) Characterization of various steel grades with respect to their response to intense plasma pulses and dust production under high flux plasma loads; (2) Modification and alloying of steels under pulsed plasma treatment aimed to improvement it working characteristics. (3) Comprehensive studies of hydrogen/helium retention in reduced activation steels modified by pulsed plasma streams in comparison with virgin materials, i.e. without plasma treatment. Experiments are including comparative studies of irradiation effects by hydrogen and multi-component plasma (H-He mixture, mixtures of light and heavy gases) on surface layer properties and material structure changes. As result of this project fulfillment the comprehensive studies of materials response to the plasma loads: surface damage; plasma contamination by erosion products, dust issues and hydrogen/helium retention in the exposed material will be performed to formulate recommendations for fusion reactor materials and for validation of predictive numerical models.

## **Interaction of high flux plasma with Reduced Activation ferritic steels: experimental and computational assessment in baseline RAFM Eurofer 97 and its advanced grades improved by thermomechanical treatment**

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To contribute to the CRP on "Plasma–Wall Interaction with Reduced Activation Steel Surfaces in Fusion Devices", we intend to perform in-depth computational analysis and validation experiments addressing how the high-flux plasma penetrates in and leads to embrittlement of the structural modifications in sub-surface region. This work will help understanding of mechanisms responsible for hydrogen penetration and retention in the surface depending on plasma-operating conditions (i.e. plasma flux and surface temperature). The experimental work will include investigation of the high flux plasma interaction with the standard grade of the RAFM steel - Eurofer97/T91 and its advanced grades being under the preparation by the novel thermo-mechanical controlled processing at Belgian Nuclear Institute (in collaboration with metallurgical industry - OCAS group). The computational work will consist of atomic scale calculations addressing the interaction of hydrogen and helium with vacancies, dislocation core, grain boundaries.

Several advanced grades have been already produced by applying thermo-mechanical-chemical (TMC) treatment. By performing primary mechanical testing it has been revealed that while preserving acceptable yield strength and ultimate tensile strength, the ductile to brittle transition temperature was shifted down to approximately -140 °C. This grade and two standard 9Cr grades (T91 and Eurofer97) have been selected for the preliminary high flux plasma exposures at Pilot-PSI linear plasma generator in Netherlands. Two target exposure temperatures have been selected, namely: 450 and 1000K. The thermal desorption spectroscopy (TDS) and scanning electron microscopy (SEM) analysis has been performed on as-exposed materials. The TDS has revealed one major release stage at 450K-500K and several minor release stages in the temperature range 700-1000K. In the case of high temperature exposure, the total retention was essentially suppressed (at least one order of magnitude) but the stage at 450K-500K remained. The SEM analysis revealed quite strong surface modifications in a form of slip bands, roughening and rarely observed blister-like surface defects (the nature is still to be confirmed). The surface modification was clearly less evident for the advanced TMC grade.

## **Study the interaction of RAFM steel with laboratory and EAST plasma conditions**

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Two Chinese institutes and one university participate in this coordination research project to study the erosion and fuel retention in RAFM steel exposure to laboratory and EAST plasma conditions. Currently, two different RAFM steels, including Eurofer 97 and CLAM (CLF-1) steel were delivered to Lanzhou institute of chemical physics, and standard polishing and outgassing procedure for CLAM steel were established according to the recommendation from IPP Garching. At the same times, magnetron sputtering was used to produce pure Fe and Cr layer co-deposited with deuterium, and deuterium retention and releasing behaviours were measured before and after deuterium plasma exposure using thermal desorption, and total retained deuterium amount in two co-deposited layers were measured as a function of incident deuterium fluence, D retention in Cr layer is two order of magnitude higher than Fe and no saturation is found in investigated fluence, for Fe co-deposited layer the total retained amount turn to saturate when the incident fluence up to  $10^{22}$  D/m<sup>2</sup>.

Using He ions damaged and undamaged CLF-1 steel samples, the permeation experiments were performed, and the permeability and diffusion coefficient of CLF-1 were achieved firstly, the results show that the defects formed during He implantation plays a role as same as permeation barrier and this effect disappears during cooling down process of permeation measurement, which can be attributed to the defects annealing during heating stage.

In the next year, the erosion and deuterium retention of CLAM steel exposed to linear plasma device will be thoroughly measured and compared with the results obtained from Eurofer, and the deuterium retention in selected samples will be investigated using both TDS and NRA, for the latter, which need to collaborate with the colleagues from IPP Garching. Also the performance of some new developed RAFM steel exposed to EAST plasma conditions will be continuously investigated. And the influence of the heavy ions damaged on the permeation properties of CLF-1 will be evaluated in ASIPP.





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