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

Summary Report of the Third Research Coordination Meeting

IAEA Headquarters, Vienna, Austria 25 – 27 March 2019

> Prepared by C. Hill

March 2019

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Abstract

Eight experts in the field of plasma-wall interaction on steel surfaces together with IAEA staff met at IAEA Headquarters 25 - 27 March 2019 for the Third Research Coordination Meeting of an IAEA Coordinated Research Project on Plasma-wall Interaction with Reducedactivation Steel Surfaces in Fusion Devices. They described progress with their research since the previous project meeting in October 2017, discussed open issues and made plans for a final project report giving details of outcomes of the CRP and for future research work relevant to the project. The proceedings of the meeting are summarized in this report.

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1. Introduction

The IAEA Coordinated Research Project (CRP) on Plasma-wall Interaction with Reduced-activation Steel Surfaces in Fusion Devices ("Steel Surfaces") is intended to enhance the database on erosion, tritium retention and tritium migration processes in fusion-relevant (reduced activation) steel surfaces. The first Research Coordination Meeting (RCM) of the CRP was held in December 2015 and the summary report is available (report INDC(NDS)-0722, August 2016). The second RCM was held in October 2017 and details of this meeting are available as report INDC(NDS)-0754 (March 2018). This previous summary report and the CRP web site provide more information about the background and the objectives of the project. Please see:

https://www-amdis.org/CRP/steel-surfaces/.

The seven research groups represented in the first and second RCMs were joined by the Institute of Plasma Physics, Chinese Academy of Sciences (ASIPP) for the final phase of 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 Tokicity, 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 plan updates are provided in Section 4. A summary of experimental results is given in a table in Appendix 1; the list of participants is in Appendix 2 and the meeting agenda is given in Appendix 3. Summaries of presentations are presented in Appendix 4.

2. Proceedings

Presentation materials for all talks are available through the meeting web page:

https://www-amdis.org/meetings/steel-surfaces-rcm3/.

The meeting was opened by A. Koning, Head of the Nuclear Data Section, and participants introduced themselves. This was followed by a review of the CRP goals and meeting objectives by the scientific secretary C. Hill, Head of the Atomic and Molecular Data Unit. The broad CRP goal is described as "Increased confidence in assessments of the role of steel as a plasma-facing material in DEMO or a Fusion power plant." It is understood that steel is not suitable as a wall material in the regions of very high heat load, but it may be attractive for other regions of the main wall if erosion and tritium retention properties are under control.

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 "Deuterium retention and erosion of CLF-1 and CLAM steels exposed to deuterium plasma", W. Jacob of Max Planck Institute for Plasma Physics, Garching, Germany on "Sputtering of EUROFER: The Role of Roughness", V. Makhlai of Institute of Plasma Physics, National Science Center "Kharkov Institute of Physics and Technology" (NSC KIPT), Kharkiv, Ukraine on "Steel-based materials surface damage and modification under high power plasma exposures", D. Nishijima of University of California at San Diego, La Jolla, USA on "Effect of He on D retention in various RAFM steels", H. Zhou of Institute of Plasma Physics, Chinese Academy of Sciences (ASIPP) on "Investigation of Deuterium Permeation and Retention in RAFM Steel", D. Terentyev of SCK-CEN Belgium Nuclear Research Centre, Mol, Belgium on "Interaction of high flux plasma with Fe and RAFM steels: experimental and computational assessment", N. Ashikawa of National Institute for Fusion Science (NIFS), Toki, Japan on "Surface modification by deuterium and helium bombardments in RAFM", and A. V. Golubeva of NRC "Kurchatov Institute", Moscow, Russia on "Deuterium retention in reduced activation ferritic/martensitic steels (RAFMS) at ELM-like pulse plasma heat loads". Summaries of the presentations are provided in Appendix 3.

The presentations and associated discussions were followed by two broad topical discussion sessions: (1) A review of the data obtained by participants in the CRP and the best way to describe and disseminate

this data; plans were made for a further coordinated comparison exercise involving standardized steel samples.

(2) On the final day participants reviewed the prospects for future work related to the goals of the CRP, including discussions on a future CRP on hydrogen permeation in fusion-relevant materials and discussed the format and content of the project's final report.

3. Discussion and Conclusions

3.1 Data description and online database

A comprehensive review of the data obtained by participants in the CRP was carried out and the relevant experimental parameters identified, with a view to the creation of a database of the results and to inform a further coordinated set of experiments on standardised steel samples (see Section 3.2). The principal parameters to be recorded are:

- Research group (affiliation, contact detail, etc.);
- Steel type (Eurofer(-X), CLAM, CLF-1, F82H, Rusfer, etc.);
- Ion flux (m⁻².s⁻¹);
- Ion fluence (m⁻²);
- Ion energy (eV);
- Sample history (description);
- Sample thickness (mm);
- Sample temperature (K, if defined);
- Sample temperature measurement location (sample backside, frontside, etc.);
- Analysis method (TDS, GDOES, NRA, SEM, TEM etc.)
- Result (sputtering yield, total amount of retained D, etc.)
- Citation (identified by DOI)
- Additional supporting materials (image files, etc.)

An online database hosted at the IAEA, initially private, but to be made open-access upon publication of the data) will be established to receive data from CRP participants and other researchers and to facilitate data aggregation and comparison.

3.2 Coordinated experiments

The meeting proposes a set of coordinated experiments on steel surfaces, CESS: Comparison Experiment for the Sputtering of Steel. The baseline goal will be to compare sputtering yields in different experimental devices. As far as possible, identical samples (with comparable histories) will be exposed to deuterium plasma or ion irradiation under comparable exposure conditions.

Participants

Contact		Experimental device
Anna GOLUBEVA, Kurchatov Institute, Russia	AG	PIM (ion beam)
Wolfgang JACOB, Max Planck Institute for Plasma Physics, Garching, Germany	WJ	SIESTA (ion beam)
Daisuke NISHIJIMA, Center for Energy Research, UCSD, USA	DH	PISCES (linear plasma)

Peng WANG, Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, China	PW	LEPS (linear plasma)
Haishan ZHOU, Institute of Plasma Physics, Chinese Academy of Sciences (ASIPP), Hefei, China	HZ	PREFACE (linear plasma)
Vadym MAKHLAI, Institute of Plasma Physics, Kharkov Institute of Physics and Technology, Ukraine	VM	QSPA (plasma gun) and DMS
Dmitry TERENTYEV, Centre d'etude de l'energie nucleaire (SCK•CEN), Mol, Belgium	DT	

Sample provision and experimental conditions

- Steel samples to be considered:
 - Pure Fe (DT)
 - Eurofer-97 (WJ)
 - Rusfer (AG)
 - CLAM (PW)
 - F82H (NA)
- All samples $10 \times 10 \times 1$ mm or $12 \times 15 \times 1$ mm; mechanical polishing, annealed at 870 K for 1 hr
- Ion energy: 200 eV
- Ion species: D
- Sample temperature (where defined): 450 K
- Ion fluence: $5 \times 10^{24} \text{ D m}^{-2}$
- Ion flux: variable (this will depend on the experimental capabilities of the devices used)

Sample analysis techniques

Certainly:

- Mass loss (to determine sputter yield)
- SEM (surface imaging before and after sputtering)
- TDS (for total retained D amount)

If possible (in decreasing order of priority):

- EBSD (before and after exposure at the same area to look for grain-dependent effects)
- EDX (to check for W enrichment at the surface)
- FIB cross sectioning (+ EDX)
- TEM
- Ion beam analysis for enrichment
- NRA for D depth profiling
- XPS depth profiling (compare before and after exposure to document changes in surface composition)

- Optical spectroscopy (during erosion to identify dynamic changes; PISCES, for example provides this facility)
- GDOES (composition depth profiling, to identify W enrichment at the surface; for example, in the kb of Peng Wang)

Time table

Possible conferences and workshops for sample exchange are:

- 17th International Conference on Plasma-Facing Materials and Components for Fusion Applications (PFMC 2019), Eindhoven, Netherlands: 21 24 May 2019
- 4th International Workshop on Models and Data for Plasma-Material Interaction in Fusion Devices (Mod-PMI 2019), at NIFS, Toki, Japan: 18 20 June 2019
- 14th International Symposium on Fusion Nuclear Technology (ISFNT 2019), Budapest, Hungary: 22 – 27 September 2019
- 19th International Conference on Fusion Reactor Materials (ICFRM 2019), La Jolla, CA, USA: 27 October – 1 November 2019

The expectation is that experimental work and analysis will be complete by the time of the 24th International Conference on Plasma-Surface Interactions in Controlled Fusion Devices (PSI 2020), to be held in Jeju, Republic of Korea from 31 May – 5 June 2020 and that a satellite meeting will be held there or at the 15th International Workshop on Hydrogen Isotopes in Fusion Reactor Materials following it in Daejeon (8 – 10 June 2020).

Assuming a set of meaningful results is obtained, an article will be submitted to a journal such as Nuclear Fusion by the end of 2020, and this will form part of the final report of the CRP.

3.3 Hydrogen retention

A summary of the experiments conducted over the duration of the CRP to March 2019 with respect to hydrogen retention and sputtering yields is given in Appendix 1.

3.4 Hydrogen permeation

In-situ hydrogen permeation measurements through F82H and CLF-1 have been done at NIFS, Japan and ASIPP, China, respectively. For the diffusion coefficient and permeability measured from gasdriven permeation experiments, no significant difference has been found between F82H and CLF-1. When plasma-driven permeation is performed, the permeation flux can be enhanced by orders of magnitude. Surface recombination coefficients for H on steels have been experimentally measured based on steady-state permeation model with a recombination-diffusion regime assumption. The recombination coefficients data have been found to be extremely sensitive to the surface conditions, e.g. impurities and incident ion energy. A systematic work on the measurement of H transport parameters for RAFM under various conditions is highly recommended. Similar permeation studies can be extended to other materials such as W and Cu alloys.

A further CRP on hydrogen permeation in fusion-relevant materials is proposed and the initial scope and considerations are given in the matrix below.

Motivation

- Reduction of uncertainty in solubilities and diffusivities for ITER baseline materials
- A better understanding of local recycling effects at the strike point / first wall regarding retention and permeation of tritium (with a focus on its importance for the tritiation of coolant)
- Assessment of advanced materials with a clear technological perspective

• Determination of the effect of surrogate radiation on hydrogen permeation properties of fusionrelevant materials

Materials

- Tungsten as a plasma-facing material
- Cu / CuCrZr-alloys as used in the monoblock pipes as part of the primary coolant circuit
- Austenitic SS 316 LN as used in pipes under the divertor an in diagnostic port components at the first wall
- RAFM as a plasma-facing material in a future demonstration power reactor

Experimental and theoretical techniques

- gas-driven permeation
- plasma/ion driven permeation
- TDS trapping effect
- e- and p+ irradiation
- FIB / TEM / PAS
- Characterisation of chemical impurities
- Support from modelling, where the tools available are mature enough: TMAP, cluster dynamics to assess irradiation effects, SRIM)
- Parameters of study
- Temperature range
- ion flux range
- energy range

Output

- Transport parameters (diffusivity, solubility)
- Estimation of permeating flux
- Impact on surface recombination and microstructure

It is expected that a Consultancy Meeting will be held later in 2019 to more precisely describe the scope and planned outcomes of the CRP, which will be subject to approval of the IAEA's Coordinated Research Activities administrative committee.

4. Work Plan Reviews

Lanzhou Institute of Chemical Physics

Using metal/deuterium co-deposited layer and helium implanted samples, a deuterium and helium depth profile measurement method was developed by glow discharge optical emission spectroscopy, the results show that GDOES can be used to evaluate the deuterium depth profile up to $20 \ \mu m$ in tungsten, and the total retention amount measured by TDS and GDOES shows the same increasing tendency. But in RAFM steel sample, due to the deuterium concentration is lower than the measurement limitation,

the total deuterium retention amount measured by GDOES shows large scatter compare with the data obtained by TDS.

The erosion of different RAFM steels including CLF-1, CLAM, EUROFER and RUSFER exposed to the same deuterium plasma batch were investigated. Grain dependent erosion morphology development was observed. W-enriched layers about 5 - 10 nm are formed at the surface of all investigated RAFM steels and proved by RBS. Different RAFM steel grades exhibit similar reduction of erosion behavior with increasing fluence. The total sputtering yield of CLF-1 steel samples exposed at temperature from 400 to 900 K shows almost as a constant.

Thermal desorption spectroscopy reveals after deuterium exposure all steel samples show only one broaden releasing peak from 450 to 870 K, and total D inventory is about $10^{18} - 10^{20}$ D/m². Similar D retention behavior of five RAFM steels was found that it decreases with increasing incident fluence and ion energy. Moreover, different cutting treatment along rolling direction leads to different D releasing behaviors.

IPP Garching

- 1. D sputtering studies were continued (see below); He irradiation has not yet been started (lack of time)
- 2. Work on the interdiffusion of Fe and W is published [1]
- 3. SDTrimSP simulations were continued and the code adapted and improve to allow a better simulation of the effect of enrichment. The result of these investigations is, that the experimentally observed reductions of the sputter yield as a function of accumulated fluence cannot be explained by tungsten enrichment alone. We suggest that surface morphology development leading to a continuous increase of surface roughness might substantially contribute to the observed decrease.
- 4. See item 3

The erosion of EUROFER steel by mono-energetic D ions was investigated in the IPP high current ion source SIESTA [2]. For EUROFER-97 a reduction of the sputter yield after high fluence bombardment with 200 eV D ions was found [3]. For higher ion energies the reduction is smaller. The existence of a W-enriched surface layer after D ion bombardment was proven by different ion beam analysis techniques. However, simulations with SDTrimSP (formerly known as TRIDYN) fail to reproduce the experimental observations quantitatively [3]. Surface morphology modifications found after ion exposures hint at a possible influence of surface roughening on the experimentally observed reduction in sputtering yields.

To allow for a modelling of surface roughness effects, the originally 1-dimensional code SDTrimSP was extended into a 3-D version (SDTrimSP-3D) [4]. This new code was benchmarked in dedicated experiments in SIESTA [5] by comparing the sputtering-fluence-dependent changes of the surface morphology of specially prepared 3-D-structured samples with code predictions. The overall agreement between model and experimental results is excellent [5].

The sputtering of EUROFER with D at 200 eV was investigated up to a fluence of 1×10^{24} D m⁻² at sample temperature of 320 K. The measured sputtering yields are in good agreement with published data [3]. SEM imaging after exposure showed a strong, grain-dependent sputtering. Some grains exhibit an extremely structured surface with a relatively regular array of cone-like pillars, while other still look perfectly flat. The rough grains had experienced much more sputtering that the flat grains. In addition, the anticipated enrichment of tungsten was verified by XPS sputter depth profiling. For this series of experiments, a fluence- dependent decrease of the sputter yield by a factor between 3 and 4 was observed. A rough assessment of the contributions of tungsten enrichment and surface morphology development led us to the conclusion, that for the here applied experimental parameters, both effects contributed about equally to the observed decrease.

[1] M. Reisner, M. Oberkofler, S. Elgeti, M. Balden, T. Höschen, M. Mayer, T. F. Silva, "Interdiffusion and phase formation at iron-tungsten interfaces", *Nuclear Materials and Energy* 19, 189-194 (2019)
[2] R. Arredondo *et al.*, "SIESTA: Ahigh current ion source for erosion and retention studies", Rev. Sci Instrum. Meth. 89, 103501 (2018)

[3] K. Sugiyama *et al.*, "Erosion of EUROFER steel by mass-selected deuterium ion bombardment", Nucl. Mater. Energy **16** 114–122 (2018)

[4] Udo von Toussaint et al., unpublished

[5] R. Arredondo et al., unpublished

NNRC Kurchatov Institute

Sputtering of Rusfer and Eurofer RAFMS was studied at 100 D⁺ plasma irradiation in a fluence range of $3 \times 10^{24} - 7 \times 10^{25}$ D/m² at 443 K. The main results are:

- 1. Results of weight loss measurements for Rusfer and Eurofer sputters at the same conditions are very close.
- 2. On the surface of both materials comb or ridges-like structure grow with a fluence.
- 3. The height of growing structures is 2.5 4 times higher at Eurofer surface at the same conditions.
- 4. The structures growing up contain fewer amounts of Fe and Cr and enriched with W as compared with bare material.

I. Samples of 4 RAFMS (Rusfer 2 mm thick, Eurofer 2 mm thick, CLAM 3 mm thick, F82H 1 mm thick) were simultaneously irradiated by pulsed high heat deuterium plasma loads of 0.3 MJ/m^2 , 1 ms and 0.6 MJ/m^2 , ms. The microstructure changes and D retention in samples were analyzed. The main results are:

- 1. At D plasma loading 0.3 MJ/m², 1 ms, 5 pulses RAFMS surfaces are flat, covered with crystallines of a size $\leq 1 \mu m$. Cracks can be found at surfaces of all materials and seems to be more pronounced at Rusfer surface.
- 2. After D plasma loading 0.6 MJ/m², 1 ms, 1 and 5 pulses surfaces of Eurofer and F82H are flat wile surfaces of Rusfer and CLAM are waved. Surfaces of all RAFMS consist of crystallines of a size $\leq 1 \mu m$.
- 3. Retention in Rusfer is higher than retention in Eurofer and CLAM and much higher than retention in F82H. Reason for smallest retention in F82H may be the fact it was the thinnest sample.
- 4. The maximum level of retention 1×10^{21} D/m² was reached in Rusfer at plasma loading above melting (0.6 MJ/m², 1 ms)
- 5. At pulsed D plasma irradiation retention in Rusfer and Eurofer is significantly higher if surface was melted.

IPP NSC KIPT Kharkov, Ukraine

The most important results of these studies can be summarized as follows: Experimental research on surface modifications of different stainless-steel (SS) grades was performed. These SS-grades are a possible options as plasma-facing materials of the DEMO-reactor first wall. The samples of the Cr₁₈Ni₁₀Ti austenite, Eurofer and Rusfer (Cr-9.7%, W-%1, Fe-base) ferritic/martensitic-steels covered by tungsten coatings were treated within the QSPA Kh-50 quasi-stationary plasma accelerator and the MPC magneto-plasma compressor. The heat load upon the sample surface was near the melting threshold (i.e. about 0.6 MJ/m², with pulse duration $\tau = 0.25$ ms in the QSPA Kh-50, and about 0.4 MJ/m², with pulse duration $\tau = 20 \ \mu s$ in the MPC). The preliminary tungsten coatings were deposited by a PVD method. The sputtering tests of the modified surfaces were also performed.

The two cycles of the plasma treatment were performed. During the first stage of such a cycle the tungsten coating of $1-2 \mu m$ in thickness should be deposited on the sample surface by the PVD method. During the second stage the coated samples should be processed with pulsed plasma streams. Possibility of the alloying of SS-surfaces with tungsten coatings was demonstrated. An increase in the tungsten concentration was observed. The tungsten phase was identified together with some lines of the Fe phase upon the treated surfaces. The presence of the α -Fe phase created good conditions for the tungsten penetration into the affected layer. The tungsten concentration achieved several wt% in the surface layer

of thickness up to 4 μ m. The maximum tungsten content of about 85 wt% (first cycle) and 27.46 wt% (second cycle) were observed in the surface layers of the Eurofer samples modified by plasma streams in the QSPA. As a result of the stresses development, some delamination of the coatings during the pulsed plasma irradiation was observed. The surface morphology was changed mostly by the melting and resolidification of a surface layer. Macro- and micro-cracks appeared also on the modified surfaces. The sputtering tests of modified surfaces were performed. Mass losses of modified Eurofer and Rusfer decreased by up to 20 %. The sputtering yield (mass losses) of samples modified by plasma streams decreased due to increasing of tungsten concentration in modified surface layer. Hydrogen outgassing from SS Cr18Ni10Ti samples saturated in steady state discharges (ion energy 0.7 keV, fluence 6×10^{24} ions/m²) and in the plasma accelerator QSPAKh-50 (ion energy 0.4 keV, fluence 5×10^{24} ion/m²) is close in value. Modified layer formation leads to essential decrease of hydrogen saturation.

Center for Energy Research (CER), University of California at San Diego

Various RAFM steels (CLF-1, Eurofer, F82H, and Rusfer) were exposed to pure D, sequential He and D, or simultaneous D and He mixed plasmas in the PISCES-A linear plasma device. The D retention in each sample was then measured using thermal desorption spectroscopy (TDS). The strong material dependence (~30 times difference between CLF-1 and F82H) and the counter-intuitive fluence dependence (a decrease in D retention with increasing the D fluence) were obtained after the pure D plasma exposure. It was shown that both dependence can be explained by the difference in the Cr content in the near-surface region: a higher Cr concentration in the near surface region leads to a higher D retention. Note that a Cr-rich surface layer was formed during outgassing at 773 K for 1 hour before the plasma exposure. The sequential He and D plasma exposure reduced the D retention compared to the pure D plasma exposure, since the Cr-rich surface layer is removed during the first pure He plasma exposure phase. High-density He bubbles were developed in the near-surface region due to the simultaneous D and He mixed plasma exposure, which are considered to reduce the D retention in the same way as for W.

SCK-CEN, Belgian Nuclear Research Centre, Mol, Belgium

The group at SCK-CEN was working on retention in pure Fe and baseline Eurofer-2 steel and optimized Eurofer (to achieve a lower ductile to brittle transition temperature). Several exposures to the high flux deuterium plasma were performed in Differ (Netherland). The exposure temperature was 450K and fluence $5 \times 10^{25} - 10^{27}$ D/m². The materials were studied by thermal desorption spectroscopy (TDS) and scanning electron microscopy and transmission electron microscopy (TEM) 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. Plastic deformation applied to pure Fe resulted in the increase of the trapping of H released at stage I, but also invoke the appearance of the second release stage around 650K. It has been discussed that this release stage is probably due to the pinning at the sub-grains formed as a result of the heavy plastic deformation. 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) in pure Fe and conventional RaFM steels. The surface modification was clearly less evident for the advanced TMC grade.

To complement the experimental studies, the atomic-scale computational analysis was performed to characterize the residence of H atoms in bcc Iron matrix and its self-interaction, interaction with vacancies and dislocations. It has been revealed that H exhibits rather weak self-interaction (binding energy of 0.22 eV), and the formation of multiple hydrogen clusters is not conducted by the release of self-interstitial Fe thus creating the thermally stable Hn-vacancy complex – contrary to the situation with Helium. The interaction energy with screw and edge dislocation was found to be 0.27 and 0.47 eV, respectively. However, the energy landscape of corresponding to the Hn cluster on the dislocation line neither revealed a possibility of the transformation of such complex into the stable cluster accommodated with the emission of kinks. Finally, the binding of H and Hn clusters with a single vacancy was assessed. The binding of a single H amounts to 0.62 eV, and two more H atoms are

bound without losing the binding strength. Fourth and fifth atoms can be further added, but the corresponding binding energy goes down to 0.2 eV. The addition of the sixth atom is no longer favourable. Hence, the growth of Hn cluster on vacancy or dislocation line should occur given the external source of free vacancies, which could be supplied thanks to intensive plastic deformation of the sub-surface region under high flux plasma exposure.

The Eurofer97-2 fully characterized grade was provided to Kurchatov and Kharkov Institutes for complimentary experiments involving plasma and heat load exposures to benchmark the performance of baseline RAFM steels.

Institute of Plasma Physics, Chinese Academy of Sciences (ASIPP)

1) Deuterium (D) transport parameters in a Chinese RAFM material: CLF-1 have been experimentally measured. D diffusivity, permeability and solubility in CLF-1 have been evaluated using gas-driven permeation (GDP) method. D plasma-driven permeation (PDP) experiments have been performed as well in a temperature range of 600 - 750 K. The PDP flux decreases with the increase of ion incident energy, due to the enhancement of D recombination coefficient at the upstream surface. The recombination coefficients can increase by 2 orders of magnitude when the sample bias is changed from 0V to -100 V.

(2) Helium (He) effects on surface morphology and D retention have been investigated. For surface morphology studies, polished F82H steel samples have been exposed to He plasmas with an ion incident energy of ~80 eV at 773 - 873 K to three exposure fluences: 6×10^{24} , 1×10^{25} and 4×10^{25} He m⁻². The RAFM steel surface is strongly modified by the low energy He plasma exposure. The W-enriched structures can be destroyed by the sequential D plasma exposure in tokamak, leading to the increase of physical sputtering yields of RAFM. High-energy He ion is pre-implanted into RAFM using an accelerator to study its effects on D retention. He-damaged layers at the surface and inside the material bulk are induced by 3.5 MeV He ion bombardments with and without energy degrader, respectively. Deeply injected He has been found to act as a permeation barrier and reduce D bulk retention.

(3) D plasma-driven permeation through a monoblock-type plasma-facing component (PFC) mock-up made by tungsten (W) and RAFM has been tested. First-of-a-kind experiments on hydrogen isotope transport through ITER-like PFC mock-ups have been demonstrated in a linear plasma facility. W armor with gaps cannot stop hydrogen isotopes permeation through RAFM into the coolant effectively. The RAFM material can act as T permeation "short cut", resulting in faster penetration speed and higher permeation flux than expected, which then may raise more complicated T contamination issue to the coolant.

5. Future Work

Lanzhou Institute of Chemical Physics

1. Continue the study of measuring the deuterium and helium retention in steel samples using glow discharge optical emission spectroscopy, comparing the deuterium depth profile in steel sample with non-exposed one.

2. Using iron and helium ions to irradiate the CLF-1 and CLAM samples to different damage level and expose to deuterium plasma, comparing the influence of irradiation damage on deuterium retention.

Center for Energy Research (CER), University of California at San Diego

1. The time evolution of the emission intensity from sputtered Cr and O atoms during plasma exposure to further clarify the effect of a Cr-rich surface layer as well as an oxide layer on the D retention;

2. In-situ LIBS (laser-induced breakdown spectroscopy) measurement of the surface composition evolution during plasma exposure;

3. Impurity (Ne, Ar, and N₂) effect on the D retention.

Institute of Plasma Physics, Chinese Academy of Sciences (ASIPP)

1. Studies on D plasma-driven permeation through steel will be continued. More systematic work will be done to evaluate the surface recombination coefficient under various plasma conditions, which is extremely important for the designs of blankets for CFETR. At the meantime, the target material will be extended to advanced materials like ODS-RAFM.

2. Efforts will be put into the R&D of feasible technology to fabricate T permeation barrier in the cooling channels of blanket and divertor of CFETR. The T permeation barrier will be made on the surface of steels and tested in linear plasma devices.

3. Hydrogen isotope retention in neutron-damaged RAFM will be studied. Recently this research has been funded by National Magnetic Confinement Fusion Science Program of China. International collaboration between ORNL in US and ASIPP has been proposed.

IPP Garching

1. Sputtering of EUROFER by He using the high current ion source SIESTA.

2. Participate in a coordinated activity to compare sputtering of different grades of RAFM steels (Exposure under well-defined experimental conditions in SIESTA).

IPP NSC KIPT Kharkov, Ukraine

Future activity in IPP NSC KIPT Kharkov, Ukraine will be focused on the following issues: 1. Comprehensive experimental studies of combined stationary and transient plasma interactions with various steel grades will be performed. Influence of consecutive irradiation of steels by the pulsed and stationary plasma streams will be evaluated.

2. Continuation of studies on hydrogen/helium retention (outgassing) in steels modified by pulsed plasma streams and in comparison with not modified materials.

NIFS, Toki-city, Japan

We intend to study D retention and interactions by helium bombardment,

Using literatures, expanding the plasma exposure conditions to collect data. For example, higher fluence, higher bombardment energy, so on. Served topics are such as follows.

1. D retention after D plasma exposure with and without He bombardments

- 2. Surface morphology of D plasma exposure and He plasma exposure
- 3. Compositions at the top surface region, which is specially tungsten enrichment by select sputterings.

In addition, we consider to ask additional produce of authorized F82H alloy by QST. Measurements of retained tritium will be planned in the future, but this will be started after 2020 (after starting JT-60SA).

NNRC Kurchatov Institute

1. Continued study of influence of damage by ELMs-like pulsed plasma heat loads on D retention in RAFMS. Plasma loads will be implemented 0.3 MJ/m2 and 0.6 MJ/m2 with a duration of 1 ms. Number of pulses will achieve 50. Microstructure will be analyzed at different stages of experiment. Fist retention after plasma loads will be investigated, second retention after gas exposure and plasma irradiation of pre-damaged samples.

2. Study of selective sputtering of CLAM and F82H irradiated with 100 eV D+ plasma at 443 K at fluencies in a range $3\times1024 \div1026$ D/m2.

3. Comparative study of temperature dependencies of selective sputtering of RAFMS (fixed fluence, varied temperature).

4. Comparative study of D retention in RAFMS at high pressures (1-24 atmospheres, 24 hours, elevated temperatures).

5. Comparative study of gas- and plasma-driven deuterium permeation through different RAFMS.

Topics 1-4 prolong works in collaboration with CRP colleagues for 2 years or more.

Topic 5 is a new one which will also go in collaboration with colleagues from RAFMS CRP.

SCK-CEN, Kurchatov, KIPT

Several baseline RAFM steels, developed in EU, Japan, Russia and China, have been exposed to high flux deuterium plasma. The exposures were performed to assess the behaviour of the steels in extreme conditions below and above the melting threshold, which could be met in the case of disruption on the first wall. The results of the exposures clearly show microstructural differences in the response of the steels when the deposited heat exceeds the melting threshold. RUSFER grade appeared to have most severe damage in terms of crack density, while F82H exhibits the least affected surface. The measurements of D retention have also shown a strong dependence on the type of steel grade. After the over-melting exposures, the highest retention was observed in RUSFER and the lowest one (one order of magnitude !) in F82H. Although four selected steel grades have essentially similar chemical composition and initial microstructure, their fabrication routes and heat treatments differ which eventually should explain the variation in the response under the plasma exposure.

To understand the correlation between the initial material microstructure with the resulting surface modification and retention, we propose to perform an in-depth microstructural analysis of the reference, thermally annealed (to mimic TDS cycle) and plasma exposed materials. The analysis will be done to assess the impact of thermal annealing and plasma exposure on the structure of lath boundaries, pocket boundaries, prior austenite boundaries, precipitate morphology and dislocation density. To clarify the penetration of the plasma-induced microstructural changes depth-dependent EBSD (electron back scatter diffraction) scans will be performed parametrically to all the exposed samples. To assess the impact of the exposures on the strength of the material, nano-indentation and micro-hardness tests will also be performed.

The presented work plan will involve close collaboration with other CRP participations, and given the considerable number of samples to be investigated, it puts a perspective for another 2-3 years work.

Appendix 1: Summary of Experiments

Contact Name	Steel Type	Ion Flux /m ⁻² .s ⁻¹	Ion Fluence /m ⁻²	Ion Energy /eV	Sample Temperature /K	Retained Deuterium /m ⁻²	Sputteri ng Yield
Terentyev	Eurofer-2 Bohler	8E+23	5E+25	50.0	450.0	_	_
Nishijima	CLF-1	1.5e+21 - 2e+21	1E+25	100.0	373.0	1.3E+21	_
Nishijima	Eurofer	1.5e+21 - 2e+21	1E+25	100.0	373.0	7.87E+19	_
Nishijima	F82H	1.5e+21 - 2e+21	1E+25	100.0	373.0	4.5E+19	_
Nishijima	Rusfer	1.5e+21 - 2e+21	1E+25	100.0	373.0	7.99E+20	_
Nishijima	CLF-1	1.5e+21 - 2e+21	1E+25	100.0	523.0	4.04E+19	_
Nishijima	Rusfer	1.5e+21 - 2e+21	2E+23	100.0	373.0	6.5E+21	_
Nishijima	Rusfer	1.5e+21 - 2e+21	2E+24	100.0	373.0	1.3E+21	_
Nishijima	Rusfer	1.5e+21 -2e+21	4E+25	100.0	373.0	1E+21	_
Zhou	CLF-1	1E+21	6E+23	3.0	550.0	2.55E+20	_
Zhou	CLF-1	6.1E+20	6E+23	5.0	570.0	1.44E+20	_
Jacob	Eurofer EUROFER-97-2 heat 993 393	5E+19	5E+23	200.0	320.0	_	0.015
Jacob	Eurofer	5E+19	9E+23	200.0	320.0	_	0.008
Jacob	Eurofer	5E+19	1E+24	200.0	320.0	_	0.006
Jacob	Eurofer	1E+19	1E+24	100.0	320.0	—	0.004
Jacob	Eurofer	1E+19	1E+24	500.0	330.0	-	0.02
Jacob	Eurofer	1E+19	1E+24	1000.0	350.0	_	0.05
Wang	CLF-1	2E+21	7E+24	180.0	450.0-470.0	_	0.02
Wang	CLAM	2E+21	7E+24	180.0	450.0 - 470.0	_	0.016
Wang	EUROFER97	2E+21	7E+24	180.0	450.0-470.0	_	0.012
Wang	Rusfer	2E+21	7E+24	180.0	450.0 - 470.0	_	0.007
Wang	CLF-1	2E+21	7E+24	70.0	450.0-470.0	_	0.004
Wang	CLAM	2E+21	7E+24	70.0	450.0-470.0	_	0.0032
Wang	EUROFER97	2E+21	7E+24	70.0	450.0 - 470.0	_	0.0028

Contact Name	Steel Type	Ion Flux /m ⁻² .s	Ion Fluence /m ⁻²	Ion Energy /eV	Sample Temperature /K	Retained Deuterium /m ⁻²	Sputteri ng Yield
Wang	Rusfer	2E+21	7E+24	70.0	450.0 - 470.0	_	0.0023
Wang	CLF-1	2E+21	2E+25	180.0	450.0 - 470.0	1.5E+18	_
Wang	CLAM	2E+21	2E+24	180.0	450.0 - 470.0	2.4E+18	_
Wang	EUROFER97	2E+21	7E+24	180.0	450.0 - 470.0	5E+18	_
Wang	Rusfer	2E+21	7E+24	180.0	450.0 - 470.0	2E+18	-
Wang	CLF-1	2E+21	7E+24	70.0	450.0 - 470.0	4.5E+18	_
Wang	CLAM	2E+21	3E+24	70.0	450.0 - 470.0	1.4E+19	-
Wang	EUROFER97	2E+21	7E+24	70.0	450.0 - 470.0	2E+18	_
Golubeva	Eurofer	3E+21	3e+24 - 7.3e+25	100.0	443.0	_	0.0125 - 0.0052
Golubeva	Rusfer	3E+21	3e+24 - 7e+25	100.0	443.0	—	$0.087 \\ -0.067$
Golubeva	Rusfer	1e+23 – 1e+24 [D pulsed high heat plasma loading, 0.3 MJ/m2, 1 ms]	in 1 pulse big uncertainty; 1 and 5 pulses (see "ion flux" column)	20.0 - 30.0	below melting	1e+20 - 3.7e+20	_
Golubeva	Eurofer	1e+23 – 1e+24 [D pulsed high heat plasma loading, 0.3 MJ/m2, 1 ms]	in 1 pulse big uncertainty; 1 and 5 pulses (see "ion flux" column)	20.0 - 30.0	below melting	1.3e+20 - 1.5e+20	_
Golubeva	F82H	1e+23 – 1e+24 [D pulsed high heat plasma loading, 0.3 MJ/m2, 1 ms]	in 1 pulse big uncertainty; 1 and 5 pulses (see "ion flux" column)	20.0 - 30.0	below melting	4.8e+19 - 1.1e+20	_
Golubeva	CLAM	1e+23 – 1e+24 [D pulsed high heat plasma loading, 0.3 MJ/m2, 1 ms]	in 1 pulse big uncertainty; 1 and 5 pulses (see "ion flux" column)	20.0 - 30.0	below melting	1.8e+20 -2.3e+20	_

Contact Name	Steel Type	Ion Flux /m ⁻² .s ⁻	Ion Fluence /m ⁻²	Ion Energy /eV	Sample Temperature /K	Retained Deuterium /m ⁻²	Sputteri ng Yield
Golubeva	Rusfer	1e+23 – 1e+24 [D pulsed high heat plasma loading, 0.6 MJ/m2, 1 ms]	in 1 pulse big uncertainty; 1 and 5 pulses (see "ion flux" column)	20.0 - 30.0	above melting	9.6e+20 - 1e+21	_
Golubeva	Eurofer	1e+23 – 1e+24 [D pulsed high heat plasma loading, 0.6 MJ/m2, 1 ms]	in 1 pulse big uncertainty; 1 and 5 pulses (see "ion flux" column)	20.0 - 30.0	above melting	2e+20 - 8e+20	_
Golubeva	F82H	1e+23 – 1e+24 [D pulsed high heat plasma loading, 0.6 MJ/m2, 1 ms]	in 1 pulse big uncertainty; 1 and 5 pulses (see "ion flux" column)	20.0 - 30.0	above melting	5.6e+19 - 7.8e+19	_
Golubeva	CLAM	1e+23 – 1e+24 [D pulsed high heat plasma loading, 0.6 MJ/m2, 1 ms]	in 1 pulse big uncertainty; 5 pulses (see "ion flux" column)	20.0 - 30.0	above melting	2.3E+20	_
Golubeva	Rusfer	_	_	_	600.0	7.6e+20 - 9e+20	_
Makhlai	Eurofer	1E+22	1E+26	30.0 - 1500.0	500.0	_	0.0037
Makhlai	Eurofer	1E+22	1E+26	30.0 - 1500.0	500.0	_	0.0034
Makhlai	Eurofer	1E+22	1E+26	30.0 - 1500.0	500.0	—	0.0029
Makhlai	Rusfer	1E+22	1E+26	30.0 - 1500.0	500.0	_	0.0027
Makhlai	Rusfer	1E+22	1E+26	30.0 - 1500.0	500.0	_	0.0024

Appendix 2: List of Participants

List of Participants

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Agenda

25 – 27 March 2019

Meeting room: M0E24

Monday, 25 March 2019

09:30 – 10:00	Arjan KONING, Christian HILL, <i>IAEA</i> : Welcome, introduction of the participants, adoption of the agenda
10:00 - 11:00	Peng WANG , Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences, China
	Deuterium retention and erosion of CLF-1 and CLAM steels exposed to deuterium plasma
11:00 – 12:00	Wolfgang JACOB , Max Planck Institute for Plasma Physics (IPP), Garching, Germany
	Sputtering of EUROFER: The Role of Roughness
12:00 – 14:00	Lunch
14:00 - 15:00	Vadym MAKHLAI, Institute of Plasma Physics, National
	Science Center "Kharkov Institute of Physics and Technology", Ukraine
	Science Center "Kharkov Institute of Physics and Technology", Ukraine Steel-based materials surface damage and modification under high power plasma exposures
15:00 – 16:00	 Science Center "Kharkov Institute of Physics and Technology", Ukraine Steel-based materials surface damage and modification under high power plasma exposures Daisuke NISHIJIMA, Center for Energy Research, UCSD, United States of America
15:00 – 16:00	 Science Center "Kharkov Institute of Physics and Technology", Ukraine Steel-based materials surface damage and modification under high power plasma exposures Daisuke NISHIJIMA, Center for Energy Research, UCSD, United States of America Effect of He on D retention in various RAFM steels
15:00 – 16:00 16:00 – 17:00	 Science Center "Kharkov Institute of Physics and Technology", Ukraine Steel-based materials surface damage and modification under high power plasma exposures Daisuke NISHIJIMA, Center for Energy Research, UCSD, United States of America Effect of He on D retention in various RAFM steels Haishan ZHOU, Institute of Plasma Physics, Chinese Academy of Sciences (ASIPP), China

Tuesday, 26 March 2019

09:00 - 10:00	Dmitry TERENTY EV , SCK•CEN, Belgium
	Interaction of high flux plasma with Fe and RAFM steels: experimental and computational assessment
10:00 - 11:00	Naoko ASHIKAWA, National Institute for Fusion Science (NIFS), Japan
	Surface modification by deuterium and helium bombardments in RAFM
11:00 – 12:00	Anna GOLUBEVA, Kurchatov Institute, Russia
11:00 - 12:00	Anna GOLUBEVA, <i>Kurchatov Institute, Russia</i> Deuterium retention in reduced activation ferritic/martencitic steek (RAFMS) at ELM-like pulse plasma heat loads
11:00 – 12:00 12:00 – 14:00	Anna GOLUBEVA, <i>Kurchatov Institute, Russia</i> Deuterium retention in reduced activation ferritic/martencitic steeks (RAFMS) at ELM-like pulse plasma heat loads Lunch

Wednesday, 27 March 2019

09:00 - 11:00	Discussion session 2 (all participants): plans for future coordinated experiments on standardized steel samples
11:00 – 11:30	Coffee Break
11:30 – 12:30	Discussion sessions 3 (all participants): outline of the meeting report and related publications; Adjournment of the meeting

Appendix 4: Presentations

Presentations

All presentation materials are available on the web page for this meeting: <u>https://www-amdis.org/meetings/steel-surfaces-rcm3/</u>

Surface modification by deuterium and helium bombardments in RAFM

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Reduced Activation Ferritic/Martensitic (RAFM) steels, such as F82H and EUROFER, are candidate materials for fusion DEMO reactor. To understand bulk fuel retention and tritium inventories of plasma-facing materials in DEMO, analyses of specimens exposed to deuterium plasmas are essential. In this study, RAFM steel specimens are exposed to low energy deuterium and helium plasmas, and variations of surface modifications and compositions are elucidated.

RAFM steels, F82H (8Cr-2W) and EUROFER (9Cr-1W), are bombarded with steady-state deuterium plasmas under conditions relevant to the first wall environment using the PlaQ facility [1, 2]. The surface temperature of the specimens during plasma exposure was measured by thermocouples and an infrared camera. It was set at 450 K. Target steels were exposed to helium pre-irradiation applied a DC-bias voltage of 200V and deuterium plasma bombardment applied a DC-bias voltage of 100V. Applied deuterium and helium fluences are 1×10^{24} D/m² and of the order of 10^{24} He/m², respectively. After the plasma exposures, specimens were analyzed with nuclear reaction analysis (NRA), microbalance, Rutherford backscattering spectroscopy (RBS), scanning electron microscopy (SEM).

Deuterium retention in the steels was measured by NRA, which was using D (He³, p) ⁴He reaction at different energies, 690 keV, 1200 keV, 1800 keV, 2400 keV, 3200 keV and 4000 keV, respectively. Amounts of deuterium retention are of the order of 10^{18} to 10^{19} D/m² at the near top surface region using an energy of 690 keV. Target specimens with helium pre-irradiation show higher deuterium retentions to compare with without helium pre-irradiations. The difference between F82H and EUROFER is almost negligible.

Surface morphologies analyzed by SEM. After deuterium plasma bombardment, a smooth plane shown. From microbalance measurement, weight loss of 10 μ g per 1 cm² was observed. But a surface on the target after helium pre-irradiation shows pin-holder like un-uniform structures. A weight loss after helium irradiation is about 60 μ g per 1 cm² and then an erosion rate by helium irradiation is higher than that after deuterium irradiation.

From cross-section SEM observation after a focus ion beam (FIB) treatment, coral structures are observed on a plasma facing side. Each length of coral structure is less than 200 nm. In the cross-section image, the plasma facing side show tungsten enrichment during the thickness of 5 nm from the top surface, measured by an energy dispersive X-ray spectrometry.

- [1] A. Manhard, et al., Plasma Sources Sci. Technol. (2011) 20 015010.
- [2] N. Ashikawa, et al., Fusion Eng. and Design, 112(2016) 236.

The authors would like to thank Dr. K. Sugiyama for NRA analysis, and Mr. J. Yagyu, Drs. M. Sato, T. Nakano, M. Fukumoto, H. Kubo and A. Sakasai in QST, Naka, for their material preparation.

Deuterium retention in reduced activation ferritic/martenitic steels (RAFMS) at ELM-like pulse plasma heat loads

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The reduced activation ferritic-martencitic steels (RAFMS) are promising structural materials for fusion. Some authors also propose to use RAFMS as plasma-facing materials in areas with relatively low plasma and heat loadings. In the present work the 2 mm thick samples of Rusfer (EK-181) and Eurofer RAFMS were irradiated at QSPA-T facility by ELM-like pulse deuterium plasma. Two levels of heat loads on the samples were selected: 0.3 MJ/m^2 and 0.6 MJ/m^2 . The pulse duration was $\sim 1 \text{ ms}$, number of pulses was in a range of 1 and 5. At the plasma heat loading of 0.3 MJ/m^2 surface layers of RAFMS was not melted but cracks appeared at surfaces of both materials. The density of cracks at Eurofer surface is about twice lower. After loading with 0.6 MJ/m^2 the surface of Rusfer samples is waved. Deuterium retention in RAFMS after plasma irradiation was investigated by thermodesorption measurements. If surface layer melts (at loading with 0.6 MJ/m^2) deuterium retention in RAFMS is several times higher than at loading below melting threshold (0.3 MJ/m^2). The maximum amount of deuterium retained in RAFMS samples was 10^{21} D/m^2 .

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Sputtering of EUROFER: The Role of Roughness

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The lifetime of a plasma-facing component is determined by sputtering due to the bombardment with energetic particles. At the first wall, the impinging particle flux is dominated by charge-exchange neutral hydrogen isotopes with energies below about 200 eV, but comprising also small fractions of high-energy species with energies up to several keV. RAFM steels contain small amounts of tungsten. Upon exposure to energetic hydrogen ions from the plasma, a W-enriched layer will be formed at the surface due to preferential sputtering. Such a W-enriched layer could lower the erosion rate to acceptable levels. We report on simulations and experiments aiming at quantifying the achievable reduction of erosion rates. In particular, we want to study the influence of surface roughening on sputtering.

The erosion of EUROFER steel by mono-energetic D ions was investigated in the IPP high current ion source SIESTA [1]. For EUROFER-97 a reduction of the sputter yield after high fluence bombardment with 200 eV D ions was found [2]. For higher ion energies the reduction is smaller. The existence of a W-enriched surface layer after D ion bombardment was proven by different ion beam analysis techniques. However, simulations with SDTrimSP (formerly known as TRIDYN) fail to reproduce the experimental observations quantitatively [2]. Surface morphology modifications found after ion exposures hint at a possible influence of surface roughening on the experimentally observed reduction in sputtering yields.

To allow for a modelling of surface roughness effects, the originally 1-dimensional code SDTrimSP was extended into a 3-D version (SDTrimSP-3D) [3]. This new code was benchmarked in dedicated experiments in SIESTA [4] by comparing the sputtering-fluence-dependent changes of the surface morphology of specially prepared 3-D-structured samples with code predictions. The overall agreement between model and experimental results is excellent [4].

The sputtering of EUROFER with D at 200 eV was investigated up to a fluence of 1×10^{24} D m⁻² at sample temperature of 320 K. The measured sputtering yields are in good agreement with published data [2].SEM imaging after exposure showed a strong, grain-dependent sputtering. Some grains exhibit an extremely structured surface with a relatively regular array of cone-like pillars, while other still look perfectly flat. The rough grains had experienced much more sputtering that the flat grains. In addition, the anticipated enrichment of tungsten was verified by XPS sputter depth profiling. For this series of experiments, a fluence- dependent decrease of the sputter yield by a factor between 3 and 4 was observed. A rough assessment of the contributions of tungsten enrichment and surface morphology development led us to the conclusion, that for the here applied experimental parameters, both effects contributed about equally to the observed decrease.

[1] R. Arredondo et al., "SIESTA: A high current ion source for erosion and retention studies", Rev. Sci Instrum. Meth. **89**, 103501 (2018)

[2] K. Sugiyama et al., "Erosion of EUROFER steel by mass-selected deuterium ion bombardment", Nucl. Mater. Energy **16** 114–122 (2018)

[3] Udo von Toussaint et al., unpublished

[4] R. Arredondo et al., unpublished

Steel-based materials surface damage and modification under high power plasma exposures

V. Makhlai^{1,2,3}, *I. Garkusha*^{1,2}, *O. Byrka*¹, *V. Voitsenya*¹, *S. Malykhin*³, *S. Herashchenko*¹, *K. Sereda*², *A. Chunadra*², *S. Surovitskiy*³, *G. Glazunov*¹, *D. Terentyev*⁴, *A. Golubeva*⁵

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Simultaneous impacts of high energy and particle loads to the material surface are typical for material performance in various extreme conditions: fusion devices (both magnetic and inertial), space apparatus in upper atmosphere, operation of turbines, nuclear engineering etc. Influence of powerful plasma impacts on number of the energy system materials, i.e. RAFM steels, coatings of various materials etc. has been discussed. Material exposures with hydrogen and helium plasma streams have been performed in several high current pulsed and quasistationary plasma accelerators providing variation of power load to the surface as well as the particle flux in vide range: energy density 1 - 25 MJ/m², particle flux up to $10^{26} - 10^{29}$ ion/m²s, pulse duration 1 - 250 µs [1, 2].

A response of the investigated materials to extreme plasma loads, which are relevant to transient events in fusion reactors, is briefly discussed. It is demonstrated that a broad combination of mechanisms of powerful plasma interactions with various materials includes not only a surface damage caused by different erosion mechanisms, but under certain conditions it may also result in a significant improvement of material properties in the near-surface surface layer of several tens μ m in thickness [1]. Some improvement of the structure and substructure of such a layer may be caused by the high-speed quenching, the shock wave formation and material alloying with plasma-and coating-species [1, 3]. The creation of unique surface structures and a considerable improvement of physical and mechanical properties of different materials can be achieved by the pulsed plasma alloying, i.e. pre-deposited coating modifications and mixing caused by the impacting plasma streams. First results on hydrogen outgassing from modified steels are also discussed [4].

- 1. I. Garkusha et al., J. Phys.: Conf. Ser. 959, 012004 (2018)
- 2. A Marchenko et al., J. Phys.: Conf. Ser. 959 012006 (2018)
- 3. V. A. Makhlaj et al., Probl. Atom. Sci. Technol. 6(106), 129-131 (2016)
- 4. M. N. Bondarenko et al., Probl. Atom. Sci. Technol. 6(118), 71-73 (2018)

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Effect of He on D retention in various RAFM steels

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Toward the use of RAFM (reduced-activation ferritic/martensitic) steels as a plasma-facing material (PFM) in future fusion devices, fundamental properties of RAFM steels as a PFM have been investigated in laboratory experiments. In this study, we have explored D retention in various RAFM steels (CLF-1, Eurofer, F82H, and Rusfer) exposed to plasmas (D fluence ~ $1x10^{25}$ m⁻², ion flux ~ $2x10^{21}$ m⁻²s⁻¹, sample temperature ~ 373 K, incident ion energy ~ 100 eV) in the PISCES-A linear plasma device. The total D retention, R_D , was measured using thermal desorption spectroscopy (TDS), taking into account both HD and D₂ signals.

As a baseline, R_D caused by pure D plasma exposure was first measured for each material. Under the same pure D plasma exposure condition, R_D was found to depend strongly on the RAFM steels: R_D (CLF-1) ~ 1.3x10²¹ m⁻², R_D (Rusfer) ~ 8.0x10²⁰ m⁻², R_D (Eurofer) ~ 7.9x10¹⁹ m⁻², and R_D (F82H) ~ 4.5x10¹⁹ m⁻². Next, simultaneous D+He mixed plasma exposure was performed with the He fluence of ~1x10²⁴ m⁻². A reduction of D retention was seen for all the RAFM steels, but with a different reduction factor: ~18x for CLF-1, ~6x for Rusfer, ~2x for Eurofer, and ~1.5x for F82H. TDS spectra show D desorption can be separated into low (< 600 K) and high (> 600 K) temperature components. It was found that the low temperature D desorption is more sensitive to (1) the type of RAFM steels, (2) the admixture of He, and (3) the sample temperature during plasma exposure, compared to the high temperature desorption.

Microstructures of the near-surface region of the plasma-exposed CLF-1 samples were observed with TEM (transmission electron microscopy). On the surface exposed to simultaneous D+He mixed plasma, high-density cone structures with the height of \sim 50-100 nm were formed, and high-density He bubbles were observed both inside and under the cones. The thickness of the He bubble layer under the cones is around 15 nm. On the other hand, a drastic change was not seen on the surface exposed to pure D plasma, except the formation of low-density large cones with the height of \sim 200 nm, which were also formed on the simultaneous D+He mixed plasma exposed surface. The high-density He bubble layer is, therefore, considered to play a key role in reducing the D retention in the same way as for W [1, 2].

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Interaction of high flux plasma with Fe and RAFM steels: experimental and computational assessment

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As a contribution to the CRP on "Plasma–Wall Interaction with Reduced Activation Steel Surfaces in Fusion Devices", over 2017-2018 we have completed the computational assessment at atomic scale to characterize the interaction of hydrogen with dislocation micostructure in BCC Fe, as prototype material for F/M steel. The impact of high flux deuterium plasma on iron and several RAFM steel samples was also studied. In both cases, the analysis was mainly dedicated to understanding of the trapping and retention of D atoms in the matrix, and microstructural changes induced by the high flux plasma.

In brief, the atomic-scale computational analysis was performed to characterize the residence of H atoms in bcc Iron matrix and its self-interaction, interaction with vacancies and dislocations. It has been revealed that H exhibits rather weak self-interaction (binding energy of 0.22 eV), and the formation of multiple hydrogen clusters is not conducted by the release of self-interstitial Fe thus creating the thermally stable Hn-vacancy complex – contrary to the situation with Helium. The interaction energy with screw and edge dislocation was found to be 0.27 and 0.47 eV, respectively. However, the energy landscape of corresponding to the Hn cluster on the dislocation line neither revealed a possibility of the transformation of such complex into the stable cluster accommodated with the emission of kinks. Finally, the binding of H and Hn clusters with a single vacancy was assessed. The binding of a single H amounts to 0.62 eV, and two more H atoms are bound without losing the binding strength. Fourth and fifth atoms can be further added, but the corresponding binding energy goes down to 0.2 eV. The addition of the sixth atom is no longer favourbale. Hence, the growth of Hn cluster on vacancy or dislocation line should occur given the external source of free vacancies, which could be supplied thanks to intensive plastic deformation of the sub-surface region under high flux plasma exposure.

On the experimental side, two sets of exposure were performed, namely: (i) pure Iron in reference and heavily deformed state; (ii) exposure of several RAFM steels including two chemically tailored grades produced specially to improve the mechanical properties and two conventional 9Cr steels - Eurofer97 and T91. Two advanced grades were 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. The exposure temperature was 450K. The thermal desorption spectroscopy (TDS), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) 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. Plastic deformation applied to pure Fe resulted in the increase of the trapping of H released at stage I, but also invoke the appearance of the second release stage around 650K. It has been discussed that this release stage is probably due to the pinning at the sub-grains formed as a result of the heavy plastic deformation. 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) in pure Fe and conventional RaFM steels. The surface modification was clearly less evident for the advanced TMC grade.

Deuterium retention and erosion of CLF-1 and CLAM steels exposed to deuterium plasma

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During last one and half year, two parts of work have been performed in Lanzhou institute of chemical physics. First, the measurement method of deuterium and helium depth profile in metal using glow discharge optical emission spectroscopy were established in our laboratory, and the deuterium and helium retention in tungsten and RAFM steel samples exposed to deuterium plasma were measured. Using co-deposited and implanted samples, a deuterium and helium depth profile measurement method was successfully developed, the results show that GDOES can be used to evaluate the deuterium depth profile up to 20 μ m in tungsten, and the total retention amount measured by TDS and GDOES shows the same increasing tendency. But in RAFM steel sample, due to the deuterium concentration is lower than the measurement limitation, the total deuterium retention amount measured by GDOES shows large scatter compare the data obtained by TDS.

The fuel retention and erosion of different reduced-activation ferritic/martensitic (RAFM) steek including CLF-1, CLAM, EUROFER and RUSFER exposed to deuterium (D) plasma were investigated. D plasma exposure was performed in a linear experimental plasma system with the incident ion energy between 30 and 180 eV per D, fluence from 10^{23} to 10^{25} D/m² and sample temperature between 300 and 900 K. After D plasma exposure, erosion structure development of surface topography was observed, but no significant difference was seen among various RAFM steels within the examined conditions. W-enriched layers are formed at the surface of all investigated RAFM steels determined by RBS. RAFM steels studied exhibited similarities in erosion and dependence on D plasma energy or fluence. The total erosion of CLF-1 and CLAM steel samples exposed at temperature from 400 to 900 K shows no obvious difference. Thermal desorption spectroscopy reveals in all steel samples only one broaden releasing peak from 450 to 800 K and total D inventory of $10^{18} - 10^{20}$ D/m². Similar D retention behavior of five RAFM steels was found that it decreases with increasing incident fluence or ion energy. Moreover, different cutting treatment along rolling direction leads to different erosion structure and D releasing behaviors.

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Investigation of Deuterium Permeation and Retention in RAFM Steel

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Reduced activation ferritic/martensitic (RAFM) steel has been selected as the candidate structural material for fusion reactors. Recently, hydrogen isotopes (H) permeation and retention in RAFM steel under plasma exposure conditions have been intensively investigated at the Institute of Plasma Physics, Chinese Academy of Sciences (ASIPP) in laboratory scale facilities as well as in the EAST tokamak.

Deuterium (D) transport parameters like diffusivity, permeability and solubility in a Chinese RAFM material: CLF-1 have been experimentally measured. The surface recombination coefficient for various D ion incident energies has been evaluated and the steady state permeation flux is found to extremely sensitive to the surface condition [1]. Helium (He) effects have been investigated as well. The RAFM steel surface can be strongly modified by the low energy helium plasma exposure [2]. Using an accelerator, high-energy He ion is pre-implanted into RAFM to study its effects on D retention. Deeply injected He has been found to act as a permeation barrier and reduce D bulk retention [3]. Finally, D plasma-driven permeation through a monoblock-type plasma-facing component (PFC) mock-up made by W and RAFM has been tested. The results suggest that W armor with gaps cannot stop hydrogen isotopes permeation through PFC into the coolant effectively. The RAFM material can act as T permeation "short cut", resulting in faster penetration speed and higher permeation flux than expected, which then may raise more complicated T contamination issue to the coolant [4].

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