Hydrogen retention in self-damaged and He-irradiated tungsten and alloys for PFC

Sabina Markelj, Primož Pelicon, Iztok Čadež, Primož Vavpetič, Anže Založnik
Jožef Stefan Institute, Association EURATOM-MESCS , Jamova 39,
SI-1000 Ljubljana, Slovenia

Olga Ogorodnikova, Thomas Schwarz-Selinger
Max-Planck-Institut für Plasmaphysik , EURATOM Association, Boltzmannstr. 2,
D-85748 Garching, Germany

EFDA-coordinated project WP13-IPH-A03-P1-01/MESCS

CRP "PWI with Irradiated Tungsten…", Vienna, November 26-28, 2013

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the accelerator laboratory at Jožef Stefan Institute

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3. *In situ* ERDA on undamaged and damaged tungsten

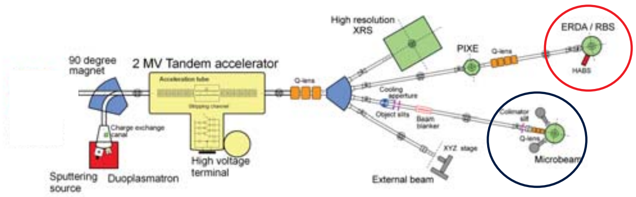
4. *In situ* NRA study on undamaged and damaged tungsten

5. Outlook for the RCP

1.Introduction:

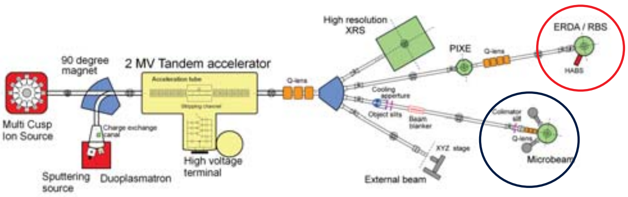
2 MV tandetron accelerator, Jožef Stefan Institute, Ljubljana, Slovenia

Two accelerator facility upgrades in 2012/2013:



Two accelerator facility upgrades in 2012/2013:

- New multicusp proton ion source enabled duoplasmatron source to be permanently configured for He beam and available for prompt ^{3}He NRA. ^{3}He consumption optimized by construction of ^{3}He/^{4}He gas mixing setup.



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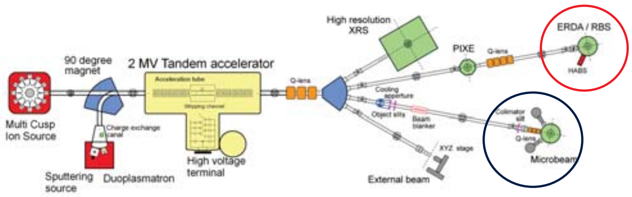
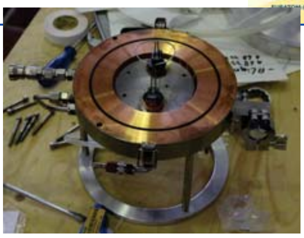
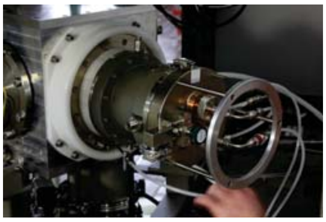
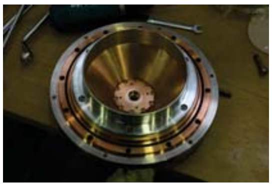
- Broad beam NRA is set and it allows prompt static and in situ D-surface measurements and experiments.



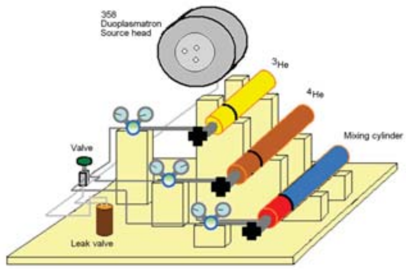






Multicusp ion source dismantled during the instalation at JSI and assembled (down left).

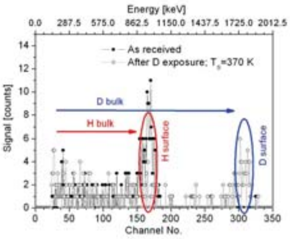
^{3}He beam formation for NRA: duoplasmatron ion source configured for He operation, positive extraction, Li exchange channel



^{3}He gas consumption strongly reduced with construction of gas mixing set-up

2. *In situ* setups for ERDA and NRA
In-situ ERDA

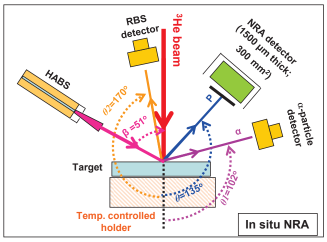
Experimental methods: Elastic Recoil Detection Analysis- ERDA
Rutherford Backscattering - RBS



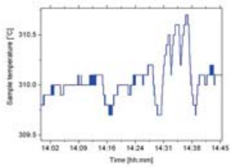
- Incoming beam: 4.3 MeV $^7Li^{2+}$
- Beam dimensions: 2 x 1 mm^2
- Hydrogen Atom Source (HABS): Typical central atom flux density at the sample was for H 1.6×10^{15} at.cm^{-2}s^{-1} and for D 1.06×10^{15} at.cm^{-2}s^{-1}.

ERDA in situ studies:
Markelj et al. JVST A 30, (2012) 041601-1
Markelj et al. Appl. Surf. Sci 282 (2013) 478

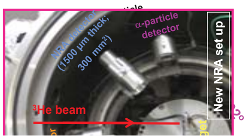
In situ NRA measurements



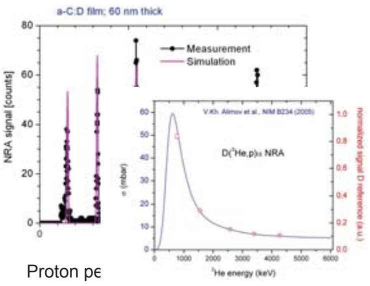
1. NRA analysis at 5 different energies – 740,1500, 2500, 3300 and 4200 keV
2. Takes about 1.5 h to go through the whole cycle, avoid too much current due to temperature increase.
3. Time of studied processes - hours/days.
4. Background vacuum monitored by Quad Mass Analyzer- 1 to 100 m/q



The new set up for NRA measurements

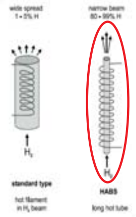


- The NRA method:
  - Analyzing protons from nuclear reaction D(^{3}He,p)^{4}He at ^{3}He energies from 650 (500) keV up to 4.5 MeV
  - D depth profile up to ε
  - D-concentration in nea layer by D(^{3}He,α)H rea lowest ion beam energ analyzed α particles, de under a shallow scatter 102° (V.Kh. Alimov, M. I J. Roth, Nucl. Instr. and Phys. Res. B 234, (2005)
- First measurements on sta C:D layer (60 nm) at five di energies (766, 1555, 2580, keV).

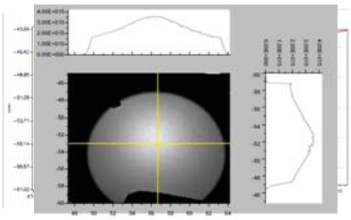


In-situ at JSI: hydrogen atom beam source - HABS

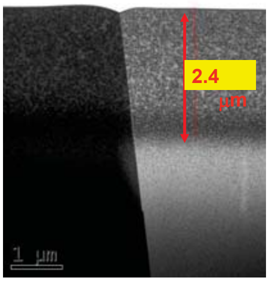
K.G. Tschersich, J.P. Fleischhauer and H. Schuler, *J. Appl. Phys.* 104, 034908 (2008);
http://www.mbe-components.com/products/gas/habs.html



1. The atom flux density was determined by the erosion of the amorphous-hydrogenated carbon (a-C:H) layer being exposed to hydrogen atoms at an elevated sample temperature [*T. Schwarz-Selinger et al., J. Vac. Sci. Technol. A. 18, (2000) 995*],
2. a-C:H temperature: 573 K in this experiment.
3. The erosion was quantified *ex-situ* by ellipsometry at IPP, Garching.



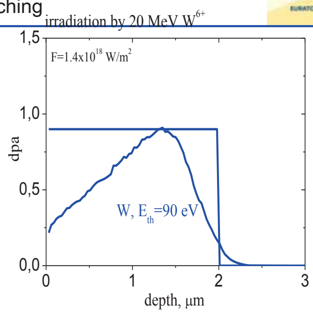
Radiation damage by W implantation: IPP, Garching



TEM for W (L. Ciupinski et al.):
Damage up to 2.4 mm

Calculation of damage profile by SRIM is in good agreement with TEM observation – small vacancy clusters produced by heavy ion irradiation

O. Ogorodnikova, H workshop (2013)

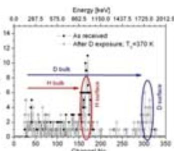


Flat depth profile was created by implantation with different energies

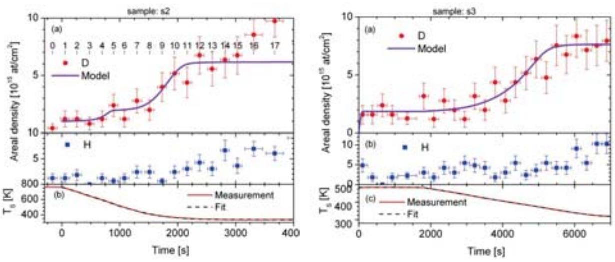
20 MeV W^{6+} F=1.4×10^{18} W^{6+}/m^2 (0.89 dpa)
8 MeV F=3.06×10^{17} W^{6+}/m^2
4 MeV F=1.97×10^{17} W^{6+}/m^2
2 MeV F=1.38×10^{17} W^{6+}/m^2

In-situ ERDA studies / undamaged W

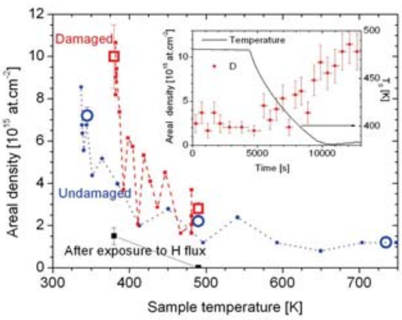
Mirror polished polycrystalline tungsten samples, with large grains perpendicular to sample surface – Plansee ITER grade
Markelj et al, Appl. Surf. Sci. 2013,



Thermoadsorption study



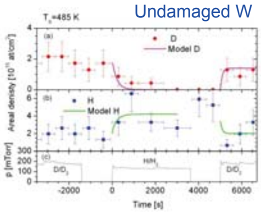
In-situ ERDA studies / damaged W



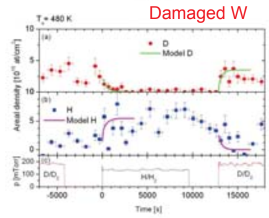
Damaged W produced by 20 MeV W ion irradiation at IPP, Garching (0,89 dpa). Markelj et al, JNM 2013

In-situ ERDA studies/isotope exchange

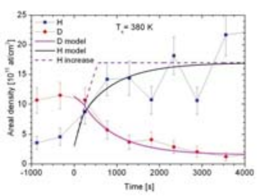
Undamaged W



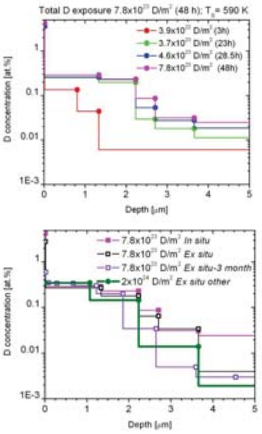
Damaged W



	H fluence [H m^{-2}]	D fluence [D m^{-2}]
Undamaged	9×10^{21}	4.1×10^{21}
Damaged	1.2×10^{22}	4.5×10^{21}



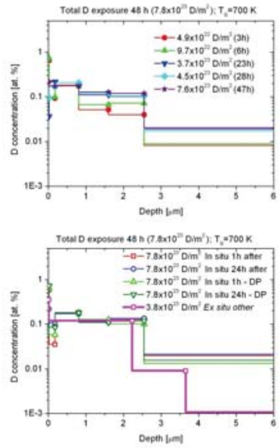
4. *In situ* NRA on undamaged and damaged tungsten



In situ/Ex situ NRA on damaged W @ 590 K

The depth profiles after the stop of atomic exposure measured *in situ* and *ex situ* are in a good agreement in damaged area (up to 2.4 µm).

In situ NRA on damaged W @ 700 K

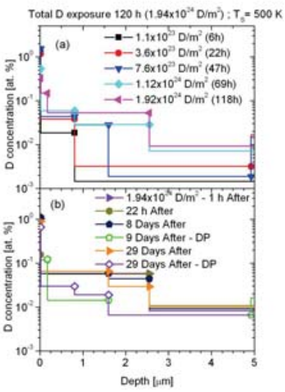


Study on damaged W @ 700 K. The absorbed D reached the final damaging depth ≈2.5 µm much faster than in the case of 600 K. After further D exposure the D concentration only increased to the final saturation concentration, which is similar as in the case of *ex situ* studies at different set up.

Probing NRA beam effect on the sample: negligible

Atomic D flux at standard analyzing ^3He position $(4.5 \pm 0.1)\times10^{18}$ D/m²s.
Different position 2 mm above (DP) - flux $(4.34 \pm 0.05)\times10^{18}$ D/m²s.
Other ex situ study - flux 3.5×10^{19} D/m²s, perpendicular impact.

In situ NRA on undamaged W - ITER grade @ 500 K



First *in situ* measurement on undamaged W showed significantly higher retention, almost one order of magnitude higher, as compared to the *ex situ* measurement obtained on the same sample after 10 days. This indicated that D diffused out after stop of exposure, indicating on the so-called dynamic retention.

Probing NRA beam effect on the sample: significant !

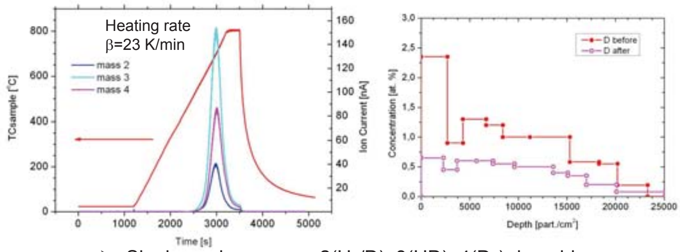
No decrease of D concentration after end of D atom exposure at standard measuring position – indication that the probing beam influences on the D retention, creating traps.
The D retention at position 2 mm above the standard analysing position (marked DP) is much lower 9 days after exposure termination – D atom fluences are similar.

Thermodesorption C:W mixed layer

Sample C:W + D_2 1.5 µm thick layer on Si substrate – sample #1, C. Lungu et al.

- Depth profile before – TPD – Depth profile after

Main desorption peak @ 700°C



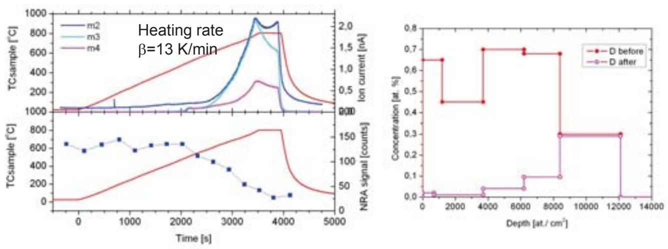
- ➢ Single peak - masses 2(H_2/D), 3(HD), 4(D_2) desorbing.
- ➢ D decreased but not completely
- ➢ W content ~ 8 at. %, Carbon ~ 92 at. % (mass ratio 1:1)

Thermodesorption C:W:Al mixed layer

Sample C:W:Al + D_2 1.5 µm thick layer on Si substrate

1. Depth profile – TPD + NRA@2.5MeV – Depth profile

D starts to decrease @ 500 °C



- ➢ No typical desorption peak, only exponential increase.

5. Outlook for the RCP

- The *in situ* measurements gave direct information about the dynamics of D migration through radiation-induced defects in W.
- Good agreement between the D depth profiles obtained *in situ* and *ex situ* on damaged W in the damaged zone was obtained. On the other hand, significant difference is observed between *in situ* and *ex situ* NRA measurements in the case of undamaged W.
- He beam implantation effect on D retention: simultaneous irradiation with high-energy (100-4500 keV) ^4He beam and atomic D beam, followed by D NRA profiling:
  looking for influence of He irradiation on the retention in damaged W, first experiments already done on undamaged tungsten.
- Study of bulk isotope exchange in damaged W – under evaluation.
- Installation of ion gun with mass filter for in-situ irradiation with low-energy ions.