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

Sabina Markej, 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

1. Introduction:
2 MV tandetron accelerator, Jožef Stefan Institute, Ljubljana, Slovenia

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.

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

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

$^3$He beam formation for NRA: duoplasmatron ion source configured for He operation, positive extraction, Li exchange channel
### 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 $^{7}$Li$^2^+$
- Beam dimensions: $2 \times 1 \text{ mm}^2$
- Hydrogen Atom Source (HABS): Typical central atom flux density at the sample was for H $1.6 \times 10^{15} \text{ at.cm}^{-2}\text{s}^{-1}$ and for D $1.06 \times 10^{15} \text{ at.cm}^{-2}\text{s}^{-1}$.

#### 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 mTorr

#### The new setup for NRA measurements

- The NRA method:
  - Analyzing protons from nuclear reaction $^{3}$He($^3$He, p)$^4$He at $^3$He energies from 650 (500) keV up to 4.5 MeV
  - D depth profile up to 8 μm in W
  - D-concentration in near-surface layer by $^{3}$He($^{3}$He, $^α$)$^4$H reaction at lowest ion beam energy – energy analyzed $^α$ particles, detected under a shallow scattering angle of 10°

#### 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

#### In-situ ERDA studies / damaged W

- Damaged W produced by 20 MeV W ion irradiation at IPP, Garching (0.89 dpa).
- H fluence: $9 \times 10^{21}$
- D fluence: $4.1 \times 10^{21}$

#### 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 / isotope exchange

- Undamaged: $9 \times 10^{21}$
- Damaged: $4.5 \times 10^{21}$

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

- Proton peaks due to $^{12}$C($^3$He, p)$^{14}$N

#### 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

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).

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: negligible
Atomic D flux at standard analyzing 3He position (4.5 ± 0.1) × 10¹⁸ D/m²s.
Different position 2 mm above (DP) - flux (4.34 ± 0.05) × 10¹⁸ D/m²s.
Other ex situ study - flux 3.5 × 10¹⁹ D/m²s, perpendicular impact.

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 + D₂ 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(H₂/D), 3(HD), 4(D₂) 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) He 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.