



Progress of W-relevant plasma-material interaction studies at ASIPP

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3rd RCM of CRP on PWI for Irradiated W and W Alloys in Fusion Devices 27-30 June 2017, Vienna, Austria

Outline

1. D retention in ion-damaged W

- D retention in Ne damaged W
- D retention after He irradiation and EAST plasma exposure
- 2. D permeation behavior in W and its alloys
- D transport parameters measurements for W and its alloys
- D gas-driven permeation in damaged W
- D plasma-driven permeation in W

3. R&D of new experimental setups for W PWI research

- A multichannel spectroscopy system for W impurity in EAST
- A new plasma-driven permeation setup: PREFACE
- A new high flux linear plasma facility: HPPX

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Progress after the 2nd RCM

1. D retention in ion-damaged W

- D retention in Ne damaged W
- D retention after He irradiation and EAST plasma exposure

D retention in Ne damaged W -- motivation

Tungsten (W): primary plasma facing material candidate

Simultaneous implantation of hydrogen isotopes and energetic neutrons
 Formation of irradiation damages throughout the materials by neutron

H isotopes retention in damaged W-PFM

From the view points of reactor safety and fuelling efficiency, understanding

of fuel retention behavior in irradiation damaged W is extremely important.

Heavy ions (like W, Fe, Cu, Si, etc.) beams are widely used to introduce irradiation damages in fusion materials such as W.

Can we simulate neutron irradiation with ions in a more proper way?



High-energy Ne-ion irradiation of W at HIRFL





Annealed W foils

- **Dimension:** Φ20x 0.05mm / 0.11mm,
- □ Annealing at 1373 K for 2 h,
- **G** SEM: Partially recrystallized,
- TEM: most dislocations annealed out; some DWs at GBs.

Irradiated in HIRFL

- Ions: 122 MeV ²⁰Ne⁺⁷,
- Fluence: 3.0×10^{20} ions/m²,
- Temperature: ~713 K,
- An energy degrader for ion incident range tailoring.

Quasi-homogeneously distributed defects in W



- An energy degrader wheel made of 30 pieces of Al-foils rotating in front of the sample;
- High energy of 122 MeV, implantation range is ~22.0 μm in W;
- **Both sides** were irradiated.

A quasi-homogeneous distribution of atomic displacement damage to 0.16 dpa within a depth of 50 μ m in W.



PALS characterizing of irradiated W

Positron Annihilation Lifetime Spectroscopy

- Open volume regions (vacancies, voids, etc.) in solids have lower electron densities; thus specimens with defects will exhibit longer positron lifetimes.
- A non-destructive technique to study the free volume in solids directly at nano-scales.
 - Positrons from ²²Na source: continuous energy spectrum, E_{max}=0.545 MeV;
 - □ Maximum incident range:

$$R_{+} = \frac{E^{1.19} \,[MeV]}{2.8\rho[g/cm^{3}] \cdot Z^{0.15}} \approx 47 \,\mu m$$

 \rightarrow defects information in the bulk material!

The curves for two irradiated W samples located significantly higher at the right hand, indicating the occurrence of long-lived lifetime components.



PALS obtained for the



□ The PALS data were analyzed based on a two-state trapping model by assuming that:

- au_1 free positrons and positrons trapped at the mono-vacancies/dislocations,
- au_2 positrons trapped at the vacancy clusters or voids.

		Disl	ocations Intermediate-sized va			cancy clusters
	Thickness of W (μm)	Lifetimes 1 (ps)	Intensities (%)	Lifetimes 2 (ps)	Intensities (%)	450 - 400 - 7
Annealed (1373 K)	50	139.6±1.7	100	/		蜜 350 -
	110	139.5±2.6	100			المعلى المعلى المعلى المعلى المعل المعلى المعلى المعل المعلى المعلى معلى المعل
Irradiated	50	118.6±3.4	59.5±1.0	406.8±5.2	40.5±1.0	සි
	110	118.9±3.6	62.7±1.3	366.4±6.1	37.3±1.3	0 20 40 60 80 100 Number of vacancies in W
Deference*.					-	T. Troev, et al., J. Phys.: Conf. Ser.

Reference*:

a. positron lifetime in defect-free W : τ_f = 105 ps

b. Correlation: positron lifetime vs. the number of vacancies clusters in W (right figure)

c. The lifetimes of positrons trapped at dislocation: slightly shorter than au_{1V}

* H.E.Schaefer, phys. stat. sol. (a) 102, 47 (1987); M. Eldrup, et al., Nucl. Instr. Meth. B 266 (2008) 3602–3606. 207 (2010) 012033

n-irradiated W/Mo:

 $\tau_2 = 350 \sim 470 \text{ ps}$

Deuterium bulk retention (D₂ gas loading)

D₂ gas loading to introduce a uniform distribution of D in the Ne-ion irradiated W

instead of plasma exposure to avoid:

1) further modification of near surface morphology by plasma irradiation;

2) larger D retention in the plasma irradiated side of the damaged W.





TDS spectra of D for annealed and Ne-ion irradiated W after D_2 gas exposure at 773 K for 2 h.

 A significant high desorption peak at ~1010 K only for two irradiated W
 ~1050 K for n-irradiated W [Hatano,et al].

 Broad D desorption spectra from 730 K to 1173 K for the Ne-ion irradiated W
 i. 730->1173 K for the n-irradiated W;

ii. 930-1050 K for the single energy W-ions irradiated W [Hatano,et al] .

A desorption shoulder at ~500-600 K ~ two orders of magnitude lower than peak 2.

Irradiation parameters:

Material: 1400 $^{\circ}$ C/2h annealed W

Sample size: ϕ 20 mm and ϕ 3 mm

Ion species: He⁺

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lon energy: 80 keV
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Irradiation temperature: RT

Fluence: 3×10^{15} ions/cm², 3×10^{16} ions/cm², 3×10^{17} ions/cm²





Microstructure after helium irradiation



The TEM images of W under various situations, (a) 3×10^{15} ions/cm², (b) 3×10^{16} ions/cm² and (c) 3×10^{17} ions/cm².

Statistical Results									
Fluence (ions/cm ²)	Peak dpa	Defect size (nm)	Defect density (m ⁻³)						
3×10 ¹⁵	0.054	11.75	2.99×10 ²¹						
3×10 ¹⁶	0.54	26.89	2.86×10 ²¹						
3×10 ¹⁷	5.4	44.9	2.95×10 ²¹						

- He+ irradiation produces large amount of dislocations.
- The size of the dislocation loop increases with irradiation fluence.
- No obvious change in the number density of dislocation loops.

D plasma exposure in EAST



Experimental details:

Position: 5 mm behind the limiter Shot number: 367 shots Total plasma exposure time: ~2000 s Plasma parameters: T_e : 5-10 eV n_e : ~1X10¹⁸ m⁻³ Sample temperature: 50-350 °C

TDS analysis after plasma exposure in EAST



- Two main desorption peaks;
- The desorption rate of D_2 at ~440 K increased with the irradiation;
- The retained amount of deuterium increased with the increase of irradiation fluence.

2. D permeation behavior in W and its alloys

- D transport parameters measurements for W and its alloys
- D gas-driven permeation in damaged W
- D plasma-driven permeation in W

D transport parameters measurements for W

Gas-driven permeation (GDP) setup



Upstream gas supply & gauge

GDP furnace & gasket-type sample

Parameters for GDP device

- Background pressure: 10⁻⁵ Pa
- Driven D₂ pressure: 10³-10⁵ Pa
- Sample temperature: RT-1000 K
- Sample sealed by VCR

couplings

• Signal monitored using QMS

Permeation of D in W: defect effects

Objectives: To measure the H transport parameters in W and to understand the defect effects on H permeation.

• Materials:

	Thermal	Thickness(µm)			
	treatment	50	100	250	
Rolled W	973 K, 2h	\checkmark		\checkmark	
Annealed W	1373 K, 2h	\checkmark	\checkmark	\checkmark	
Recrystallized W	1673 K, 2h			\checkmark	

TEM observation of dislocations



- **Results of GDP experiments**
- The higher the density of defects, the lower the measured diffusivity;
- Permeability is not affected by dislocations



Permeation of D in W: second phase effects

Objective:

To measure the effects of ZrC/La_2O_3 addition phases on H permeation through W.

Materials

W-0.5 wt.% ZrC (by Xie et al.): powder ball milling + sintering + rolling

□ W-1 wt.% La₂O₃ (by Yan et al.): liquid–liquid doping + sintering + rolling

Rolled W@ 973k,2h

- Results of GDP experiments
- □ The effective diffusivity of D in ZrC/La₂O₃-dispersion strengthened W is higher than that in pure W;
- The permeability is almost the same for these W materials, indicating small amount of dispersed phases do not apparently affect the H permeation flux.





D gas-driven permeation in damaged W

2x1.7MV tandem accelerator in Peking Uni.





Actually, GDP experiments has been performed for He damaged W as well. But unfortunately, must samples were broken when we try to seal them...

Deuterium GDP through irradiated W



- Due to the defects (irradiation-induced & intrinsic) in W, it takes much longer time for the 1st permeation run to reach a steady state permeation.
- □ After the 1st permeation, all the irreversible traps are fully filled by D; thus the 2nd/3rd runs can quickly reach steady state.

Diffusion coefficient of D in irradiated W

Effective diffusivity was measured from the permeation transient curve with a time-lag method.



The higher the irradiation dose (i.e., irradiation defects), the lower the effective diffusivity is measured for the 1st run;

• The effective diffusivities for the 2nd /3rd run approach those from un-damaged W.

Pressure dependence of GDP

Deuterium GDP through a irradiated W of ~30 dpa at different temperatures.



A linear relation between GDP flux and the square-root of upstream pressure has been found at all the temperatures examined in this work.

GDP is diffusion-limited for all samples.

Permeability of D in irradiated W



The values of permeability, measured independently by different researchers, are within the same order of magnitude in 800–1000 K.

When there is thermodynamic equilibrium between H in traps and H in solution:

$$C_s/z = C_t/(C_T - C_t)\exp(-E_t/kT)$$

We can get:

$$D_{eff} = \frac{D_L}{1 + \frac{C_T}{z} \exp(\frac{E_t}{kT})}$$
$$S_{eff} = C_s + C_t = C_s [1 + \frac{C_T}{z} \exp\left(\frac{E_t}{kT}\right)]$$

Thus:

$$\boldsymbol{P}_{eff} = \boldsymbol{D}_{eff} * \boldsymbol{S}_{eff} = \boldsymbol{D}_L * \boldsymbol{C}_s = \boldsymbol{D}_L * \boldsymbol{S}_L = \boldsymbol{P}$$

[G. Federici' review, NF, 2001]

The irradiation induced voids in bulk W did not significantly affect the effective permeability of hydrogen.

Plasma-driven permeation through W

Preliminary results (obtained in this month)



3. R&D of new experimental setups for W PWI research

- A multichannel spectroscopy system for W impurity in EAST
- A new plasma-driven permeation setup: PREFACE
- A new high flux linear plasma facility: HPPX

Multichannel spectroscopy system in EAST



- 22 lines-of-sight (LOS) in outer divetor and 17 LOSs in inner divertor enable poloidal profile measurement of the divertor plasma spectroscopy in visible range with a 13 mm poloidal resolution on the surface.
- Both multichannel spectrometer and filtered photomultiplier tubes (PMTs) are employed to accommodate good spectral resolution (0.02 nm) and fast tracking (up to 20 kHz) of the line radiation in transient incidents e.g. ELMs.





H. Mao et al. Rev. Sci. Instrum. 2017

NBI & ECRH Heating Impacts on W source



The impurity fluxes, ion saturation current (Js) and T_e at divertor target plane are increased during NBI and ECRH heating phase, therefore enhancing the W sputtering

W Sputtering Enhanced by Ne Seeding



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Upgrade plan of EAST lower divertor (Early 2019-)

Mission

- SSO -1000s; withstand heat flux > 10MWm⁻²
- W-Divertor Physics
- W/Cu divertor with active water cooling
 - Monoblock structure in the strike point region
 - Flat-type structure for the dome plates





- Compatible with engineering and EAST in-vessel requirements.
- A new cryo-pump will be added for long-pulse particle/impurity exhaust.
- > Plasma can be accommodated with δ =0.4-0.6
 - Normally with OSP locating on the horizontal plate, to avoid the W sputtering in the far SOL region.
 - Physics exp. ops with OSP on the vertical plate.



After Prof. Y. Song's SOFE2017 talk

The PREFACE facility

Permeation and Retention Evaluation FACility for fusion Experiments (PREFACE)

- Background: ~ 10⁻⁶ Pa
- Max ECR power : 6 kW @ 2.45 GHz
- Plasma diameter:
 40 mm at the limiter
 30-70 mm at the target





PREFACE

Plasma parameters in PREFACE

 Diagnostics: Hiden Langmuir probe Avantes spectrometer (197-717 nm) Thermocouples Parameters: Te: 2-5 eV ne: 10¹⁶-10¹⁷ m⁻³ Ion flux: 10²⁰-10²¹ m⁻²s⁻¹



Plasma-driven permeation setup



W sample for PDP studies

OMS

HPPX (Helicon Physics Prototype eXperiment)



Plasma source

- 50 kW RF @ 13.56MHz
- 100 kW ECH @ 2.45GHz

Expected plasma parameters:

- Ne: ~10¹⁹ m⁻³
- Te: 1-20 eV
- Plasma column : 100 mm
- Ion flux: ~1x10²³ m⁻² s⁻¹

Vacuum chamber





Actively cooled W target and dump

All the in-vessel components are actively cooled to handle the steadystate high heat flux.



Target



Dump



Current status

First plasma (Ar): Dec. 2016 at low densities





Current status:





Summary

- D retention studies have been done for high energy Ne/He ion irradiated W by gas absorption, plasma exposure and thermal desorption spectroscopy.
- D transport parameters like D and P have been measured for W and its alloys. Preliminary plasma-driven permeation experiments through W have been performed.
- New facilities are under development to support further Wrelevant PWI research at ASIPP.

Thank you for your attention ! Welcome your collaborations !

Formation of dislocation loops in the irradiated W

Annealed W



Irradiated W

- Annealed W: dislocations aligned to form polygonized dislocation walls (WDs) near grain boundaries.
- Irradiated W: additional features like dislocation loops of 10-20 nm, which are rearranged linearly to form strings (coalescence reactions)

Observation of dislocations with HRTEM





HRTEM: High resolution TEM FFT :Fast Fourier Transform IFFT: Inverse FFT

- □ Beam direction B=[001];
- □ Fig.(c) presents the corresponding plane of (110) as circled in (b) :
- mark "2-4" & "6": perfect dislocation loops;
- ◆ mark "5" :

Frank loops, interstitial-type.

W[110]: $a/\sqrt{2} = 3.165/1.414=0.2238$ nm Sizes of the loops: ~1-2 nm

Both large (~10-20 nm) and small (~1-2 nm) dislocation loops may contribute to the peak 1 desorption in TDS for the Ne-ion irradiated W.

Formation of cavities in the irradiated W

*TEM resolution for voids: ~ 1nm

Under-focus

Over-focus



☐ High density of voids with an average diameter of ~1 nm are evenly distributed in the large observed area.

These voids are probably the main contributor to the longer positron lifetime components.

2. TDS & GDP experiments



Upstream gas supply & gauge

GDP furnace & gasket-type sample

TDS experiment

Background : $\sim 10^{-5}$ Pa Heating rate: 10 K/min Temperature range: RT to 1273 K High resolution(for D₂ and He) Calibrated by standard leaks

GDP experiment

Background: ~10⁻⁵ Pa Upper gas pressure: 10²-10⁵ Pa Sample temperature: 300-1000 K