IAEA CRP 3rd Research Coordination Meeting on June27-30, 2017

Deuterium behavior in HFIR neutron- irradiated tungsten under US-Japan PHENIX program

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Outlines

Motivation

Progress on D retention in neutron-irradiated tungsten
 TDS and NRA in HFIR neutron-irradiated single crystal tungsten

Status update

- Tritium Plasma Experiment (TPE) Upgrade
- Surface/bulk diagnostics upgrade for low activation & tritium contaminated samples at STAR facility
- TMAP integration into MELCOR Fusion
- RB* neutron irradiation with thermal neutron shielding under PHENIX
- Future plan

INL's motivation for irradiated tungsten

- Safety concern of in-vessel tritium source term in FNSF and DEMO
 - Tungsten (tungsten alloy) is the leading candidate for solid PFC option
 - T retention in <u>bulk</u> neutron-irradiated W (W alloy) at elevated temperature



- Leverage the unique capabilities in INL and US-Japan collaboration
 - Irradiate tungsten (tungsten alloy) samples at High Flux Isotope Reactor, ORNL
 - Expose neutron-irradiated samples at Tritium Plasma Experiment, INL
 - Post-irradiation (and post-exposure) examination with thermal desorption spectroscopy, nuclear reaction analysis, glow-discharge optical emission spectroscopy, positron annihilation spectroscopy
 - Simulate with reaction-diffusion code, Tritium Migration Analysis Program



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- D retention in n-irradiated SCW at DEMO-relevant elevated temp.
 - Specimens:
 - Material:

- Single crystal (100) tungsten from Goodfellow USA
- Size: 4.0x4.0x0.5 mm³
- Neutron irradiation condition in HFIR, ORNL:
 - Capsule: TB-300-2, TB-500-2, and TB-650-3
 - Neutron fluence: 0.52 x 10²⁵ n/m² (E > 0.1 MeV)
 - Dose: 0.1 dpa
 - HFIR irradiation temp.: 360, 690, and 760°C
- Plasma exposure condition in TPE, INL
 - TPE exposed temp.: 400, 600, and 700°C
 - Plasma exposed area: $\pi(3.88)^2 \text{ mm}^2$
 - D ion flux density: $(7.0-9.0) \times 10^{21}$ D m⁻²s⁻¹ (D only plasma)
 - D ion fluence: 5.0 x 10²⁵ D m⁻²
 - D ion energy: ~ 100 eV (biased to -100 V with plasma potential -5~0 V)
 - 2 samples (A and B) were exposed to similar ion flux density at each temperature
- A-samples (TPE-TDS study) :
 - TDS (10°C/min to 900°C) were performed within 24 hours in INL
- B-samples (TPE-NRA study):
 - NRA (up to 3 micron) were performed within 2 months at SNL-NM







- Results form A-samples (TPE-TDS study) :
 - TDS conditions:
 - TDS were performed within 24 hours after TPE exposure to 5.0 x 10^{25} D m⁻²
 - TDS started when the vacuum pressure is less than 1.3x 10⁻⁵ Pa (1.0x 10⁻⁷ Torr)
 - Ramp rate of 10°C/min to 900°C, and 30 min hold at 900°C



JAPAN-US JOINT PROJECT NRA results from HFIR irradiated 0.1 dpa SCW

• Nuclear reaction analysis (NRA) by Bill Wampler (SNL-MN)

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Sample ID	HFIR n irr. temp.[K]	TPE exp. temp.[K]	D concentration at 1E-6 m [D/W]	D concentration at > 3E-6 m [D/W]	ion induced damage?	D desorbed in ramp-down?
W53B (0.1dpa)	633	673	3.0E-03	~ 2.0E-03	Yes	Νο
W55B (0.1dpa)	963	873	5.0.E-05	~ 2.0E-04	No	Yes
W26B (0.1dpa)	1033	973	< 1.0E-05	~ 1.0E-04	Νο	Yes

TMAP simulation of HFIR irradiated 0.1 dpa SCW



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TMAP simulation of HFIR irradiated 0.1 dpa SCW

Fitting parameters

- Detrapping energy: E_{det} [eV]
- Trap concentration: n_{trap} [T/W] 2.
- desorption flux [D/m²-s] 3. Integrated fraction of D in normal distribution for source rate ≈ (1 - reflection coefficient)

Simulation results

- High reflection coefficient (>0.90) was required to reduce retention
- Good fit for W53 (400°C) case
- High detrapping energy (>2.30 eV) was required to fit D behavior for W55 (600°C) and W26 (700°C).



Sample ID	TPE exp. temp. [°C]	TPE exp. temp.[K]	exp. D. conc. at 3E-6 m [D/W]	reflection coefficien: R [ND]	detraping energy: E _{det} [eV]	Trap conc.: n _{trap} [T/W]	sim. D. conc. at > 3E-6 m [D/W]	NOTE
W53 (0.1dpa)	400	673	~2.0E-03	0.90	1.80	2.0E-03	2.0E-03	good agreement
W55 (0.1dpa)	600	873	~ 2.0E-04	0.99	2.30	2.0E-04	2.0E-04	OK, but high E _{det}
W26 (0.1dpa)	700	973	~ 1.0E-04	0.99	2.60	2.0E-04	2.0E-04	OK, but high E _{det}

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Reference on detrapping energy in irradiated tungsten

Definitions

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- 1. Detrapping energy: E_{det} [eV] = $E_{bin} + E_d$
- 2. Biding energy: E_{bin} [eV]
- 3. Activation energy for H diffusion in W: E_d [eV]

• $E_d = 0.39 \text{ eV}$

• O.V. Ogorodnikova, JAP 2015

– Dislocations:	$0.4 \le E_{bin} [eV] \le 0.8$
– Vacancy:	$0.8 \le E_{bin} [eV] \le 1.4$
– Vacancy cluster:	$1.4 \le E_{bin} [eV] \le 2.2$

Sample ID	TPE exp. temp. [°C]	detraping energy: E _{det} [eV]	binding energy: E _{bin} [eV]	possible trap site
W53 (0.1dpa)	400	1.80	1.41	vacancy or vacancy cluster
W55 (0.1dpa)	600	2.30	1.91	vacancy cluster
W26 (0.1dpa)	700	2.60	2.21	vacancy cluster? (too high E _{bin})



FIG. 6. Schematic potential energy diagram for hydrogen chemisorption, solution, and trapping at different trapping sites.



FIG. 10. Fine spectrum of H binding energy in polycrystalline W according to DFT calculations without and with ZPE (Refs. 36, 38, and 39) and according to "adsorption model" in comparison with the rate equation modelling of experimental TDS data in assumption of single binding energy of deuterium with each defect.^{1,4,12–14}

TMAP simulation of TDS up to 1500 °C (1773 K)

Objective

- TDS experiment ended at 900°C (1173 K) → T0 profile
- To estimate required temperature to desorb all the D in W53 sample

Simulation condition

TPE exp.

temp.

[°C]

400

Sample

ID

W53

(0.1dpa)

- 1. Same R, E_{det} , and n_{trap} are used
- 2. 2800 sec. ramp-down with time constant T_{const} = 1800 [s]
- Start TDS (10K/min to 1873K) at 10,000 s. → T2 profile

TPE

exp.

temp.[K]

673

exp. D. conc.

at 3E-6 m

[D/W]

~2.0E-03



2.0E-03

2.0E-03

1.80

0.90

~ 1400K

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TMAP simulation for extremely high ion fluence

Objective

- Simulate extremely high ion fluence
 - 2.0E27, 4.0E27, and 2.0E28 D $m^{\text{-}2}$
- Compare with Doerner (NME 2016)

Simulation condition

- 1. Same *R*, E_{det} , and n_{trap} are used
 - R = 0.90
 - *E_{det}* = 1.80 eV
 - *n_{trap}* = 2.0E-3
- 2. Increase exposure time to reach 2.0E27, 4.0E27, and 2.0E28 D $m^{\text{-}2}$
- 3. Ramp-down for 2800 s with time constant T_{const} = 1800 [s]
- 4. TDS (10K/min to 1873K) for 9000 s
 → T2 profile

Results

 1200°C (1473K) is required to desorb all the D atoms from n-irradiated SCW exposed high fluence (up to 2.0E28 D m⁻²) 400°C (673K) D plasma



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Comparison with Doerner et al. [Nucl. Mater. Ener. 9 (2016) 89-92]

Doerner et al. (NME 2016)

- Exposed PCW at 640K to extremely high ion fluence
 - 2.0E27, 4.0E27, and 2.0E28 D m⁻²
- Performed TDS with 18 K/min to 1340K.
- All the D atoms desorbed by 1173K.

Comparison results

- Similar fluence dependence ~ fluence^(0.50-0.59)
- > x10 difference in D retention between 0.1 dpa SCW and
 - 0 dpa PCW





Retention in tungsten resulting from extremely high fluence plasma exposure R.P. Doerner', M.J. Baldwin, T.C. Lynch, J.H. Yu

enter for Energy Research, UCSD, La Jolla, CA 92093-0417, USA



Time (s)



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Future plan

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TPE - Tritium Plasma Experiment

- TPE is a unique linear plasma device in four elements:
 - Tritium plasma (< 500 Ci per discharge),
 - Divertor-relevant high-flux plasma (>10^{22} m^{-2} s^{-1}) ,
 - Moderately radioactive (< 1 mSr/hr @ 30 cm) materials handling
 - Beryllium handling
- Utilizes two containments other than its SS vacuum vessel
 - Ventilated enclosure (as a high contamination area/HCA boundary)
 - Ventilated PermaCon room (as a contamination area/CA boundary for T)
- TPE achieved its first deuterium plasma using the new control center outside of the CA after a significant three-year upgrade in Nov. 2015.



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Simple estimate for expected ion flux density and heat flux after TPE upgrade

 With new power input capability, a simple linear scaling estimate showed that the TPE will be capable of reaching

>1.0×10¹⁹ m⁻³ $-n_{e,max}$ $-\Gamma_{max}$ >1.0×10²³ m⁻² s⁻¹ >1 MW m⁻² $-q_{heat}$

Table 2 Summary of expected discharge performance, ion flux density, heat flux with new power supplies.

Parameters	TPE (old PS)	TPE upgrade (new PS)	
I _{dis} ^{max} [ADC]	100	>200	
P _{dis} ^{max} [kW]	10	>20	
V _{bias} ^{max} [VDC]	-200	-600	
$\Gamma_i^{\rm max} [{ m m}^{-2} { m s}^{-1}]$	$0.4 imes 10^{23}$	>1.0 × 10 ²³	
$q_{\rm heat}$ ^{max} [MW m ⁻²]	0.6	>1	

Ref: M. Shimada et.al., FED 2016



(I_{PS}^{max})^{new}

(V max, I max) new

PISCES-A [Goebel et.al. RSI '85] TPE [Shimada et.al. RSI TPE '12

700

600

500

400

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(a)



Improvement of Bulk & Surface Diagnostic Capabilities at STAR for Enhancing Tritium and Nuclear PMI Sciences

Challenge

- Limited availability of bulk and surface diagnostics for <u>neutron-irradiated</u> and <u>tritium contaminated</u> samples

Significance and Impact

This improvement of bulk and surface diagnostics at STAR facility (DOE less than Hazard Category III nuclear facility) will allow us to characterize bulk and surface of neutron-irradiated and tritium contaminated samples (for the first time) to advance fusion nuclear sciences and fusion safety.



X-ray photoelectron spectroscopy (FES funded for relocation/setup)





Scanning Auger Microscopy (FES funded for relocation/setup)



Coincidence Doppler and Positron Lifetime Spectroscopy (INL funded)

Glow Discharge Optical Emission Spectrometry (INL funded)

> Reference: M. Shimada, C. N. Taylor, R. J. Pawelko, L. C. Cadwallader & B. J. Merrill, "Tritium Plasma Experiment Upgrade and Improvement of Surface Diagnostic Capabilities at STAR Facility for Enhancing Tritium and Nuclear PMI Sciences", *Fusion Science and Technology*, http://dx.doi.org/10.1080/15361055.2017.1293422

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New surface diagnostics at STAR

Glow discharge optical emission spectroscopy

- Quantitative elemental depth profile analysis
- Nanometer resolution
- Can analyze 10s or even 100s µm sample depth
- Installation completed in FY16

X-ray photoelectron spectroscopy

- Surface sensitive chemical analysis
- Excellent for quantification
- Particularly useful for surface effects related to permeation
- Installation completed in June 2017
- Scanning Auger electron spectroscopy
 - AES provides elemental characterization
 - Scanning mode allows for microscopy
 - Installation completed in June 2017

NOTE: all three diagnostics are capable of handling low activation and tritium-exposed materials.



Glow discharge optical emission spectroscopy

Technique

- Elemental characterization with depth profiling.
- Analyzed 10s of elements simultaneously.
- Nanometer depth resolution.
- Sputtering rates of nm/sec.

Specifications

- Capable of differentiating H and D.
- Realtime depth measurements.
- Monochromator can be used to look at unknown element.

GD-OES will measured the D depth profiling from bulk (>> 10µm) neutronirradiated tungsten exposed to plasma

AIP ADVANCES 7, 055305 (2017)

Direct depth distribution measurement of deuterium in bulk tungsten exposed to high-flux plasma

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(Received 21 March 2017; accepted 1 May 2017; published online 8 May 2017)

Understanding tritium retention and permeation in plasma-facing components is critical for fusion safety and fuel cycle control. Glow discharge optical emission spectroscopy (GD-OES) is shown to be an effective tool to reveal the depth profile of deuterium in tungsten. Results confirm the detection of deuterium. A \sim 46 µm depth profile revealed that the deuterium content decreased precipitously in the first 7 µm, and detectable amounts were observed to depths in excess of 20 µm. The large probing depth of GD-OES (up to 100s of µm) enables studies not previously accessible to the more conventional techniques for investigating deuterium retention. Of particular applicability is the use of GD-OES to measure the depth profile for experiments where high deuterium concentration in the bulk material is expected: deuterium retention in neutron irradiated materials, and ultra-high deuterium fluences in burning plasma environment. © 2017 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/). [http://dx.doi.org/10.1063/1.4983384]

One of very few surface analysis techniques capable of measuring hydrogen.





FIG. 3. Deuterium depth profile in tungsten exposed to deuterium plasma, shown in linear scale (a). Plotting the smoothed data (50 point averaging) semi-log scale in (b) indicates that smaller quantities of deuterium are found up to >20 μ m in the sample.

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X-ray photoelectron microscopy (XPS) and Scanning Auger electron spectro/microscopy (SAM)

- XPS
 - Technique
 - Excellent chemical sensitivity.
 - Expansive libraries.
 - Capable of detecting elements, Li and larger.
 - Specifications
 - X-ray monochromator for high resolution XPS scans.
 - Multiple x-ray sources.
 - Sputtering ion gun for depth profiling.
- SAM
 - Technique
 - Excellent elemental sensitivity.
 - Limited quantification.
 - Rastering electron beam + Secondary electron detector = SEM
 - Specifications
 - LaB6 filament.
 - Sputtering ion gun for depth profiling.





Digital microscopy

- Technique
 - Digital optical microscope
- Specifications
 - 20x 2000x magnification
 - 3D profilometry
 - Particle counting
- Complement other diagnostics by providing a quick high-mag look at surfaces.



Multi-Angle Observation

Real-Time Measurement

Visualize Surfaces in 3D



INL MELCOR for Fusion Safety Code

- MELCOR an NRC code developed by SNL-NM and is fully integrated, engineering level thermalhydraulics computer code that models the progression of accidents in fission, including a spectrum of accident phenomena such as reactor cooling system and containment fluid flow, heat transfer, and aerosol transport MELCOR for fusion has been
- MELCOR for fusion has been modified to model fusion reactors including fusion relevant coolants (PbLi, Li, FLiBe, He, etc.), material oxidation, etc.
- A version of this code was used to license ITER





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Surface heat and particle fluxes of 10 MW/m² and

 5×10^{23} ions/m²-s, W thickness of 8 mm, water

MELCOR-TMAP Code Development

- A development effort to merge MELCOR and TMAP to create a more complete tool for analyzing fusion accidents began in 2015
- A paper describing the initial application of this code to a DEMO relevant water-cooled tungsten divertor has been published^{*}

Single trap test for un-irradiated tungsten. Simple eq.** predicts 220 h break through.





MELCOR-TMAP Code Development (CONT.)

- H, D, T, He (liquid metal) and H₂, D₂, T₂, HT, DT, and HD (vapor) transport equations were recently added to the MELCOR-TMAP code
- An improved PbLi Equation of State was also developed and a SnLi EOS is under development
- An initial code version capable of modeling a fusion reactor with a tungsten divertor and a liquid metal cooled blanket is presently undergoing internal testing at INL.
- A paper on this code version will be presented at ISFNT-13, September 2017
- Future improvements under consideration are dust resuspension and explosion models and different coolants allow per given heat transport loop

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PHENIX Task 3 (Tritium behavior in n-irradiated W)

Challenges:

- Unavailability of high-flux 14 MeV neutron source.
- Unavailability of simultaneous neutron and plasma irradiation capability

Research approach:

- Use of available fission reactor (e.g. High Flux Isotope Reactor/HFIR).
 - Relatively high-flux fast neutron (>0.1 MeV) available to simulate neutron damage
 - High-flux thermal neutron accelerates solid transmutation and increases activation
- Use of thermal neutron-shielding (e.g. cadmium or gadolinium)
 - To minimize thermal neutron and simulate fast fusion neutron spectrum.
- Use of linear plasma device to study <u>sequential</u> neutron-plasma irradiation.
 - TPE can handle tritium neutron-irradiated material.

Neutron irradiation under US-Japan TITAN (2007-2012) & PHENIX (2013-2018):

- Neutron-irradiation with thermal neutron-shielding at RB* position in HFIR, ORNL
 - 500, 800 and 1100 °C irradiation temperature up to 1.0 dpa
- Deuterium plasma exposure in TPE, INL
 - 500, 800 and 1100 °C plasma exposure temperature
 - > 10^{22} m⁻²s⁻¹ D ion flux up to 10^{27} m⁻² D ion fluence
- Post irradiation examination (PIE) at INL, SNL-NM, ORNL.
 - Nuclear reaction analysis (NRA) for D depth profiling
 - Thermal desorption spectroscopy for total D retention
 - XPS, SAM, PAS, GD-OES for surface/bulk characterization.



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Neutron energy spectrum with Gd thermal neutron shielding



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Joyo HP

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Capsule design, specimen matrix and irradiation schedule

RB19J capsule design

- Cost sharing with JAEA-ORNL F82H irradiation program (300 °C zone)
- 500, 800, 1100 °C temperature zone for PHENIX tungsten irradiation

Specimen matrix for PHENIX Task 3: "Tritium behavior in irradiated tungsten"

- D6TQ : 6 mm OD, 0.25mm thick discs
- : 6 mm OD, 0.5 mm thick discs (standard) : ~ x80 each temp. zone D6TH
- D6T1 : 6 mm OD, 1.0 mm thick discs : ~ x10 each temp. zone
- D10TQ : 10 mm OD, 0.25 mm thick discs : ~ x15 each temp. zone
- D10TH : 10 mm OD, 0.5 mm thick discs

 $: \sim x10$ each temp. zone

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- : \sim x10 each temp. zone

RB19J irradiation completed

- Started: Cycle 466 (June, 2016)
- Ended: Cycle 469 (December, 2016)





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Future plan

Neutron-irradiated W specimen exposed to TPE

- Displacement damage vs. irradiation temperature
 - TITAN ('07-'12) focused on low temperature (<500°C) high dose (<< 1 dpa) in pure W
 - PHENIX ('13-'18) aims at high temperature (>500°C) high dose (> 1 dpa) in W and W alloy





PIE plans in PHENIX Task 3

1) PIE at ORNL (sharing data from Task 2)

- Positron Annihilation Spectroscopy coincidence lifetime (PAS-CL) for defect characterization
- Transmission Electron Spectroscopy (TEM) for defect characterization

2) PIE at STAR, INL

- Plasma-driven D/T/He implantation with TPE for D/T/He implantation
- Gas-driven T permeation with TGAP for T permeation
- Deuterium gas exposure with SGAS for D absorption
- PAS Doppler broadening measurement (PAS-DB) for defect characterization
- Thermal desorption spectroscopy (TDS) for total D/T/He retention measure't
- X-ray photoelectron spectroscopy (XPS) for surface chemical state
- Scanning Auger Microscopy (SAM) for surface elemental composition
- Glow discharge optical emission spectroscopy (GD-OES) for depth profiling
- 3) Nuclear reaction analysis (NRA) at SNL-MN (Bill Wampler)
- 4) Supporting PIE at JA Oarai Center
 - PAS-DB, PAS-CL, Compact Divertor Plasma Simulator (CDPS), TDS etc.

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• <u>TPE experimental condition:</u>

- $-n_{e,max}$
- $0.5 \times 10^{19} \text{ m}^{-3}$ $-\Gamma_{max}$ 0.5×10²³ m⁻² s⁻¹
- $-q_{heat}$ < 1.0 MW m⁻²
- $-T_{sample}$ <700 °C
- $-E_{ion}$ up to 600 eV
- Plasma species: He, D, T

- \rightarrow > 1.0×10¹⁹ m⁻³ (plan ^{*1})
- → > 1.0×10^{23} m⁻² s⁻¹ (plan ^{*1})
- \rightarrow > 1.0 MW m⁻² (plan ^{*1})
- \rightarrow up to 1100 °C (plan *2)

- PIE plans *3
 - D6TH specimens (Task 3 standard specimen)
 - Temperature (500-1100 °C), ion flux (1e22-1e23 m⁻²s⁻¹), ion fluence (1e26-1e28) m⁻²) dependence on plasma-driven D/T/He retention
 - D6TQ, D6TH, and D6T1 specimens
 - Thickness dependence on plasma-driven D/T/He retention
 - Thickness dependence on gas-driven D retention
 - D10TQ and D10TH specimens
 - Thickness dependence in gas-driven T permeation

NOTE:

*1) New source holder was designed based on UCSD PISCES-A, and is being fabricated at INL.

- *2) New sample holder is being designed based on UCSD PISCES-A.
- *3) Actual test matrix will be determined based on the number of intact HFIR irradiated samples after its shipment to INL.

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