

2012 PFC & MASCO meeting
Session 6: PMI Laboratory Experiments

“PFC activities in STAR”

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Outlines

1. Motivation of PFC work in Fusion Safety Program, INL
2. Recent progress/update in PFC activities
 - Completion of Ion implantation system refurbishment
 - Progress on TMAP modification
 - Progress on retention study in HFIR neutron-irradiated tungsten
3. Summary and future work

Motivation of PFC work in Fusion Safety Program, INL is:

- To understand tritium behavior in fusion reactor materials for blanket and divertor components because tritium retention and permeation determine in-vessel inventory levels and ex-vessel release quantities.
- Accurately predicting “*in-vessel inventory source term*” is a key safety issue for licensing future DEMOs and fusion reactors in off-normal and accident scenarios.
- FSP’s PFC work supports: 1) the US/Japan TITAN collaborative agreement and 2) *the International Energy Agency’s (IEA) agreement on the Environmental, Safety and Economic Aspects of Fusion Power (ESEFP) by utilizing an ion implantation device (IIX) and linear plasma device (TPE) with some data analysis performed with the tritium migration analysis program (TMAP) code.*
- ***Why “Safety” is important to PFC research?***
 - *The recent decision by the ITER organization to exclude carbon from the tritium phase of operations is based on predictions of unacceptable levels of tritium retained in co-deposited carbon layers. This is the example of how safety plays a major role in the PFC material choice.*

Motivation of PFC work in Fusion Safety Program, INL is:

“To understand tritium retention in tungsten under nuclear burning plasma environment (e.g. in neutron-irradiated PFCs)”

— Ion Implantation Experiment (IIX)

- Low D ion flux (10^{18} - 10^{20} m⁻² s⁻¹), high energy (500-1500 eV) ion implantation exp.
- Unique ion implantation device that can handle neutron-irradiated materials
- To investigate the flux and fluence dependence in tritium behavior in PFCs.

— Tritium Plasma Experiment (TPE)

- Divertor relevant high D ion flux (10^{20} - 10^{22} m⁻² s⁻¹), low energy (< 250 eV) linear plasma device
- Unique high-flux linear plasma device that can handle both tritium and neutron-irradiated materials
- To investigate tritium behavior in PFC under divertor-relevant plasma conditions.

— Tritium Migration Analysis Program (TMAP)

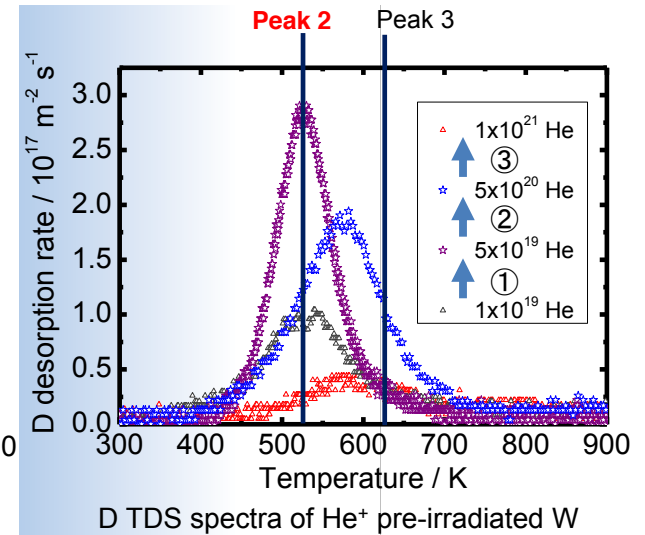
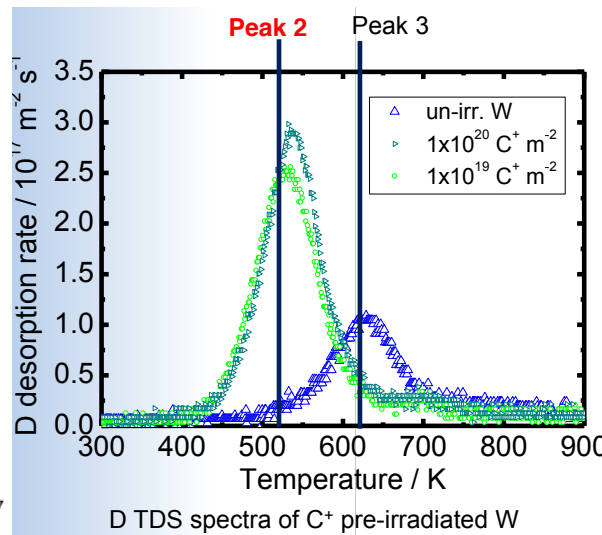
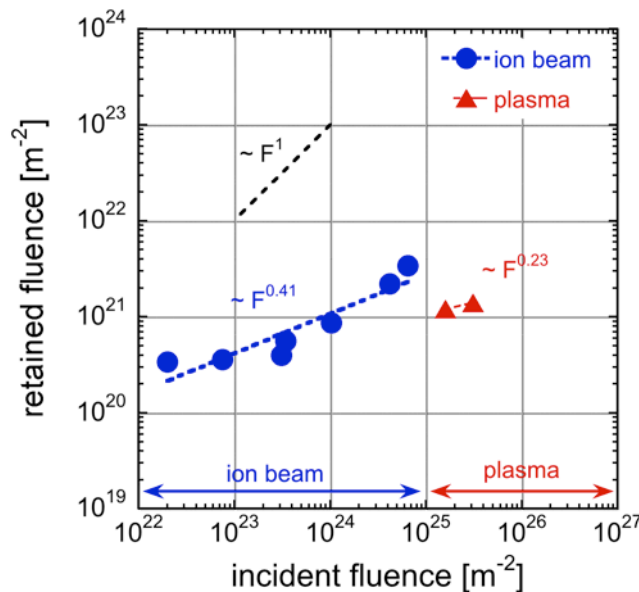
- Developed at INL to analyze tritium behavior (retention, permeation and loss) in fusion reactor structures, systems, and confinement rooms during normal operation and accident conditions using one dimensional (1-D) thermal and mass-diffusion equations including trapping of the tritium on defects within structural materials

Completion of ion implantation system refurbishment:

IIX, built by R. Anderl in the 90's, produced many important PFC results in 90's. Recently IIX has been reassembled/refurbished and is now ready to study the fundamental science of hydrogen isotope behavior in neutron-irradiated PFCs.

- Completion of reassembly and refurbishment (Dec. 2010)
- System verification (June 2011)
- Fully operational (June 2011-)

Ion beam and linear plasma devices cover 5 orders in magnitude of ion fluence



Progress on recent TMAP trapping model improvements:

- TMAP7 presently only allows three possible traps.
- TMAP4 was modified to allow the use of as many trap types as needed to verify the conclusions previously obtained by two separate TMAP7 calculations
- While the approach of modeling multiple trap types, each of a specific energy, can be used to reproduce TPE TDS results, it seems more reasonable to conclude that neutron damage would produce a more continuous distribution of traps with respect to trap energy
- To investigate this possibility, this same version of TMAP4 was modified to treat any specified distribution of possible trap site energies; in particular, lognormal distributions. The implementation of this capability followed that of a dust resuspension model recently developed at INL for the MELCOR code¹
- Both modifications were applied to TPE TDS results for W targets irradiated to 0.025 dpa after exposure to a TPE plasma at a target temperature of 200°C

¹B. J. Merrill & P. W. Humrickhouse, Fusion Engineering and Design 86 (2011) 2686–2689.

Progress on recent TMAP trapping model improvements:

- Two new TMAP trapping model was developed in TMAP4 by B. Merrill
 - Multiple trap model
 - Bimodal Lognormal trap model
- Initial results are promising when compared to complex TDS spectrum of neutron-irradiated tungsten, but more validation is needed

TMAP Lognormal Trap Model for Simulating Neutron-irradiated W

A lognormal distribution has the density function with trap energy (E) is:

$$\phi(E; \mu, \sigma) = \frac{1}{(2\pi)^{1/2} \sigma E} \exp \left\{ -\frac{[\ln(E) - \mu]^2}{2\sigma^2} \right\}$$

where μ is the lognormal mean and σ is the lognormal standard deviation. The concentrations of traps and trapped D₂ (#/m³) are:

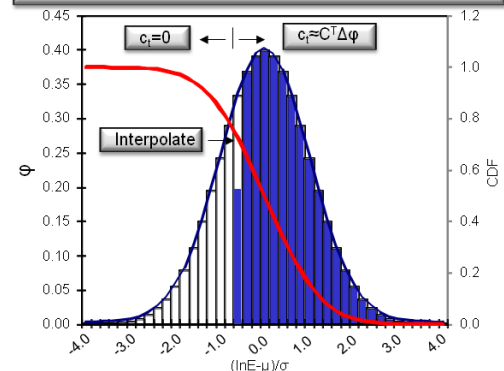
$$C^T = \int_{-\infty}^{\infty} C^T \phi(E) dE$$

$$c_i = \int_{-\infty}^{\infty} c_i(E) dE; \quad \frac{\partial c_i}{\partial t} = \int_{-\infty}^{\infty} \frac{\partial c_i(E)}{\partial t} dE$$

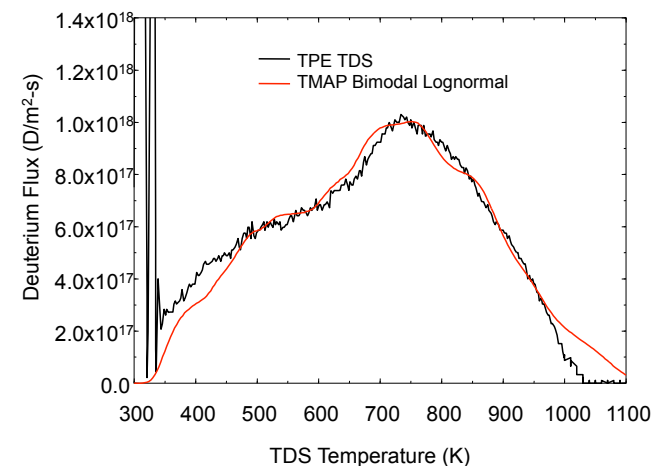
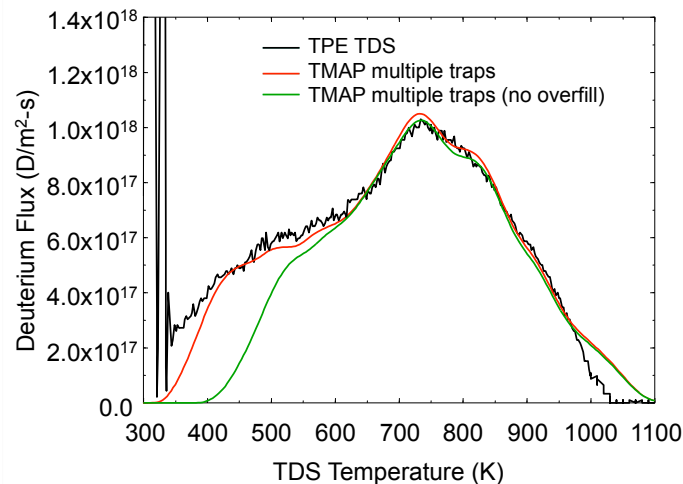
$$\frac{\partial c_i}{\partial t} = \int_{-\infty}^{\infty} \left(\frac{\alpha_i c_m}{N} (C^T \phi(E) - c_i(E)) - \alpha_r(E) c_i(E) \right) dE$$

$$\cong \sum_{j=1}^N \left(\frac{\alpha_i c_m}{N} (C^T \Delta \phi_j - c_{i,j}) - \alpha_{r,j} c_{i,j} \right)$$

Simplification¹: fill traps right to left (accounting for equilibrium). Only tracking filling a single trap.

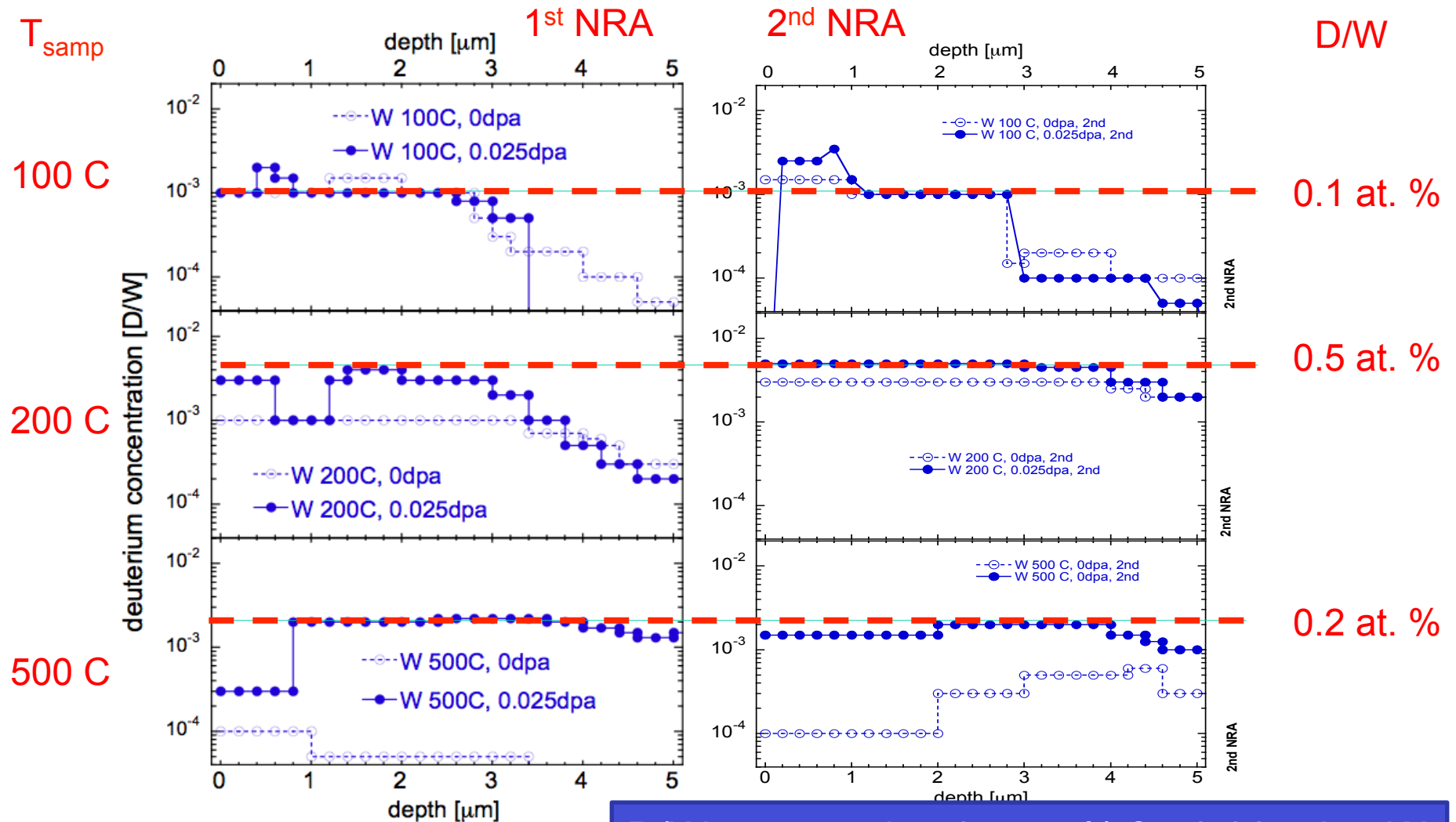


¹ B. J. Merrill & P. W. Humrickhouse, Fusion Engineering and Design 86 (2011) 2686–2689.



Progress on retention study in HFIR neutron-irradiated W

Investigation of saturation in trapping concentration via NRA



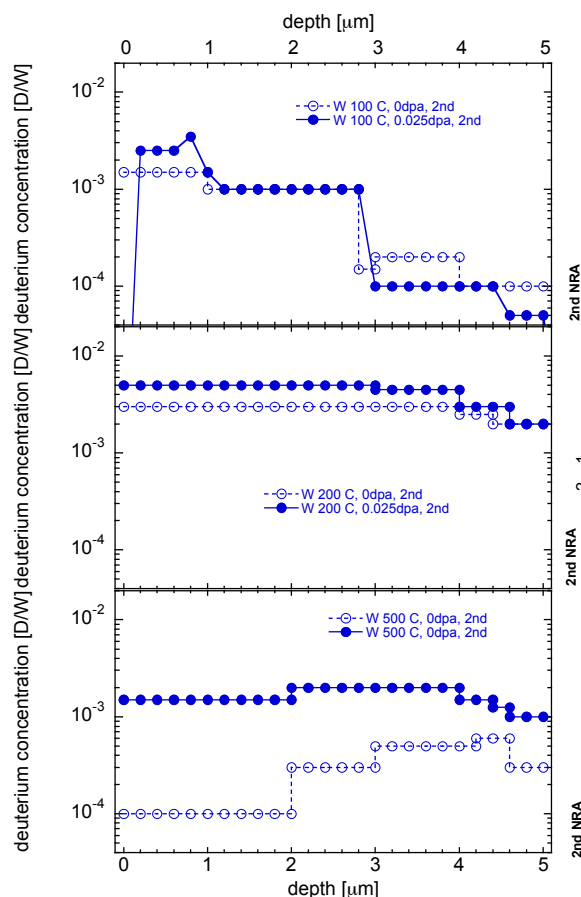
D/W saturated at 0.5 at. % for 0.025 dpa W

Progress on retention study in HFIR neutron-irradiated W

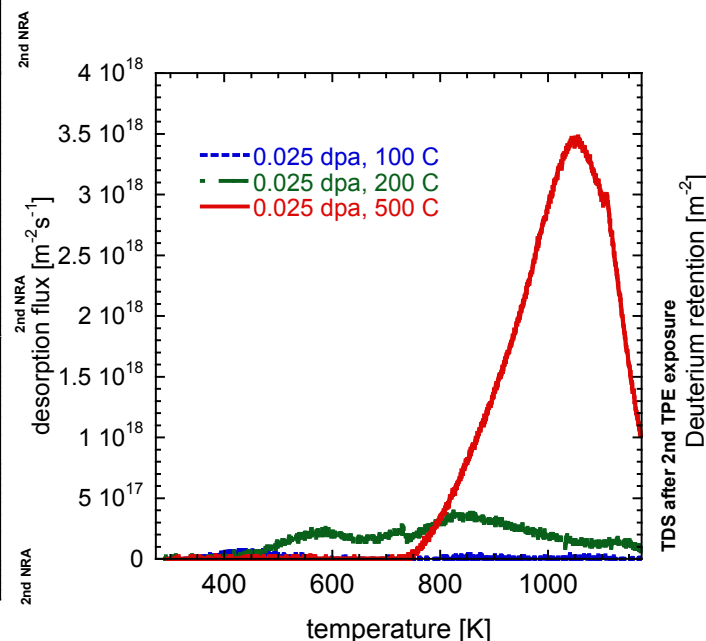
Experimental procedure:

- 1st TPE (@INL) → 1st NRA (@U of Wisc.) → 2nd TPE → 2nd NRA → final TDS
- Flux: $5 \times 10^{21} \text{ m}^{-2} \text{ s}^{-1}$, Fluence: (4-5) $\times 10^{25} \text{ m}^{-2}$ each TPE exposure, (8-10) $\times 10^{25} \text{ m}^{-2}$ in total fluence
- 6 specimens: 0 dpa and 0.025 dpa at 100, 200, and 500 C, Ion energy: 100 eV

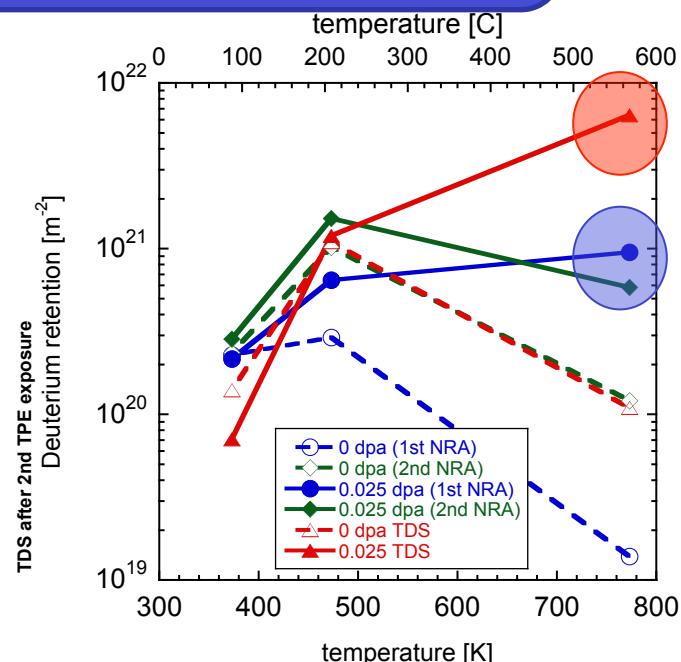
Large retention at 500 C
Discrepancy between TDS and NRA at 500 C
indicates that D is migrated and trapped in bulk
($> 5 \mu\text{m}$) → 50-100 μm ?



2nd NRA result



TDS after 2nd TPE exposure



TDS vs. NRA

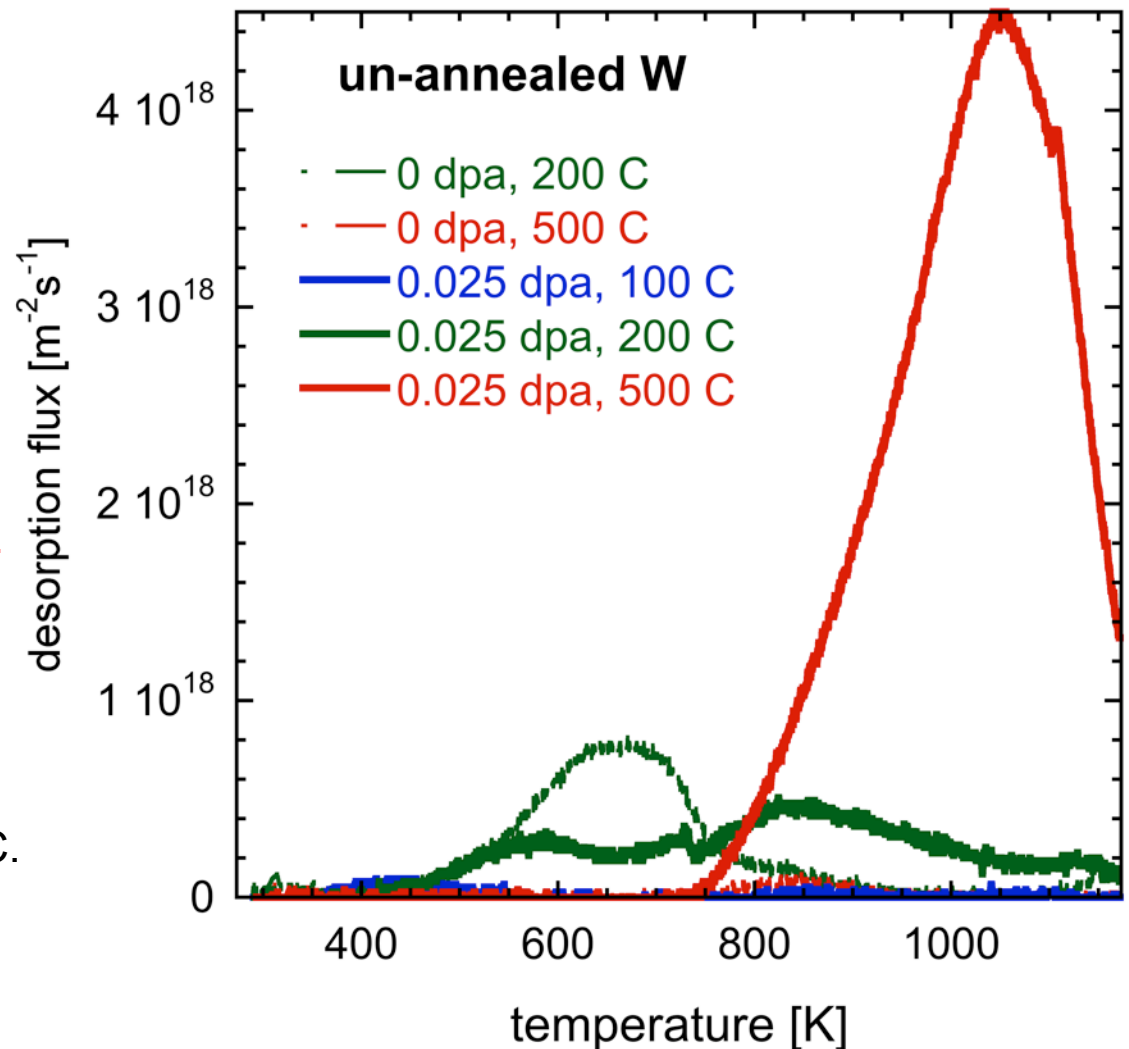
Thermal desorption spectra of pre-TDS W

Plasma exposure conditions:

- Exposure temp.: 100C, 200C, 500C
- Ion fluence: $(1.0-1.2) \times 10^{26} \text{ m}^{-2}$
- Incident ion energy: 100 eV
- Time Interval between TPE exposure and TDS: 600 days
- TPE → NRA → TPE → NRA → TDS

Experimental observations:

- D depth profile saturates at 0.5 at. % D/W
- Large D retention in 0.025 dpa, 500C
- 10 more D in TDS than NRA
→ D may be trapped up to 50 μm
- No D retention in 0.025 dpa, 100C.
- Very small D retention in 0.025 dpa, 200C
- Peak positions are all different
→ difficult to model with TMAP



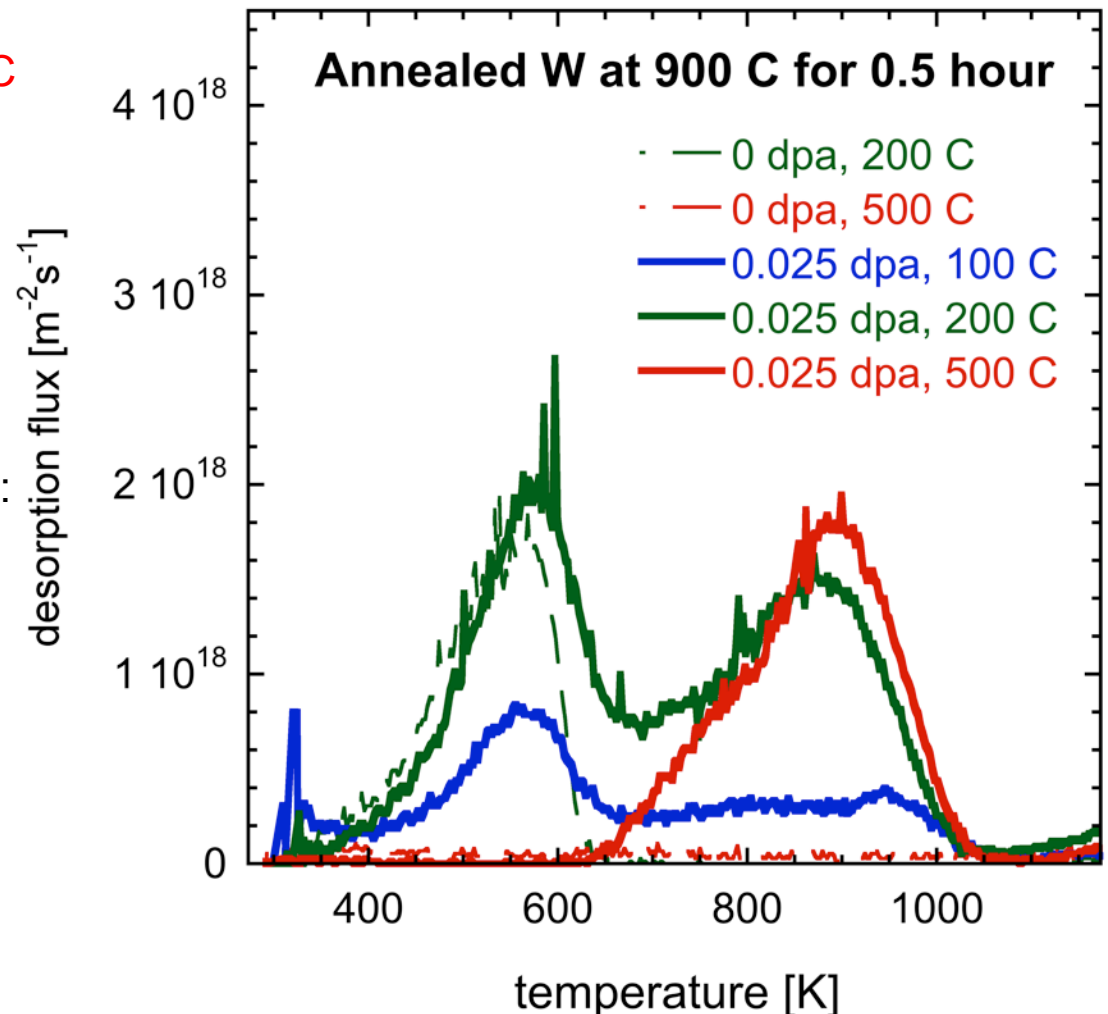
Thermal desorption spectra of post-TDS W

Plasma exposure conditions:

- Exposure temp.: 100C, 200C, 500C
- Ion fluence: $(1.0-1.2) \times 10^{26} \text{ m}^{-2}$
- Incident ion energy: 100 eV
- Time Interval between TPE exposure and TDS: < 24 hours
- No NRA

Experimental observations:

- Distinctive two peaks are observed:
 - Low temp. peak: 400-700 K
 - High temp. peak: 600-1050K
- Sum of 0 dpa 200 C (Low temp. peak) and 0.025 dpa 500C (High temp. peak) can be explained well with the two peaks observed for 0.025 dpa, 200 C.
- Shape spikes are observed at the beginning of heat-up for 0.025 dpa, 100 C. (this was thought to be due to oxide formation, but there should be no oxide after annealing)



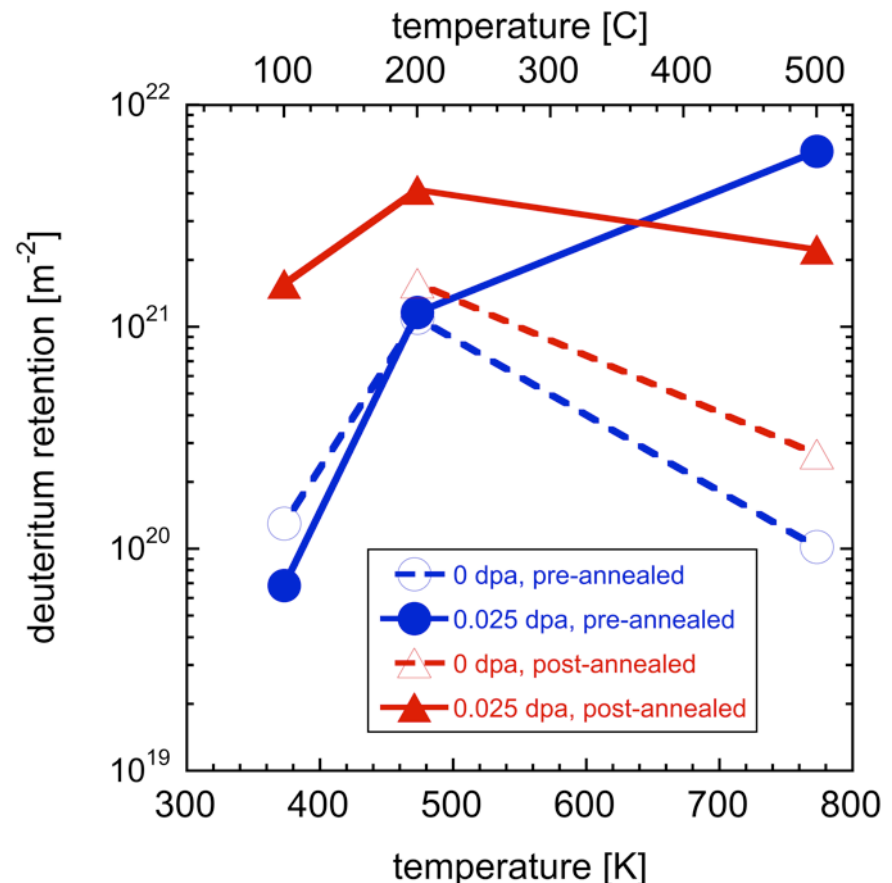
Comparison of post-annealed with pre-annealed

Plasma exposure conditions:

- Exposure temp.: 100C, 200C, 500C
- Ion fluence: $(1.0-1.4) \times 10^{26} \text{ m}^{-2}$
- Incident Ion energy: 100 eV
- Time Interval between TPE and TDS:
 - Pre-anneal: $\sim 600 \text{ day}^*$
 - Post-anneal: $< 1 \text{ day}$

Experimental observations:

- D retention for 0.025 dpa 100C increased by a factor of **x 10**
- D retention for 0.025 dpa 200C increased by a factor of **x 4**
- D retention for 0.025 dpa 500C decreased by **2/3**
- Annealing at 900 C for 0.5 hour suppressed the high temperature peak, but enhanced the D retention for the low temperature peak.
- Possible mechanism:
 - Vacancy migration to form voids
 - Bubble formation from the voids



900 C anneal decreased the vacancy density, increased the void density, and then increased nucleation site for bubble formation

- Low temp. peak: Bubble formation in void
- High temp. peak: Trapping in vacancy

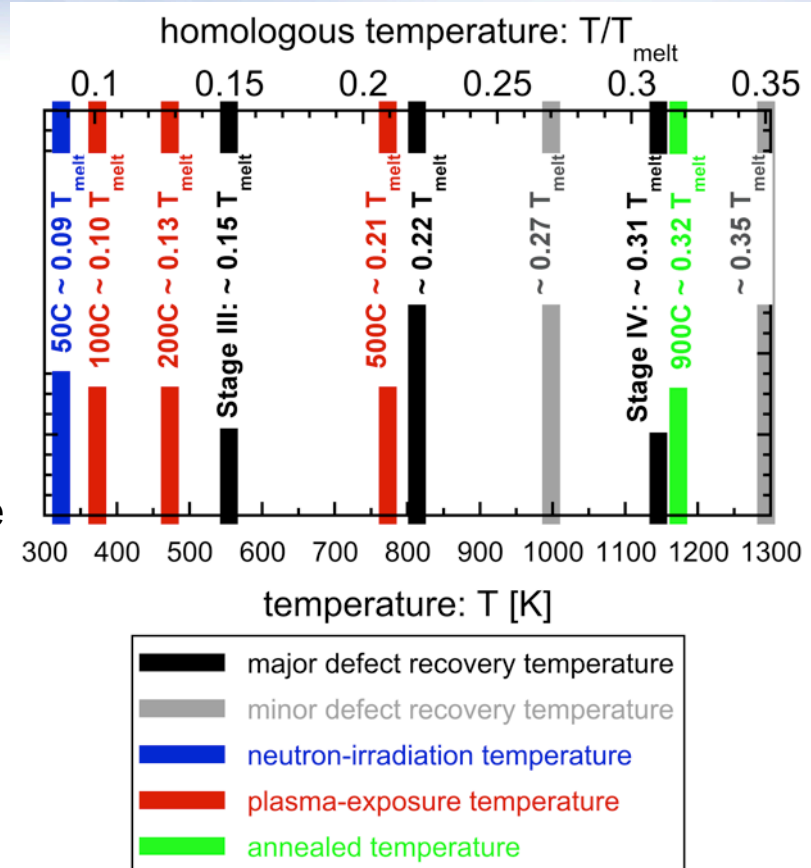
Temperature history of neutron-irradiated W

In the previous study (Pre-Annealed/TDS):

- 1) Irradiated at 50-80 C to 0.025 dpa at High Flux Isotope Reactor (HFIR), ORNL
- 2) Exposed at 100, 200, and 500C to deuterium plasma ($\sim 5 \times 10^{25} \text{ m}^{-2}$ ion fluence) at TPE, INL
- 3) Exposed to ^3He beam at RT to measure D depth profile up to 5 μm via Nuclear Reaction Analysis (NRA) at U. Wisconsin-Madison.
- 4) Repeat 2) and 3) one more time.
- 5) Heated up with 10 C/min to 900 C to measure D retention via Thermal Desorption Spectroscopy (TDS), and then held at 900 C for 0.5 hour for annealing

In this study (Post-Annealed/TDS)

- 1) Exposed at 100, 200, and 500 C to deuterium plasma ($\sim 5 \times 10^{25} \text{ m}^{-2}$ ion fluence) in TPE, INL
- 2) Heated up with 10 C/min to 900 C to measure D retention via TDS, and then held at 900 C for 0.5 hour



Stage III (0.15 T_m): SIA migration
 Stage IV (0.31 T_m): Vacancy migration
 900 C anneal can decrease the vacancy density and increase the void density

Ref: Keys, L.K. and Moteff, J., JNM '70 and PR '68

Conclusions

- Refurbishment of ion implantation system is completed
 - Ion beam and linear plasma devices cover 5 orders of magnitude in fluence
- Two new TMAP trapping models (multiple trap, and bimodal lognormal trap) were developed in TMAP4.
 - The initial results are promising to model complex TDS spectrum of neutron-irradiated tungsten, but more validation against additional data is needed.
- Very reproducible TDS spectrum were obtained for annealed/TDS'd at 900 C for 0.5 hour, with distinctive two peaks (Low temp. peak: 400-700 K and high temp. peak: 600-1050K) observed.
 - Annealing at 900 C for 0.5 hour suppressed the high temp peak by 2/3, but increased the D retention in the low temp. peak by a factor of 4.
 - Annealing at 900 C decreased the vacancy density, increased the void density, and then increased nucleation site for bubble formation
 - “High temperature peak (600-1050K) is the D desorption from the vacancy created by neutron-irradiation.”
 - “Low temperature peak (400-700K) is the D desorption from the bubbles formed in the voids.”

Conclusions.....Future work

If my conclusions are correct:

- “High temperature peak (600-1050K) is the D desorption from the vacancy created by neutron-irradiation.”
- “Low temperature peak (400-700K) is the D desorption from the bubble formed in the void.”

It's known that

- The detrapping energy from vacancy is ~ 1.4 eV.

➔ High temperature peak (600-1050K) should be ~ 1.4 eV (instead of 1.7-1.8 eV)

Brand new fit with the vacancy assumption of 1.5 eV, with 0.04 at. % D/W up to 100 micron fits the experiment of Y105 (0.025 dpa, 500C) well

