

**32<sup>nd</sup> Tritium Focus Group Meeting:**

***Tritium research activities in  
Safety and Tritium Applied Research (STAR)  
facility, Idaho National Laboratory***

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*April 25<sup>th</sup> 2013, Germantown, MD*

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# Outlines

1. Motivation of tritium research activity in STAR facility
2. Unique capabilities in STAR facility
3. Research highlights from tritium retention in HFIR neutron-irradiated tungsten



STAR facility

## ***Fusion Safety Program (FSP), Idaho National Laboratory***

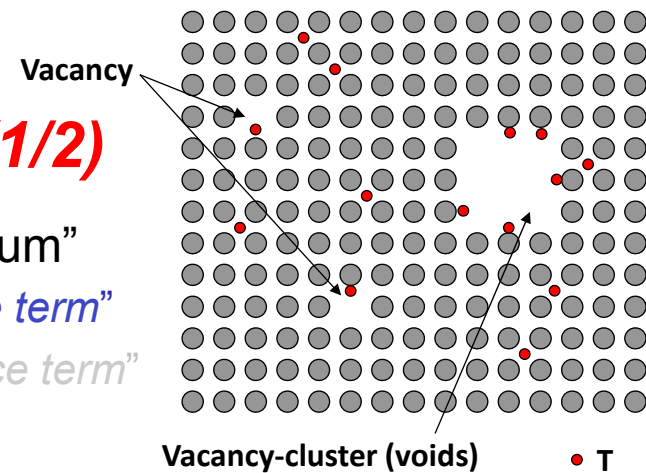
- FSP is supported by US Department of Energy (DOE) Office of Fusion Energy Sciences (FES) under Office of Science (SC)
- FSP consists of two (Analytical and Experimental) parts
  - Fusion Safety Analytical Research:
    - Fusion Safety Code Development, Risk Assessment Analysis, Dust Safety, ARIES Design support, ITER TBM Safety, Tritium Migration Analysis Program (TMAP) development, and International Energy Agency (IEA) implementing agreement on Environmental, Safety and Economic Aspects of Fusion Power (ESEFP) support
  - Fusion Safety Experimental Research:
    - Tritium research with Tritium Plasma Experiment and Ion Implantation Experiment, SNL/CA collaboration support and STAR operation management, Tritium breeder and coolant applications for blanket technology, IEA ESEFP supports, and US-Japan TITAN/PHENIX program support
  - Work for Others:
    - ITER Reliability, Availability, Maintainability, and Inspectability (RAMI)
    - Beryllium Dust Explosion Experiment
    - DOE Nuclear Energy (NE), Very High Temperature Reactor (VHTR)

## ***Safety and Tritium Applied Research (STAR) facility***

- Designated a US DOE **National User Facility** at the Idaho National Laboratory
- Classified as a Radiological facility, and it is restricted to a facility total tritium inventory of less than 16,000 Ci, to remain below a DOE Hazard Category 3 Non-Reactor Nuclear Facility
- Specializes in: tritium, activated materials (neutron-irradiated tungsten), advanced coolant (FLiBe, PbLi, He), and Be
- Both tritium and non-tritium fusion safety research are investigated:
  1. Interactions of tritium and deuterium with plasma-facing component (PFC) materials utilizing divertor relevant high-flux ( $>10^{22} \text{ m}^{-2}\text{s}^{-1}$ ) linear plasma device, Tritium Plasma Experiment, and low-flux high-energy Ion Implantation Experiment, and Tritium Migration Analysis Program (TMAP)
  2. Tritium breeder and coolant applications for blanket technology (tritium solubility and permeability in lead lithium eutectic)
  3. Fusion safety issues (beryllium dust explosion and steam reactivity)

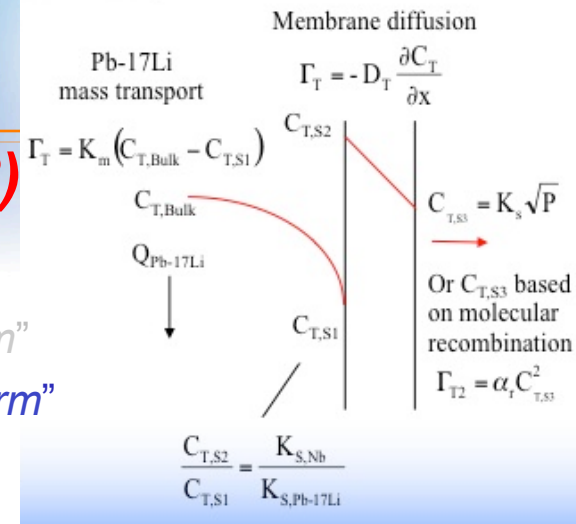
## Tritium related safety issues in fusion (1/2)

- 50% of fusion fuel is radioactive hydrogen isotope, “tritium”
  1. Tritium retention determines “*in-vessel inventory source term*”
  2. Tritium permeation determines “*ex-vessel release source term*”
- “Tritium retention in plasma facing components”
  - Diffusivity of hydrogen isotope in metal is very large, making it difficult to contain tritium (e.g.  $D_H \sim 10^{-8} \text{ m}^2\text{s}^{-1}$  in Fe at RT  $\rightarrow$  H will diffuse 1 mm in Fe in 100 sec at RT.)
  - The plasma facing components (PFCs) will subject to intense fusion fast neutrons to  $> 10$  dpa in DEMO/future reactor. (e.g. 0.7 dpa for W divertor in ITER)
  - Radiation damages will be created by 14 MeV throughout PFCs thickness.
  - Tritium is trapped in radiation damages (vacancy, vacancy-cluster, void etc.) in bulk PFCs
  - Large amount of tritium can be trapped in vacancy-cluster as gas form, leading to bubble formation, and blister formation in metal
  - Tritium behavior in the fusion nuclear environment is not fully understood
- STAR facility operates the Tritium Plasma Experiment (TPE):
  - the only existing high-flux linear plasma device that can handle both tritium and neutron-irradiated materials in the world fusion community



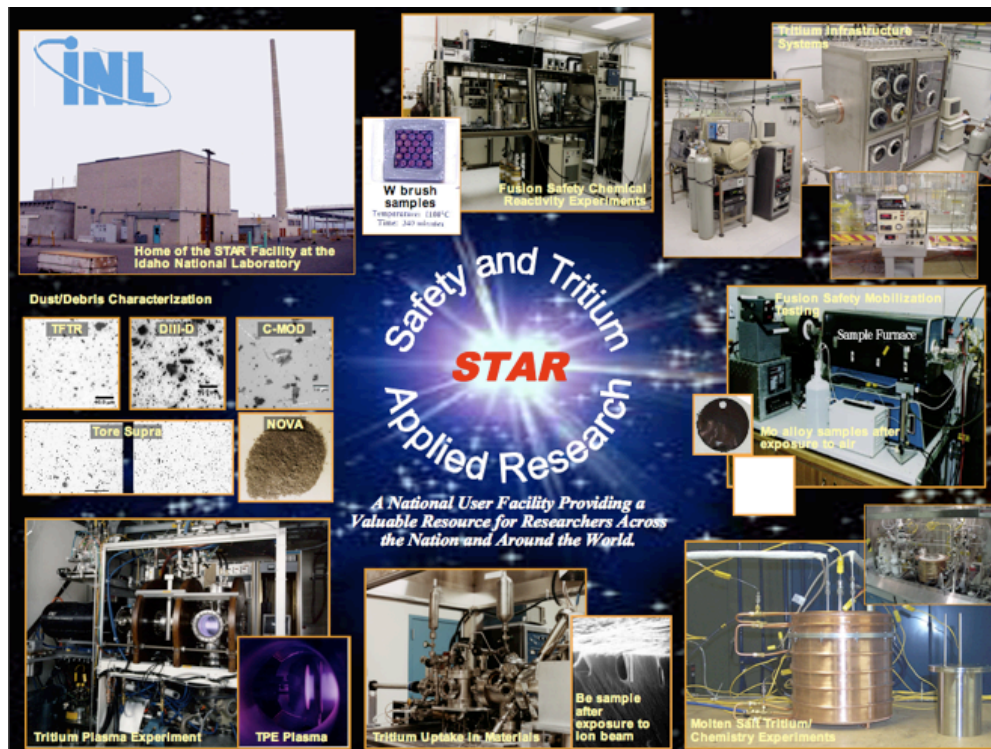
## Tritium related safety issues in fusion (2/2)

- 50% of fusion fuel is radioactive hydrogen isotope, “tritium”
  - Tritium retention determines “*in-vessel inventory source term*”
  - Tritium permeation determines “*ex-vessel release source term*”
- “Tritium permeation in blanket/structural/barrier materials”
  - Mass transport properties (e.g. diffusivity, solubility, and permeability) of tritium at realistic blanket conditions (e.g. low tritium partial pressure < 1000 Pa) is important for tritium blanket system design, but the data is very limited.
  - Tritium permeation barrier materials can reduce the release to the environment, however, the performance of tritium permeation barrier materials (e.g. ceramics) is unknown under fusion nuclear environments due to strong radiation field and displacement damage.
  - Tritium behavior in blanket/structural/barrier materials at realistic blanket conditions (e.g. low tritium partial pressure < 1000 Pa) is not fully understood**
- STAR facility operates the Tritium Heat Exchanger (THX) experiment and the Tritium Lead Lithium Eutectic (TLLE) experiment:**
  - Designed to measure the mass transport properties of tritium at realistic blanket conditions (e.g. low tritium partial pressure < 1000 Pa) in metal and liquid breeder material (e.g. PbLi and FLiBe) with tubular (THX) and disc/liquid metal (TLLE) sample



# Outlines

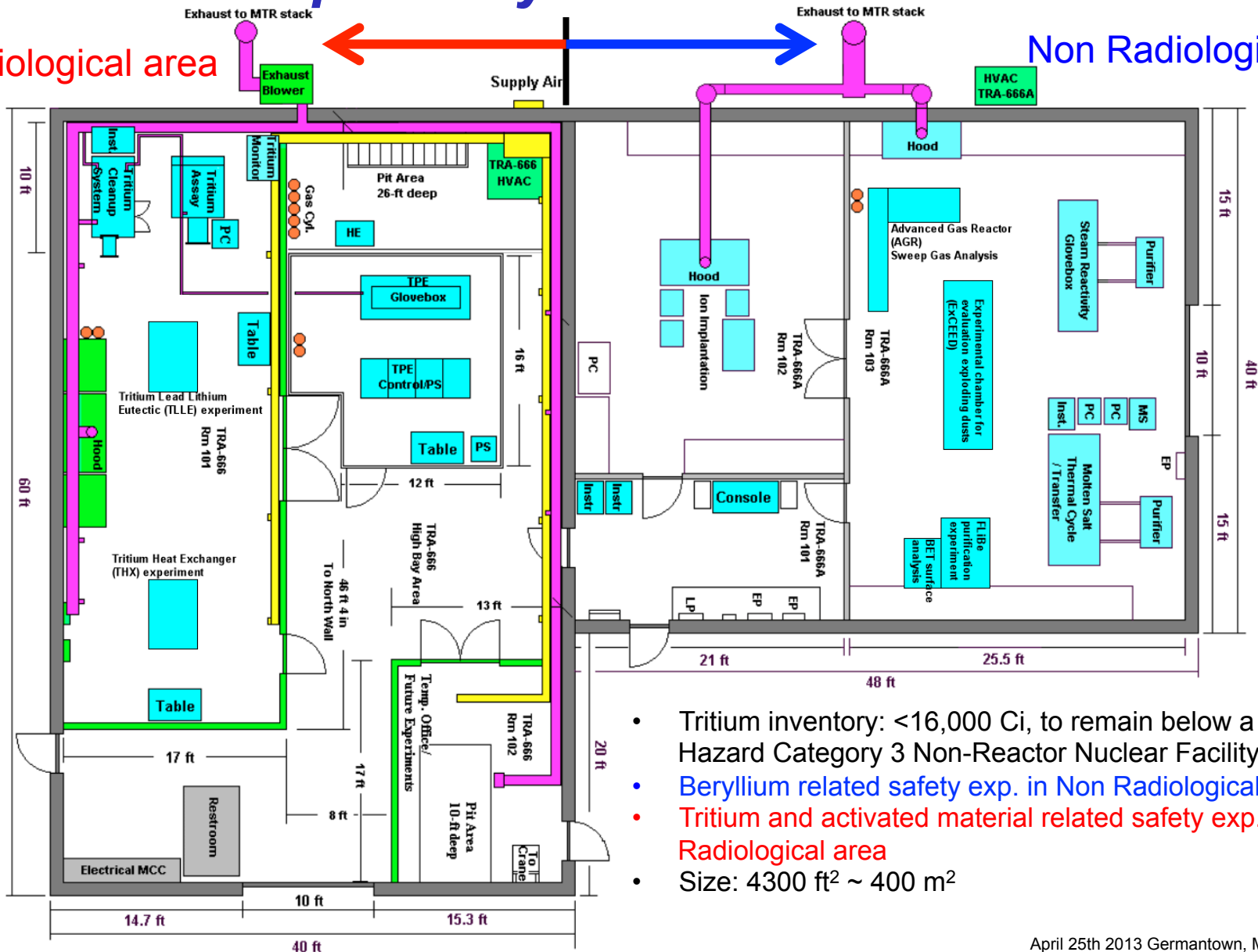
1. Motivation of tritium research activity in STAR facility
2. Unique capabilities in STAR facility
3. Research highlights from tritium retention in HFIR neutron-irradiated tungsten



# STAR Floorplan Layout

Radiological area

Non Radiological area



- Tritium inventory: <16,000 Ci, to remain below a DOE Hazard Category 3 Non-Reactor Nuclear Facility
- Beryllium related safety exp. in Non Radiological area
- Tritium and activated material related safety exp. in Radiological area
- Size: 4300 ft<sup>2</sup> ~ 400 m<sup>2</sup>



# Experimental infrastructures in STAR

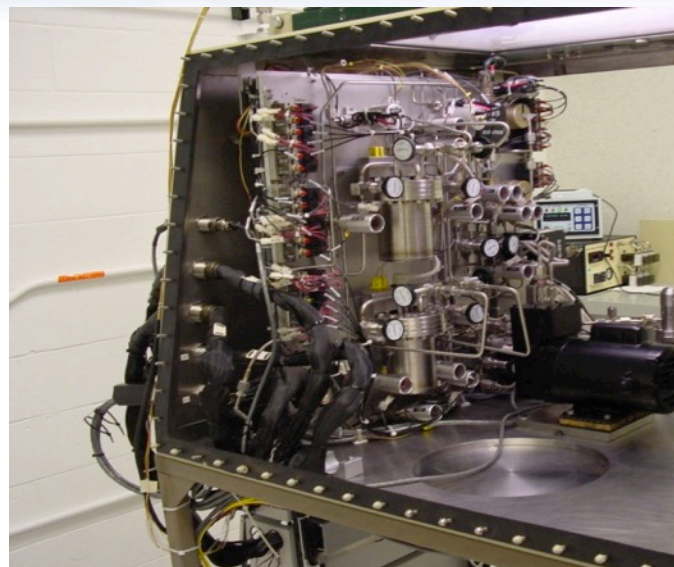
## Non Radiological area:

1. Three inert atmosphere gloveboxes
  - Preparation, purification and testing of FLiBe ( $2 \text{ LiF} \cdot \text{BeF}_2$ )
  - Steam oxidation for fusion safety
  - Beryllium dust explosion for fusion safety
2. One ventilated enclosure
  - High-energy (up to 3000 eV) Ion Implantation Experiment (IIX)
3. One class-A ventilation hood
4. Others:
  - Advanced Graphite Capsule (AGC) and Advanced Gas Reactor (AGR) Exhaust Gas Analysis Experiment

## Radiological area:

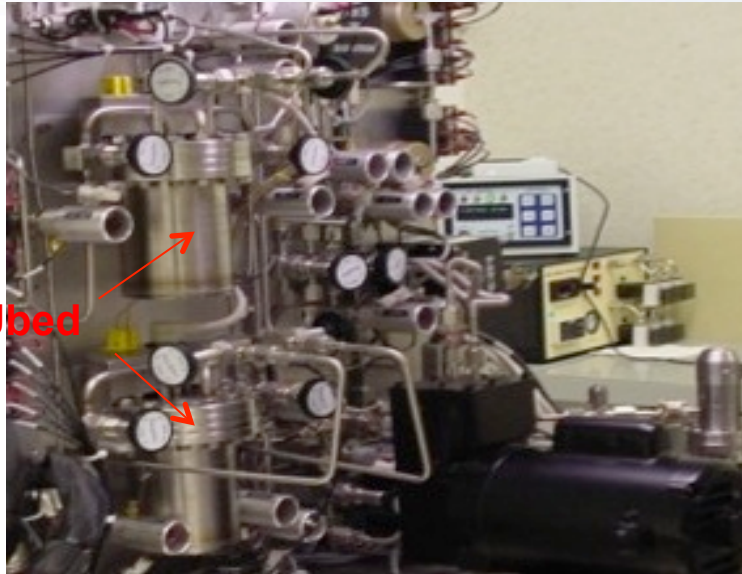
1. One inert atmosphere glovebox for high concentration tritium
  - Tritium Storage and Assay System (SAS)
2. Five ventilated enclosures
  - Tritium Plasma Experiment (TPE): unique linear plasma device to test neutron-irradiated fusion material with tritium plasma
  - Tritium permeation measurement for fission/fusion materials (THX)
  - Tritium lead lithium eutectic (TLLE) experiment
  - Diamond Wire Saw (DWS) for tritium depth profiling
  - Tritium Cleanup System (TCS) for tritium effluent decontamination
3. One class-A ventilation hood for tritium use

# STAR Storage and Assay System (SAS)



- Designed to store and transfer/supply tritium for various experimental needs
- Utilizes two 50 gram depleted uranium beds to store maximum inventory (15000 Ci) of tritium
- Enclosed in an inert (argon) atmosphere glovebox for high concentration tritium
- Current tritium inventory is approximately 2750 Ci in the SAS

# Tritium shipment from Savannah River Site (SRS)

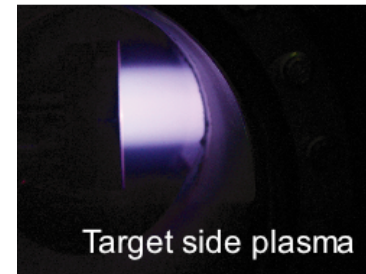
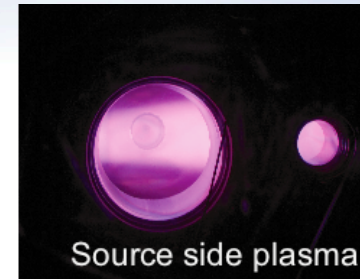
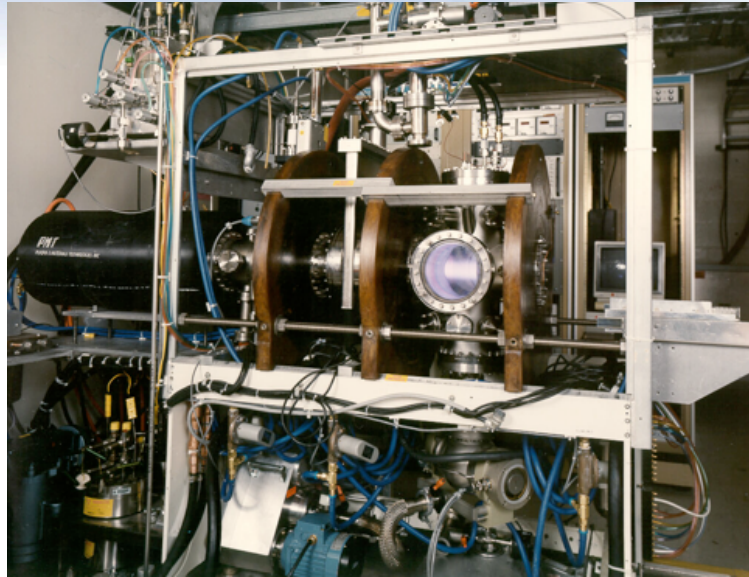
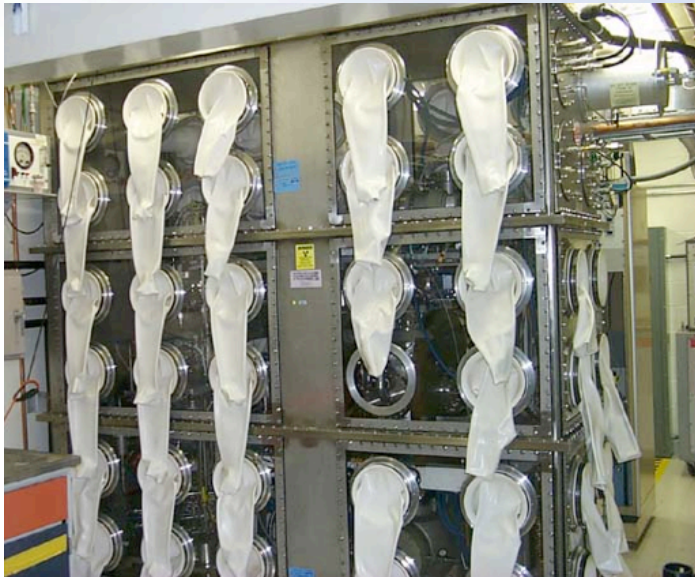


50 g Ubed  
(x2)



- Two CTCV-2 tritium cylinders (TCV-14 and TCV-42) were certified at the Savannah River Site (SRS) on 10/08/2012. The certification expires on 10/08/2015. The cylinders are currently stored at SRS.
- Tritium shipments from the SRS limited to 1000 Ci with standard type-A shipping container
- The last tritium shipment from the SRS arrived on 11/18/2010. Two cylinders contains ~ 950 Ci of tritium each ~ 1900 Ci total.
- Current tritium inventory is approximately 2750 Ci in the SAS

# Tritium Plasma Experiment - TPE



## Unique capabilities

- TPE is contained within double enclosure (PermaCon Box and Glovebox)
- Tritium use (max. T inventory: < 1.5g in STAR)
- Handling of “neutron-irradiated materials”
- Cutting tritiated material in Glovebox
- Plasma-driven tritium permeation capability (under development)

## Research Goals

- High Z metal R&D for ITER
  - Material under direct plasma exposure
  - Low dose neutron irradiated material
  - Impact of blistering on retention
- High Z metal R&D for DEMO and FNSF
  - High dose neutron irradiation effects on tritium retention and tritium permeation

# Brief history of TPE and host tritium facilities (1/2)

## I. 1983-early 1990's: Tritium Research Laboratory (TRL), SNL-Livermore

- Established as the **Tritium Plasma eXperiment (TPX)** and operated for 10 years
- RF driven plasma (390 MHz) up to 200 W; axially magnetized to  $\sim 150$  G, plasma density  $\sim 3 \times 10^{11}$  ions/cm<sup>3</sup>, Te  $\sim 10$  eV, on-sample ion flux 10 mA/cm<sup>2</sup>
- Performance: T throughput  $\sim 0.1$ g/day; experiment placed in a high-velocity ventilation hood for T contamination control; pumping system exhausted to TRL vacuum effluent recovery system, diagnostics included Langmuir probes, QMS (plasma species and permeation species), in-situ AES
- Decision was made to upgrade the TPX, and then close the TRL in 1992

## II. early 1990's-2002: Tritium Systems Test Assembly (TSTA) at LANL

- Rename as the **Tritium Plasma Experiment (TPE)**, and upgraded to hot cathode reflex arc w/ LaB<sub>6</sub> source, returned to tritium operation in 1995, and operated for 7 yrs
- Performance: Increased maximum T throughput to  $\sim 0.5$  g/hr; direct-feed of T from TSTA facility, or local T source from a U-Bed; T effluent captured on U-Beds, ion fluxes up to 1 A/cm<sup>2</sup> and 100 - 200 eV energy, increased pumping speed to 2200 l/s, diagnostics included Langmuir probes, QMS.
- System placed in a glovebox with atmosphere T monitoring and purge gas control
- Decision was made to close the TSTA and relocate the TPE.

## ***Brief history of TPE and host tritium facilities (2/2)***

### ***III. 2002-present: Safety and Tritium Applied Research (STAR) facility, INL***

- Tritium contamination level as high as 300,000 dpm / 100 cm<sup>2</sup> located within instrument racks and power supply chassis (CA limit is 10,000 dpm / 100 cm<sup>2</sup>).
- Decontamination efforts unsuccessful at reducing levels below CA limit.
- Substantial facility modifications were made to build a PermaCon enclosure (CA boundary), re-route and expand electrical service, modify facility ventilation, extend the fire suppression system into the PermaCon.
- Returned to deuterium operation in 2005, and returned to tritium operation in 2009.
- Performance: maximum T throughput ~ 0.05g/day; experiment placed in a ventilated enclosure (HCA boundary) and Permacon enclosure (CA boundary); local T source from a 300 cc cylinder; T effluent captured on U-Beds, ion fluxes up to 1 A/cm<sup>2</sup> and 100 - 200 eV energy, decreased pumping speed to 900 l/s, diagnostics included Langmuir probes, QMS, and optical spectrometers.
- ***New capabilities at STAR:***
  - Handling of “neutron-irradiated materials”
  - Cutting tritiated material in ventilated enclosure
  - Plasma-driven tritium permeation capability (under development)

# Comparison of plasma parameters among Existing and proposed US Linear Plasma Devices

	<b>PISCES-B (UCSD)</b>	<b>TPE (INL)</b>	<b>PMTS (ORNL)</b>
Deuterium ion flux: $\Gamma_i$ ( $\text{m}^{-2}\text{s}^{-1}$ )	$10^{21}\text{--}10^{23}$	$10^{20} - 3.7 \times 10^{22}$	$>10^{23}$
Incident ion energy: $E_i$ (eV)	20–300 (bias)	50–200 (bias)	??
Electron temperature: $T_e$ (eV)	4 – 40	5 – 20	3-50
Ion temperature: $T_i$ (eV)	2 – 5	2 - 5	1 - 200
Electron density: $n_e$ ( $\text{m}^{-3}$ )	$10^{18}\text{--}10^{19}$	$10^{16} - 3.5 \times 10^{18}$	$10^{18}\text{--}3 \times 10^{19}$
Max. heat flux: $P_{\text{max}}$ ( $\text{MW}/\text{m}^2$ )	5	~1.2	20
Plasma diameter (mm)	75	50	120
Max. specimen size	$\phi \sim 25.4$ mm disc	$\phi \sim 50.8$ mm disc	100 x 100 mm plate
Pulse length (s)	Steady state	Steady state	Pulse and Steady state
Activated targets	No	Yes	Yes
Tritium	No	Yes	No
Beryllium	Yes	Yes/No*	Yes
Permeation capability	No	Yes**	No
Ion incident angle	Normal	Normal***	Inclined and Normal
Plasma source (cathode)	Reflex arc ( $\text{LaB}_6$ )	Reflex arc ( $\text{LaB}_6$ )	Helicon (no cathode)
Year of operation	Since 1988	Since 1989	Proposed phase
Unique capabilities	In-situ surface analysis, transient surface heating, beryllium testing	Tritium use and diagnostics, neutron irradiated materials	Electrodeless plasma (Helicon + ECH + ICH) minimizes plasma contamination by impurity

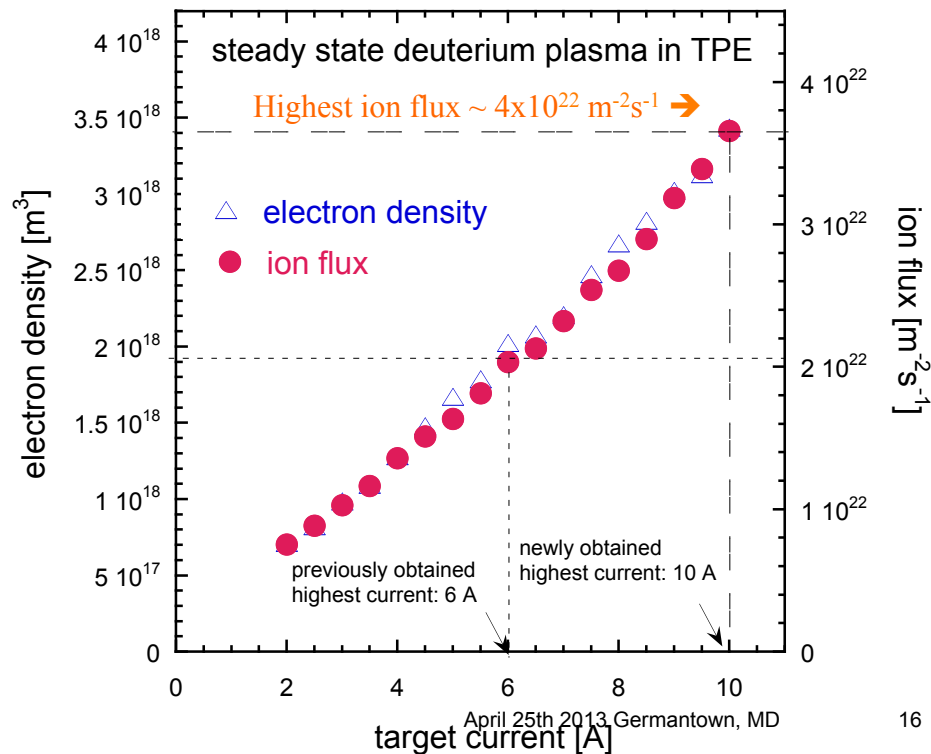
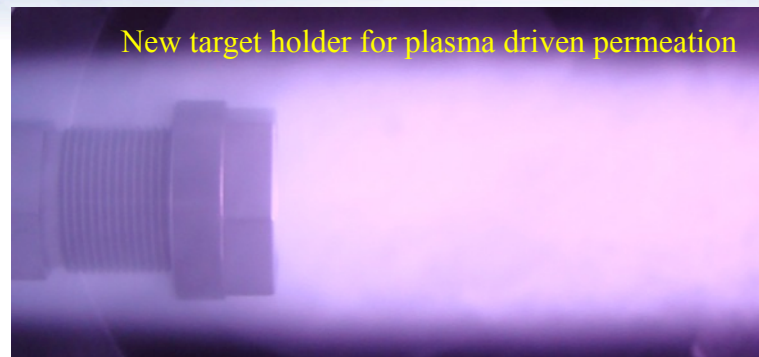
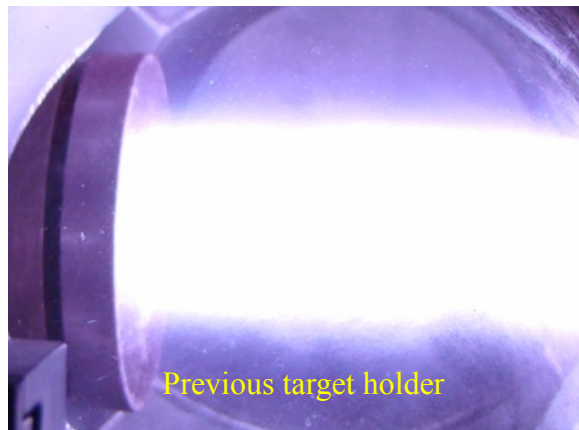
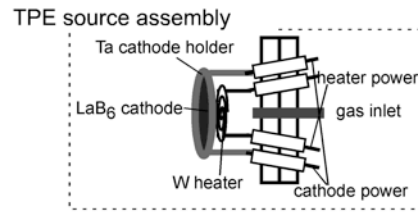
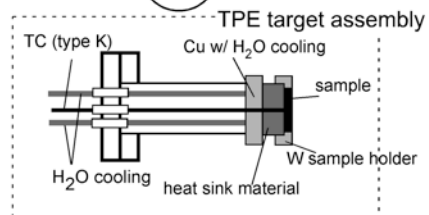
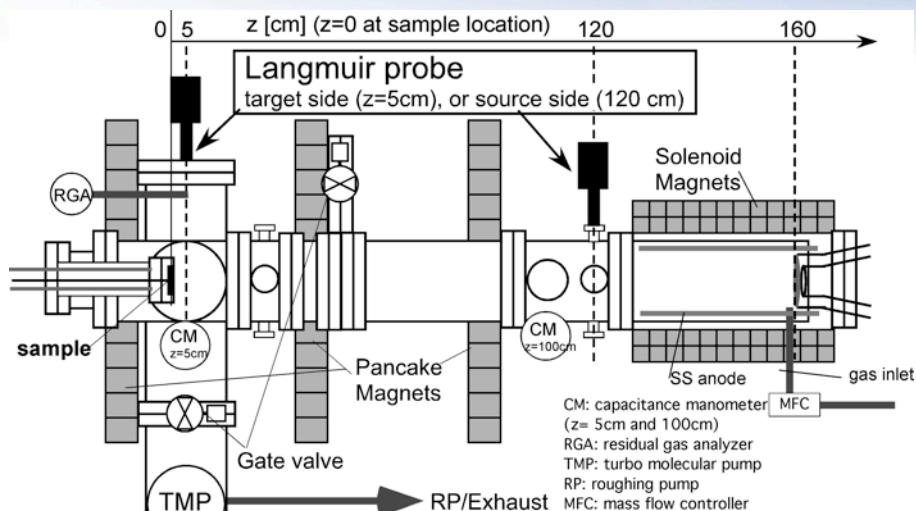
NOTES:

\*: Beryllium has been extensively tested in TPE during its tenure at TSTA, LANL in 90's, but it has not been actively tested in INL.

\*\* : Tritium plasma-driven permeation capability is under development with the SNL/CA collaboration April 25th 2013 Germantown, MD

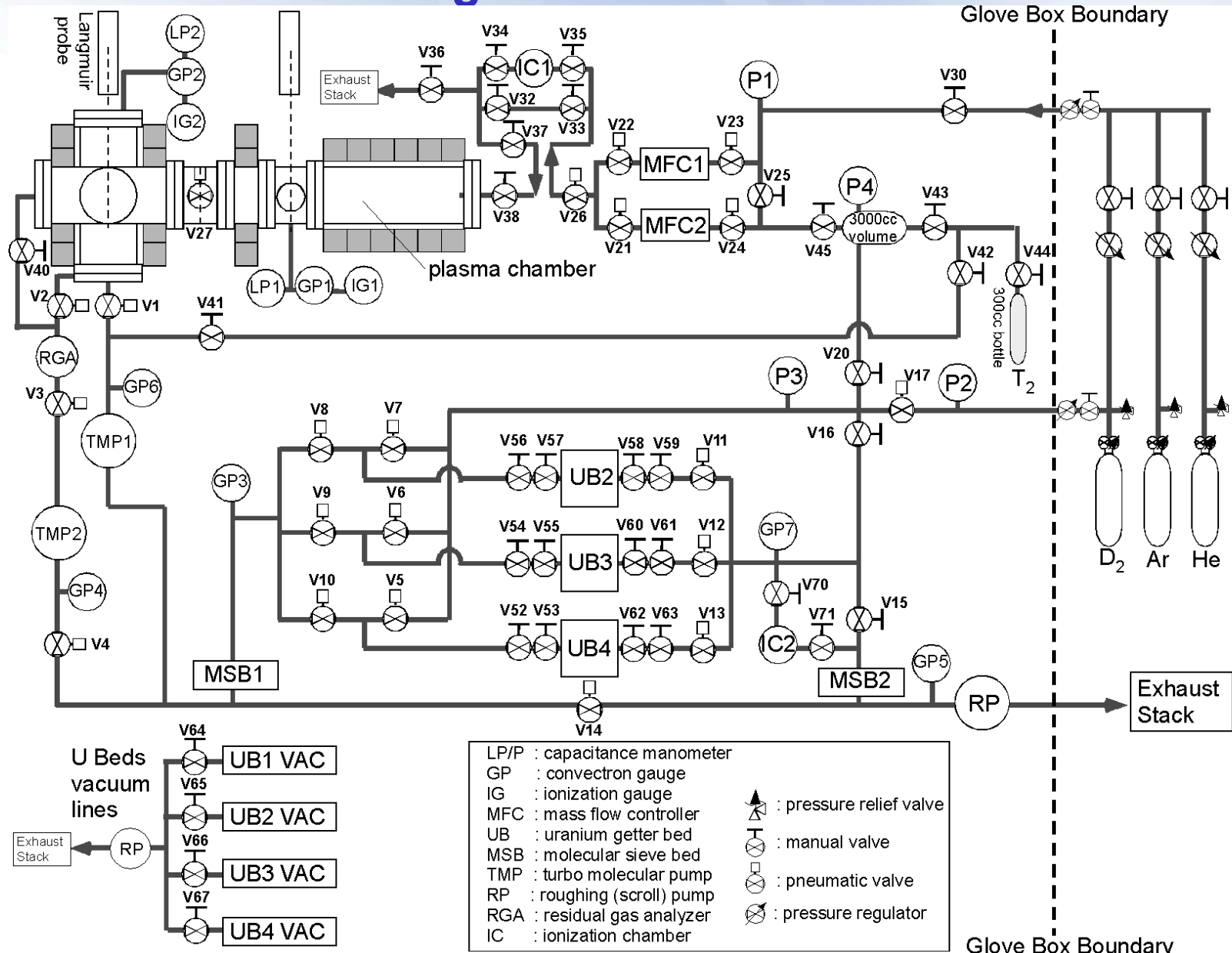
\*\*\*: Incident angle can be varied upon target holder design, and the current target holder is designed for normal incidence only.

# Plasma characterization in TPE



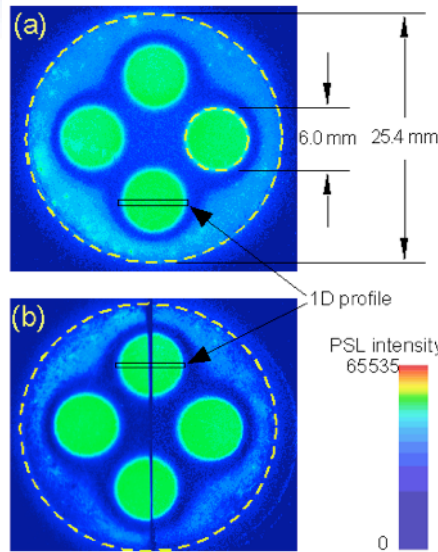


# Schematic/flow diagram of TPE

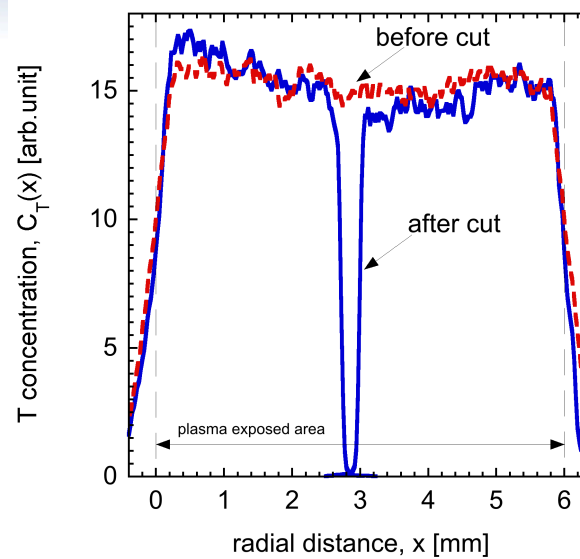


# Unique experiment with trace amount of tritium in TPE (1/2)

Imaging plate image of tungsten disc sample ( $\phi=25.4\text{mm}$ , 3mm) exposed to 0.5%  $T_2/D_2$  plasma for 2 hours.



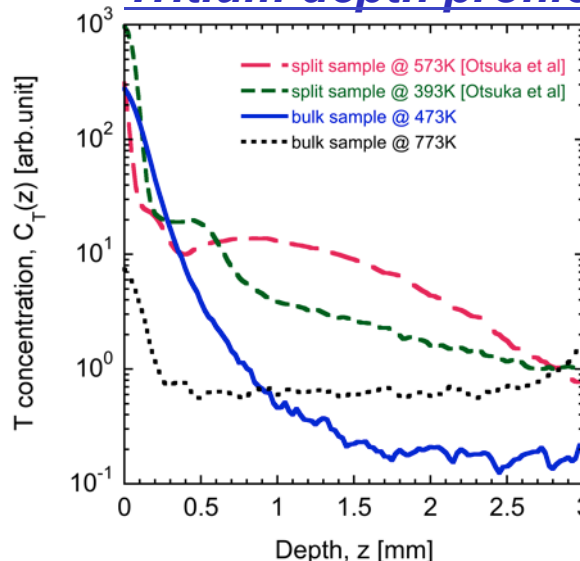
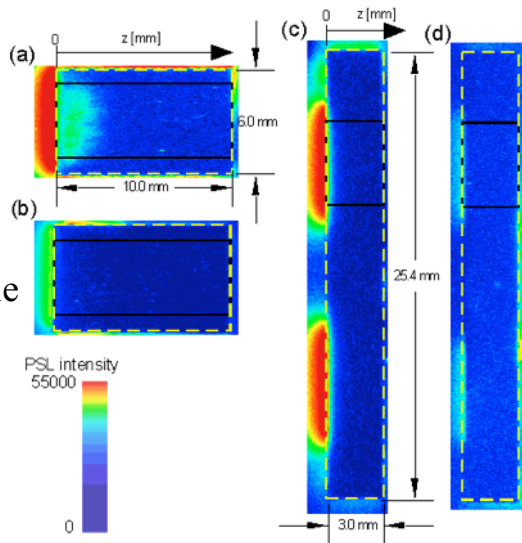
The sample cut in half by diamond wire saw



Imaging plate radial profiles of surface tritium conc. before and after cut

## Tritium depth profile in PFC:

Imaging plate of cross-sectional surface to measure tritium depth profile

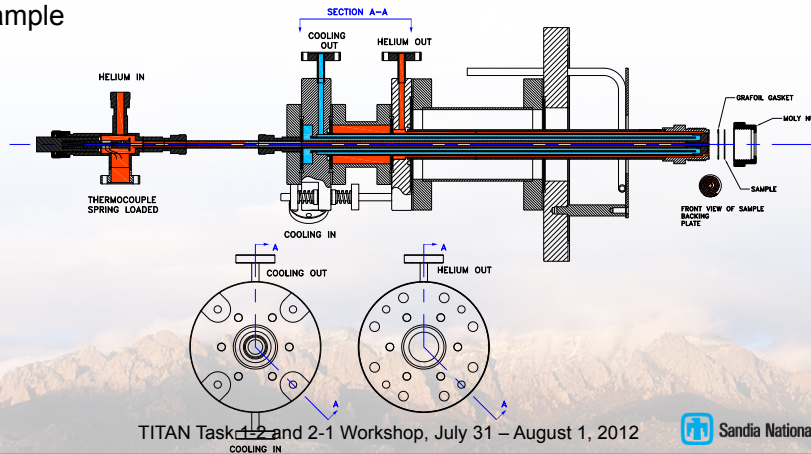


Imaging plate depth profiles of tritium concentration at 200C and 500C

# Unique experiment with trace amount of tritium in TPE (2/2)

## Tritium Permeation Stage Design Activities

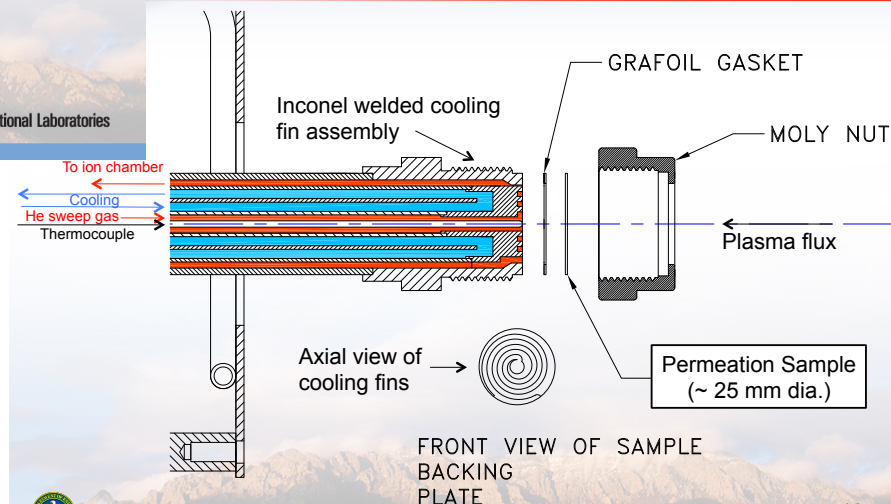
- 3<sup>rd</sup> generation layout of permeation stage includes several new features
  - Helium flow gas to capture permeating D/T between cooling fins and sample
  - Sample is sealed on downstream side (Grafoil gasket)
  - High pressure bellows allow provide variable loading of cooling fins to sample



TITAN Task 1-2 and 2-1 Workshop, July 31 – August 1, 2012

Sandia National Laboratories

## Expanded View of Sample Region



## Plasma-driven tritium permeation:

- Use helium as the sweep gas, and utilizes 1000 cc ion chamber to measure  $> 10^{-6}$  Ci/m<sup>3</sup>.
- Tritium enhanced the detection sensitivity
- Under development with SNL-CA collaboration

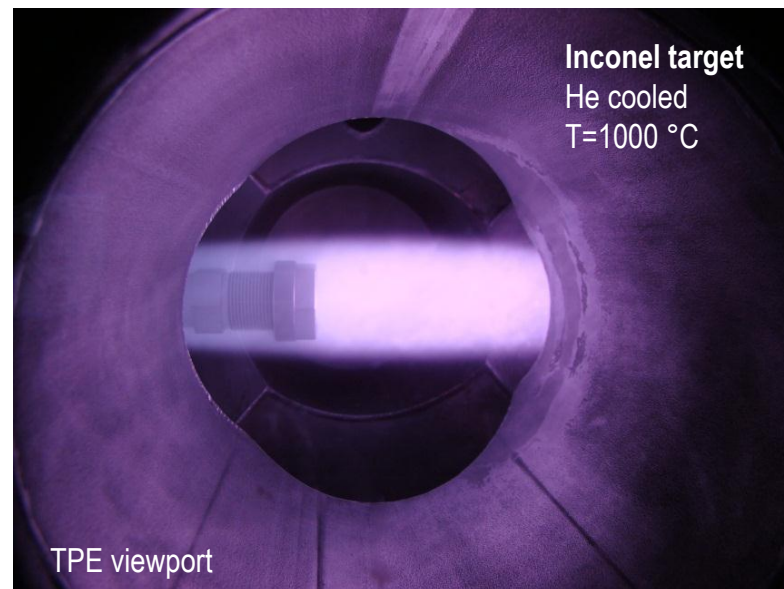
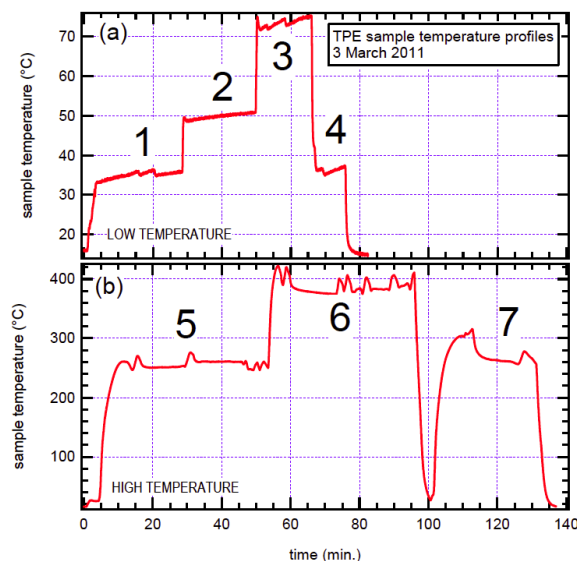
# Unique experiment with trace amount of tritium in TPE (2/2)

Collaboration with SNL/CA (R. Kolasinski leads)

- Key challenge for plasma-driven permeation: stable operation at high temperature.
- Developed two retention stages (Cu and Inconel) to test new design concept.
- Leveraged concentric cooling channel design from PISCES.
- Successful testing Inconel target to  $T=1000$  °C using He cooling.



**Cu target**  
Water cooled  
 $T < 500$  °C

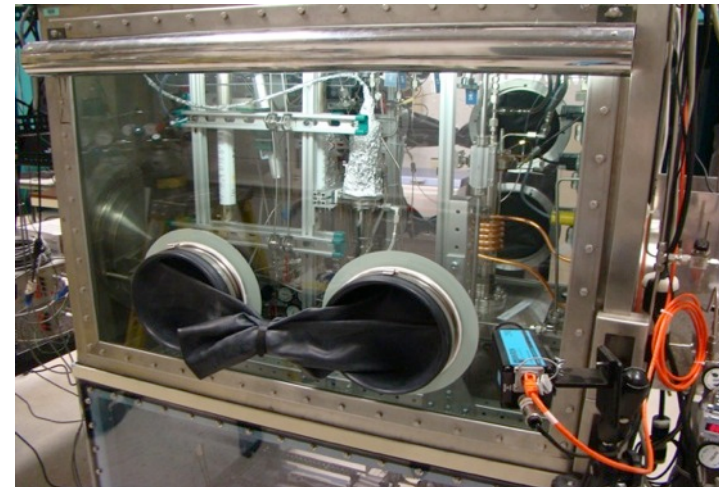
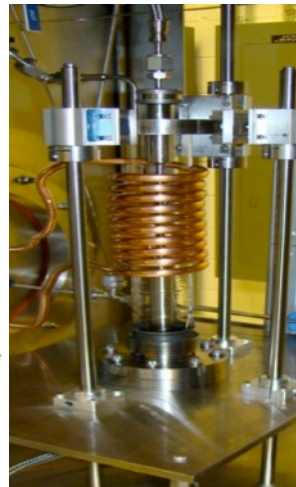
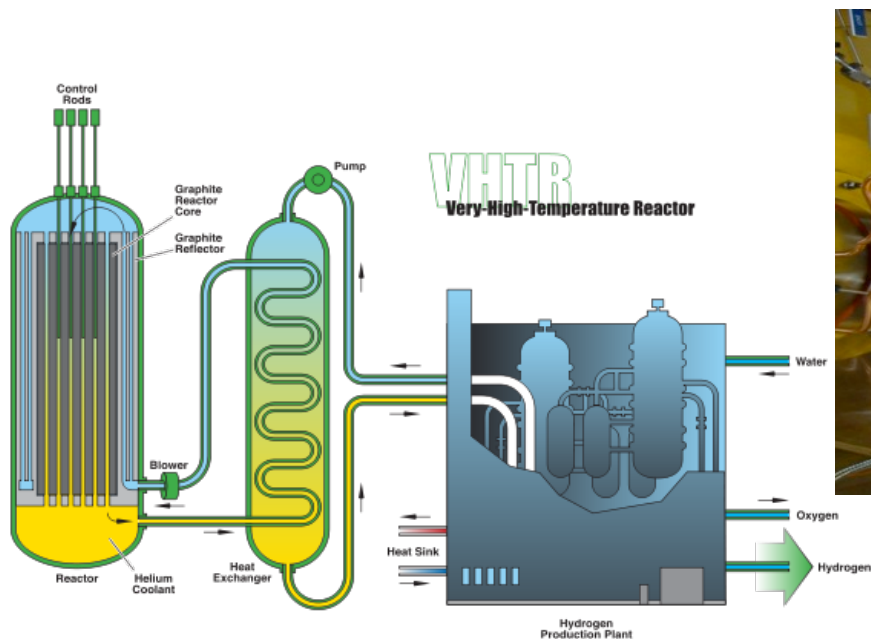


**Inconel target**  
He cooled  
 $T=1000$  °C

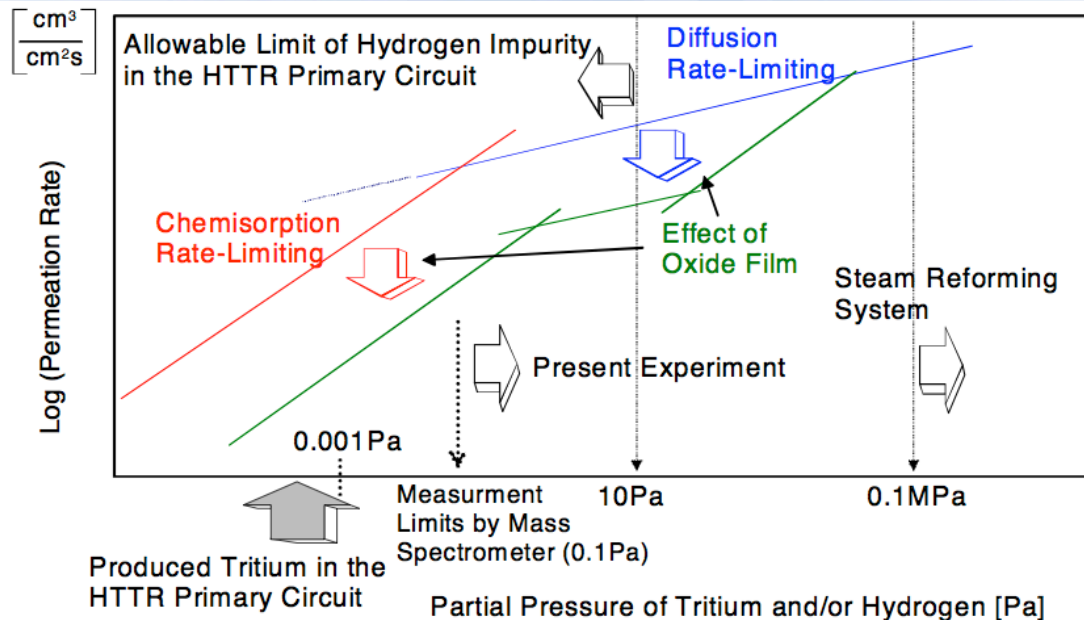
TPE viewport

# Tritium Heat Exchanger (THX) Experiment

- Tritium permeation apparatus was built in support for DOE NE NGNP/VHTR design
- Designed to measure tritium permeation rate through the candidate materials for VHTR IHX **at low tritium partial pressure conditions (ppb – ppm) in the primary loop.**
- Underlying physics for tritium permeation in **the transition regime between diffusion limited and surface limited regimes** is complex and there exists a surface oxide effect on permeation
- Designed to test a tubular shaped specimen **up to 1000 C**
- Available to measure tritium permeability in fusion materials as well.



# Motivation of this THX



## Tritium behavior in metal:

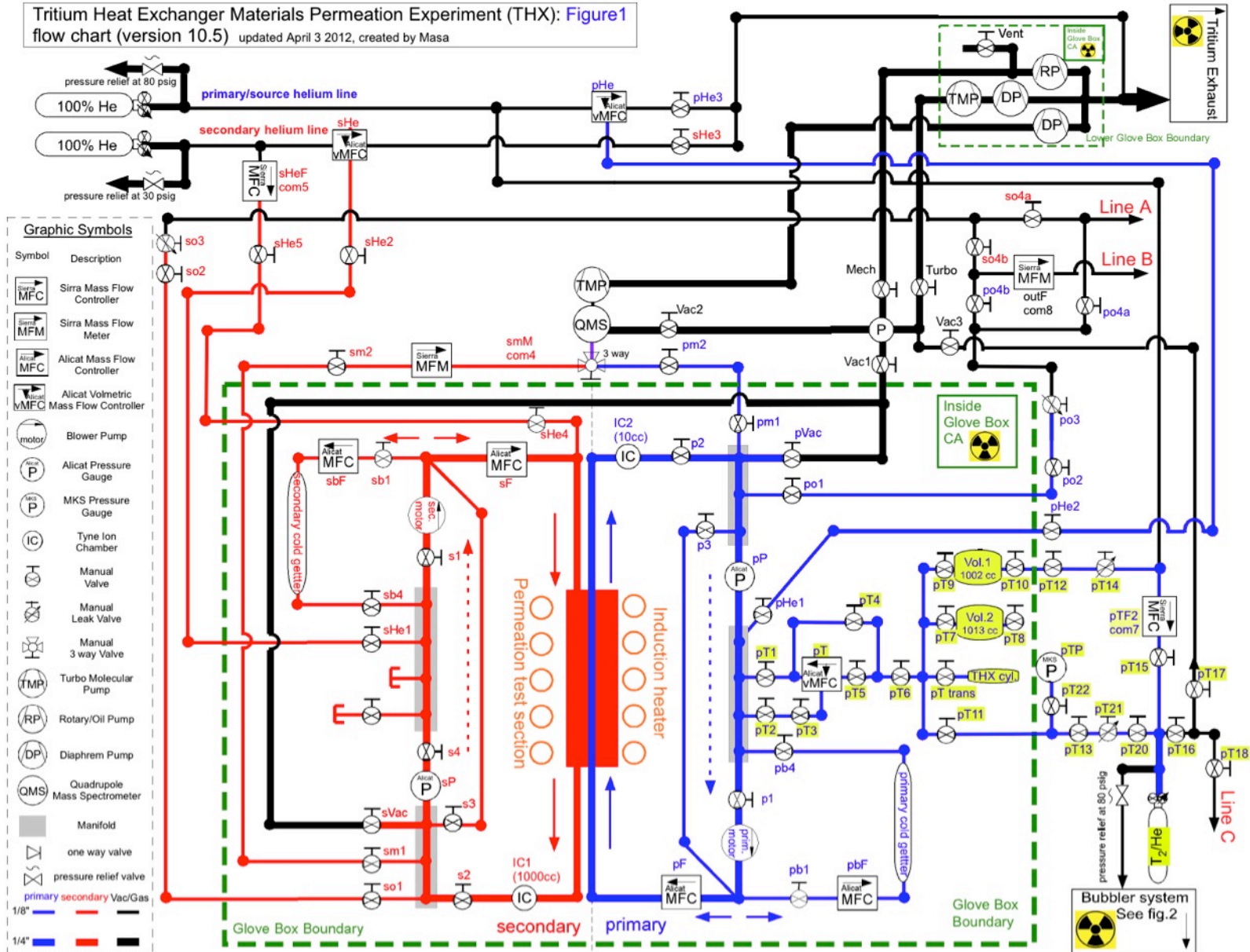
- ✓ At high partial pressures, diffusion rate-limited permeation, in which the permeation flux is proportional to the square root of pressure, is expected.
- ✓ In the intermediate pressure range, effect of oxide film appears depending on surface conditions.
- ✓ At low pressures, chemisorption (surface)-limited permeation is expected, the flux is proportional to the pressure (the relationship is linear).
- ✓ Use of tritium enhances the detection sensitivity up to ppt ( $10^{-7}$  Pa) in ion chamber, allowing us to investigate the low tritium partial pressure range

Reference: T. Takeda et. al. "Study of tritium/hydrogen permeation in the HTTR hydrogen production system"

7<sup>th</sup> International Conference on Nuclear Engineering, ICNE-7102, (1999)

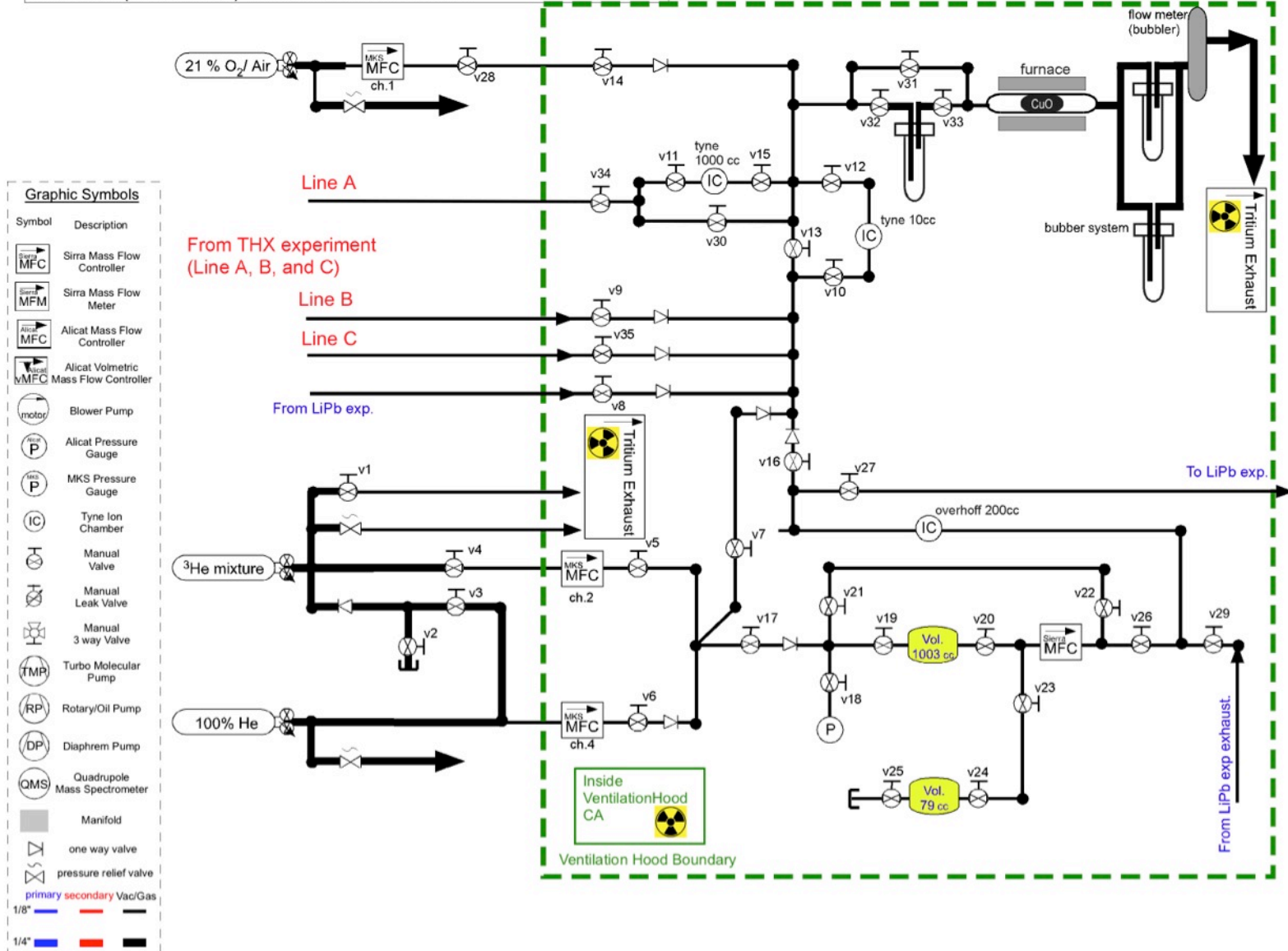
# Schematic/flow diagram of THX (1/2)

Tritium Heat Exchanger Materials Permeation Experiment (THX): Figure 1 flow chart (version 10.5) updated April 3 2012, created by Masa



# Schematic/flow diagram of THX (2/2)

Tritium Heat Exchanger Materials Permeation Experiment (THX): Figure 2  
 flow chart (version 10.5) updated April 3 2012, created by Masa





# Results from THX (1/2)

## Incoloy 800H

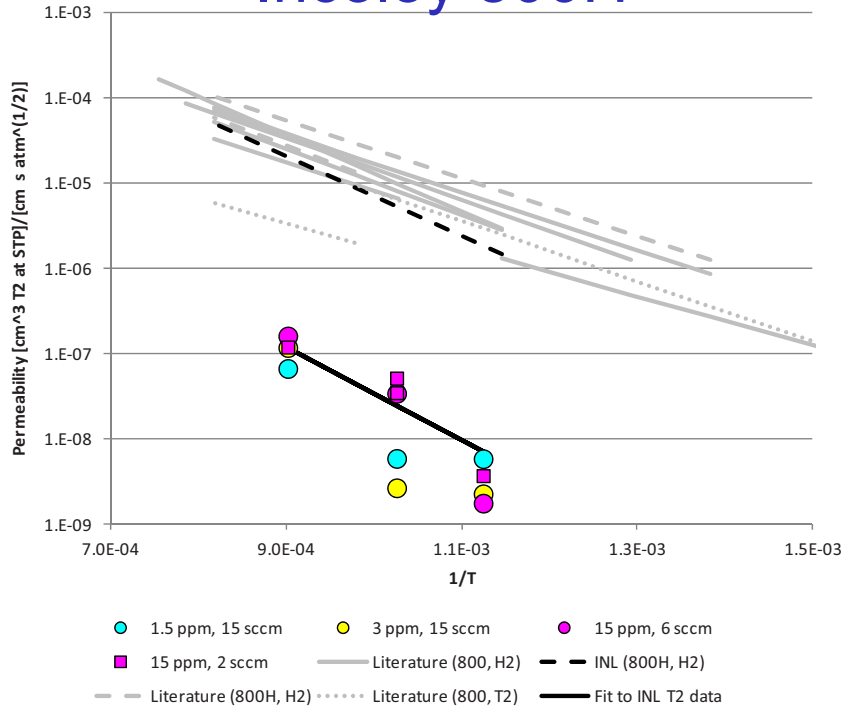


Figure 12. Arrhenius plot of Incoloy 800H tritium permeability (FY 11) with literature data.

## Inconel 617

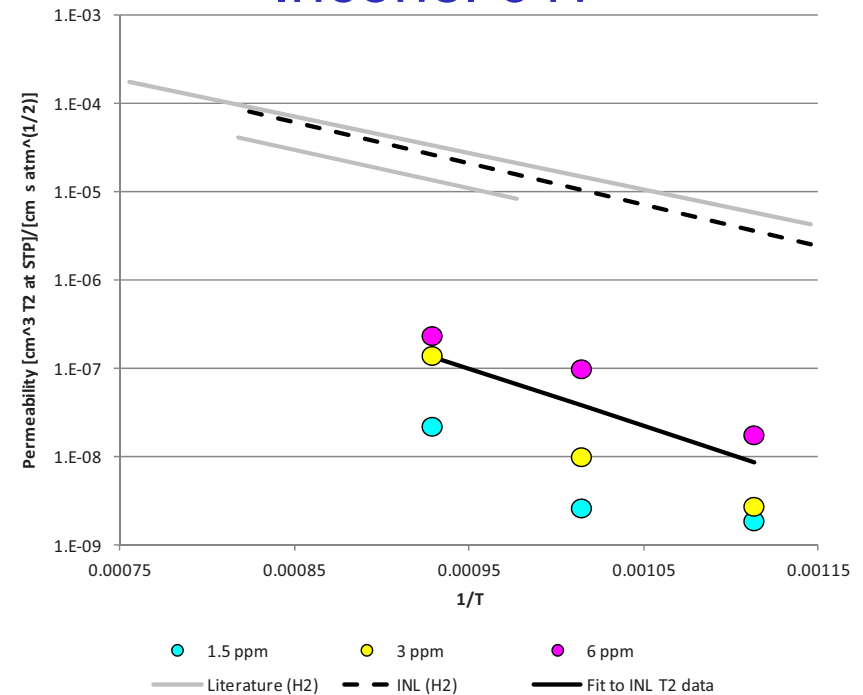


Figure 14. Arrhenius plot of Inconel 617 tritium permeability (FY 11) with literature data.

At high partial pressures (most of literature data), diffusion-limited permeation, in which the permeation flux is proportional to the square root of pressure, is expected.

# Results from THX (2/2)

## Incoloy 800H

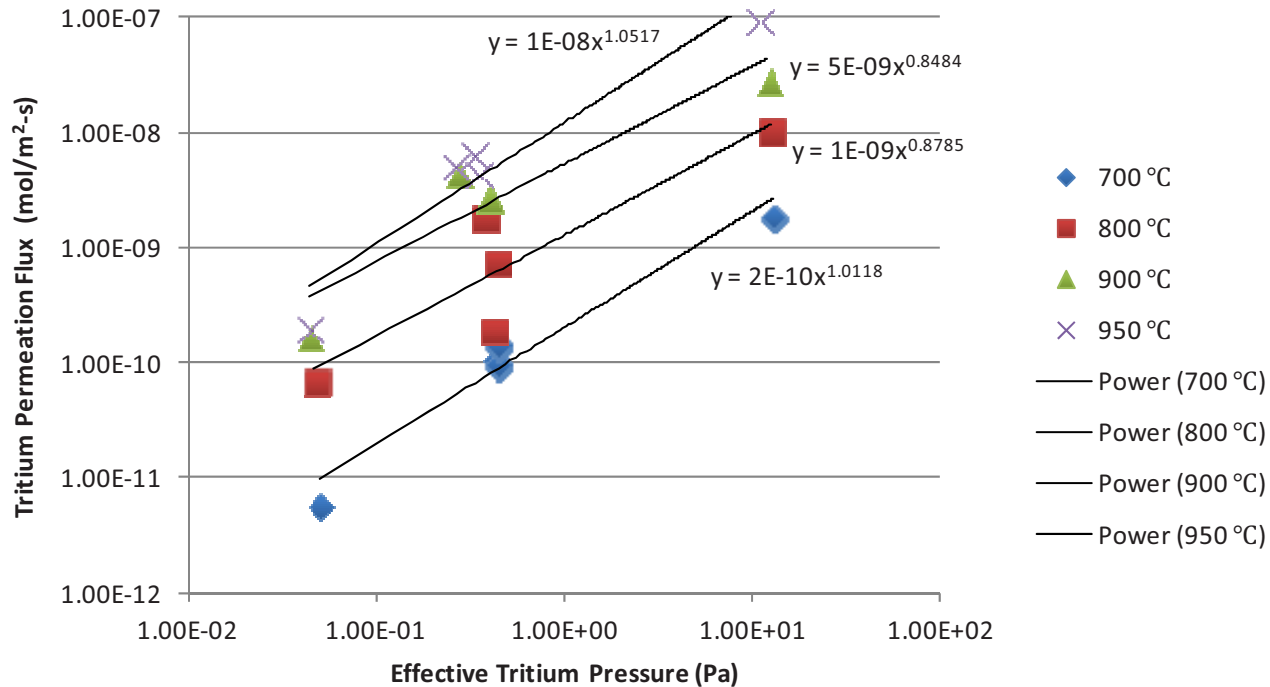
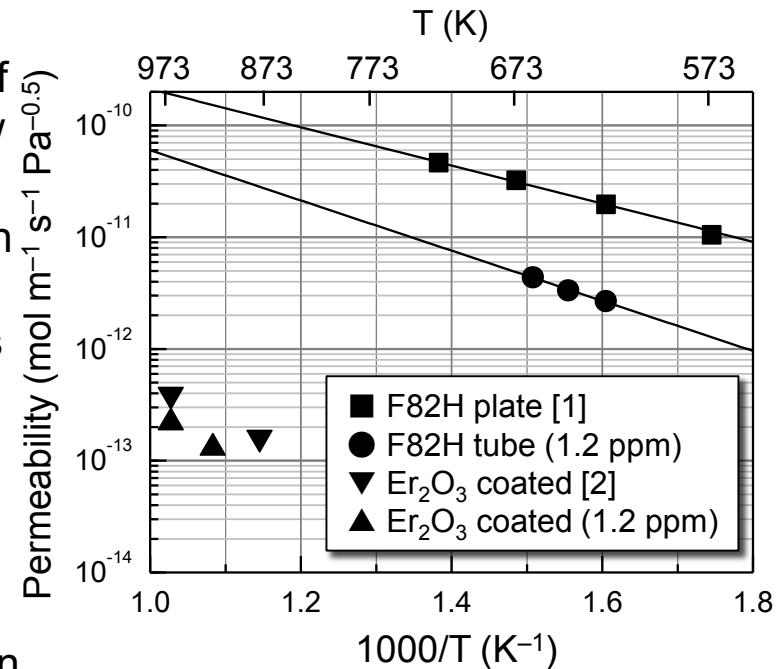


Figure 16. Tritium permeation flux versus effective tritium pressure (FY 12) at four different (peak) temperatures.

At low pressures, surface-limited permeation is expected, the flux is proportional to the pressure (the relationship is linear).

# Tritium permeability measurement in F82H and tritium permeation barrier materials for fusion application

- Motivation is to utilize high detection sensitivity of tritium for low tritium permeation rate through low permeable erbium oxide coated F82H
- Investigate tritium permeability/permeation rate in the temperature range 300 to 700°C and at primary concentrations of 0.1 to 100 (atom) parts per million tritium in helium (partial pressures of  $<10^{-7}$  atm)
- Low partial pressure data ( $<10$  ppm) provide some evidence that permeation has become surface-limited
- Permeation experiments have been performed on  $\text{Er}_2\text{O}_3$  coated F82H (reduced activation ferritic steel) sample at 500–750 °C with 1.2 ppm tritium
- At 750 °C, the coated sample indicated three orders of magnitude lower permeability than that of F82H substrate

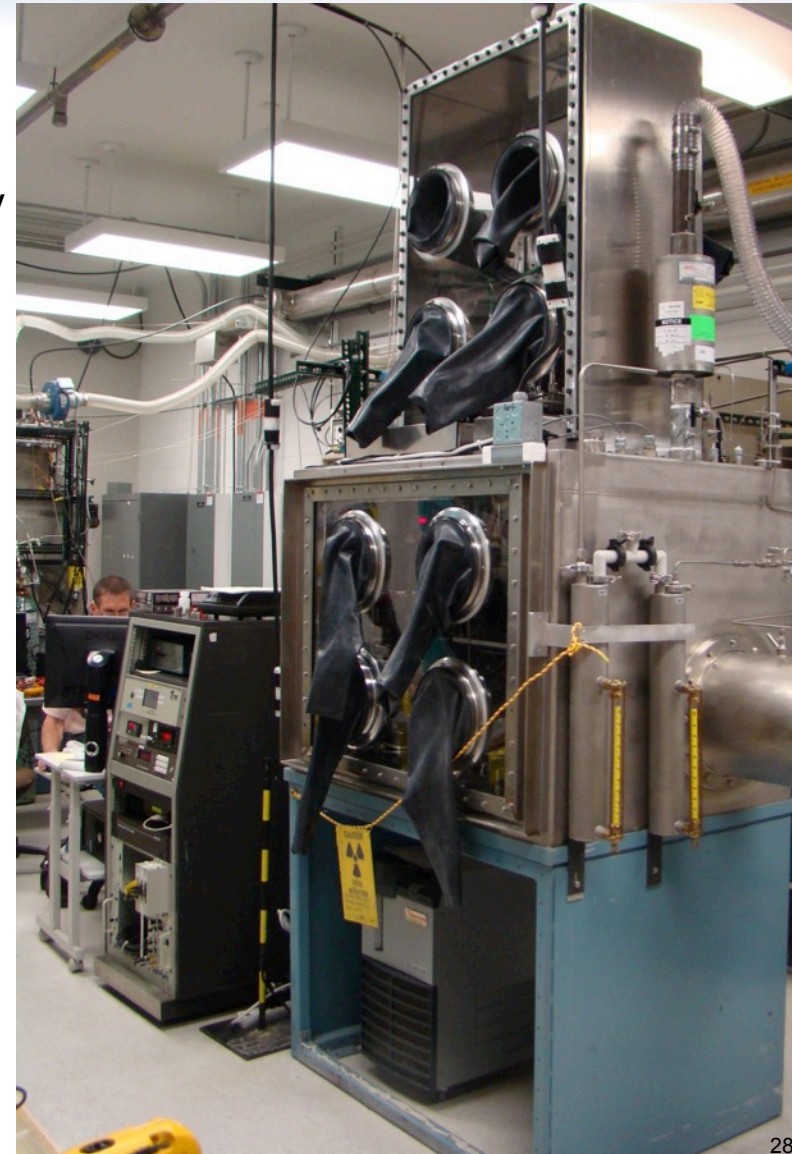
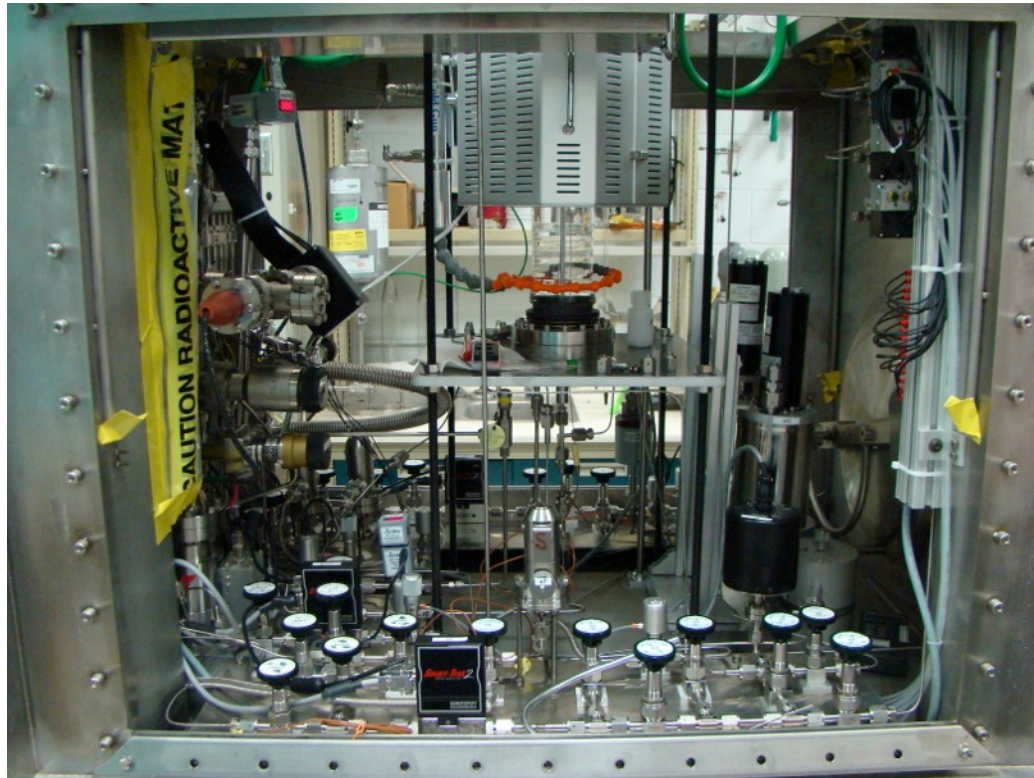


[2] T. Chikada, *et al.*, Fusion Eng. Des. 85 (2010) 1537–1541.

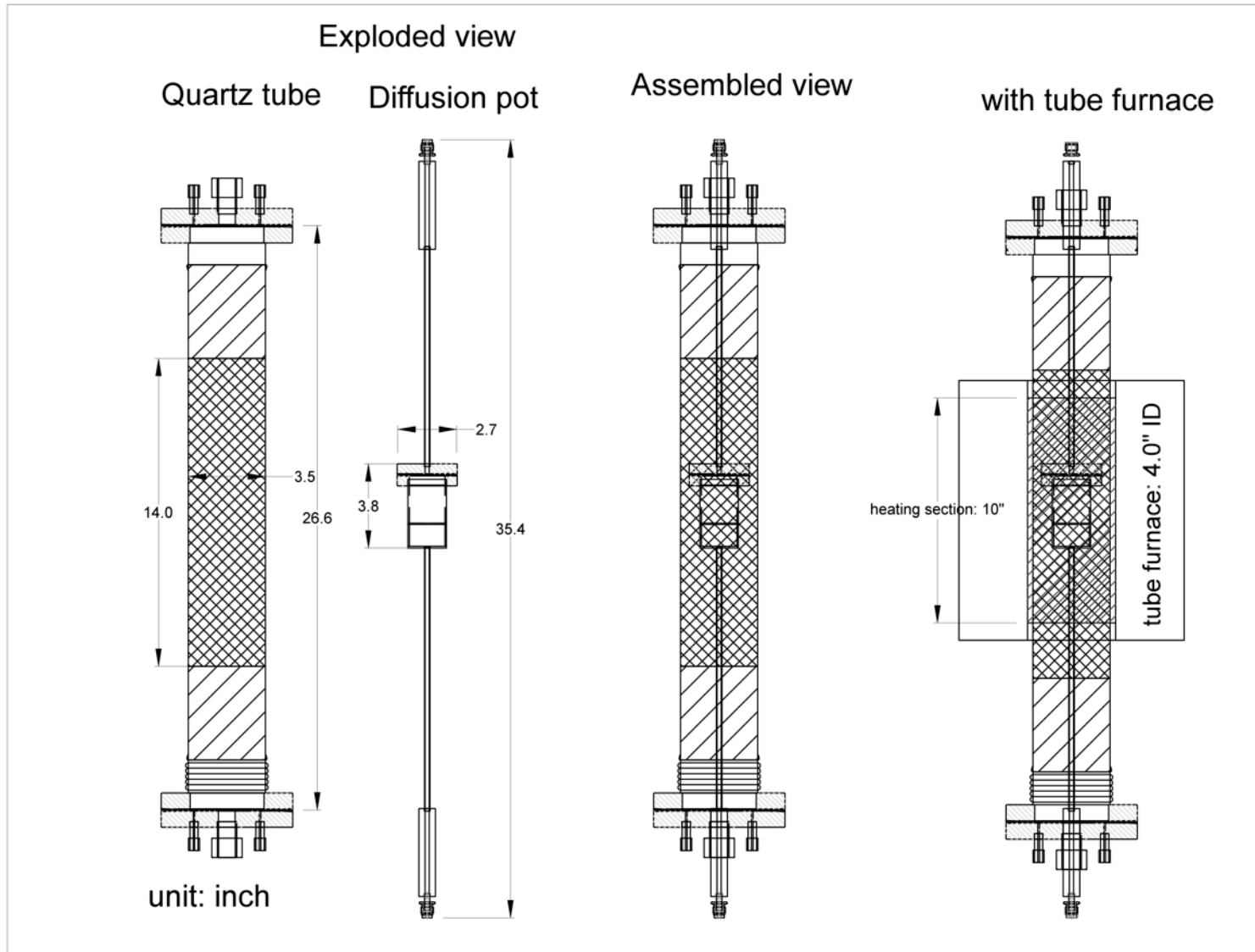
# Tritium Lead Lithium Eutectic (TLLE) experiment

To measure the mass transport parameters (diffusivity, solubility, and permeability) of tritium

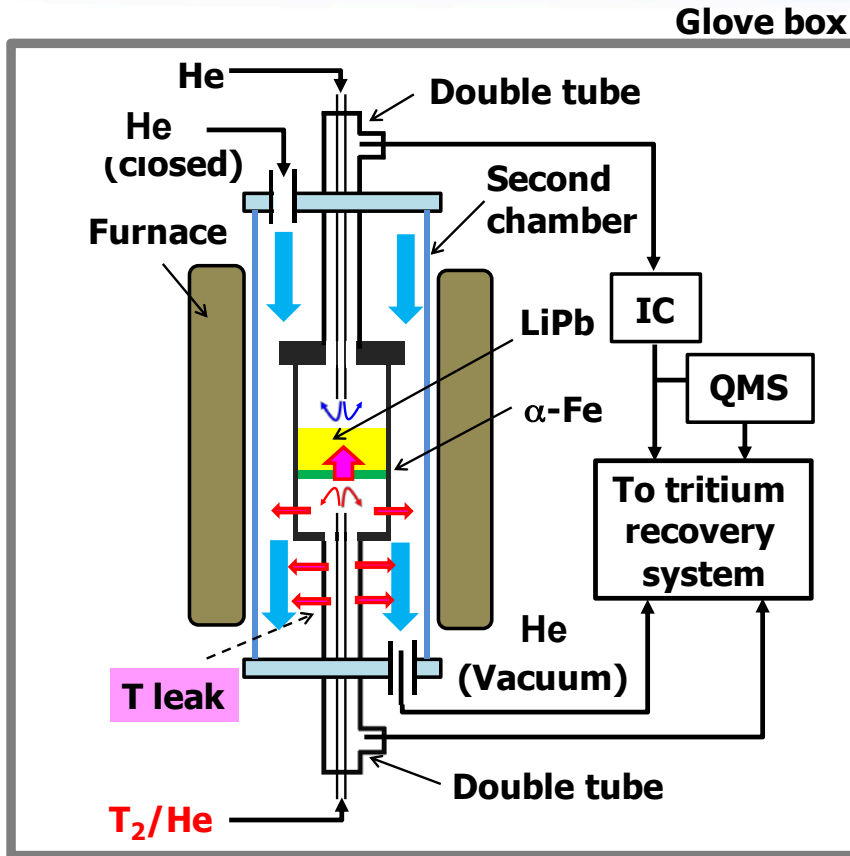
- Construction completed on Feb. 2013
- System verification and hydrogen test is under way
- Initial tritium campaign starts on May 2013



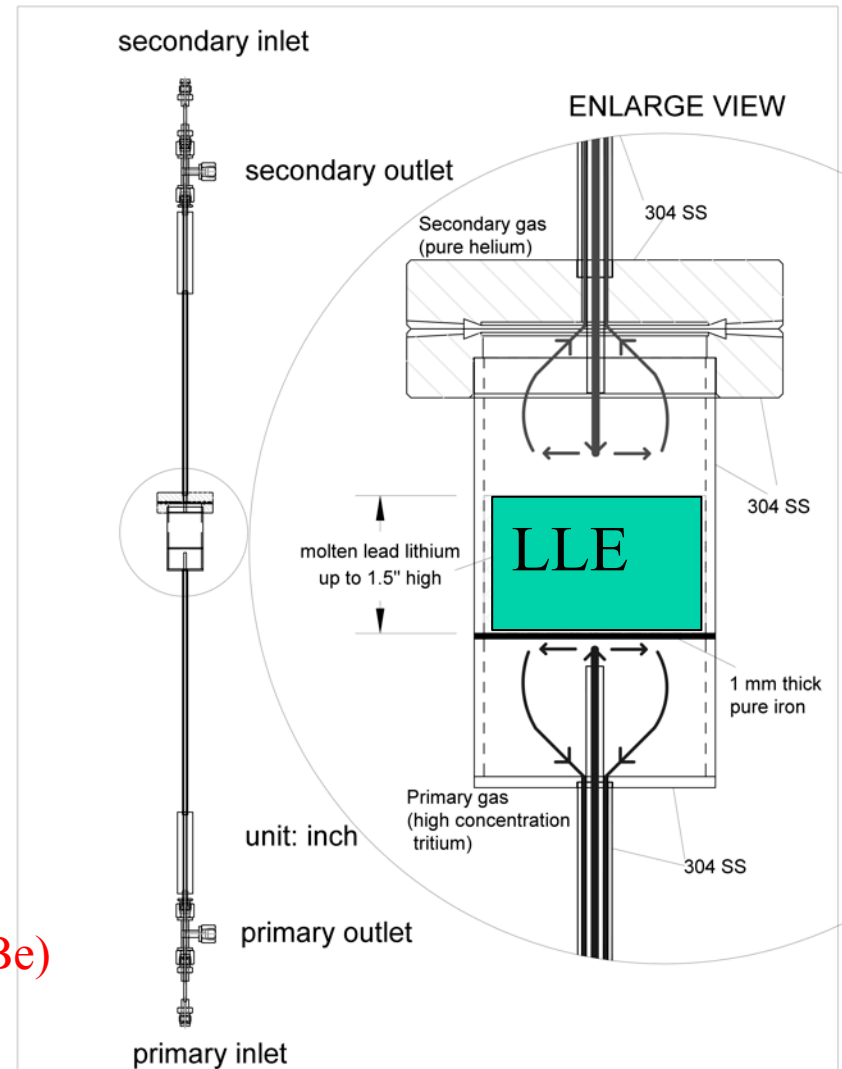
# Detail of TLLE experiment



# Detail of TLLE experiment



This test section can be easily modified to test tritium permeation in liquid metal and salt (FLiBe) as well as disc shape metal in high temperature (up to 1000 C)



# Schematic/flow diagram of TLLE

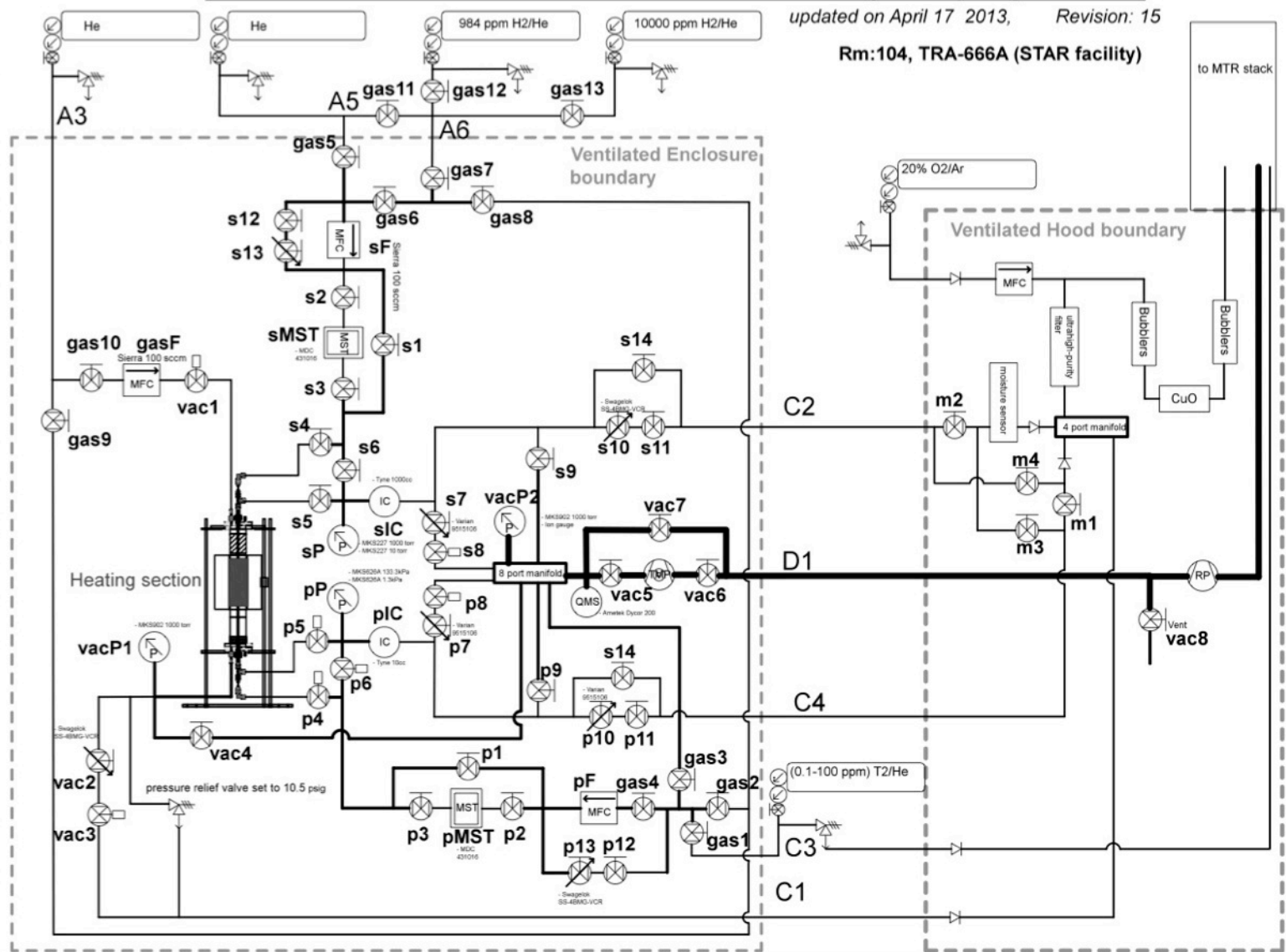
## Schematic of Tritium Lead Lithium Eutectic (TLLE) experiment

updated on April 17 2013, Revision: 15

Rm:104, TRA-666A (STAR facility)

### Graphic Symbols

Symbols	Descriptions
	Mass Flow Controller
	Molecular Sieve Trap
	Ion Chamber
	Turbo Molecular Pump
	Rotary Vane Oil Pump
	Quadrupole Mass Spectrometer
	Pressure Gauge
	Pressure Regulator
	Manual Valve
	Manual Leak Valve
	Pneumatic Valve
	Pressure Relief Valve
	Check Valve
	1/8" OD SS tube
	3/8" OD SS tube
	1/2" or 1.5" OD SS tube



## Outlines

1. Motivation of tritium research activity in STAR facility
2. Unique capabilities in STAR facility
3. Research highlights from tritium retention in HFIR neutron-irradiated tungsten

### NOTE:

This research was carried out under the US-Japan collaboration, *Tritium, irradiation, and thermofluid for America and Nippon (TITAN) project (April 2007-March 2013) task 2-1*





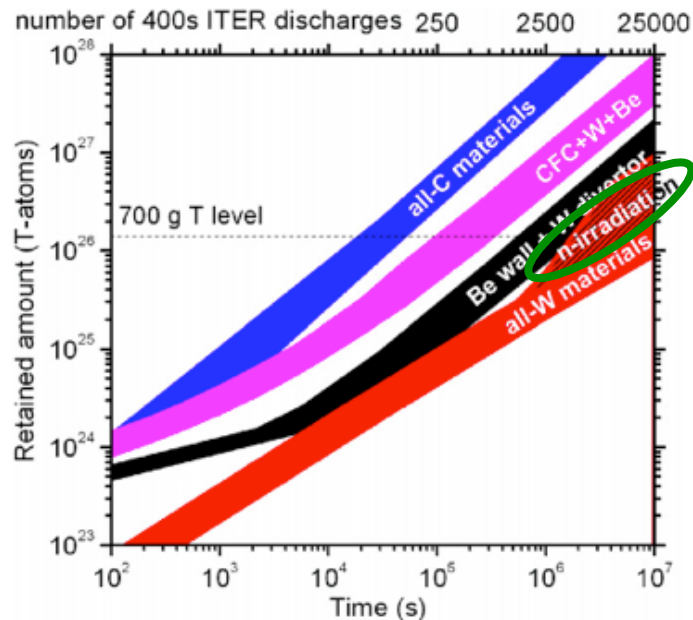
## Past/present/future US-Japan collaboration:

- I. JFY2001-2006 (Apr.2001-Mar.2007):
  - *The second Japan/US Program on Irradiation Test for Fusion Research (JUPITER-II)*
    - Corrosion and purification of molten salt (FLiBe)
    - Mass transport of tritium in FLiBe
- II. JFY2007-2012 (Apr.2007-Mar.2013):
  - *Tritium, irradiation, and thermofluid for America and Nippon (TITAN)*
    - Mass transport of tritium in lead lithium eutectic and development of tritium permeation barrier materials
    - Tritium retention in HFIR neutron-irradiated tungsten
- III. JFY2013-2018 (Apr.2013-Mar.2019):
  - *PFC evaluation by tritium plasma, heat, and neutron irradiation experiment (PHENIX)*
    - Tritium behavior (retention, diffusion, and permeation) in HFIR neutron-irradiated tungsten

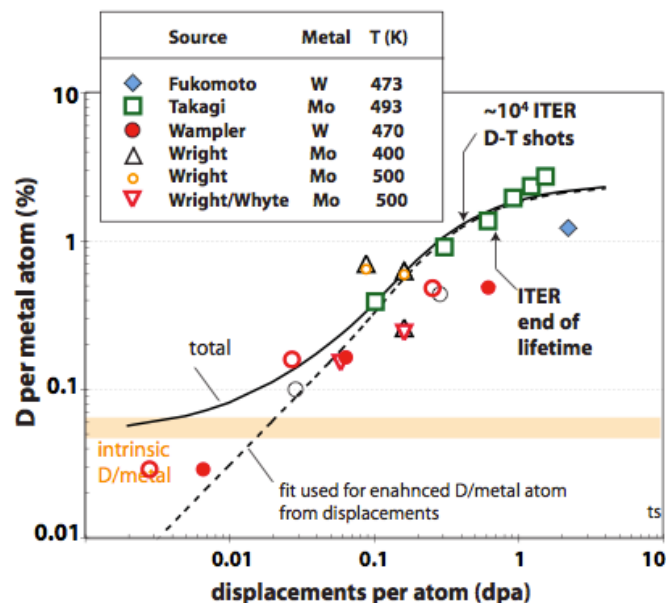
# “In-vessel tritium source term” in neutron-irradiated PFC

- Tungsten, a candidate PFC for the divertor in ITER, is expected to receive a neutron dose of 0.7 dpa by the end of operation in ITER, and >10 dpa in FNSF and DEMO.
- High energy ion beams have been used to simulate displacement damages by 14 MeV fusion neutron, and provided us three trends in damaged-tungsten:
  1. The trap concentration will most likely saturate at > 1 dpa
  2. T will most likely stay with in a few micro meters from the surface
  3. Very small D retention from damaged W at high exposure temperature (> 500C= 773 K)

Reference: J. Roth et. al. PPCF 2008



Reference: B.Lipschultz et. al. MIT report 2010

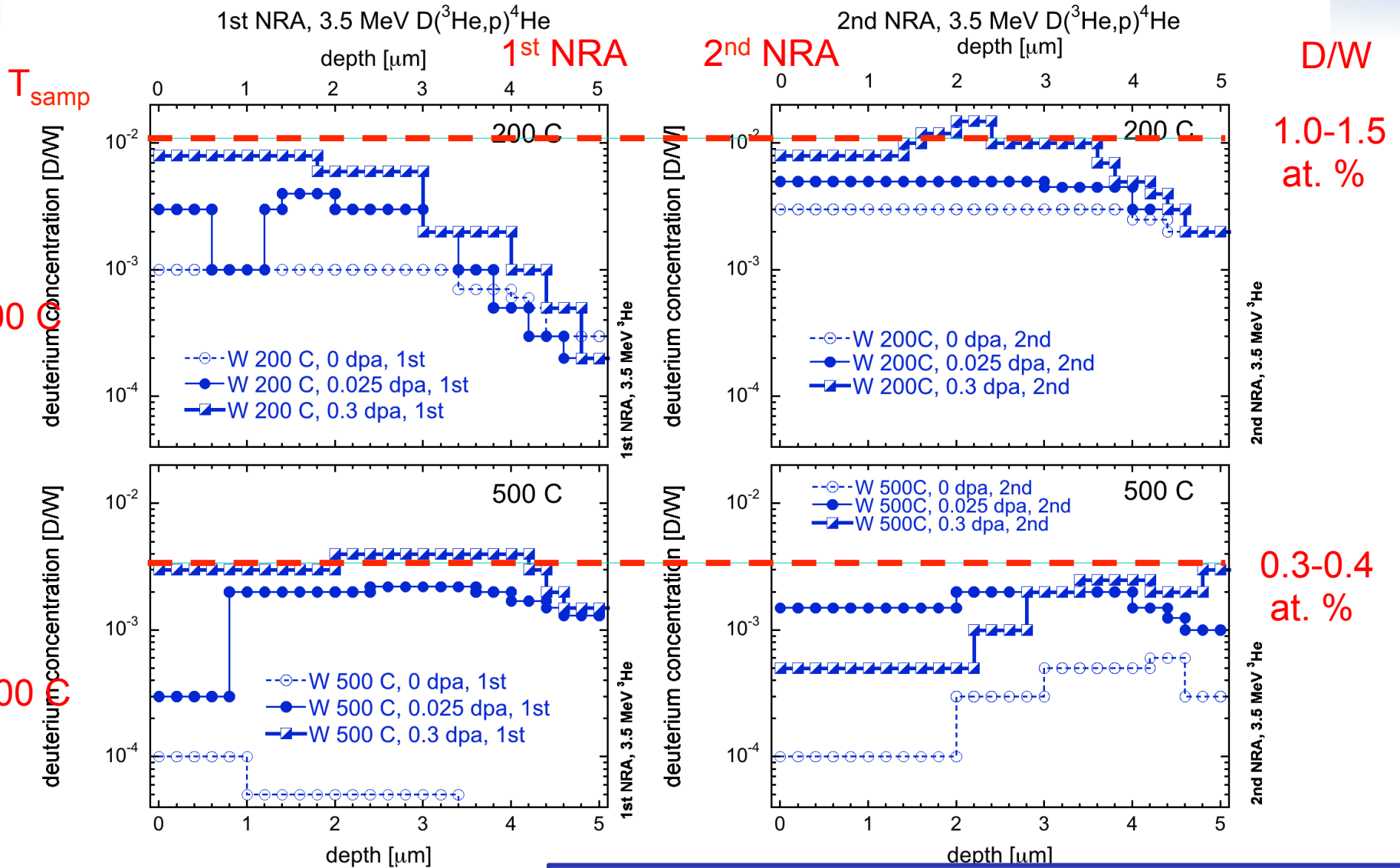


## **Motivations of TITAN task 2-1 and PHENIX task 3:**

- To understand tritium retention in neutron-irradiated tungsten utilizing two unique capabilities in the US:
  - High Flux Isotope Reactor (HFIR), ORNL
    - One of the highest flux reactor-based sources of neutrons in the US
    - One of the highest steady-state neutron fluxes of any research reactor in the world.
  - Tritium Plasma Experiment (TPE), INL
    - The only existing high-flux linear plasma device that can handle both tritium and neutron-irradiated materials
    - The only device that can investigate the tritium behavior in neutron-irradiated PFCs in the world fusion community at this moment
- Past US-Japan project TITAN task 2-1 investigated deuterium retention in neutron-irradiated tungsten
- Current US-Japan PHENIX task 3 investigates deuterium/tritium retention (and permeation) in neutron-irradiated tungsten (tungsten coated and/or tungsten alloys) under deuterium/tritium/helium plasma



# Progress on retention study in HFIR neutron-irradiated W



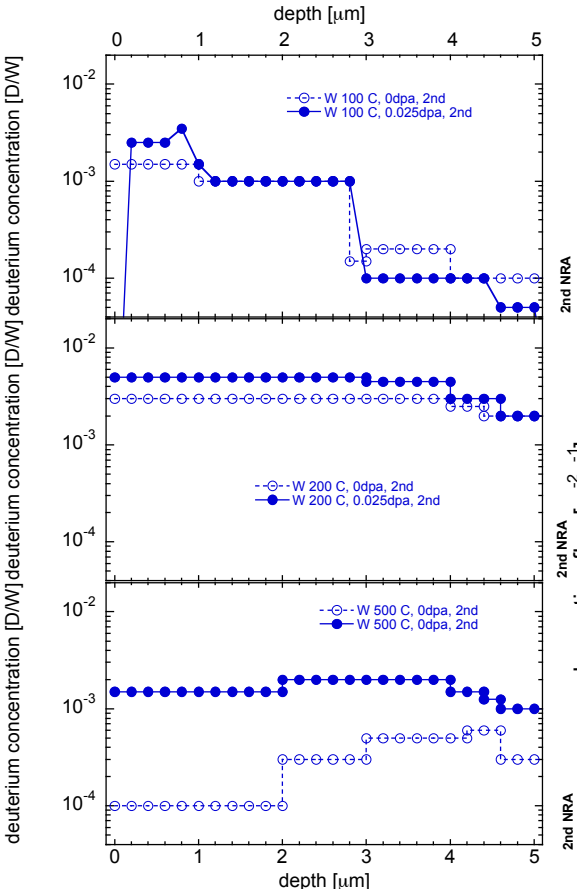
D/W saturated at 1.0~1.5 at. % for 0.3 dpa W<sup>36</sup>

# Observations from 0.025 dpa neutron-irradiated tungsten

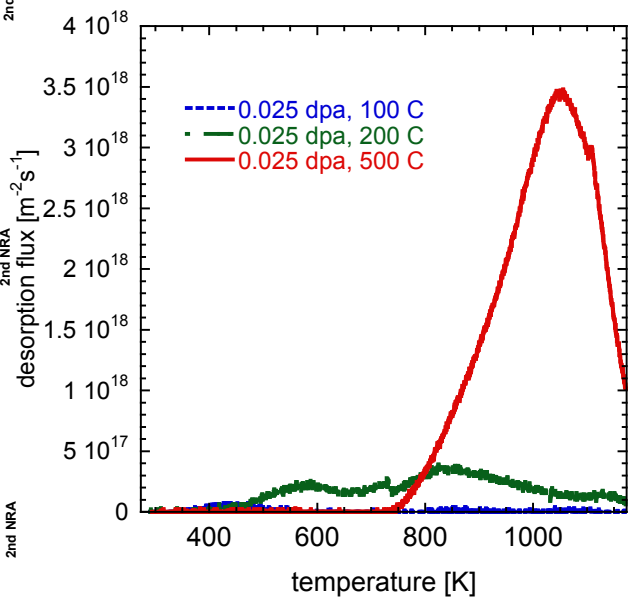
Experimental procedure:

- 1<sup>st</sup> TPE (@INL) → 1<sup>st</sup> NRA (@U of Wisc.) → 2<sup>nd</sup> TPE → 2<sup>nd</sup> 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

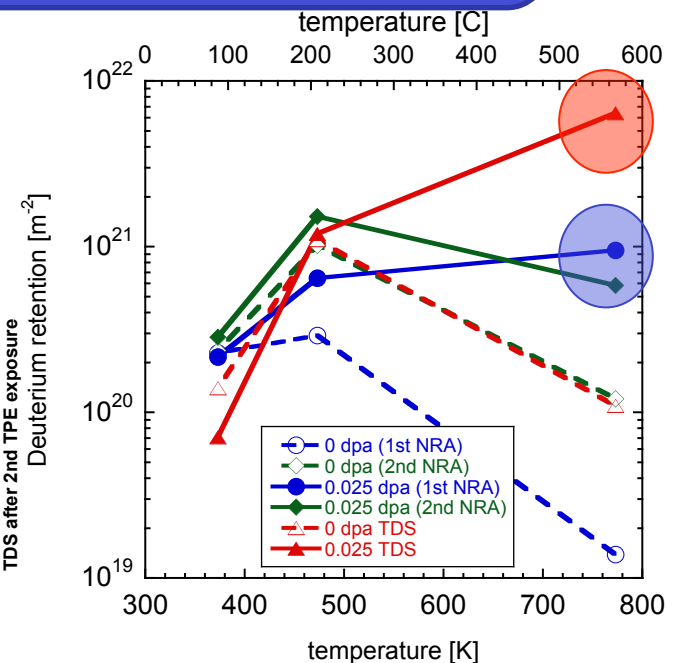
Large retention at 500 C  
 Discrepancy between TDS and NRA at 500 C  
 indicates that D is migrated and trapped in bulk  
 ( $> 5 \mu\text{m}$ ) → 50-100  $\mu\text{m}$  ?



2<sup>nd</sup> NRA result



TDS after 2<sup>nd</sup> TPE exposure

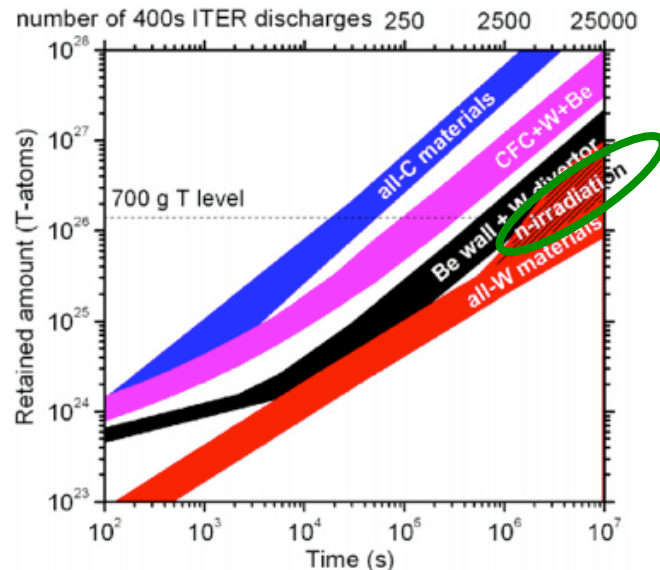


TDS vs. NRA

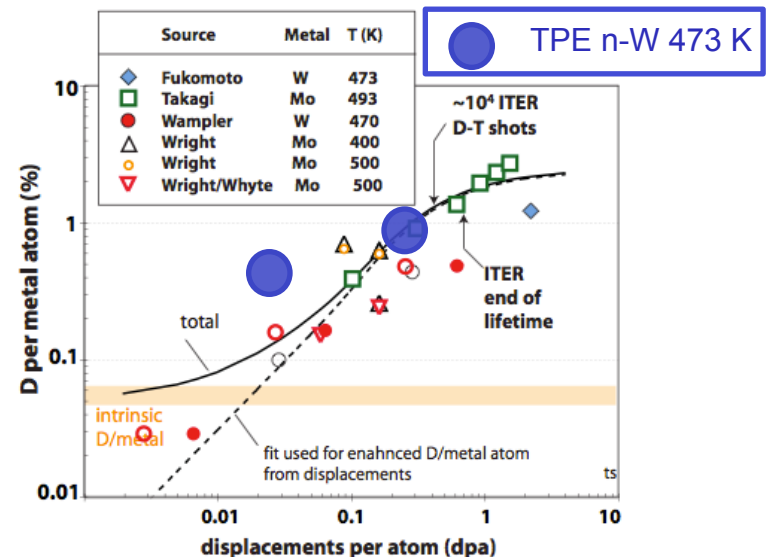
# “In-vessel tritium source term” in neutron-irradiated PFC

- Tungsten, a candidate PFC for the divertor in ITER, is expected to receive a neutron dose of 0.7 dpa by the end of operation in ITER, and >10 dpa in FNSF and DEMO
- High energy ion beams have been used to simulate displacement damage by 14 MeV fusion neutrons, and provided us three trends in damaged-tungsten:
  1. The trap concentration will most likely saturate at > 1 dpa → We need to evaluate this at > 1 dpa.
  2. T will most likely stay within a few micrometers → Questionable?
  3. Very small D retention from damaged W at high exposure temperature (> 500°C = 773 K) → Questionable?
- TITAN task 2-1 results questioned two out of three trends above.

Reference: J. Roth et. al. PPCF 2008



Reference: B.Lipschultz et. al. MIT report 2010



## ***Key issues identified in TITAN task 2-1:***

- Key issues identified through the past 6 years in task 2-1:
  1. (0.1-1.5 at.% D/W) deuterium concentration even in low-moderate dose (0.025 - 0.3 dpa) HFIR neutron-irradiated tungsten
  2. Deep (>10  $\mu\text{m}$ ) migration and trapping of deuterium and resulting high deuterium retention at high plasma exposure temperature (500 C)

## *Summary:*

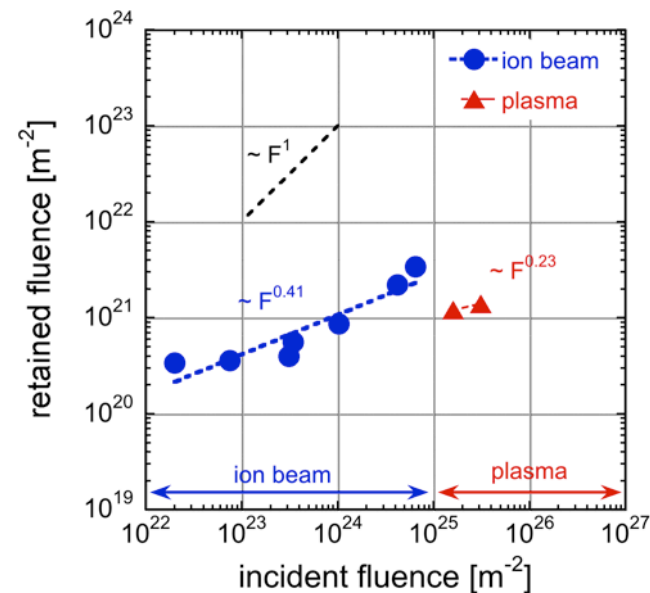
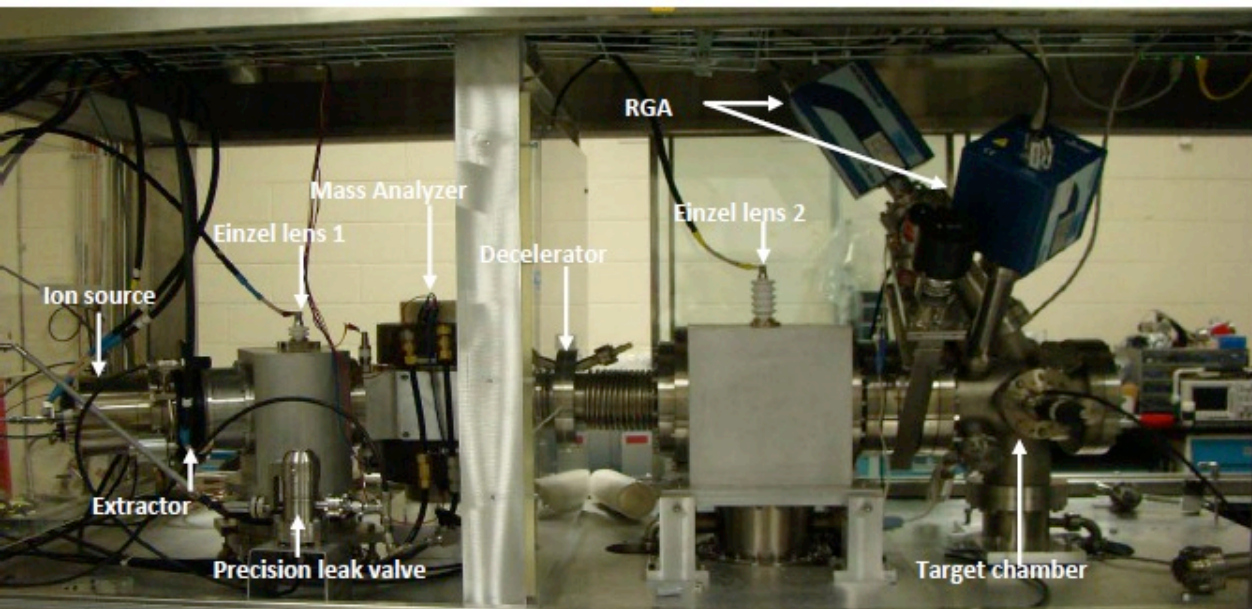
- The STAR facility, ATR complex continue to support FES, NE and ITER research activities for the purpose of demonstrating the scientific & technological feasibility of fission/fusion energy
- The STAR facility possesses the unique capabilities to handle tritium, beryllium, and activated materials
- TPE/STAR is leading the research area in tritium behavior in radiation damaged fusion reactor materials
- FSP/STAR continues to support the US-Japan collaboration (April 2013-March 2019) utilizing the unique linear plasma device, the Tritium Plasma Experiment (TPE)



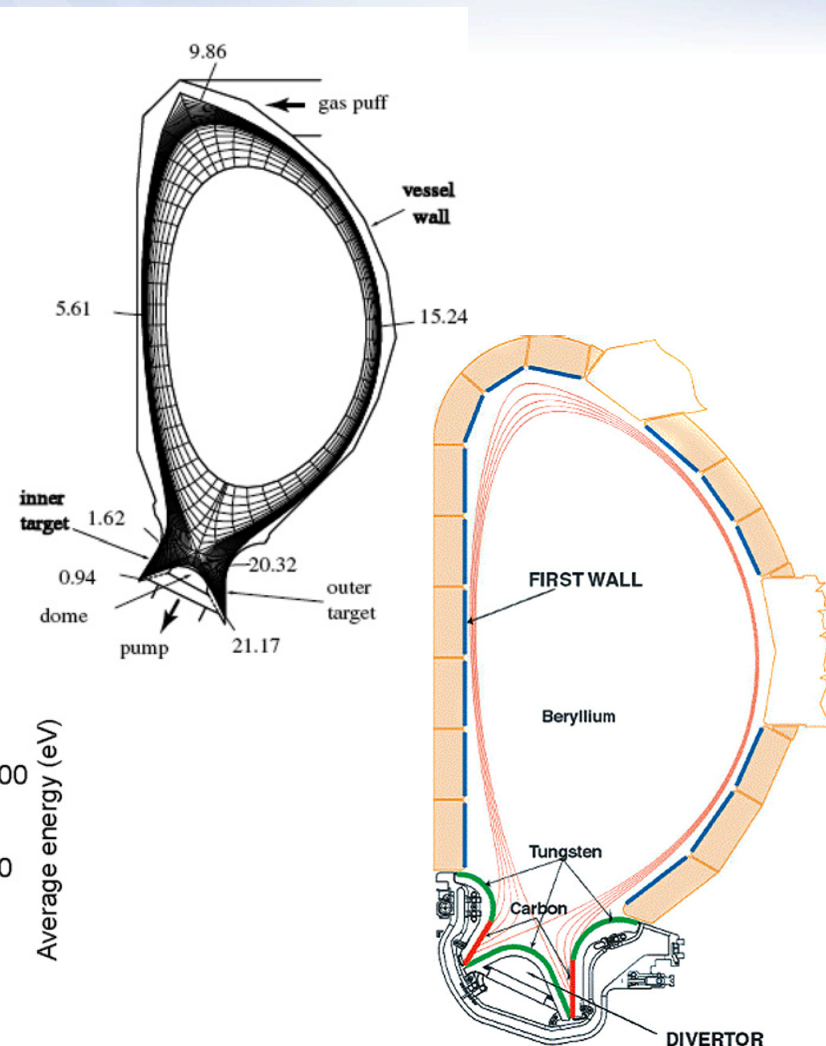
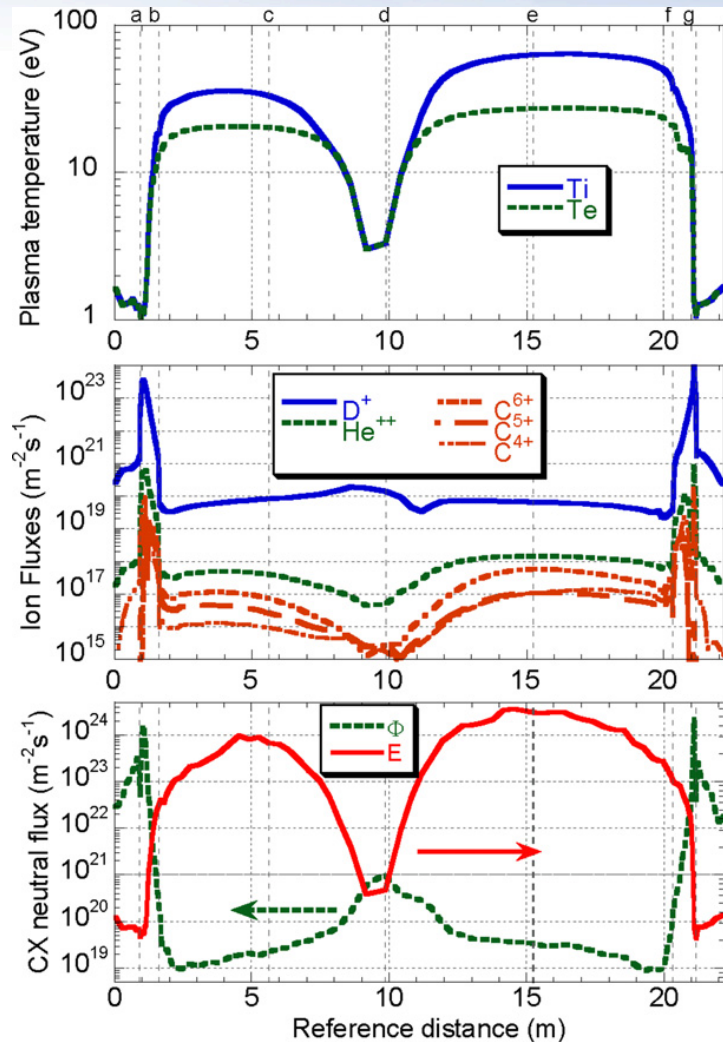
# *Support slides*

# Ion Implantation experiment (IIX)

- Designated to investigate hydrogen isotope behavior on PFCs under low ion flux and fluence conditions (flux:  $<10^{20} \text{ m}^{-2}\text{s}^{-1}$ , fluence:  $10^{22}\text{-}10^{25} \text{ m}^{-2}$ )
- Utilized to benchmark experiment for TMAP development/modification
- Combined with the fluence range of TPE, FSP can investigate tritium behavior in 5 orders of magnitude in ion fluence ( $10^{22} < \Gamma_{D,ion} [\text{m}^{-2}] < 10^{27}$ )



# ITER neutral and ion flux profiles



- $\text{D}^+$  ion flux ranges in 5 orders of magnitudes (from  $10^{19}$ - $10^{23} \text{ m}^{-2}\text{s}^{-1}$ )

Ref: Roth et al 2008

# Methodology of the retention study

## Methodology:

- Small (6 mm in diameter) pure tungsten specimens were irradiated by fission neutrons to 0.025 dpa and 0.3 dpa at 50-70 C (reactor coolant temperature) in HFIR, ORNL, and then were sent to STAR
- Neutron-irradiated tungsten was exposed to a deuterium plasma in TPE at 100, 200, and 500 C
- Specimens were sent to UW-Madison for Nuclear Reaction Analysis (NRA)
- TPE exposure and NRA were repeated, and then final thermal desorption spectroscopy (TDS) was performed.

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

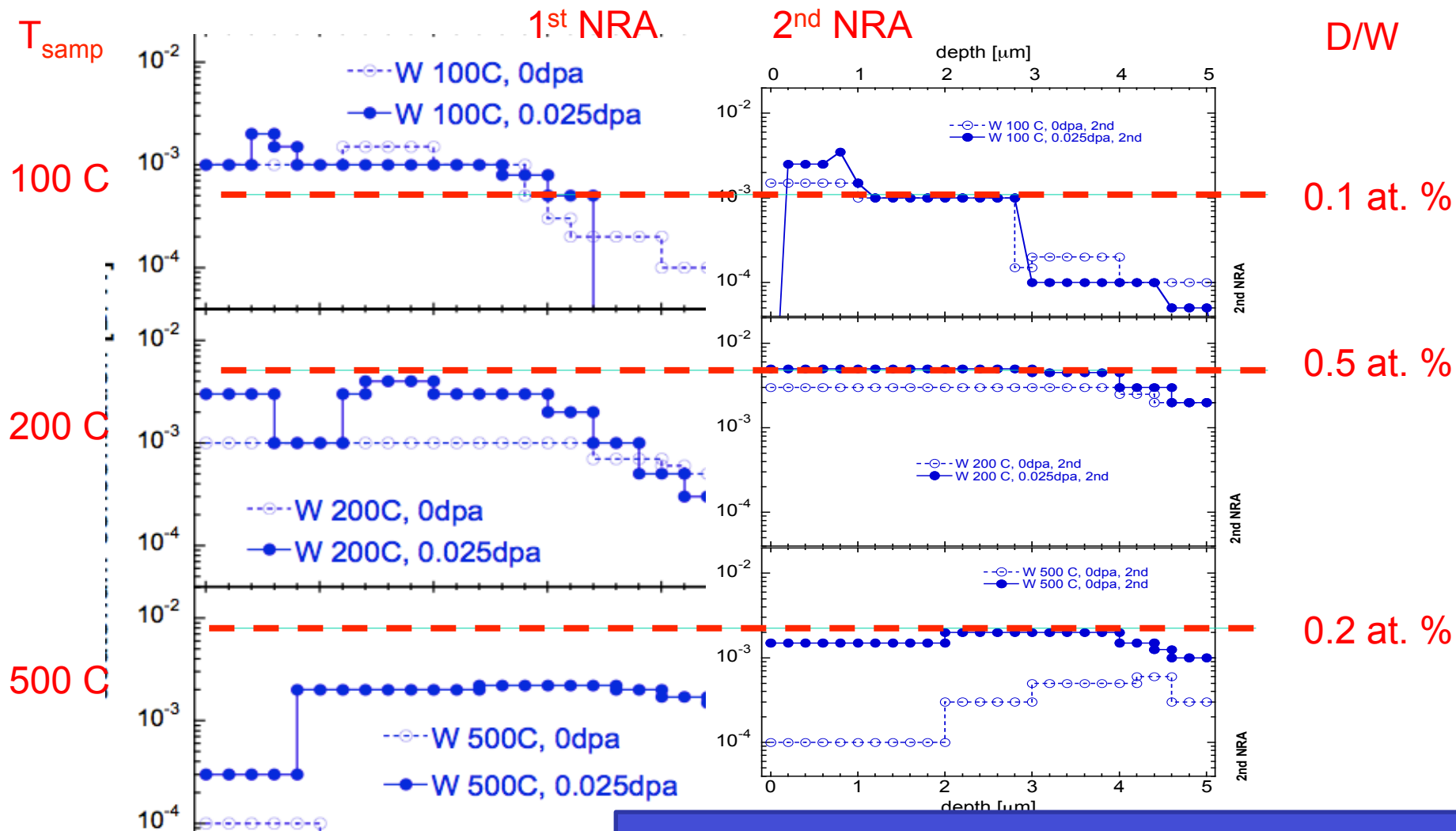
- 1) Irradiated at 50 C to 0.025 dpa at the High Flux Isotope Reactor (HFIR), ORNL
- 2) Exposed at 100, 200, and 500 C to a deuterium plasma ( $\sim 5 \times 10^{25} \text{ m}^{-2}$  ion fluence) at TPE, INL
- 3) Exposed to  $^3\text{He}$  beam at RT to measure D depth profile up to 5  $\mu\text{m}$  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 the second study (Post-Annealed/TDS)

- 1) Exposed at 100, 200, and 500C to a deuterium plasma ( $\sim 5 \times 10^{25} \text{ m}^{-2}$  ion fluence) at 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

# Observations from 0.025 dpa neutron-irradiated tungsten

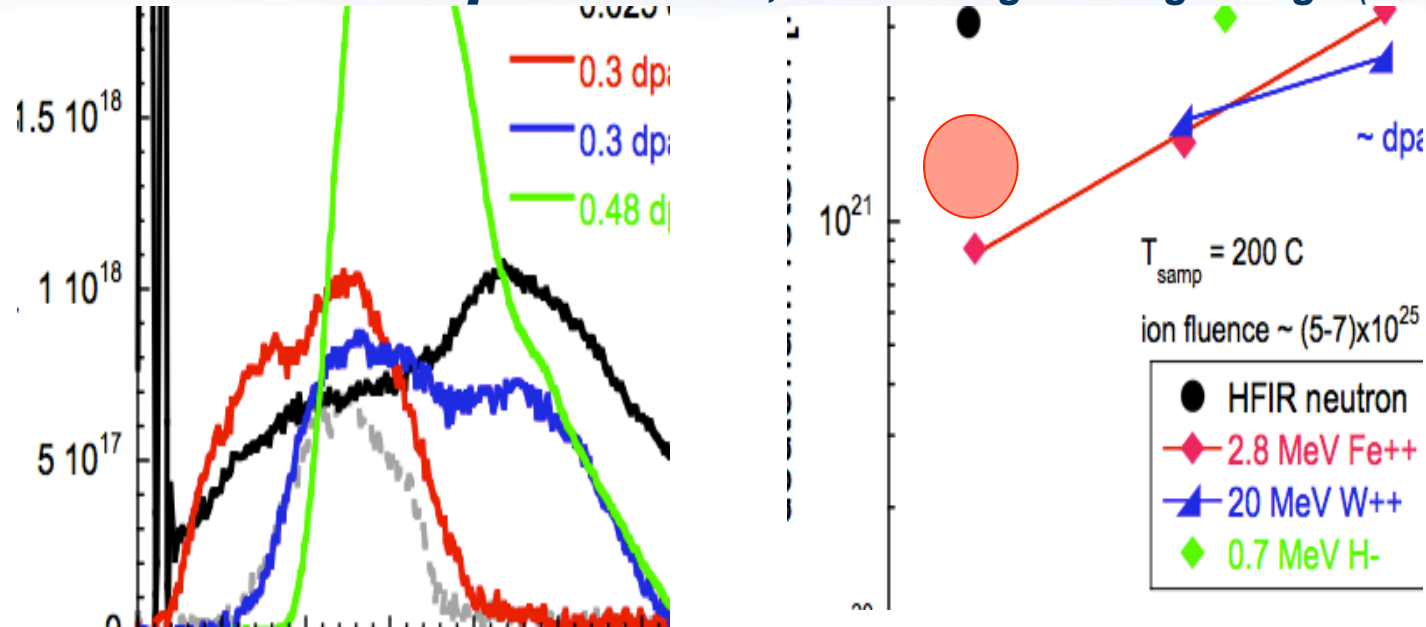
## Investigation of saturation in trapping concentration via NRA



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

# Observations from 0.025 dpa neutron-irradiated tungsten

[Shimada et al., Fusion Engineering Design (2012)]



- High energy ion-damaged samples
  - 2.8 MeV Fe<sup>2+</sup> (0.027, 0.3, 3.0 dpa) provided by T. Oda, The Univ. of Tokyo
  - 20 MeV W<sup>4+</sup> (0.3, 3.0 dpa) provided by B. Tyburska-Puschel, IPP
  - 700 keV H<sup>-</sup> (0.48 dpa) provided by Y. Ueda, Osaka Univ.
- Comparison of ion-damaged with neutron-irradiated W shows:
  - HFIR neutron produces the broad TDS spectrum (300-1000 K)
  - Fe<sup>++</sup> reproduces the lower temperature TDS peaks (300-700 K)
  - H<sup>-</sup> and W<sup>++</sup> reproduce the medium temperature TDS peaks (450-900 K)
- D retention from 0.025 dpa HFIR neutron is similar to that from 3.0 dpa Fe<sup>++</sup> and W<sup>++</sup>
  - Despite the 2 orders of magnitude difference in dpa

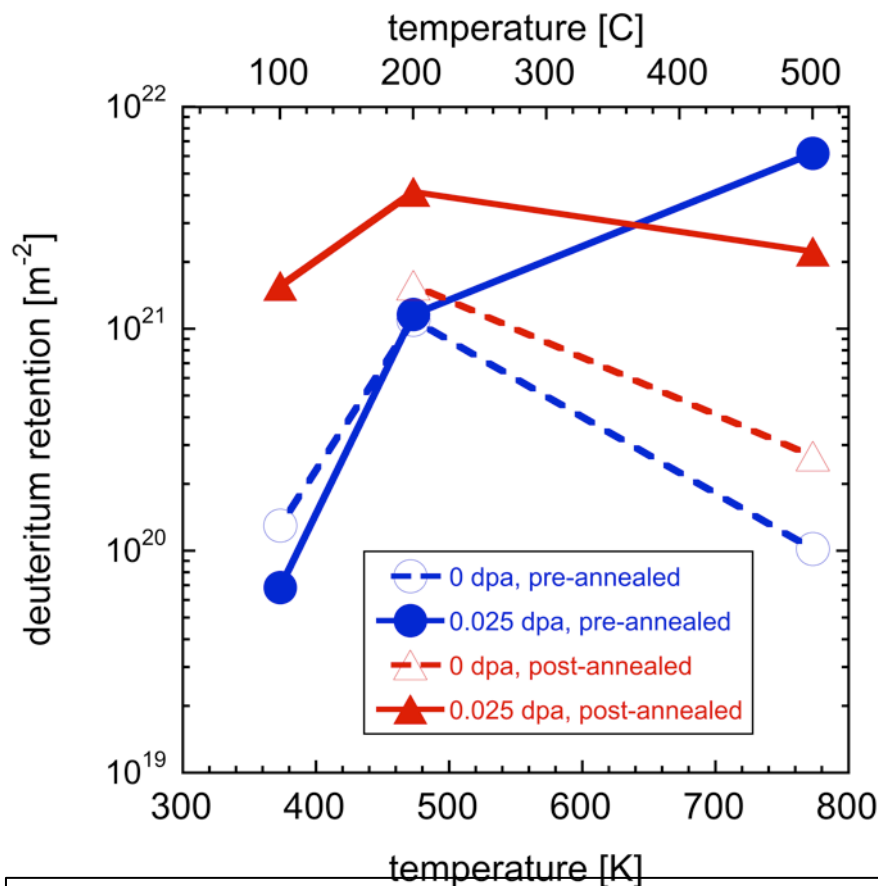
# Comparison of post-annealed with pre-annealed

## Plasma exposure conditions:

- Exposure temp.: 100, 200, 500 C
- 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:

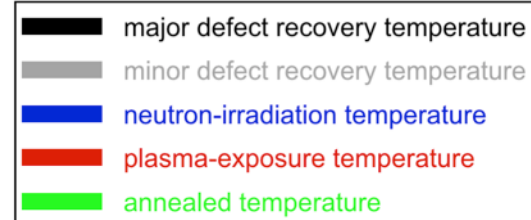
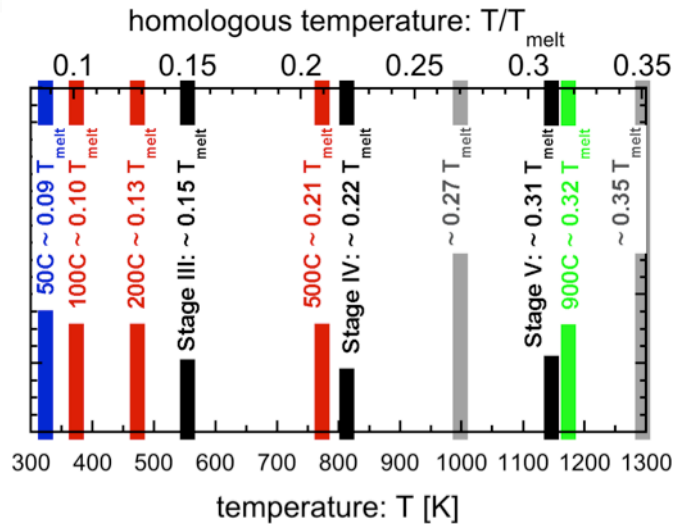
- D retention for 0.025 dpa 100 C increased by a factor of **x 10**
- D retention for 0.025 dpa 200 C increased by a factor of **x 4**
- D retention for 0.025 dpa 500 C decreased by **2/3**
- Annealing at 900 C for 0.5 hour suppressed the high temperature peak, but enhanced the D retention for the low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



900 C anneal decreased the densities of vacancy, ( $V_{4-10}$  and  $V_{11-16}$ ) vacancy clusters, increased the ( $V_{40-60}$ ) void density, and then increased nucleation sites for bubble formation

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

# Possible trapping mechanism in n-irradiated W



Stage III (0.15 T<sub>m</sub>): Vacancy migration  
 Stage V (0.31 T<sub>m</sub>): Vacancy cluster migration

Ref: H. Schultz, Mater. Sci. Eng. '91

- Near surface (< a few  $\mu\text{m}$ ) :
  - High concentration of D by intense plasma is required for bubble formation
    - High solute D conc. (near surface) + Void/Vacancy cluster ( $V_{40-60}$ )  $\rightarrow$  bubble formation
- In the bulk (> 10  $\mu\text{m}$ ) :
  - Trapping at not annealed defect (vacancy clusters)
    - Vacancy, di-vacancy, and Vacancy cluster ( $V_{4-10}$ ) should be annealed at 900 C
    - Vacancy cluster ( $V_{11-16}$ ) started to be annealed around 900 C
    - Void/Vacancy cluster ( $V_{40-60}$ ) can not be annealed at 900 C
  - Deeper migration and trapping at higher temperatures
    - This can only be observed by neutron-irradiation W due to uniform damage creation



## *Subtask review of activities in INL for task 2-1*

- Low-dose (0.025 dpa), low-temperature (50-70 C) neutron-irradiated W shows:
  - Similar depth profiles after 2<sup>nd</sup> plasma exposure
  - Saturated deuterium concentration (D/W) of 0.5 at. %
  - High deuterium retention ( $6 \times 10^{21} \text{ m}^{-2}$ ) and deep deuterium penetration at 500 C
  - Distinctive two peaks after annealing at 900 C for 0.5 hour
- Medium-dose (0.3 dpa), low-temperature (50-70 C) neutron-irradiated W shows:
  - Saturated deuterium concentration (D/W) of 1.0-1.5 at. %
- The deuterium concentration (D/W) trend observed (by NRA) for HFIR neutron-irradiated W was similar to that from high energy ion-beam study
  - ➔ High energy ion-beam study is valuable tool to characterize deuterium concentration
- The total retention trend observed (by TDS) for HFIR neutron-irradiated W was different (~ a factor of x10) from that of the high energy ion-beam study
  - ➔ Deuterium was observed to be migrated and trapped in bulk (tens of microns) at 500 C
  - ➔ NRA alone can not be used for estimating tritium retention in neutron-irradiated tungsten

## *Subtask review of activities in INL for task 2-1*

- Possible trapping mechanism(s) with the current understanding of the recovery temperature:
  - Exposure at 500 C decreased the number densities of vacancy and di-vacancy and increased that of small vacancy cluster ( $V_{4-10}$ )
  - Annealing at 900 C decreased the number densities of small vacancy cluster ( $V_{4-10}$ ) and middle size vacancy cluster ( $V_{11-16}$ ), and increased that of Void/Vacancy cluster ( $V_{40-60}$ )
  - Void/Vacancy cluster ( $V_{40-60}$ ) is nucleation site for bubble formation near the surface
  - “High temperature peak (600-1050K) is the D desorption from the vacancy clusters ( $V_{4-10}$  and  $V_{11-16}$ ) in the bulk”
  - “Low temperature peak (400-700 K) is the D desorption from the bubble formed in the void near surface.”
- More tungsten R&D in materials group and PWI (including PHENIX program) are needed to help understand the trapping mechanisms in neutron-irradiated tungsten.
- Deeper bulk (> tens of micro meter) tritium retention and its mitigation (e.g. He effects and/or W fuzz) needed to be investigated.