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Dynamic Measurements of Deuterium Retention in Tungsten During Exposure to Plasma

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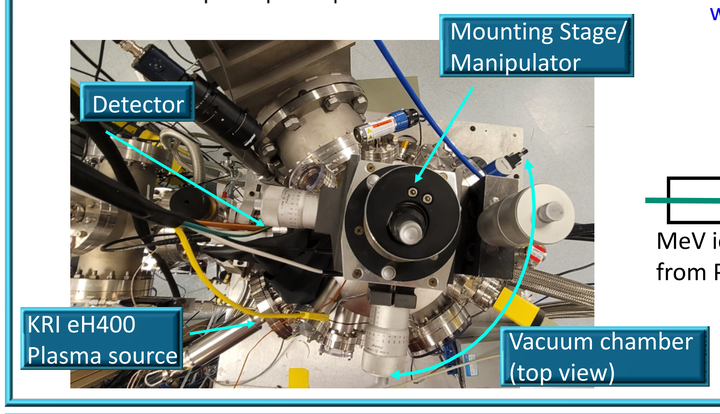
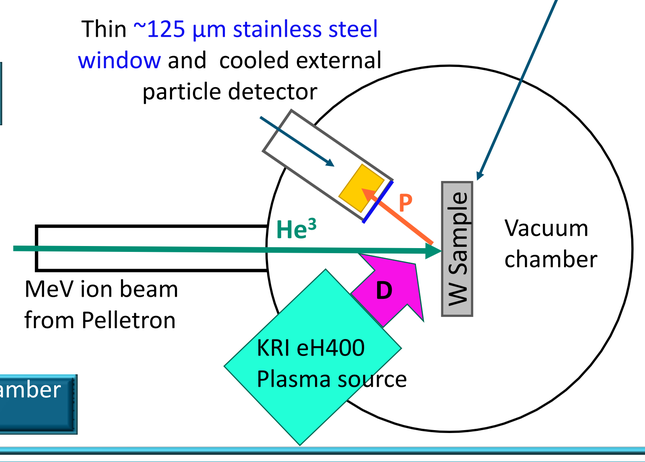
INTRODUCTION

To investigate materials, under fusion relevant environments, the Ion Beam Laboratory (IBL) at Sandia National Laboratories has developed a new endstation in which materials can be exposed to a high flux ($\sim 10^{20}\,\mathrm{D}\cdot\mathrm{m}^{-2}\ \mathrm{s}^{-1}$) of low energy (~100 eV) deuterium (D) ions and MeV ions from an NEC Pelletron ion accelerator. This new capability allows for dynamic measurements of near-surface D content in materials during exposure to plasma using D(He^3,p) He^4 nuclear reaction analysis (NRA). By doing in-situ and in-operando measurements we can examine physical mechanisms in real time such as implantation and diffusion, trapping at lattice defects and gas-phase precipitation, all which contribute to D retention and are strongly temperature dependent.

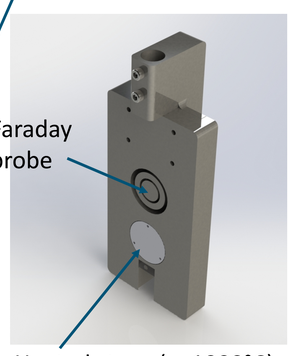
EXPRIMENTAL SETUP

- Using an NEC Pelletron accelerator we produce a beam of He^3 ions
- Ions are transported through the beamline into the (Fusion Energy Sciences) FES chamber
- The He^3 ion beam is incident on the tungsten(W) sample located on the mounting stage inside the chamber
- 0.8 MeV He^3 ion beam from NEC Pelletron produces ~12 MeV protons from D(He^3,p)He^4 nuclear reaction with D in target.
- Protons are counted by a particle detector, the accumulated yield is proportional to D content.
- Detection limit: 10^{18} D m^{-2} or 10 appm
- Detection depth: up to 3 µm in W





Mounting Stage



Capable of sparking plasmas comprised of
- Argon
- Helium
- Deuterium
- Or a mix of gases

CHARACTERIZATION OF PLASMA FLUX

Vacuum Conditions

Operation of the new plasma source was optimized by incorporation of a 1200 l/s turbo pump in combination with a 8 in Helix cryogen pump.
- Vacuum ~ 2×10^{-7} Torr
- In operation vacuum of ~ 1.5×10^{-3} Torr with up to 150 sccm of D_2 gas

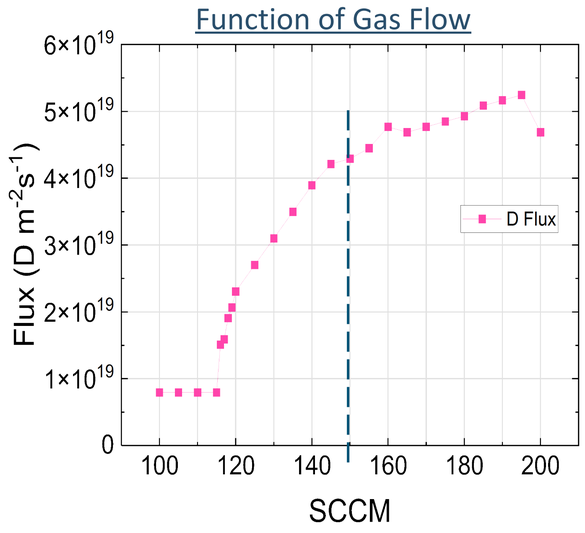
Plasma Flux Determination

- Plasma current is measured using the Faraday probe above the sample holder.
- The current is converted to a flux reading where **r** is equal to the diameter of the Faraday probe surface in meters (0.005).

$$\Phi = \frac{\text{Faraday Probe Current } [\frac{C}{s}]}{\pi \times \mathbf{r}^2 \times 1.602\times10^{19}\ C} = \frac{\text{Ions}}{m^2\cdot s}$$

- Max flux: ~ $10^{20}\ \mathrm{D}\cdot\mathrm{m}^{-2}\mathrm{s}^{-1}$
- Energy of D from plasma: ~ 100 eV [1]

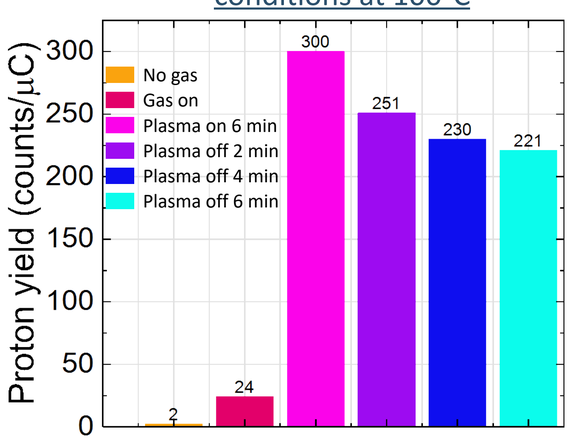
Deuterium Plasma Flux as a Function of Gas Flow



Flux quickly increases as a function of gas flow (between 110 sccm to 140 sccm) and then tapers off. As a result, 150 sccm gas flow was chosen during experiments.

PRELIMINARY RESULTS: Deuterium retention

1. Measured deuterium for different exposure conditions at 100°C



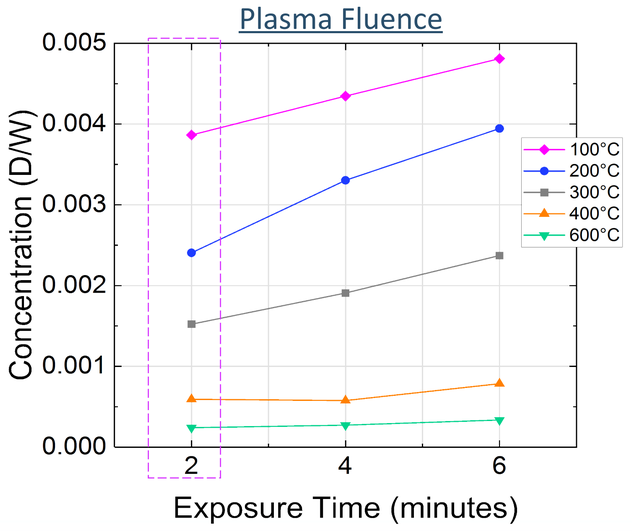
- Deuterium begins to diffuse outward quickly after the plasma is turned off.
- This demonstrates the need and benefit of in-operando NRA measurements where diffusion at shorter time scales can be studied.
- Traditional NRA is performed up to months after exposure

2. Deuterium Reference Counts

$$N_W = Y_W \cdot \left(\frac{N_{D_{ref}}}{Y_{D_{ref}}}\right)$$

- Deuterium reference (0.5µm ErHD) yielded 23,592 counts per µC for a D content $N_D = 1.43 \times 10^{22}\ \mathrm{D}\cdot\mathrm{m}^{-2}$. [2]
- By using the equation above we can convert our counts during experiments, to an areal density of $\mathrm{D}\cdot\mathrm{m}^{-2}$

3. Deuterium Content as a Function of Plasma Fluence



We observed evidence of temperature dependent deuterium retention. As temperature increases, the near-surface retained deuterium decreases. The deuterium decrease is even more prominent at high temperature where there is little to no significant retained deuterium changes from 2 minutes to 6 minutes.

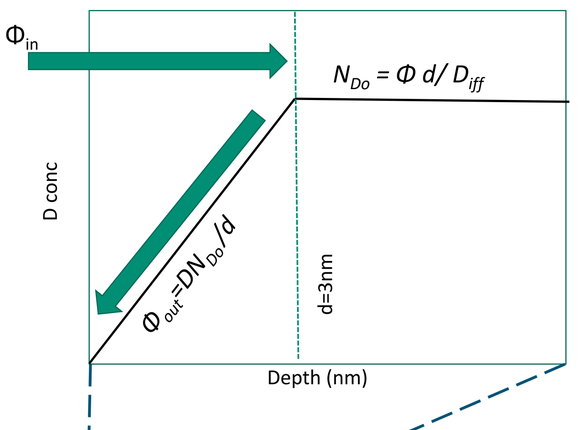
MODEL: Implantation from plasma with diffusion-limited release from surface

Near-surface concentration $N_{Do} = \Phi\ d/\ D_{iff}$
d ~ 3 nm range for 100 eV ion energy (SRIM)

Flux of deuterium from plasma:
$\Phi_{in} \sim 0.6 \times 10^{16}\ \mathrm{D}\cdot\mathrm{cm}^{-2}\ \mathrm{s}^{-1}$

Steady state:
$\Phi_{in} = \Phi_{out} = D_{iff}\ N_{Do}\ /d$

diffusion length $x_o = \sqrt{D_{iff}\ t}$
$D_{iff} = D_o \exp(-Q_D/kT)$
D_o = 4.1e-3 cm^2/s [3]
Q_D= 0.39 eV [3]

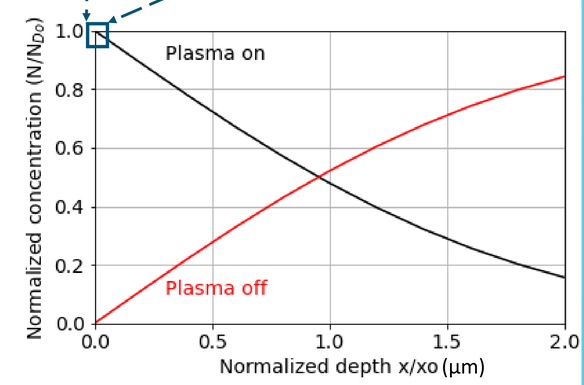


Deuterium concentration (N_d) in solution vs depth

(i.e., assumes no trapped or precipitated deuterium)

Plasma on:
Initial: $N_D(x>0,t=0) = 0$
Boundary: $N_D(x=0,t>0) = N_{Do}$
$N_D(x,t) = N_{Do}\ (1 - \mathrm{erf}(x/2x_o))$

Plasma off:
Initial: $N_D(x>0,t=0) = N_{Do}$
Boundary: $N_D(x=0,t>0) = 0$
$N_D(x,t) = N_{Do}\ \mathrm{erf}(x/2x_o)$



NRA yield vs depth & He^3 energy

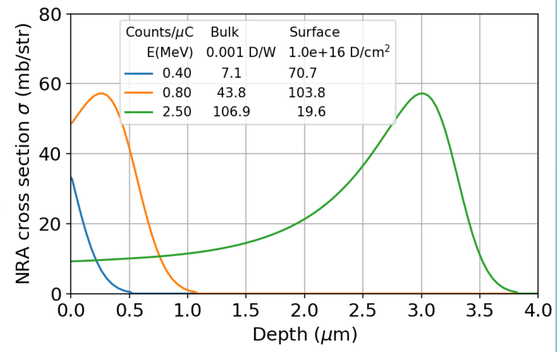
$$Y(t) = (\Omega N_i) \int \sigma(x)\ N_D(x,t)\ dx$$

Measures deuterium within:
- 0.1 µm with 0.4 MeV He^3
- 0.5 µm with 0.8 MeV He^3
- 3.0 µm with 2.5 MeV He^3

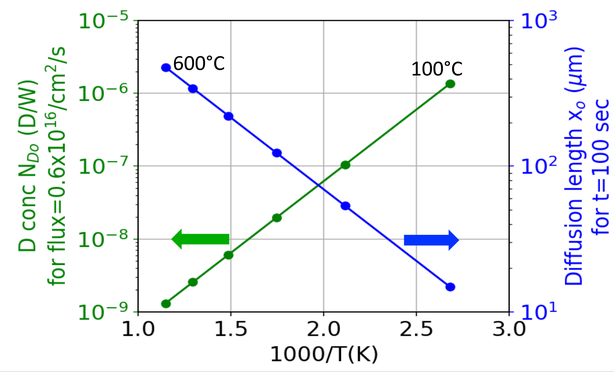
Can distinguish bulk D from surface D from yield vs He3 energy:

Uniform D conc $N_D = N_{do}$
$$Y = (\Omega N_i)\ N_{Do} \int \sigma(x)\ dx$$

Surface D areal density A_D
$$Y = (\Omega N_i)\ A_D\ \sigma(0)$$



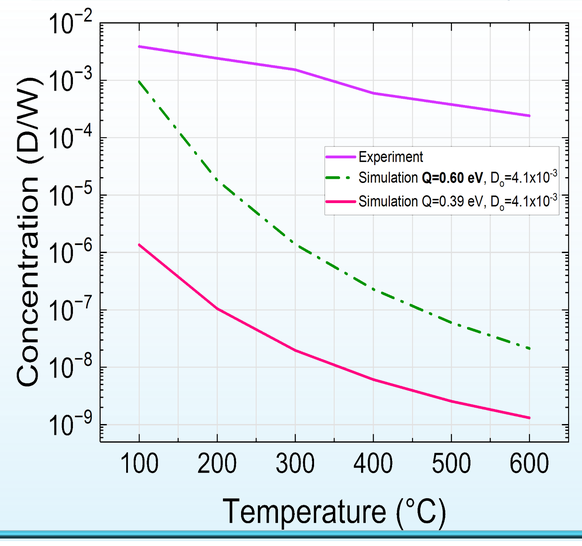
As temperature increases, deuterium concentration decreases and diffusion length increases.



EXPERIMENTS VS MODEL COMPARISON

- Both experimental and simulated D to W concentrations decrease with increasing temperature.
- However, Simulated concentrations are much lower than experimentally observed ones.
- Diffusion coefficients need to be much smaller for concentrations to increase and more closely simulate experimental results.

Comparison of Experimental and Simulated D/W concentrations for 2 minute exposure



CONCLUSION

In this work we have discussed coupling of a plasma source to the existing Ion Beam accelerator and demonstrated a new capability at the IBL. We can dynamically measure deuterium in materials during and after exposure to plasma as a function of temperature, to study response to fusion-plasma environment. Experimental results support modeling work where near surface deuterium has been observed to decrease as a function of increasing temperature. However, given this is a simple model in its initial development stage, the diffusion coefficient used is 2 to 3 orders of magnitude too large when compared to experimental results. Future simulations will include smaller diffusion coefficients that can more closely simulate experimental results.

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[1] A high-flux low-energy hydrogen ion beam using an end-Hall ion source, Jacqueline van Veldhoven, Edwin te Sligte, Jochem Janssen, ICCC
[2] Round robin analyses of hydrogen isotope thin films standards , J. Banks et. al. NIMB 219-220 (2004) 444-449
[3] Solution and diffusion of hydrogen in tungsten, R. Frauenfelder, JVST 6 (1969) 338-397

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