Deuterium Retention in Tungsten-Coated Reduced Activation Ferritic/Martensitic Steel

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Background

Tungsten (W)

Good candidate as plasma-facing material (PFM) in fusion reactor

Bulk W is heavy → **Development of tungsten coating** on PFM (such as F82H)

H isotope retention/ desorption

- Influences density control of fusion plasma, and safety operation of reactor(T inventory).
- **Greatly depends on the material (composition, microstructure).**

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Objectives

Deuterium Retention in Reduced Activation Ferritic/Martensitic Steel with Sputter-Deposited tungsten layer

- ✓ To evaluate the deuterium retention/desorption behaviour of tungsten coating on reduced activation ferritic/martensitic steel (F82H).
- ✓ To study the mechanism of deuterium trapping in tungsten film and its relation with the desorption behaviour.

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Sample

Tungsten-coated F82H (W/F82H)

Deposited by rf magnetron sputtering
W film thickness 1: 50 nm (thin)
W film thickness 2: 1 μm (thick)
Surface Area: 20 mm x 5 mm
W film density: 18.8 g/cm^3

F82H (manufactured by JAEA)

Sample size:
30 mm x 5 mm x 0.2 mm

Tungsten (W) sheet

99.95% (Nilaco Corporation)
30 mm x 5 mm

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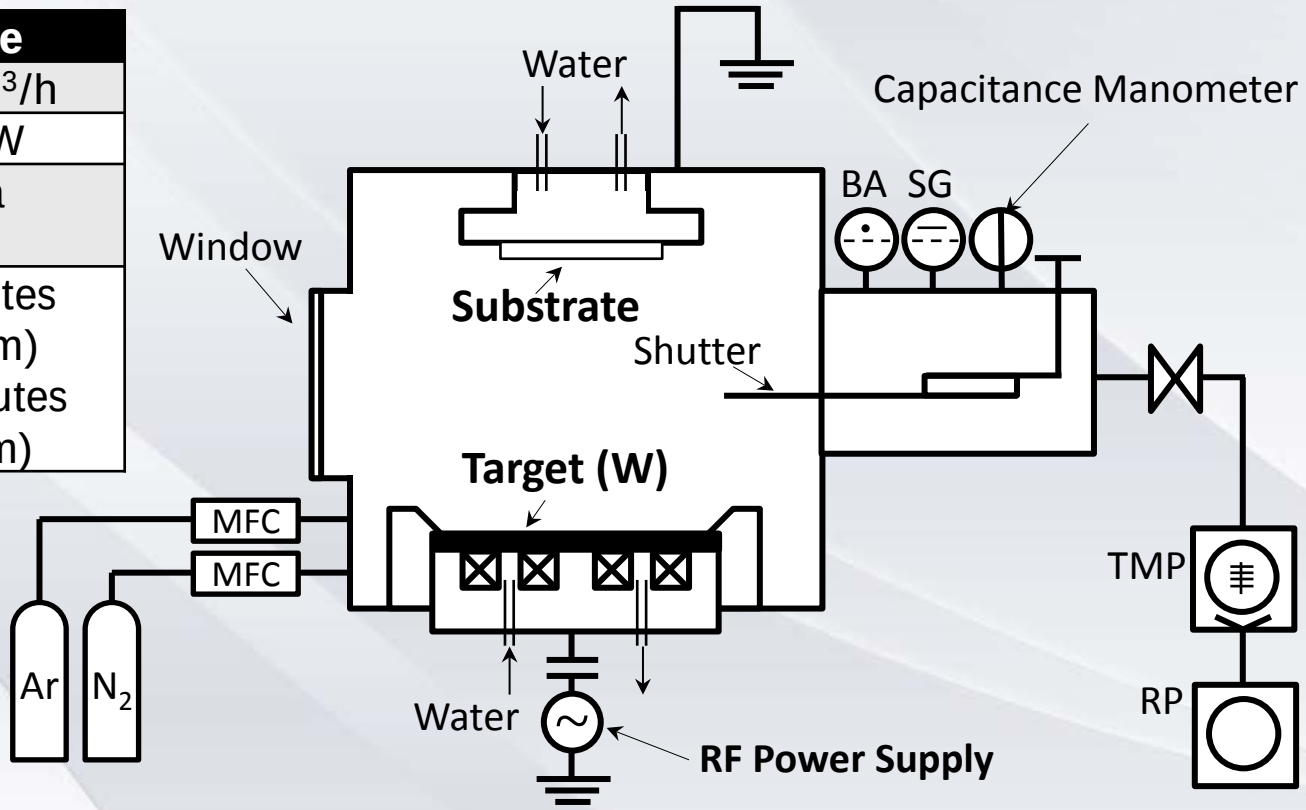
RF Magnetron Sputtering Device

Parameter	Value
Ar flow rate	0.02 m^3/h
Power	400 W
Working Pressure	3 Pa
Deposition time	4 minutes (50 nm) 60 minutes (1 mm)



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Results of XPS analysis (W/F82H)

Element	Composition (at.%)
W	80-81
C	11.5-12.4
N	3.2-3.5
O	4.3-4.7

- ✓ Metal-W and WO_2 in coating interior
- ✓ Surface impurities of O

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Experimental Procedure

Degassing

TDS Apparatus

Degassed at 600°C for 30 mins

Deuterium Ion Irradiation

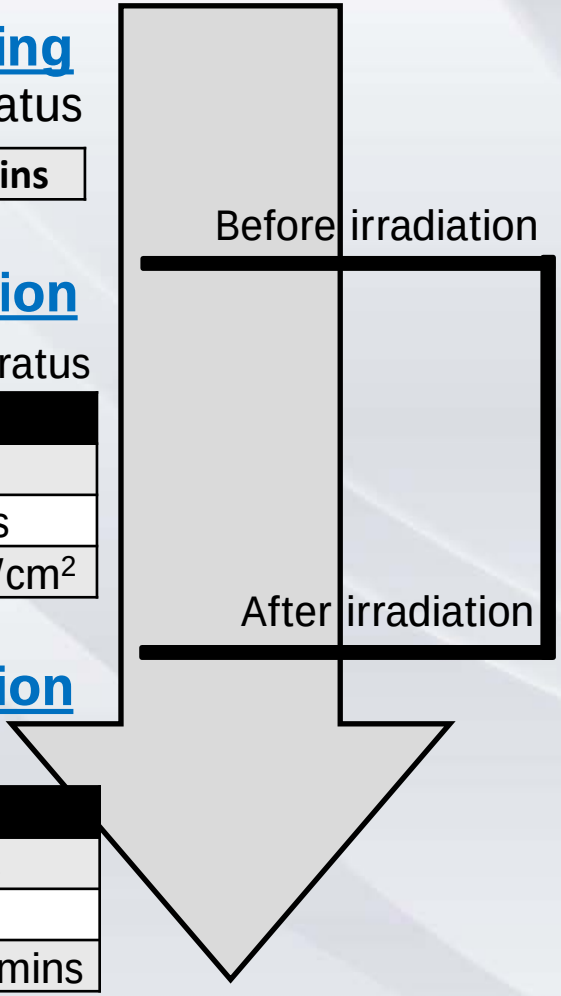
ECR Ion Irradiation Apparatus

Parameter	Value
Temperature	RT
Energy	5 keV D_3^+ ions
Fluence	(0.5 – 2) x10^{18} D/cm^2

Retention Evaluation

TDS Apparatus

Parameter	Value
Heating	RT to 800°C
Heating Rate	0.5 K/s
Hold up	At 800°C for 30 mins



Surface Observation

Scanning Electron Microscopy (SEM)

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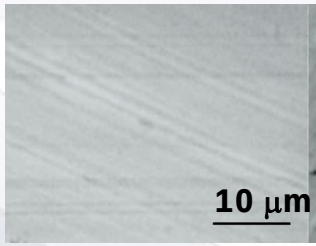
Surface Morphologies of Thin W Film

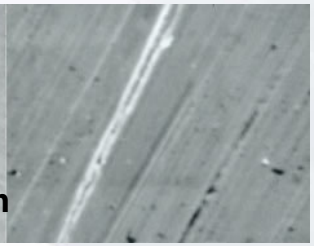
Before irradiation

F82H



Linear pattern

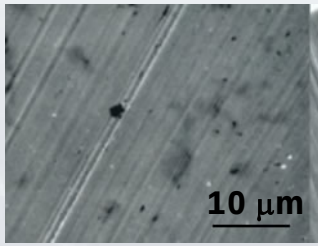
Thin W/F82H



Linear pattern
(~1 μm distance)
Initial cracks (porous)

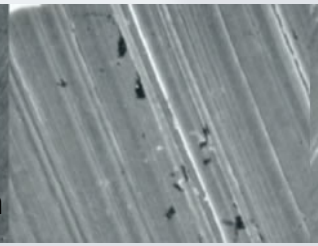
After irradiation

0.5 x10^{18}D/cm^2

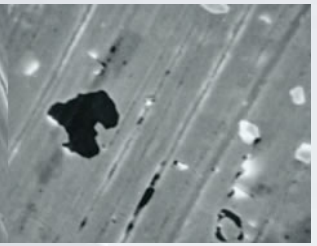


Still some cracks for both fluences
No significant difference

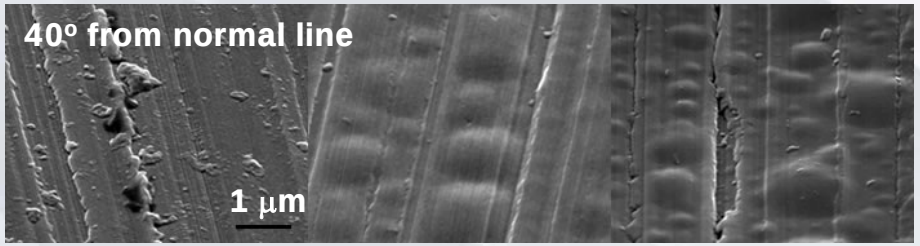
1 x10^{18}D/cm^2



2 x10^{18}D/cm^2



Larger cracks
Up to 7 μm



No blisters

Small circular blisters ~1 μm max

- Blister size → limited by F82H linear pattern acting as grain boundaries
- Film exfoliation → Film is too thin to hold accumulated gas, leading to **blister burst**

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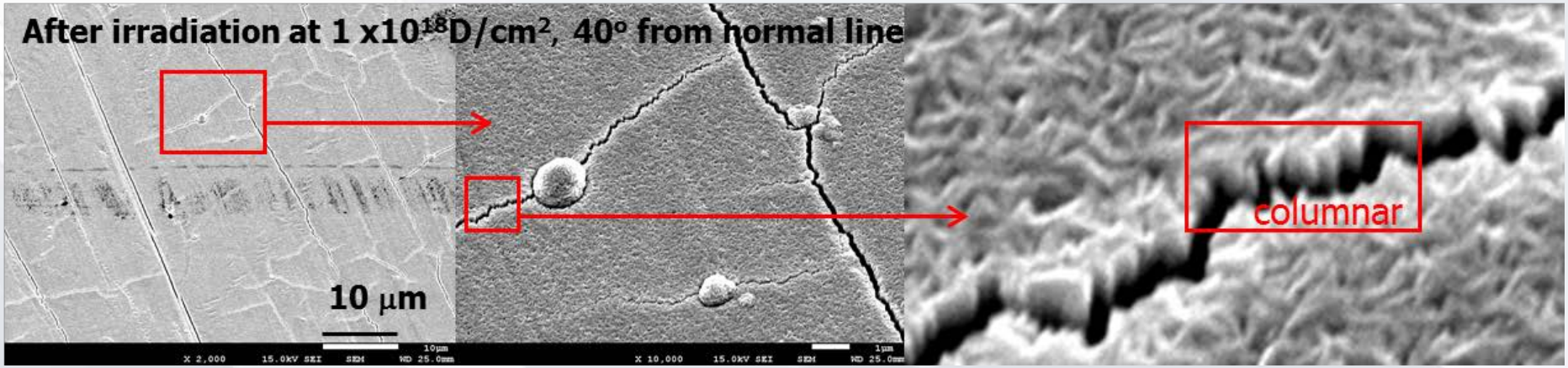
Surface Morphologies of Thick W Film



- Film cracks due to tensile stress
- Columnar pattern

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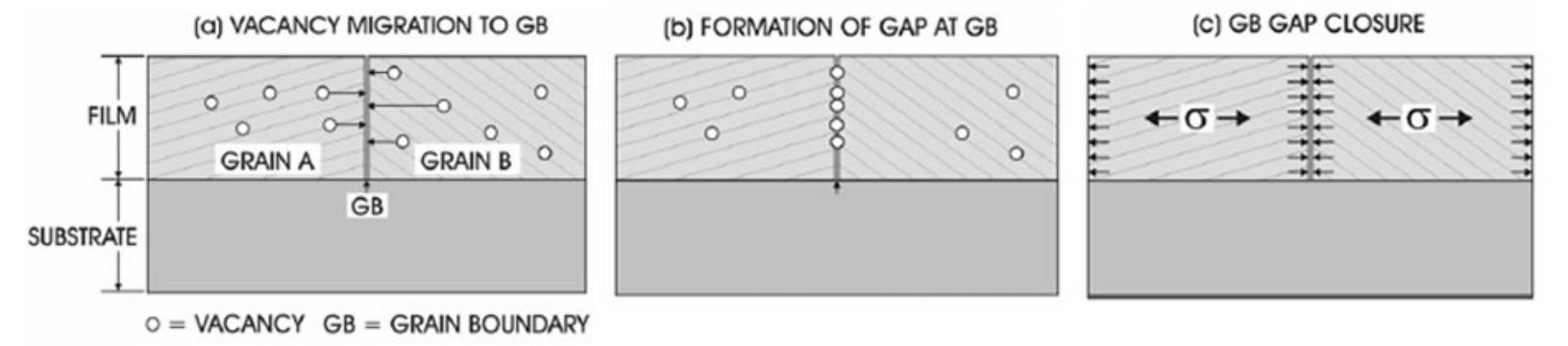
Surface Morphologies of Thick W Film

Film Tensile Stress

Common stress in deposited film [1]



(a) **Intrinsic/Ion-induced vacancy migration** from the bulk to grain boundaries.
(b) Excess vacancies at GB is annihilated to maintain the equilibrium vacancy concentration at RT.
(c) The shrinkage is countered by the substrate, thus giving bi-axial tensile stress to the film.

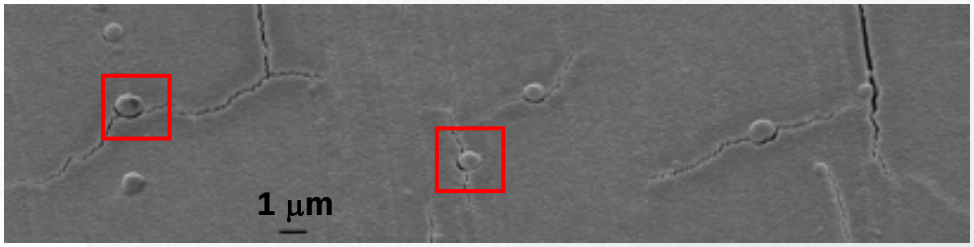
When the tensile stress reaches the film yield strength, plastic deformation occurs.

[1] S. Nakahara, S. Ahmed, D.N. Buckley, and T. Tanaka Ahmed. ECS Transactions, 2 (6) (2007) 167-183.

Surface Morphologies of Thick W Film

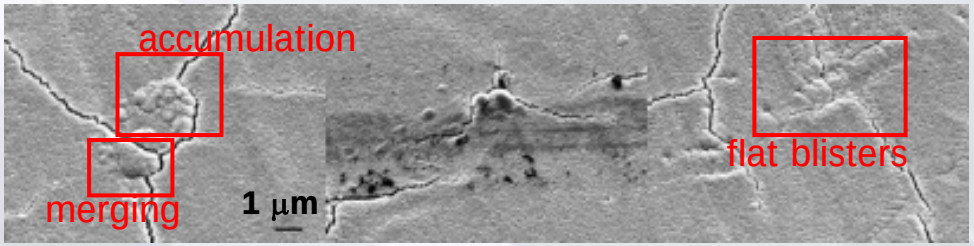
After irradiation (40° from normal line)

0.5 x10^{18} D/cm^2



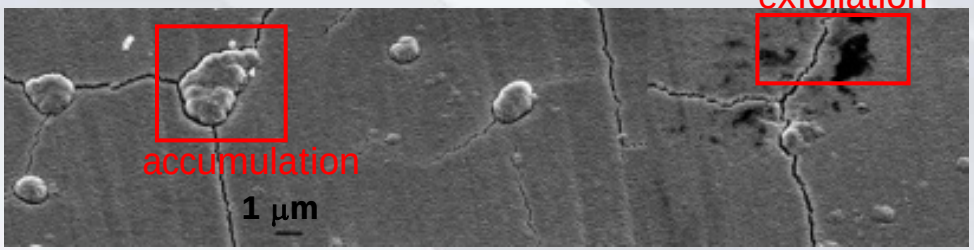
Circular blisters with size about 1 μm

1 x10^{18} D/cm^2



Circular blisters with size about 1 μm
Blister growth by accumulation
Blister growth by merging
Flat blisters due to small cracks

2 x10^{18} D/cm^2



Further blister growth was allowed
Clear large blisters with size more than 1 μm
Film exfoliation
Crack growth

Blister Locations

- Cracks (many)
- Throughout the surface/Bulk location (small portion)

Blister growth through vacancy migration

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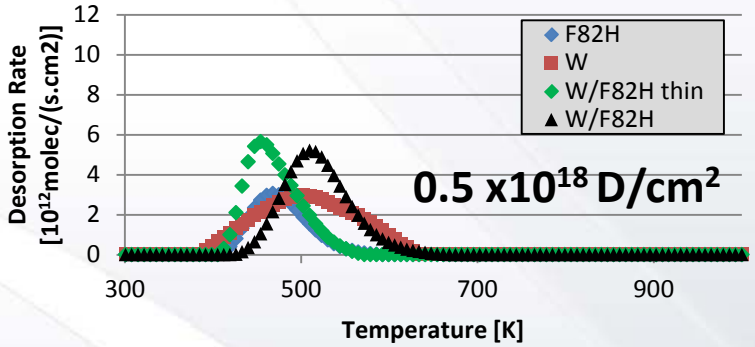
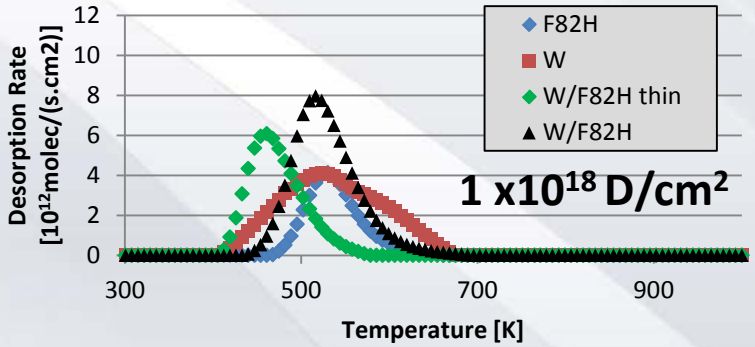
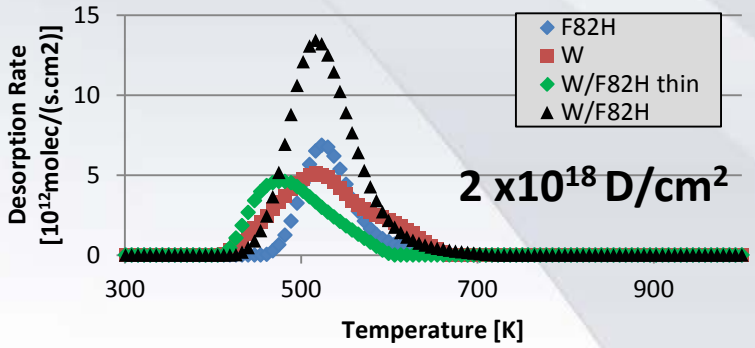
Thermal desorption Spectra

D_2 spectra after irradiation







F82H

- Peak at 520 K
- Common peak of F82H: 523 K (trapping at martensite laths)
- Peak shift at low fluence (480 K) → impurities

Pure W

- Peak around 500-520 K
- Shoulder around ~600 K

Thick W Film (W/F82H)

- Peak around 500-520 K
- No shoulder around ~600 K

Thin W Film (W/F82H thin)

- Peak around 480-500 K
- No shoulder (same shape as thick W film)

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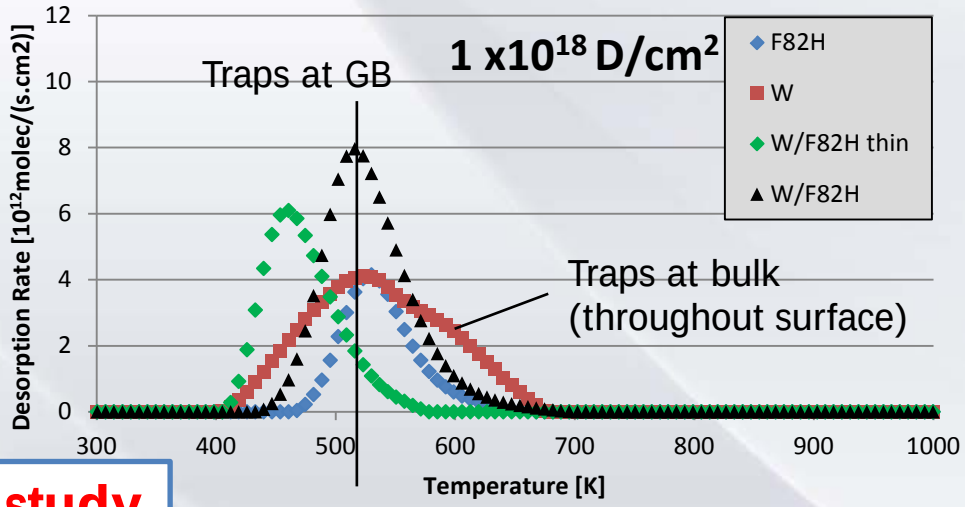
Desorption Peaks in W

Common Traps in W

- ✓ **Low temp. traps (500-700 K)**
  Due to vacancies, surface impurities
- ✓ **High temp. traps (900-1000 K)**
  Due to voids in deeper section



D_2 Peaks Obtained in present study

- W peak at 640 K is attributed to vacancies throughout the material (bulk) [2]
- Thin W/F82H peak shifted to low temperature region due to blister burst [3]
- No ~600 K shoulder for W/F82H due to the vacancy migration from bulk to GB

[2] M. Poon, A.A. Haasz, J.W. Davis, J. Nucl. Mater 374 (2008) 390-402.
[3] W.M. Shu, K. Isobe, T. Yamanashi. Fusion. Eng. Des. 83 (2008) 1044-1048.

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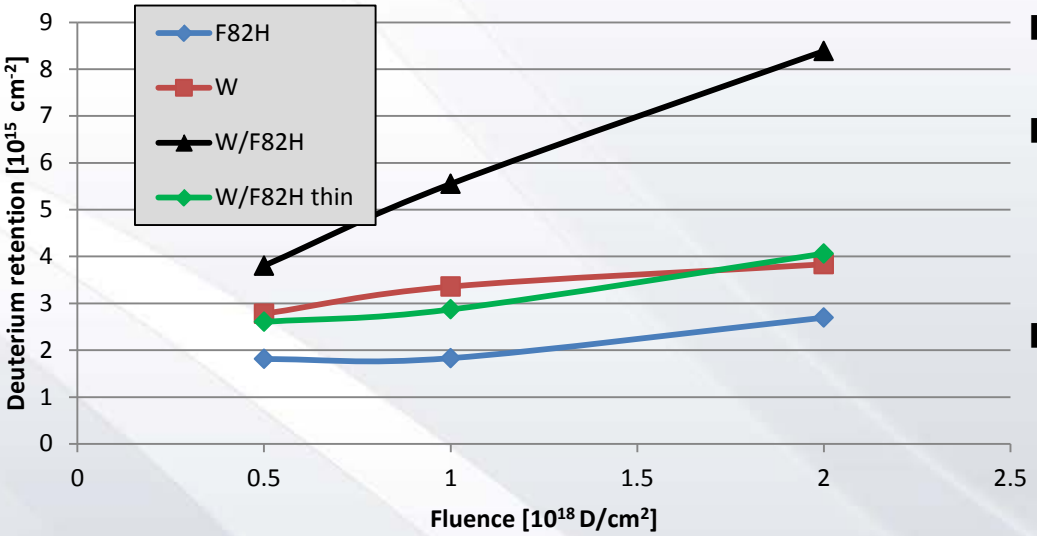
Deuterium retention

Total D Retention



- Retention increases with fluence
- Retention in both W/F82H is higher than in F82H due to porous structure of the film
- Retention in thin W/F82H is similar to that in pure W, but retention in thick W/F82H is drastically higher

Thin W Film

- The available traps inside the film might be already occupied. → saturation behavior may be reached.
- Limitation of blister growth → low retention

Thick W Film

- Molecular D accumulate inside pores and cracks through the porous path → large retention
- Further growth of blisters → large retention

Different structure → Different D retention

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Conclusion

Deuterium Retention in Tungsten-Coated Reduced Activation Ferritic/Martensitic Steel

- ✓ Different retention/desorption between the thin and thick film was caused by the different structures.
- ✓ Deuterium atoms were retained in a large amount because of the porous structure inside the W film.
- ✓ The production of a dense W film might minimize deuterium retention.

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