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A Comparison of Near Surface Precipitation in Tungsten Alloys Due to High Flux Plasma Exposure

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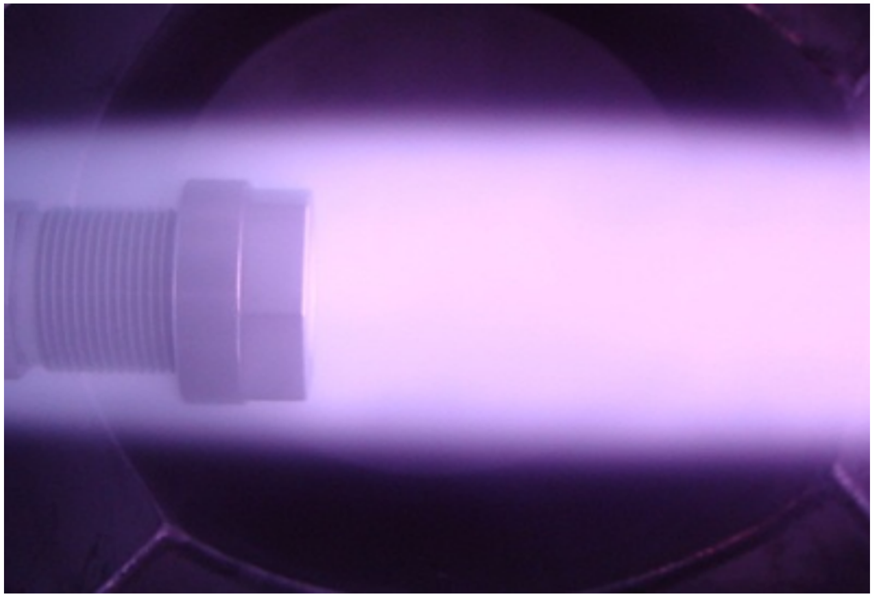
Question: Can tungsten microstructure affect hydrogen precipitation due to plasma exposure?

- Precipitation affects hydrogen migration through material
- Bubble growth mechanisms depend on microstructure
- Higher concentration of grain boundaries could affect diffusion
- Growth mechanisms are critical to developing realistic models

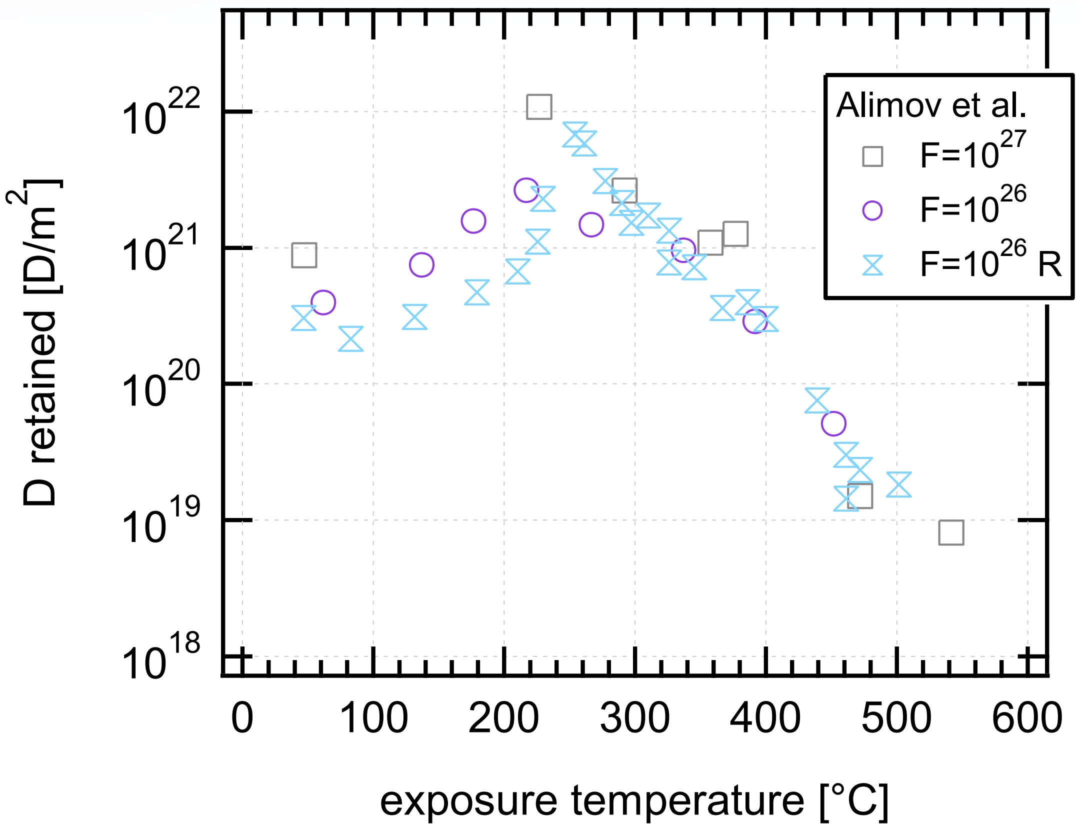


ITER-grade W Exposure Conditions

exposure type	ion energy [eV]	duration [min]	flux (Γ_i) [m^{-2} s^{-1}]	fluence (Φ) [m^{-2}]
LF	100	60	4.9×10^{21}	1.8×10^{25}
HF	100	120	1.5×10^{22}	1.1×10^{26}

- TPE plasma exposures at INL
- Microscopy at Shizuoka

Compare Retention with Measurements Obtained in Other Laboratories



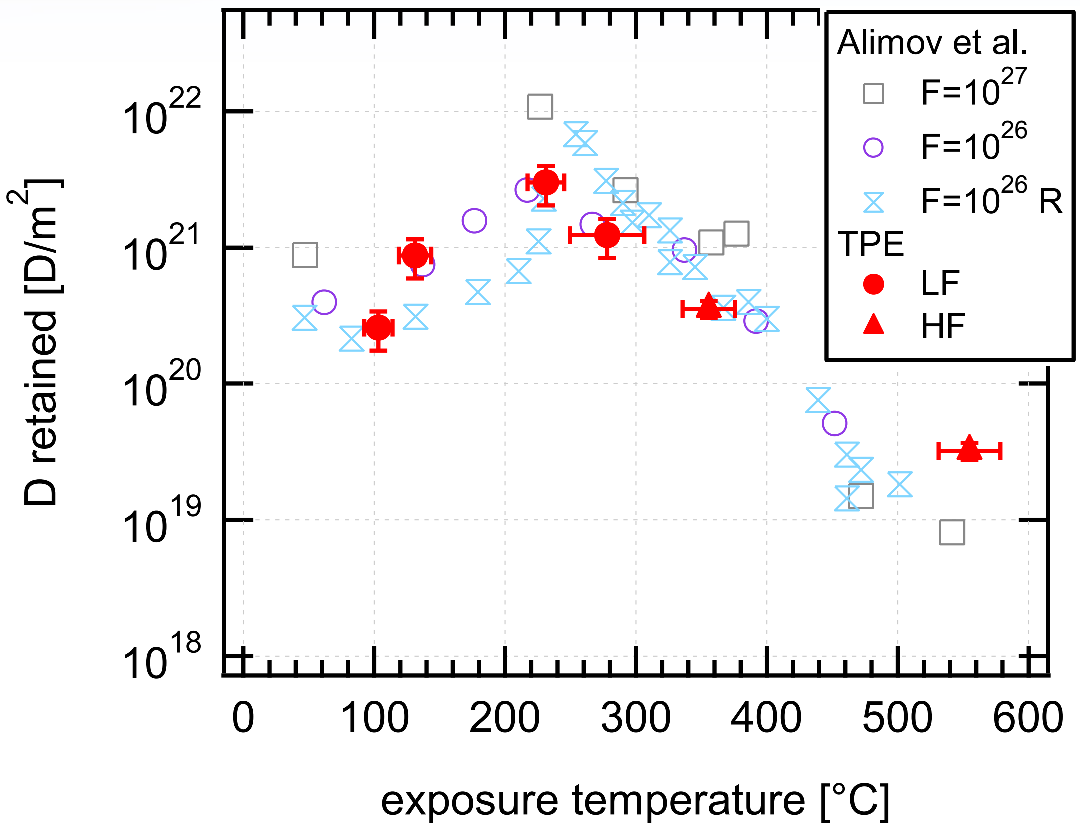
V. Kh. Alimov, et al. *J. Nucl. Mater.* **420** (2012) 519.

Previous work by Alimov et al:

- ITER-grade W
- E=38 eV
- $\Gamma_i = 10^{22}$ D m^{-2} s^{-1}

Comparable exposure conditions

Compare Retention with Measurements Obtained in Other Laboratories



Previous work by Alimov et al:

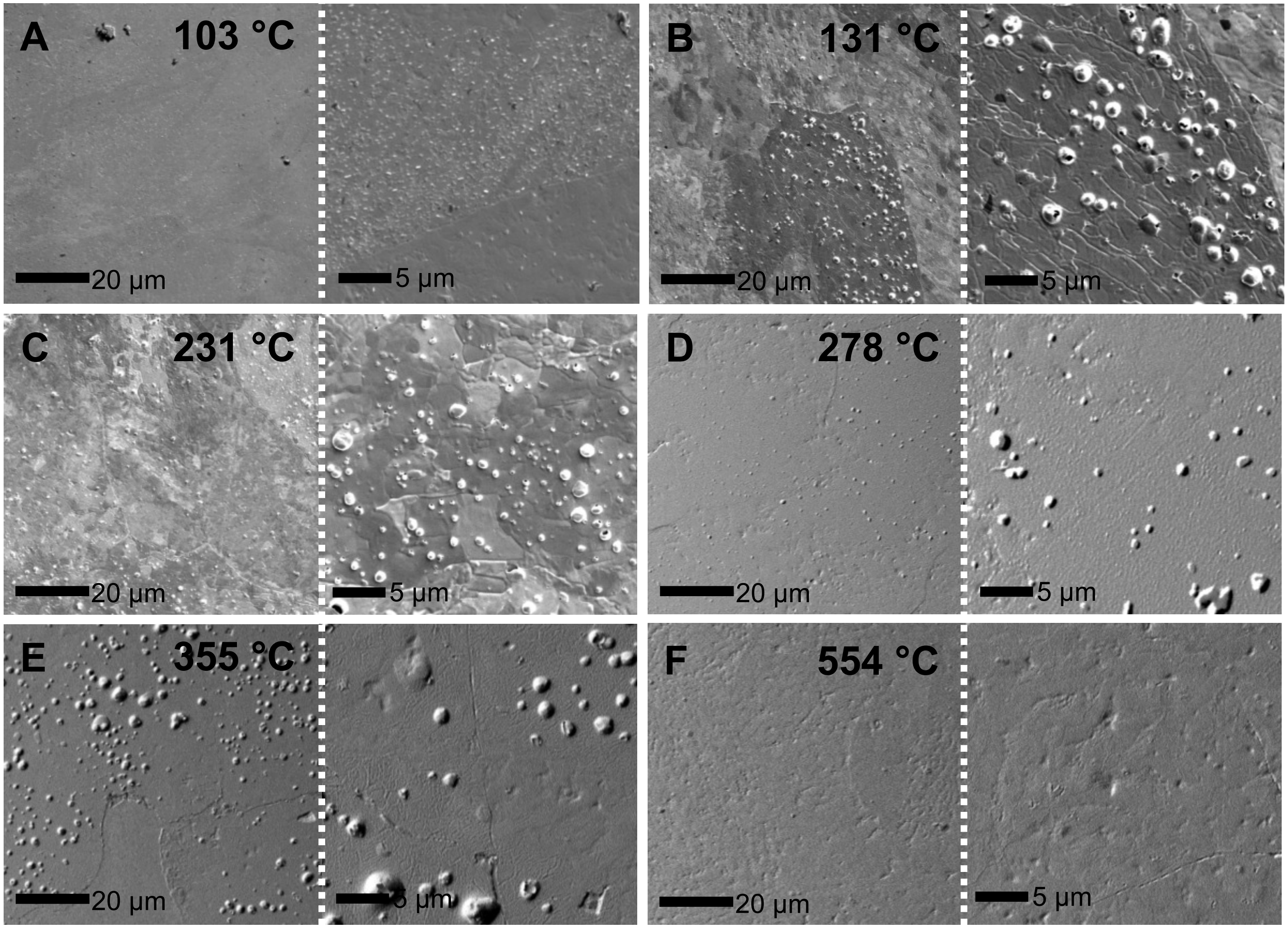
- ITER-grade W
- E=38 eV
- $\Gamma_i = 10^{22}$ D m^{-2} s^{-1}

Comparable exposure conditions

TPE retention measurements (by TDS):

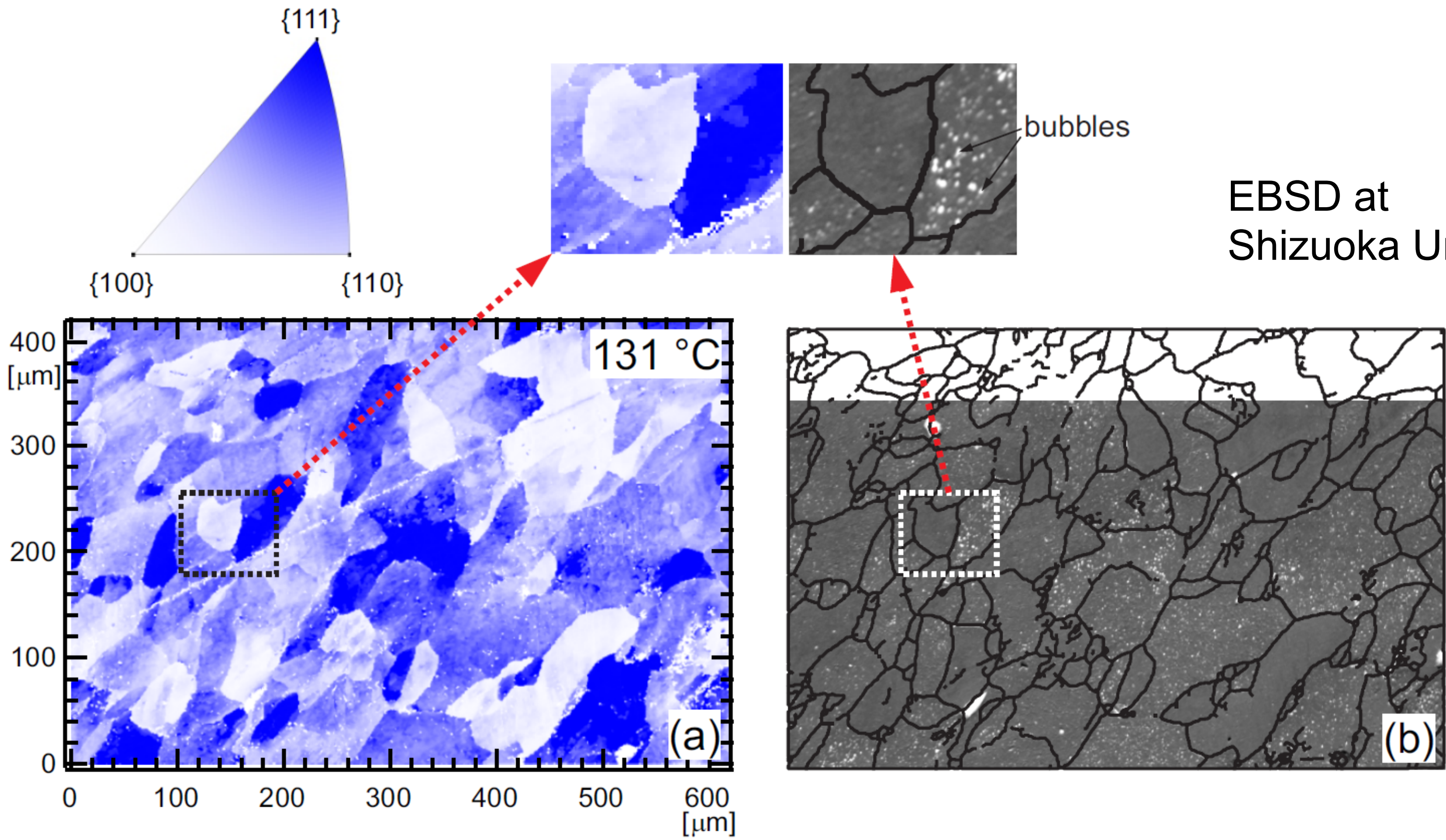
- Correspond closely with Toyama/IPP measurements.
- Confirm accepted retention temperature dependence.

Surface Morphology Variation with Temperature



- Key features:
- Non-uniform coverage
- Bubbles are small (<10 µm dia.) compared with warm-rolled W material
- Growth minimized at low and high temperature

Electron Backscatter Diffraction Clarifies Grain Orientation Dependence of Nucleation

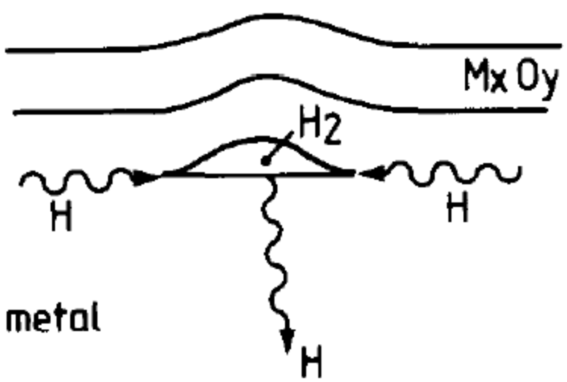


EBSD at
Shizuoka University

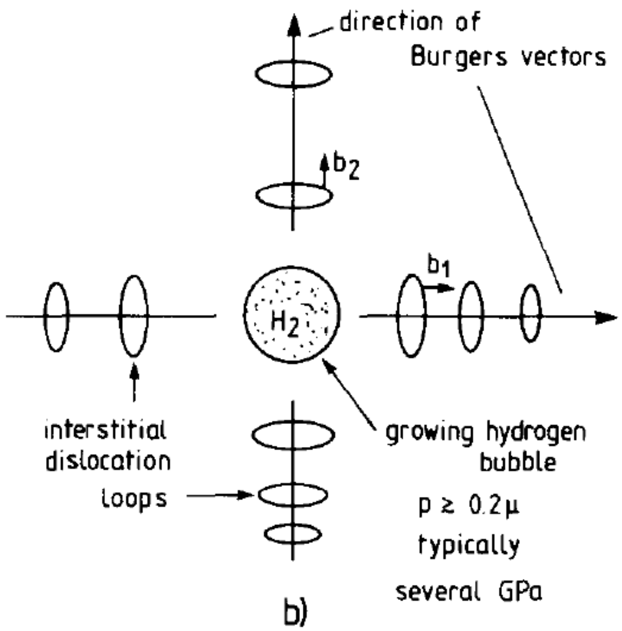
Bubbles nucleate on grains aligned with {110}<111> slip systems

What Bubble Growth Mechanisms are Active in Tungsten During Plasma Exposure?

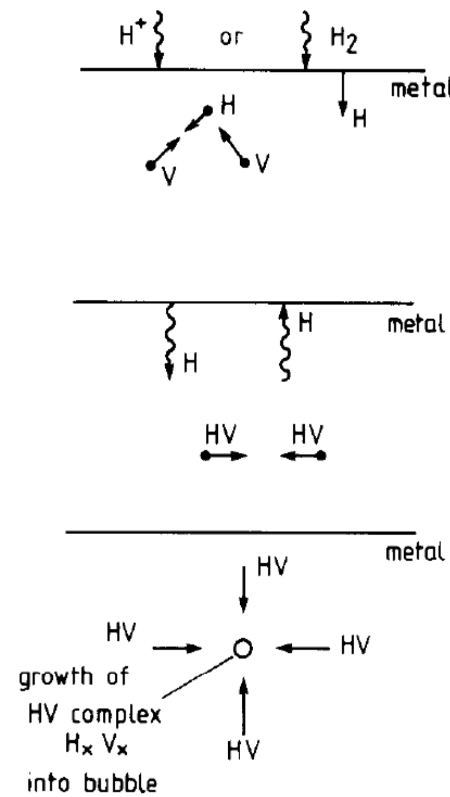
near-surface plastic deformation



dislocation loop punching

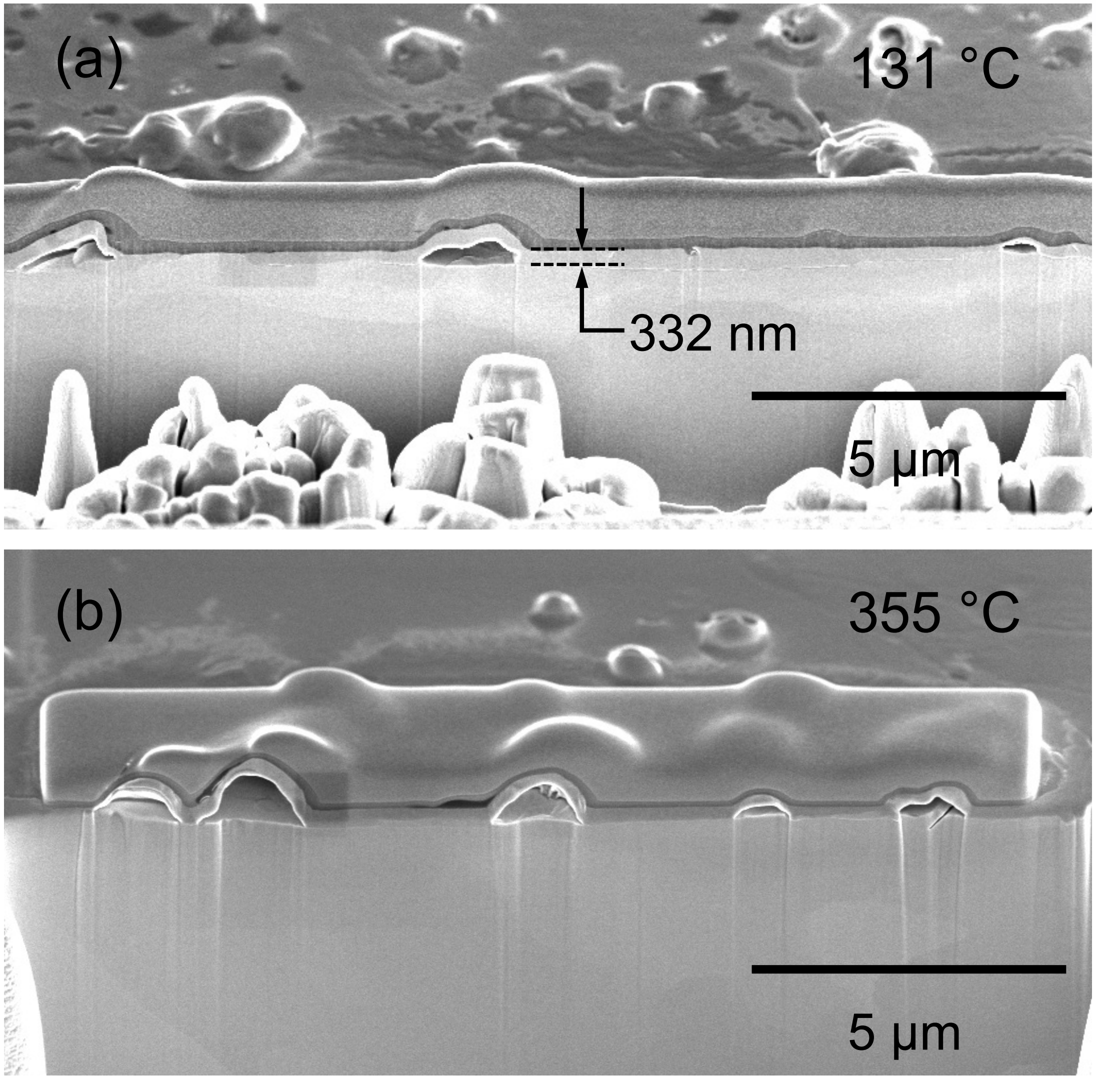


vacancy clustering

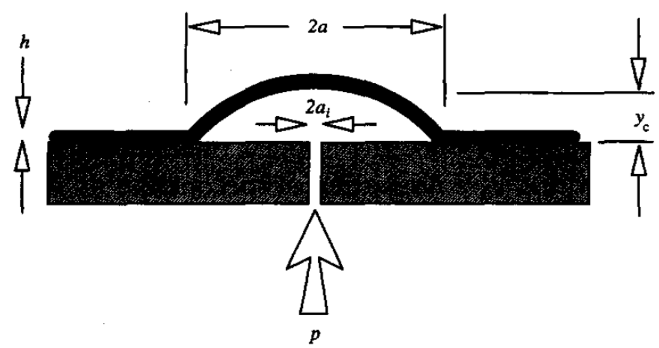


Figures from: J. B. Condon & T. Schober, *J. Nucl. Mater.* **207** (1993) 1.

Focused Ion Beam Profiling Reveals Precipitates Expand Through Crack Extension



- Nucleation near the surface is favored by crack extension: platelet-shaped cracks
- Expansion due to internal gas pressure (> 1 GPa)



$$p \geq (4T(Eh)^{1/3}/5C_1C_2)^{3/4}/r_b$$

K. –T. Wan and Y. –W. Mai, Acta. Metal. Mater. **43**, 4109 (1995).

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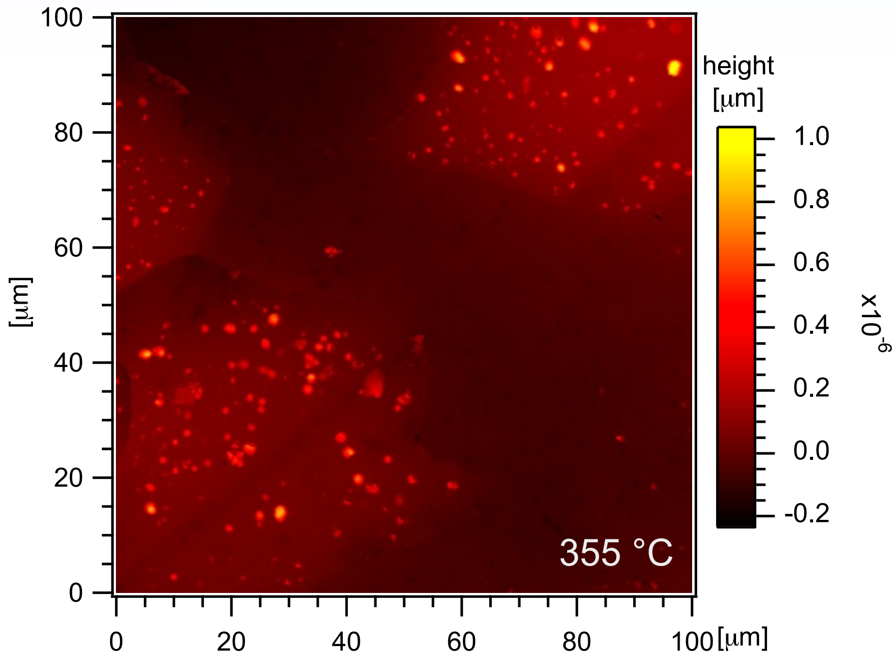
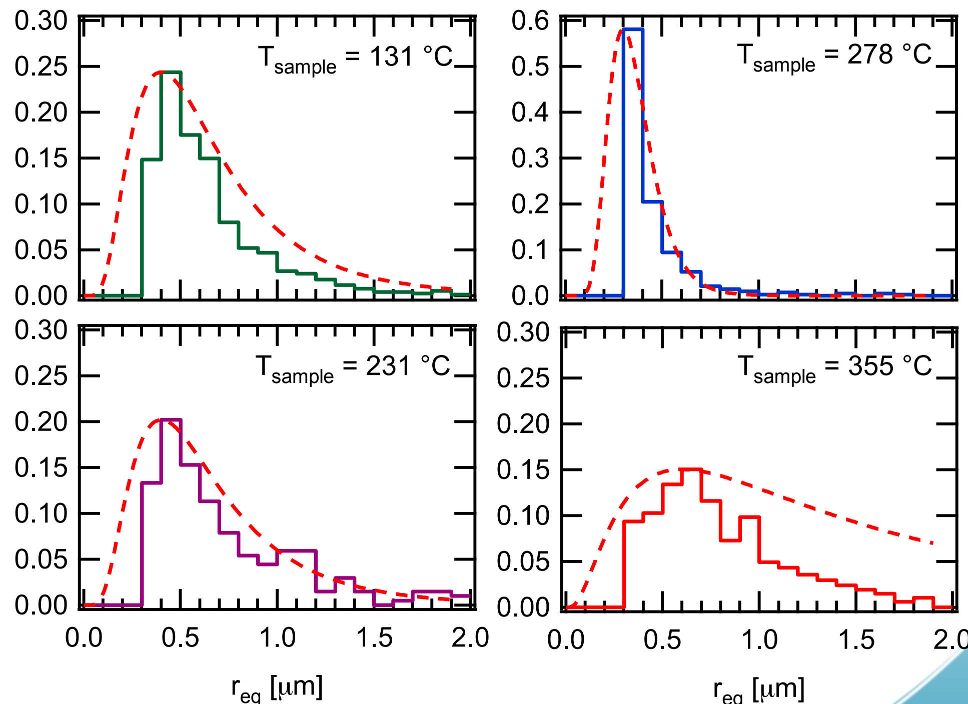
Atomic Force Microscopy Reveals Details of Surface Structure

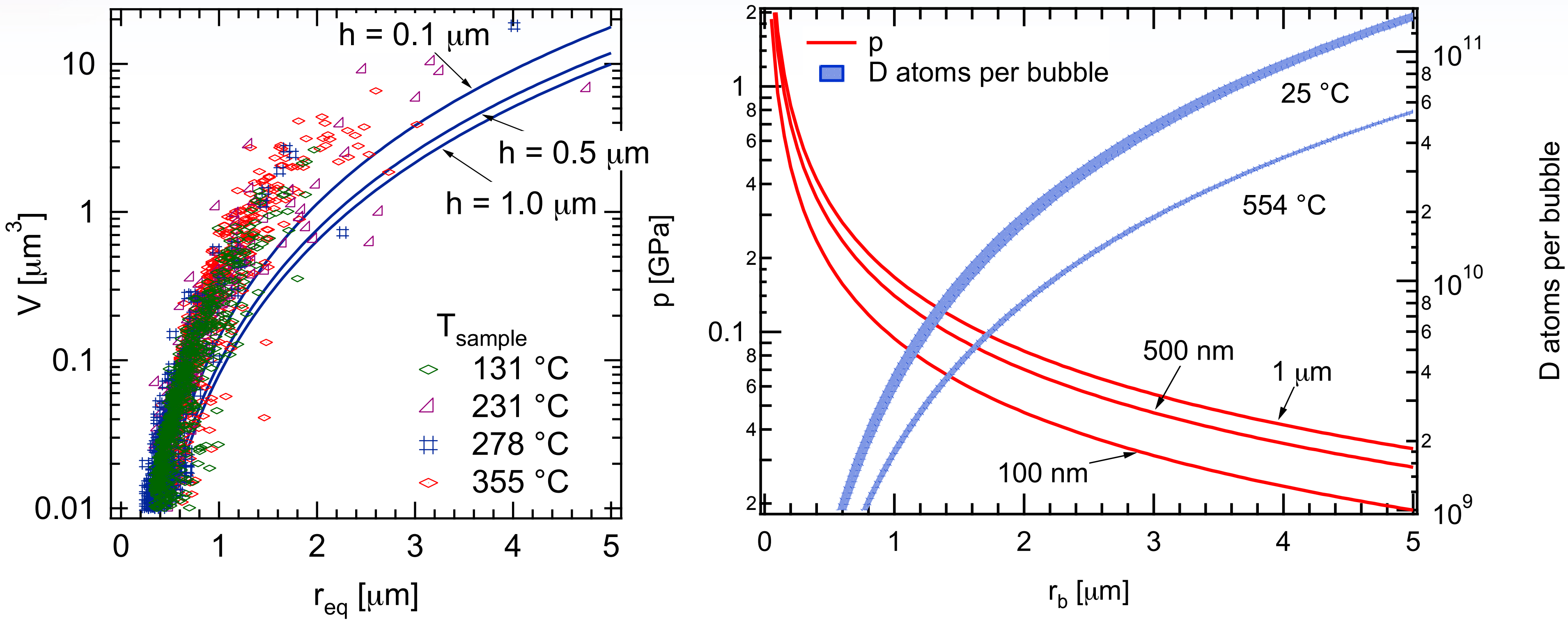


- Atomic force microscopy provides information on the shape of the deformed surface
- Individual bubbles identified and analyzed automatically.

corresponding bubble size distributions



Atomic Force Microscopy Quantifies Bubble Size and Distribution of Deuterium Therein

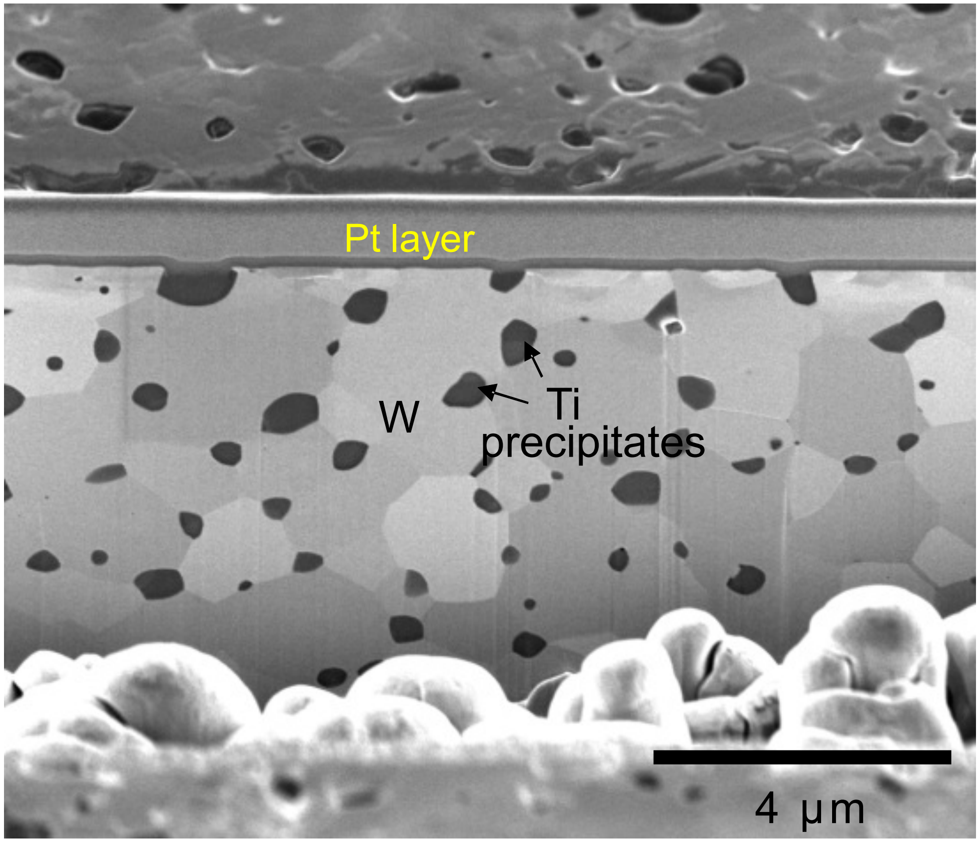


Bubble sizes correlate well with model predictions, log-normal distribution

Conclusions:

- Projected D trapped in bubbles: 2% - 28% of total inventory
- Precipitates unable to prevent deep diffusion in ITER-W
- Publication: R. Kolasinski, et al. *J. Appl. Phys.* ***118*** *(2015) 073301.*

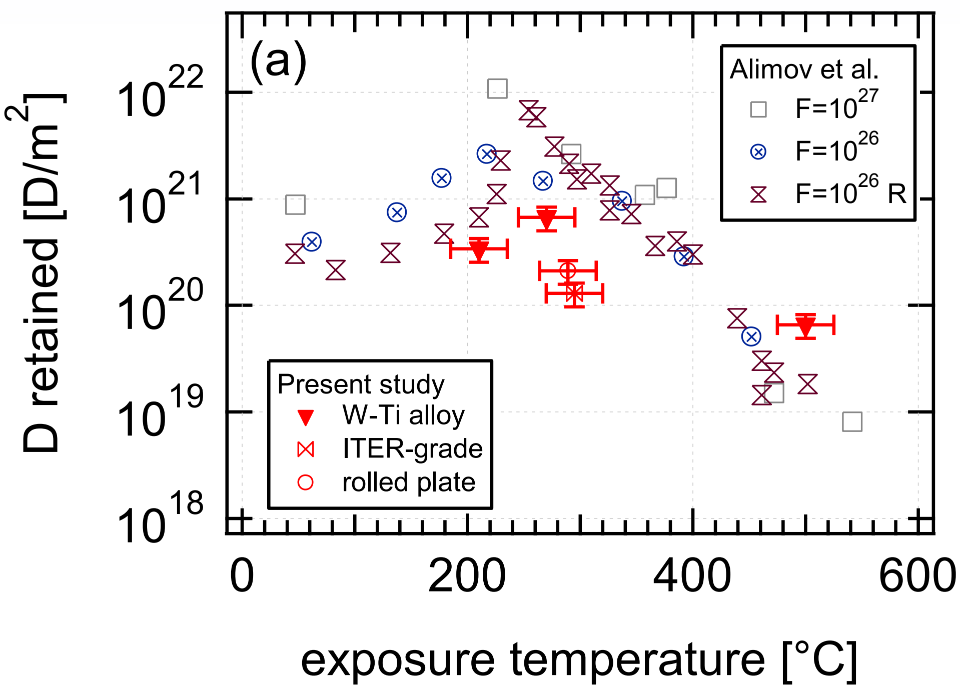
Plasma Retention Measurements Performed On Dispersoid Strengthened UFG Tungsten



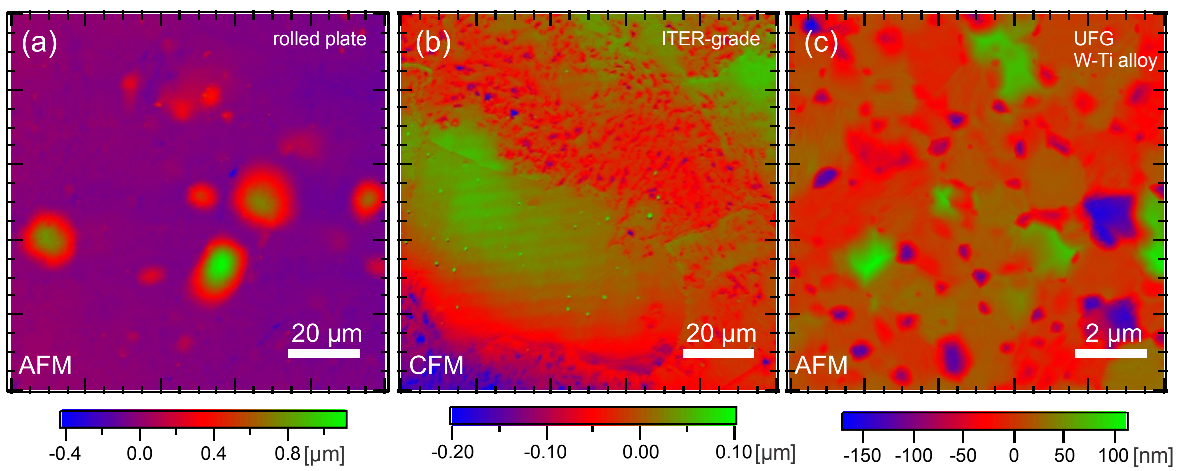
- W-Ti alloy developed at Univ. of Utah (Zak Fang)
- Microstructure offers possible improved strength and resistance to neutron damage
- **Response to plasma unknown**
- Collaboration with UCSD for high-flux deuterium plasma exposure (PISCES-A)

Use high-energy planetary ball milling: W 10-30 nm dia.
Compaction by uni-axial press and sintering at relatively low T under H_2
Further consolidation by rapid heating

UFG W Exhibits Improved Resistance to Surface Modification, With Modestly Increased D Retention

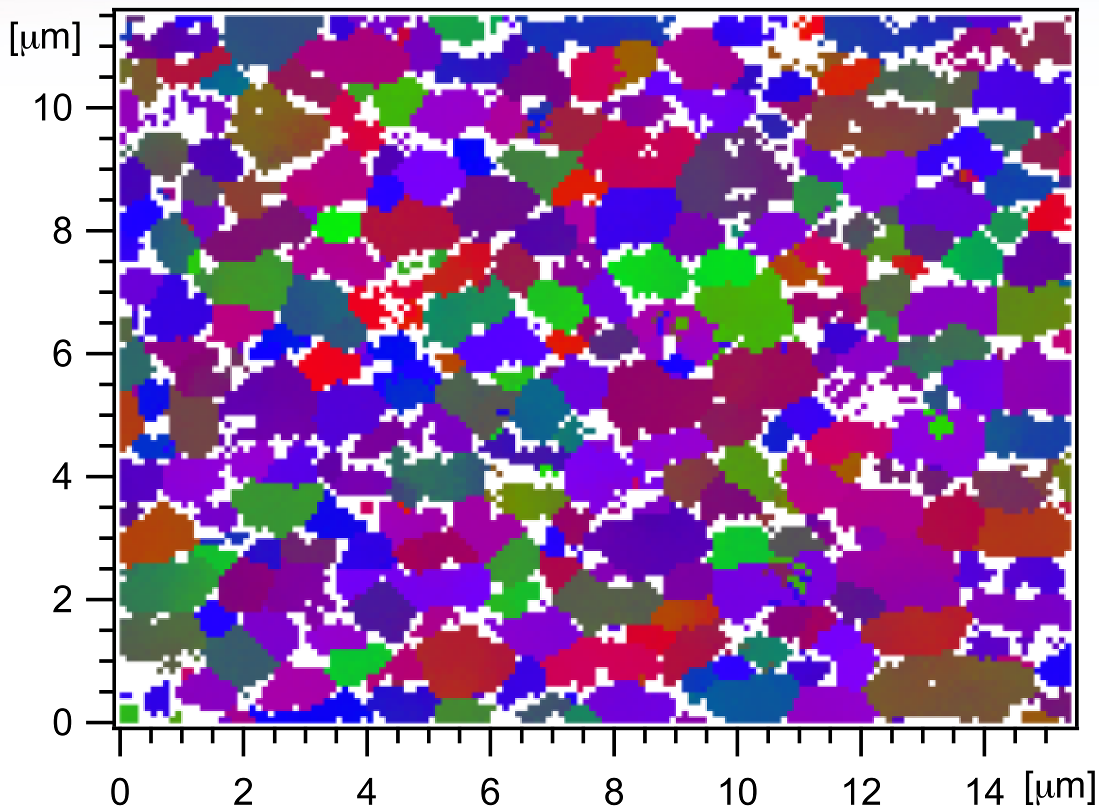


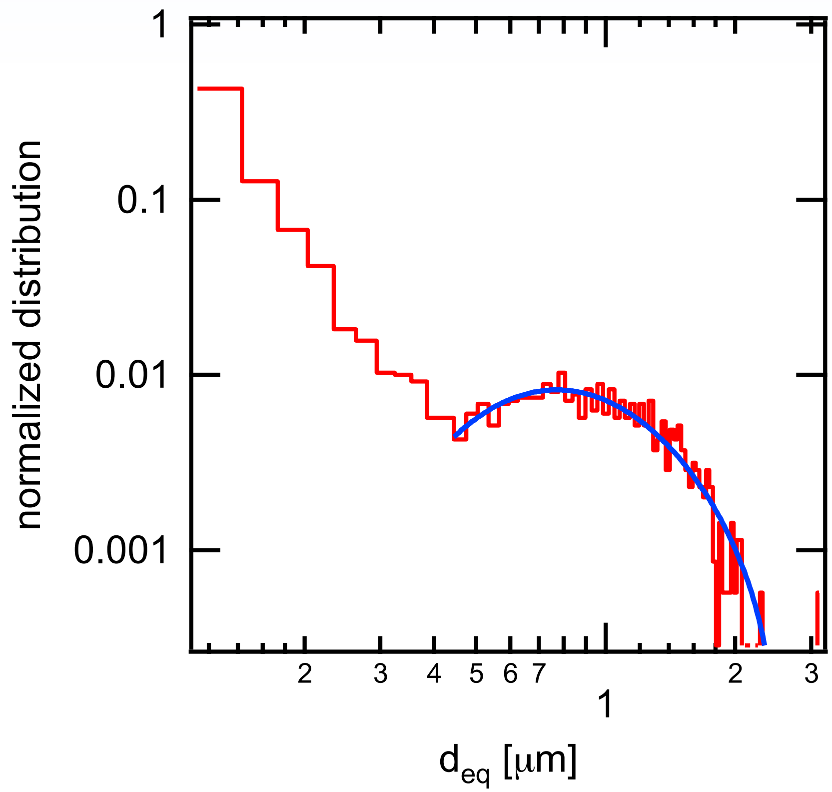
- Retention ~ 3 x higher than reference polycrystalline W
- Within typical range of variability for W grades
- Surface modification negligible

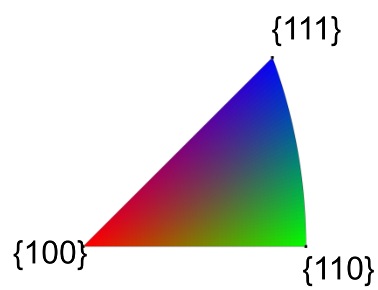


R. D. Kolasinski, D. A. Buchenauer, R. P. Doerner, et al., J. Vac. Sci. Technol. (submitted).

EBSD of UFG W Exhibits Random Grain Orientation With Grain Size Distribution Peaking < 1 μm

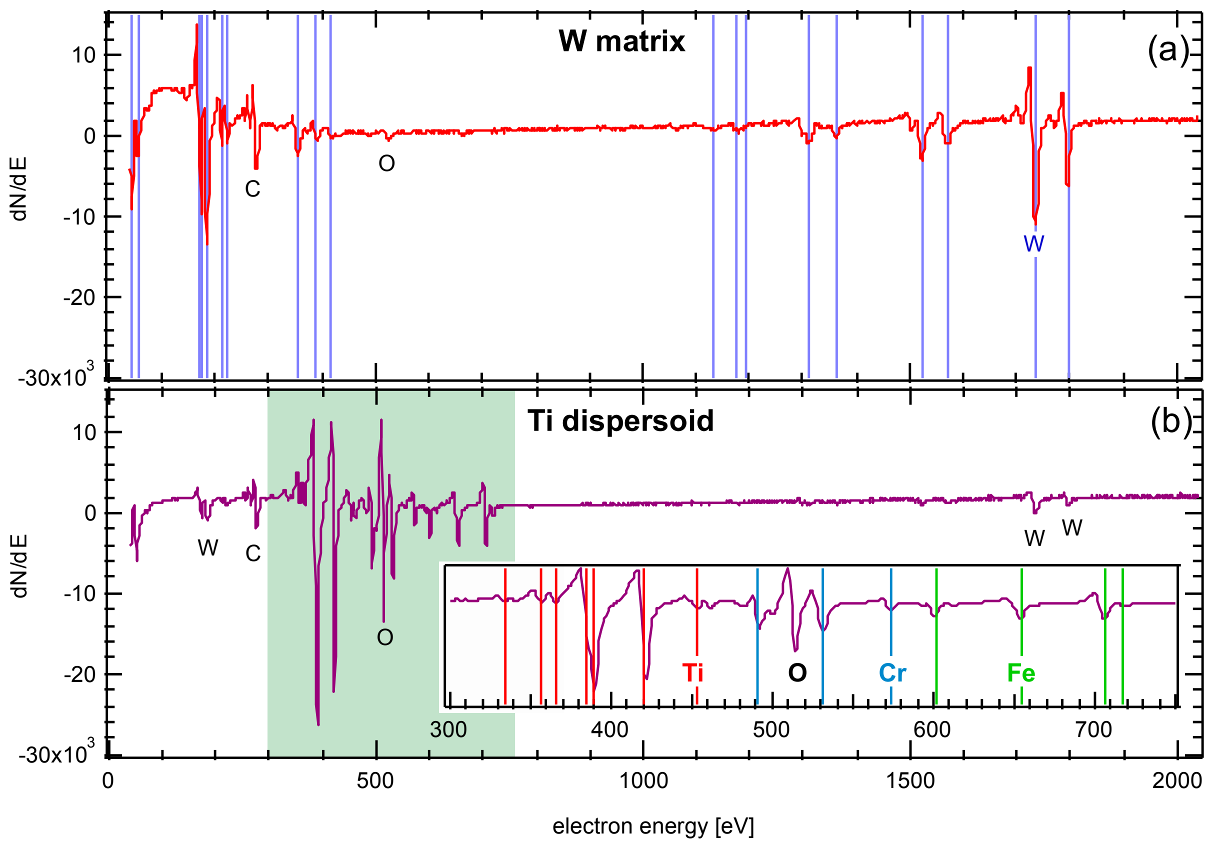






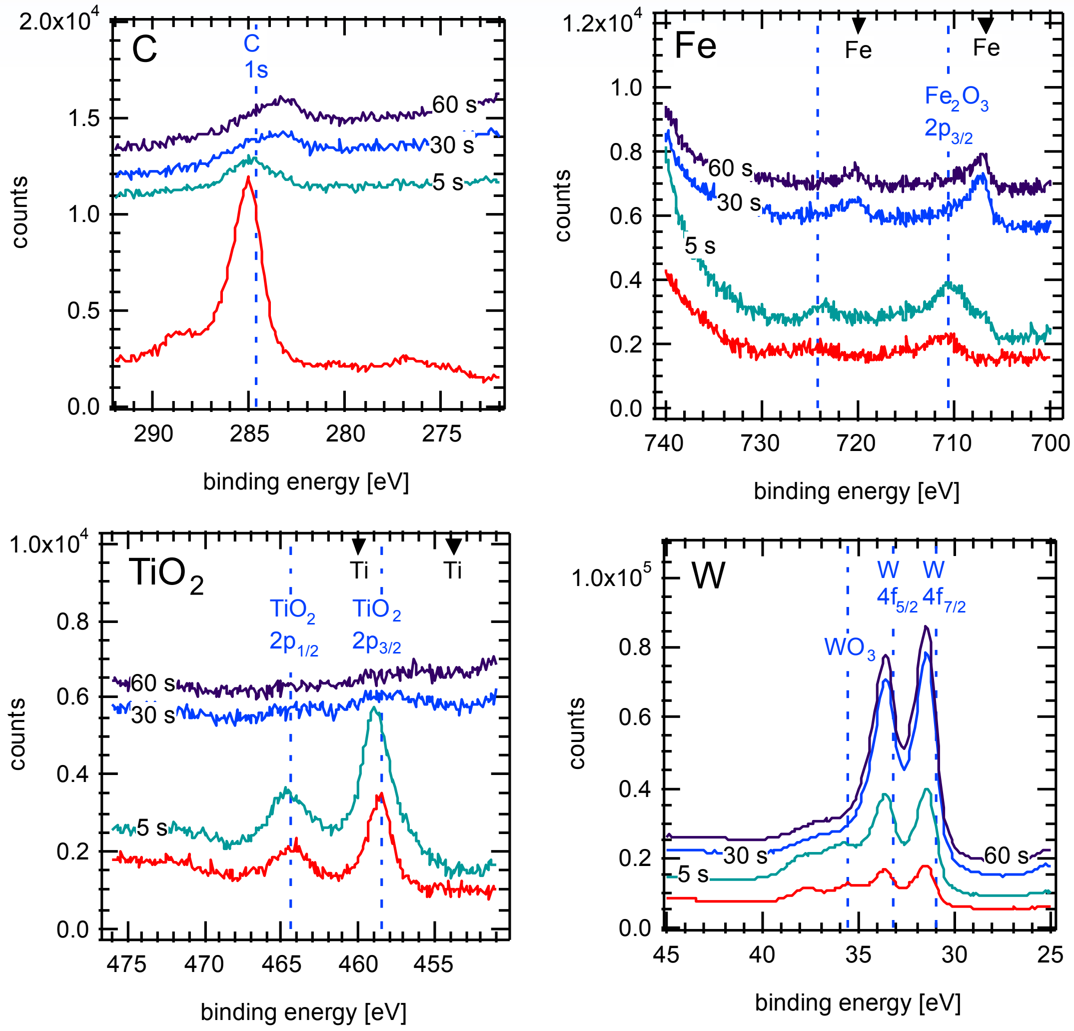
EBSD Map performed at Shizuoka University
Map obtained in 0.1 μm steps

Auger Electron Spectroscopy Indicates the Presence of Oxygen in the Ti Dispersoids



Surface prepared by sputter cleaning

XPS Shift Confirms TiO_2 and W Phases

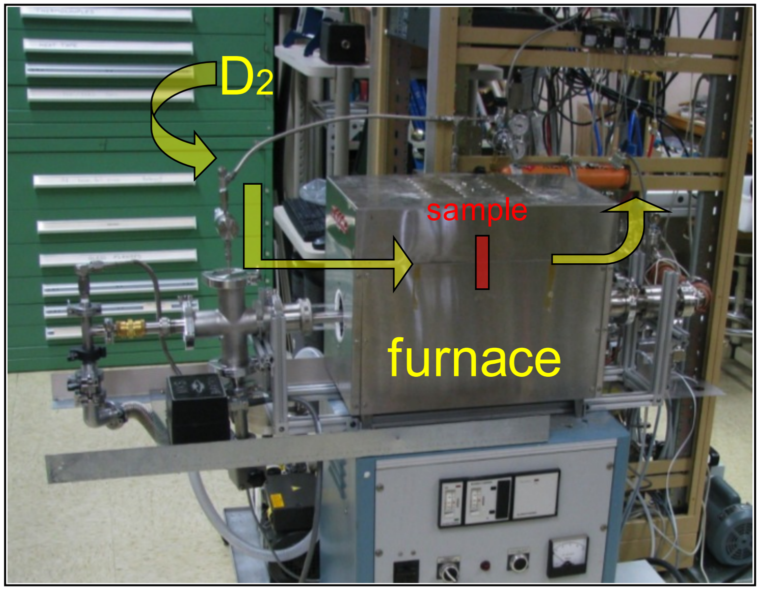


XPS performed at Shizuoka University
Sputtering time is seconds are noted

Permeation Experiments at Sandia California

- **Deuterium gas driven permeation capabilities presently in use at SNL**
  - 1st generation (150 < T < 500 °C) used stainless steel construction (VCR seals), evacuated quartz outer tube to reduce D_2 bypass, and low flow to prevent surface contamination

Materials studied: stainless steels, steel alloys, welds, aluminum alloys, nickel, AM steel

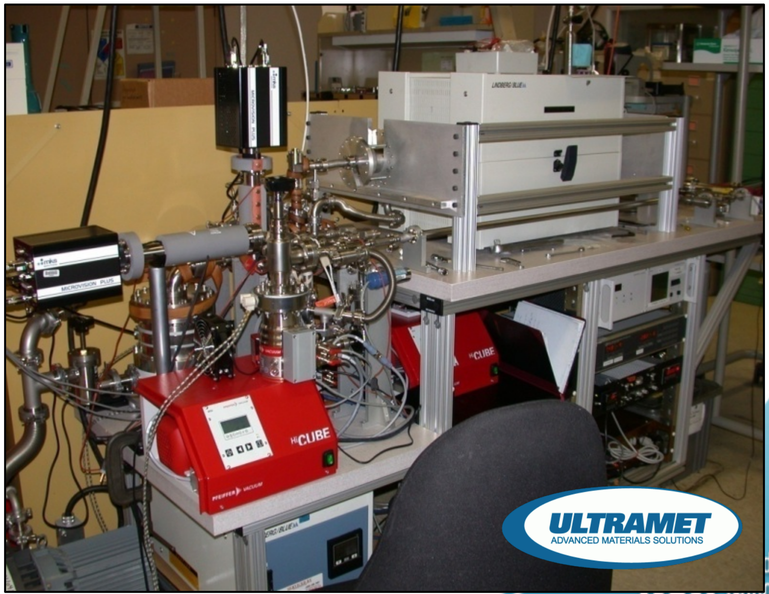


1st Generation System

  - 2nd generation (50 < T < 1150 °C) uses Al_2O_3 construction and soft, pressure loaded seals for brittle specimens (funded by “Work For Others” program to measure SiC permeation barriers for fusion blankets)

Materials studied: stainless steels, SiC, tungsten

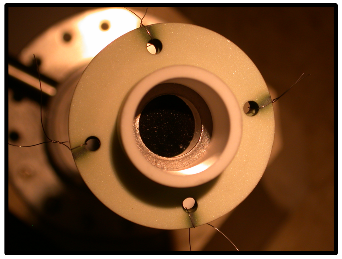
$P_{SiC} < 10^{-12}$ mol H^2 m^{-1} s^{-1} $MPa^{-0.5}$



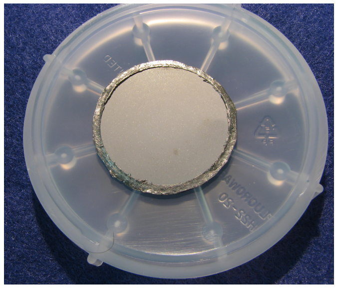
2nd Generation System

Permeation of Tungsten and Tungsten Alloys

- **Successful sealing to tungsten materials in the HT system was achieved by applying pressure above W DBTT (≥ 500 °C) ➔ Increased utilization of HT system**
  - ITER grade tungsten (October – September 2014) – PHENIX
  - High purity tungsten foil (May 2015)
  - Fe^{3+} implanted ITER tungsten (June 2015) – PHENIX
  - Dispersoid strengthened ultra-fine grained (UFG) tungsten (July 2015)
    - Collaboration with Zak Fang (University of Utah)
    - Repeat of UFG tungsten (August 2015)
  - Several calibrations were needed to understand data.

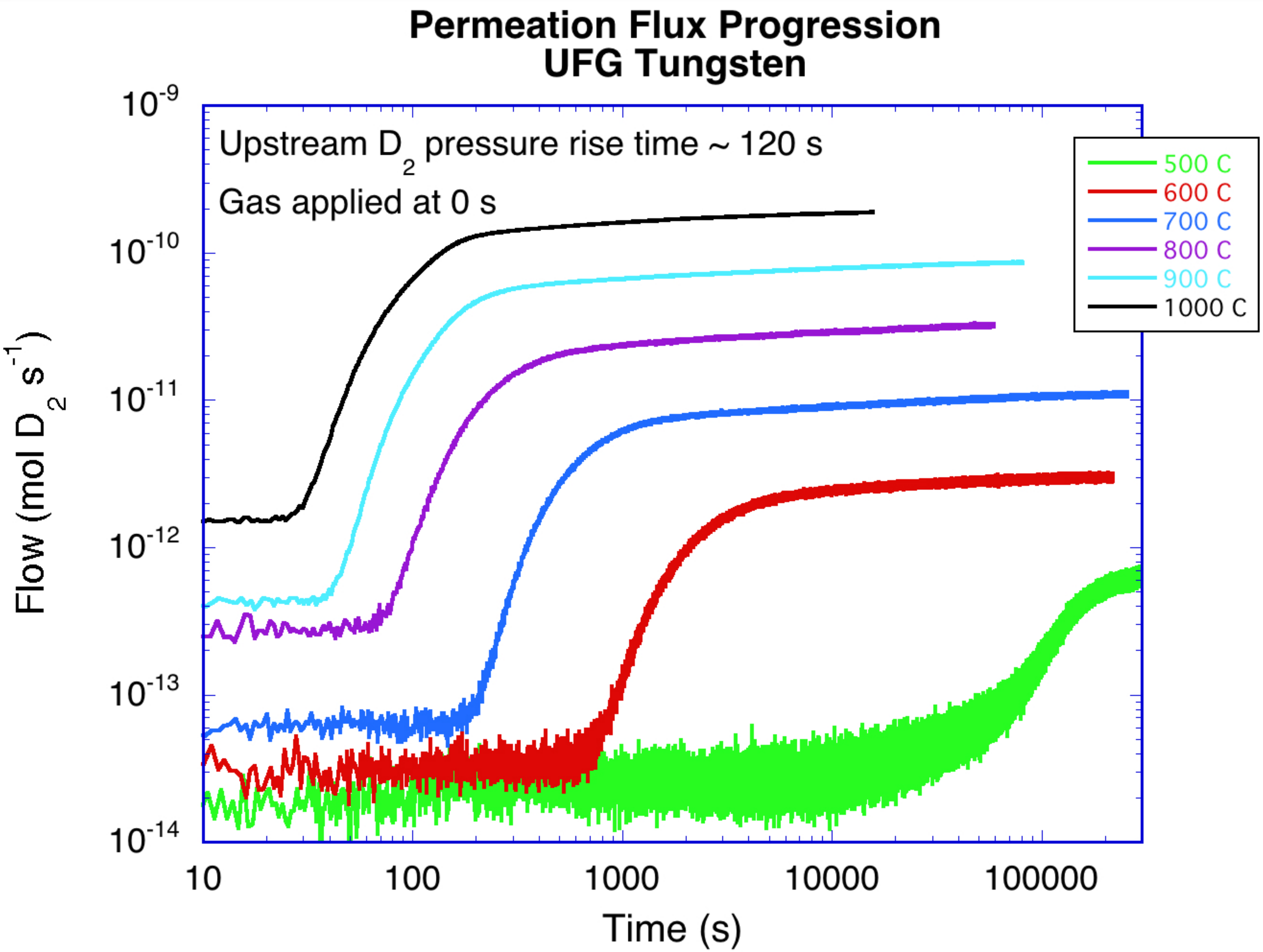


- **Surface analysis to examine the samples for oxides and carbides**
  - Auger Electron Spectroscopy
  - X-Ray Fluorescence

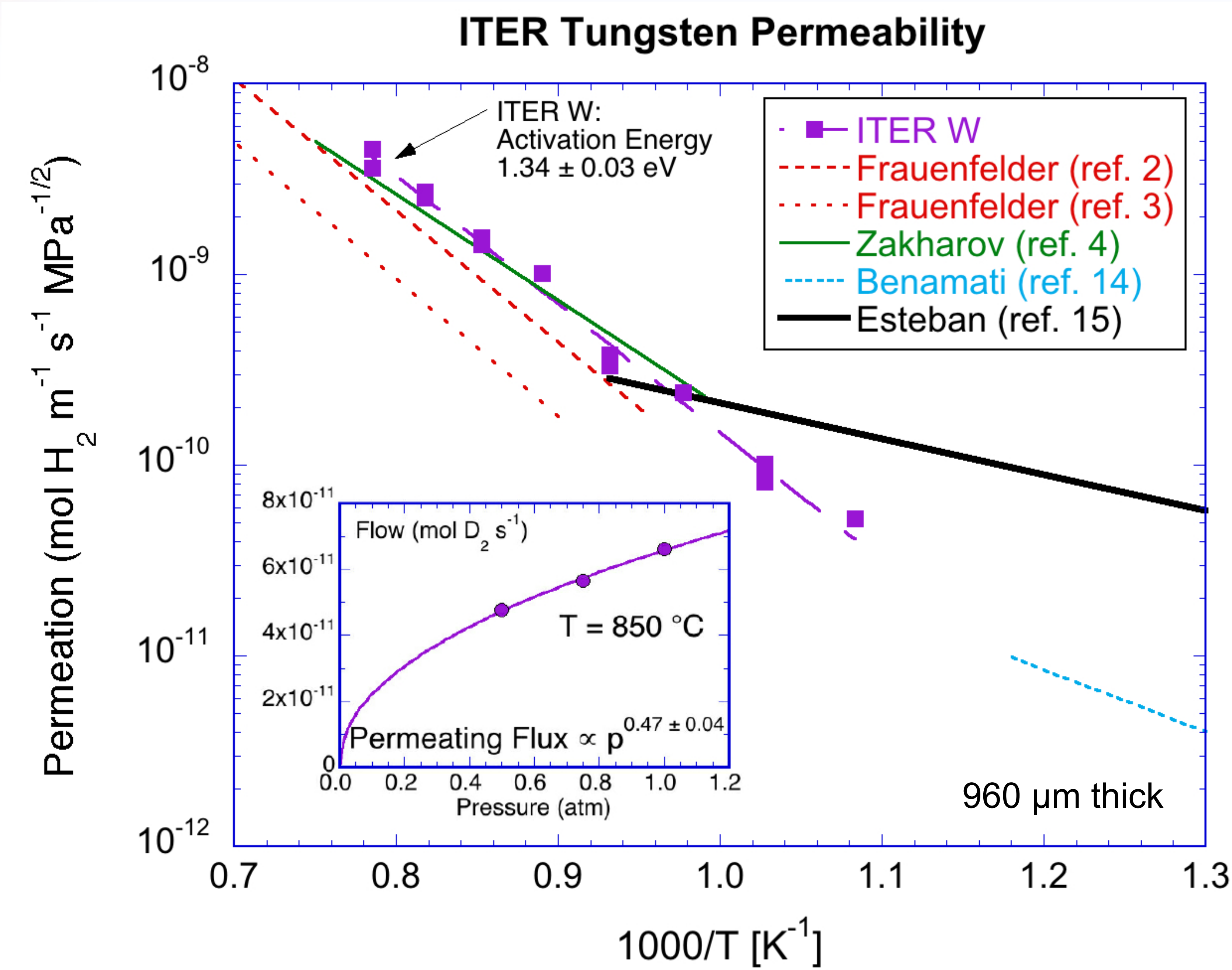


- **Design of HT system and tungsten results presented at ISFNT-12**
  - D. Buchenauer, et al., submitted to Fusion Engineering and Design

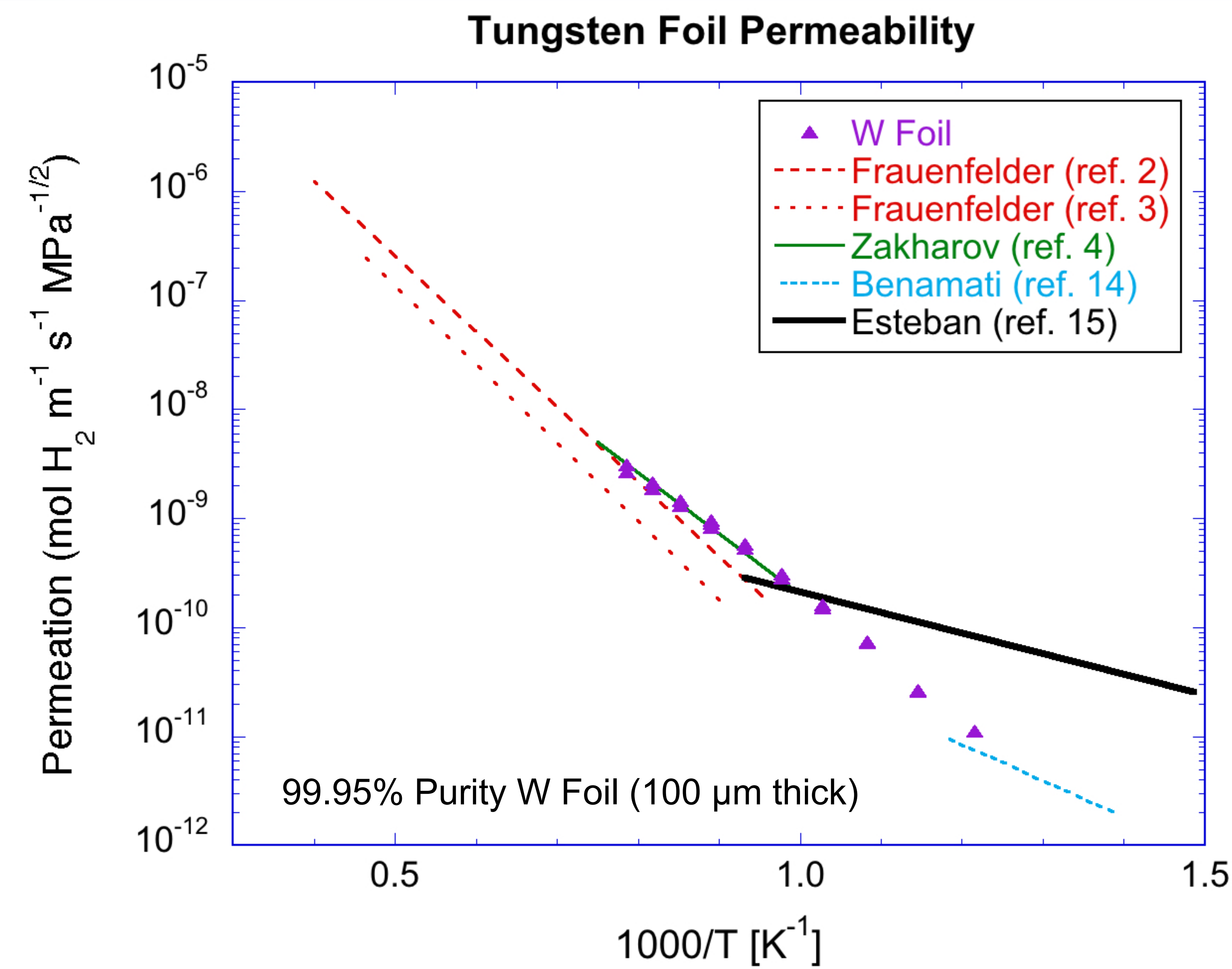
Example of Permeation Curves (Going Up in T)



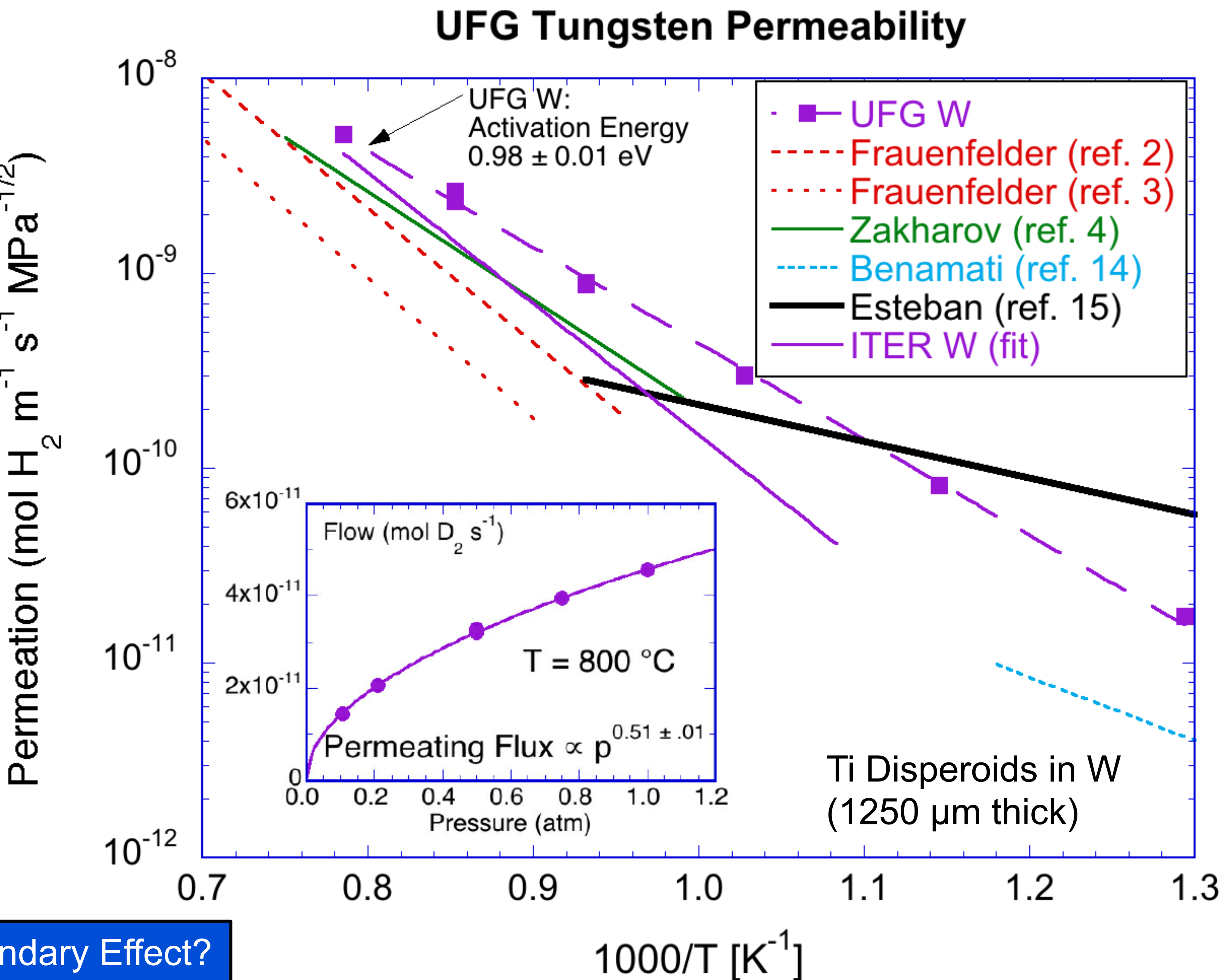
Gas Permeation Through ITER Tungsten



Thin High Purity Tungsten Foil Used to Compare With Literature Values



Higher Permeability Observed in Dispersoid-Strengthened Ultra Fine-Grained (UFG) Tungsten



Grain Boundary Effect?
Pipe Diffusion?

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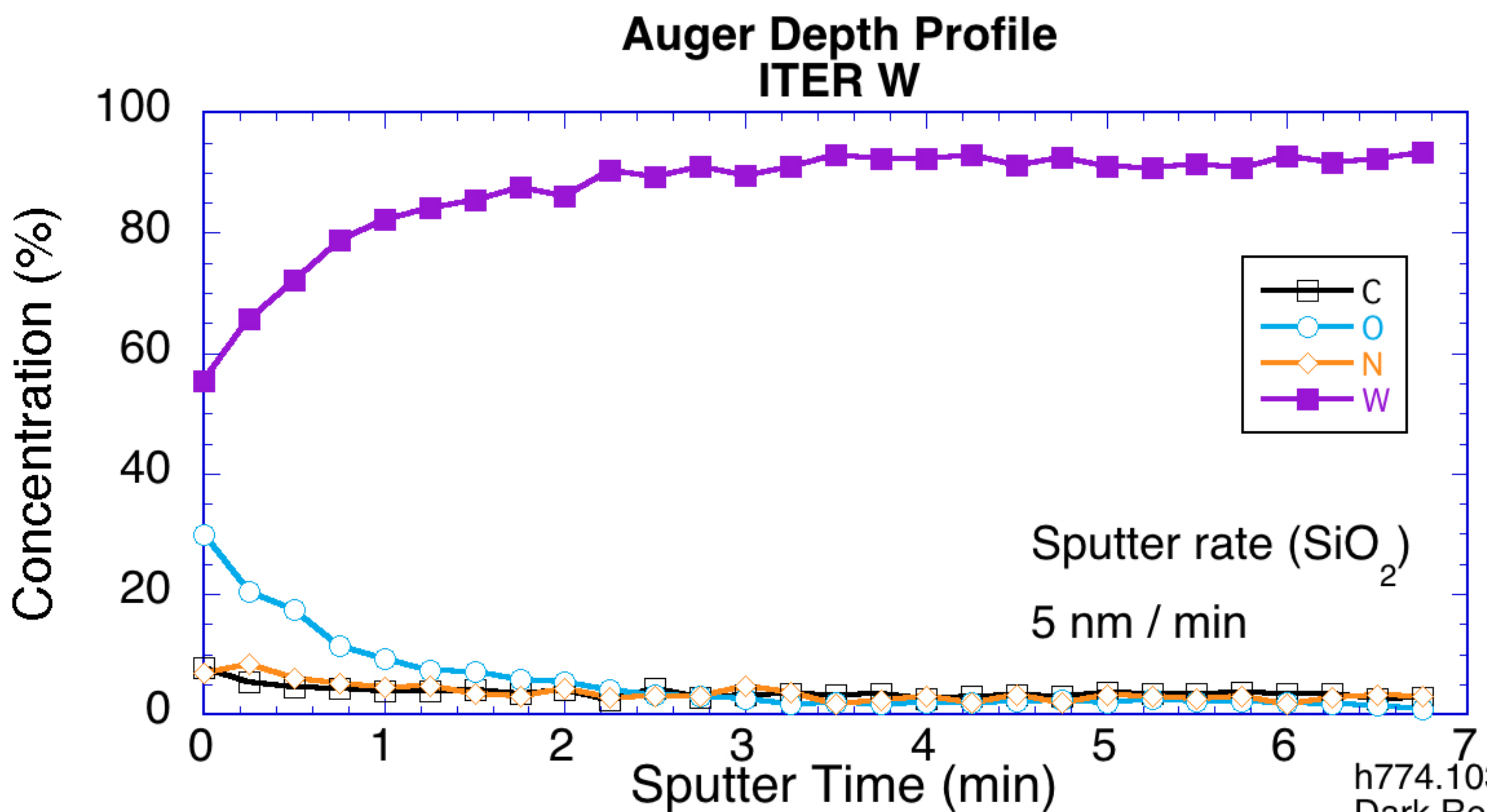
Auger Electron Spectroscopy (AES) Depth Profiling



h774.103_1, Point 1
Dark Region

960 μm thick

Note: Detection limit is a few atomic percent

Summary

- Measurements of deuterium retention and surface modification by precipitation show a dependence on the tungsten microstructure. Bubble formation depends on grain orientation and is suppressed in the dispersoid-strengthened ultra-fine grained tungsten.
- A model of crack expansion by gas pressure agrees well with bubble size distributions obtained by atomic force microscopy (ITER grade W). Estimates of the deuterium retained compared with thermal desorption indicate most deuterium is trapped at other defects.
- The first hydrogen compatibility tests of UFG tungsten indicate moderately higher retention. The use of different dispersoid materials is now under consideration.
- The measured permeation of ITER tungsten compares well with commonly cited literature values over the temperature range 500 – 1000 °C and the activation energy is similar to that obtained by Frauenfelder and Zakharov (although Estebahn's activation energy is much lower).
- Dispersoid strengthened UFG tungsten shows higher permeation and a somewhat lower activation energy. At the temperatures expected in a reactor divertor, however, the difference in permeation is negligible.

Permeation references in following slide

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Permeation References

[1] D. Buchenauer, R. Kolasinski, M. Shimada, D. Donovan, D. Youchison, and B. Merrill, Fus. Eng. Design 89 (2014) 1014..
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[14] G. Benamati, E. Serra, and C.H. Wu, J. Nuc. Mater. 283-287 (2000) 1033.
[15] G.A. Esteban, A. Perujo, L.A. Sedano, and K. Douglas, J. Nuc. Mater. 295 (2001) 49.
[20] A. Manhard, S. Kapser, and L. Gao, J. Nuc. Mat. 463 (2015) 1057.
[23] E.A. Aitken, H.C. Brassfie, P.K. Conn, E.C. Dudersta, and R.E. Fryxell, Trans. Metall. Soc. of AME, 239 (1967) 1565..

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