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PHENIX Research at SNL

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Permeation Experiments on Tungsten Materials
Surface Modification and Retention of Tungsten Materials by Plasma Exposure
Sample Preparation for RB19J Sub-Capsules

PHENIX Task 3 Workshop, Sapporo, Japan, September 21-22, 2015

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NNSA National Nuclear Security Administration

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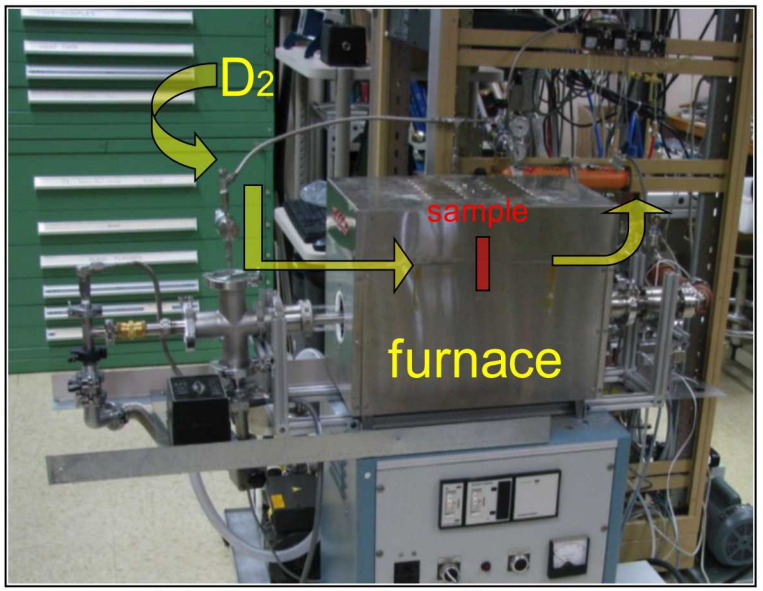
Permeation Experiments at Sandia California

- **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

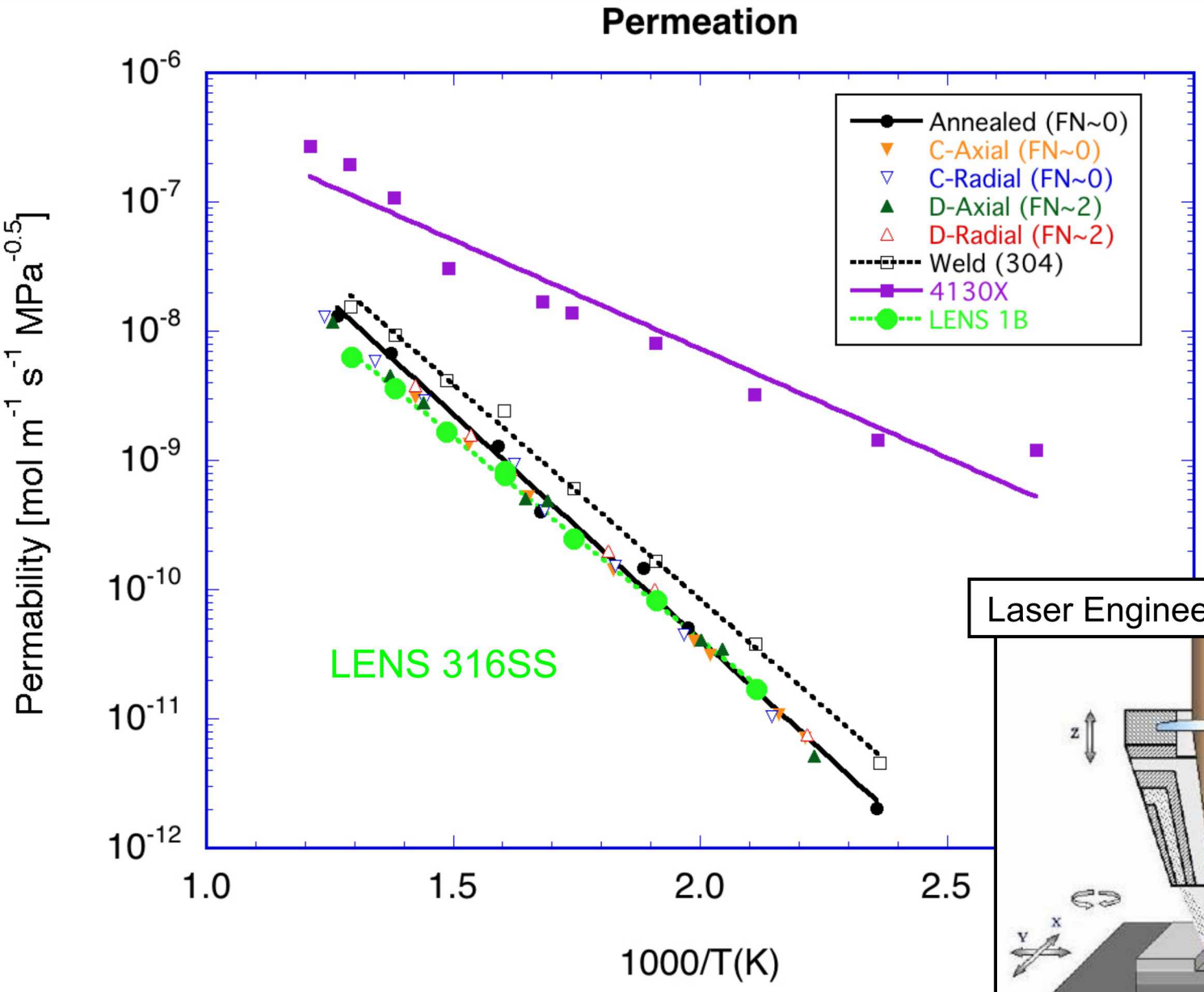
  - 2nd generation (50 < T < 1150 °C) uses Al_2O_3 construction and soft, pressure loaded seals for brittle specimens (funded by "Work For Others" program to measure SiC permeation barriers for fusion blankets)

Materials studied: stainless steels, SiC, tungsten

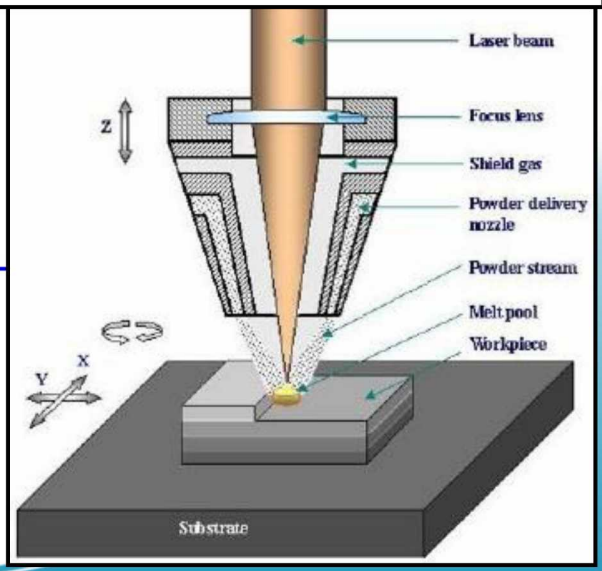
$P_{SiC} < 10^{-12}$ mol H^2 m^{-1} s^{-1} $MPa^{-0.5}$

2nd Generation System

Low Temperature Permeation System Studying Steels Fabricated by Additive Manufacturing

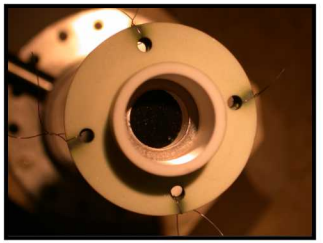


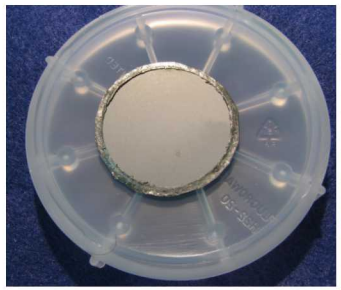
Laser Engineered Net Shaping



Permeation of Tungsten and Tungsten Alloys

- **Successful sealing to tungsten materials in the HT system was achieve by sealing at elevated temperatures (≥ 500 °C) ➔ Increased utilization of HT system**
  - ITER grade tungsten (October – September 2014) - PHENIX
    - Professor Oya supplied ALMT sample
    - Dr. Otsuka and Professor Yamauchi's assignments
  - High purity tungsten foil (May 2015)
  - Fe^{3+} implanted ITER tungsten (June 2015)
    - Dr. Chikada's assignment
  - 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**
  - Publication: D. Buchenauer, et al., submitted to Fusion Engineering and Design

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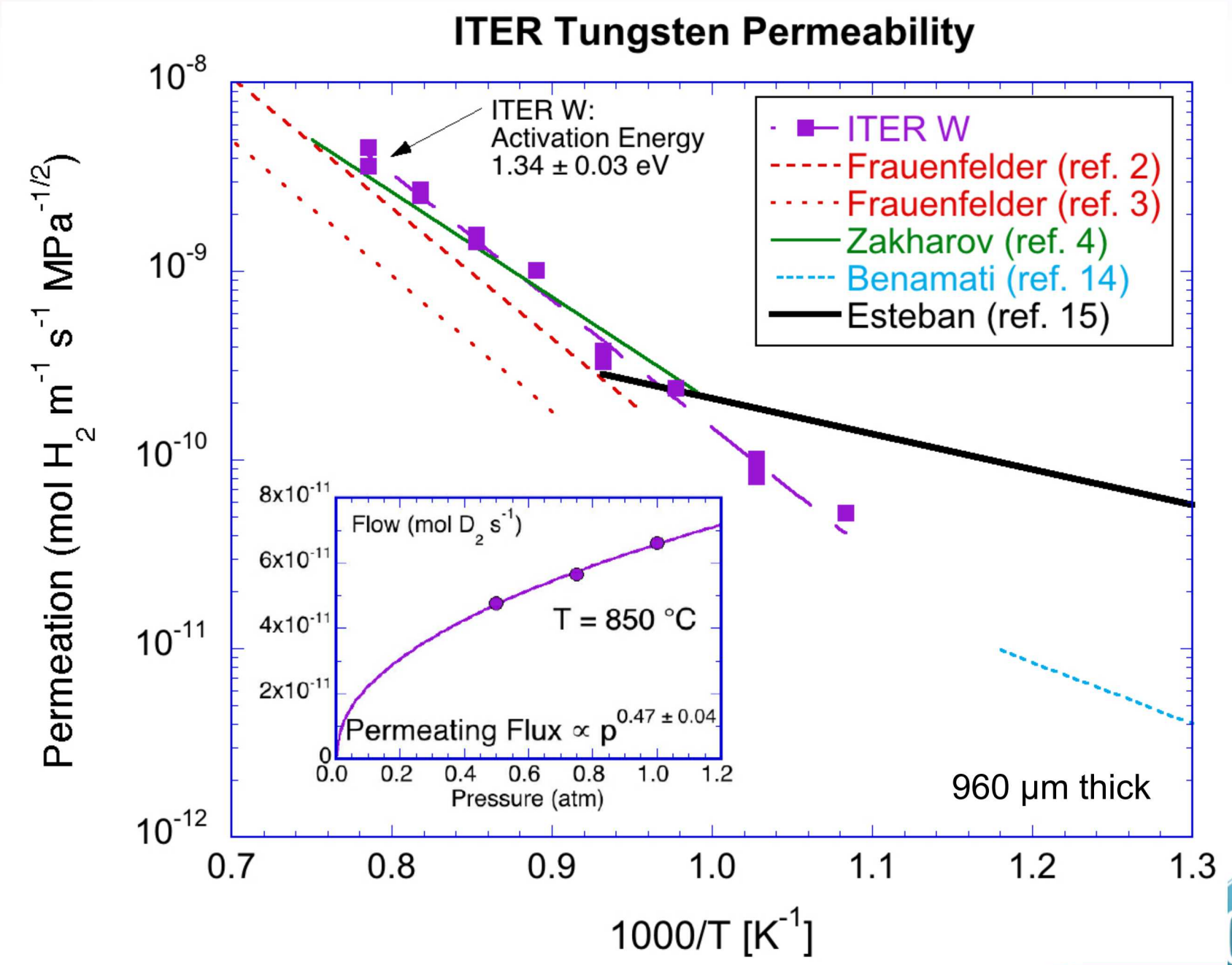
Gas Permeation Through ITER Tungsten



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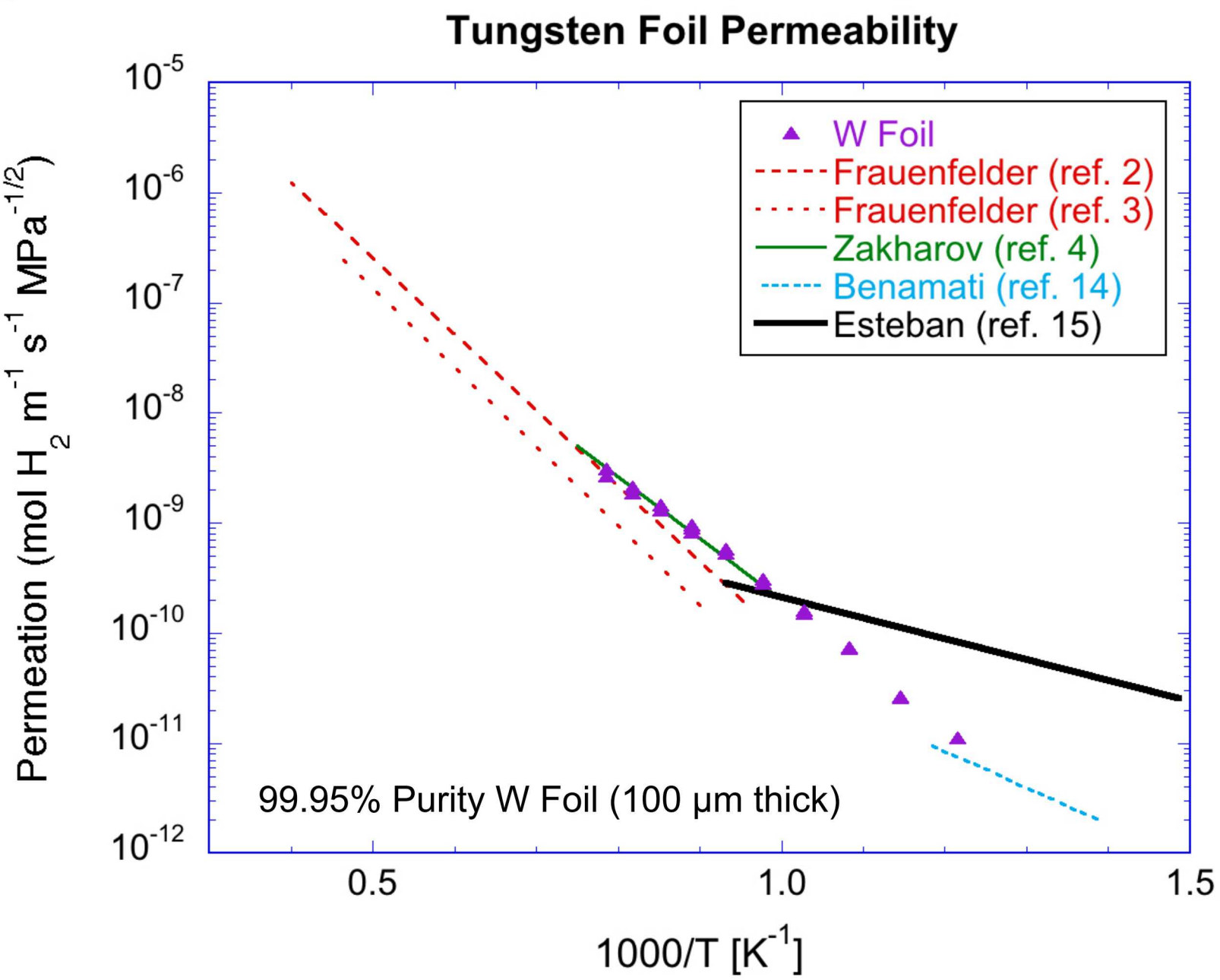
Thin High Purity Tungsten Foil Used to Compare With Literature Values



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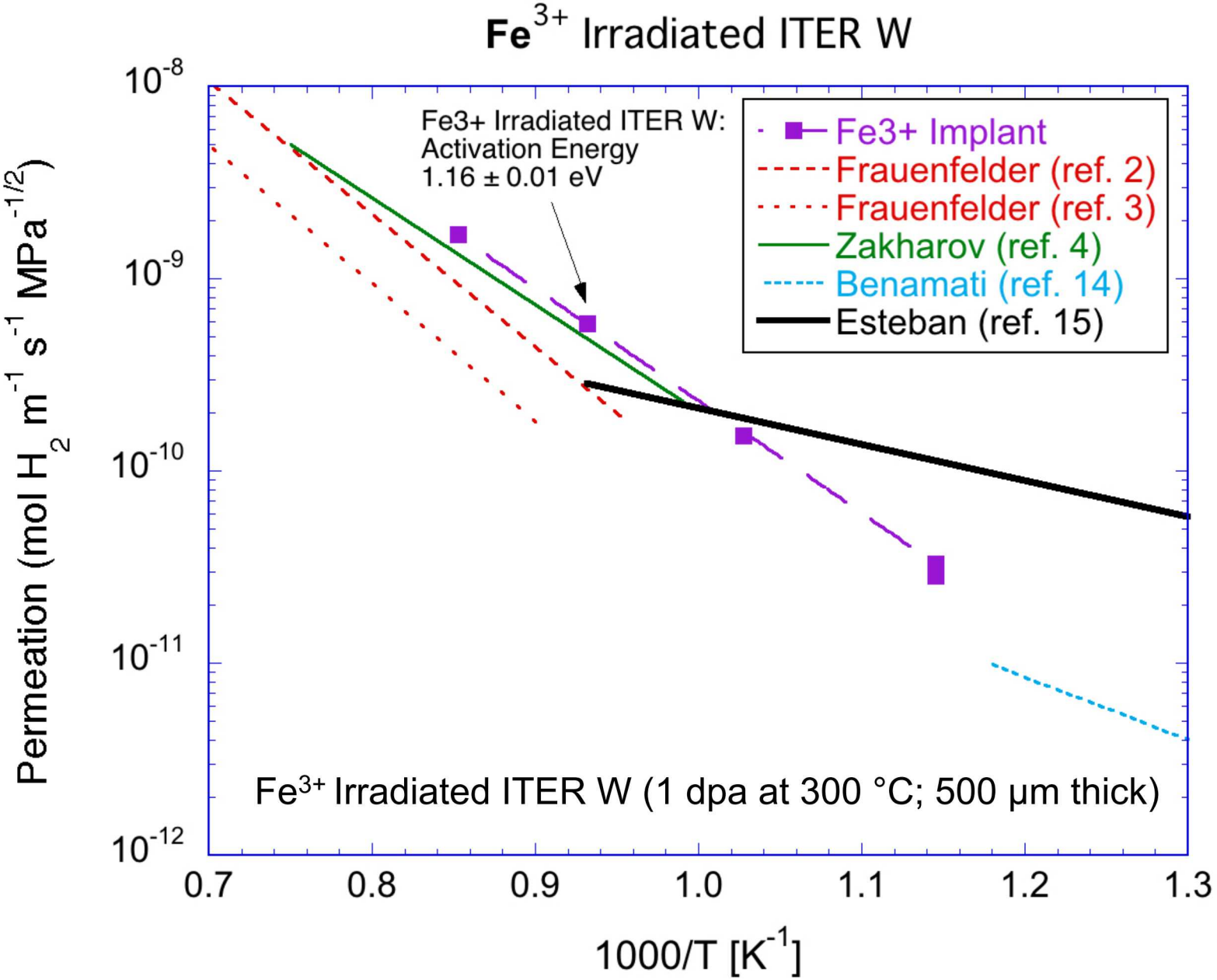
ITER Tungsten Damaged by Fe^{3+} Irradiation at RT Exhibits Permeability Similar to ITER W



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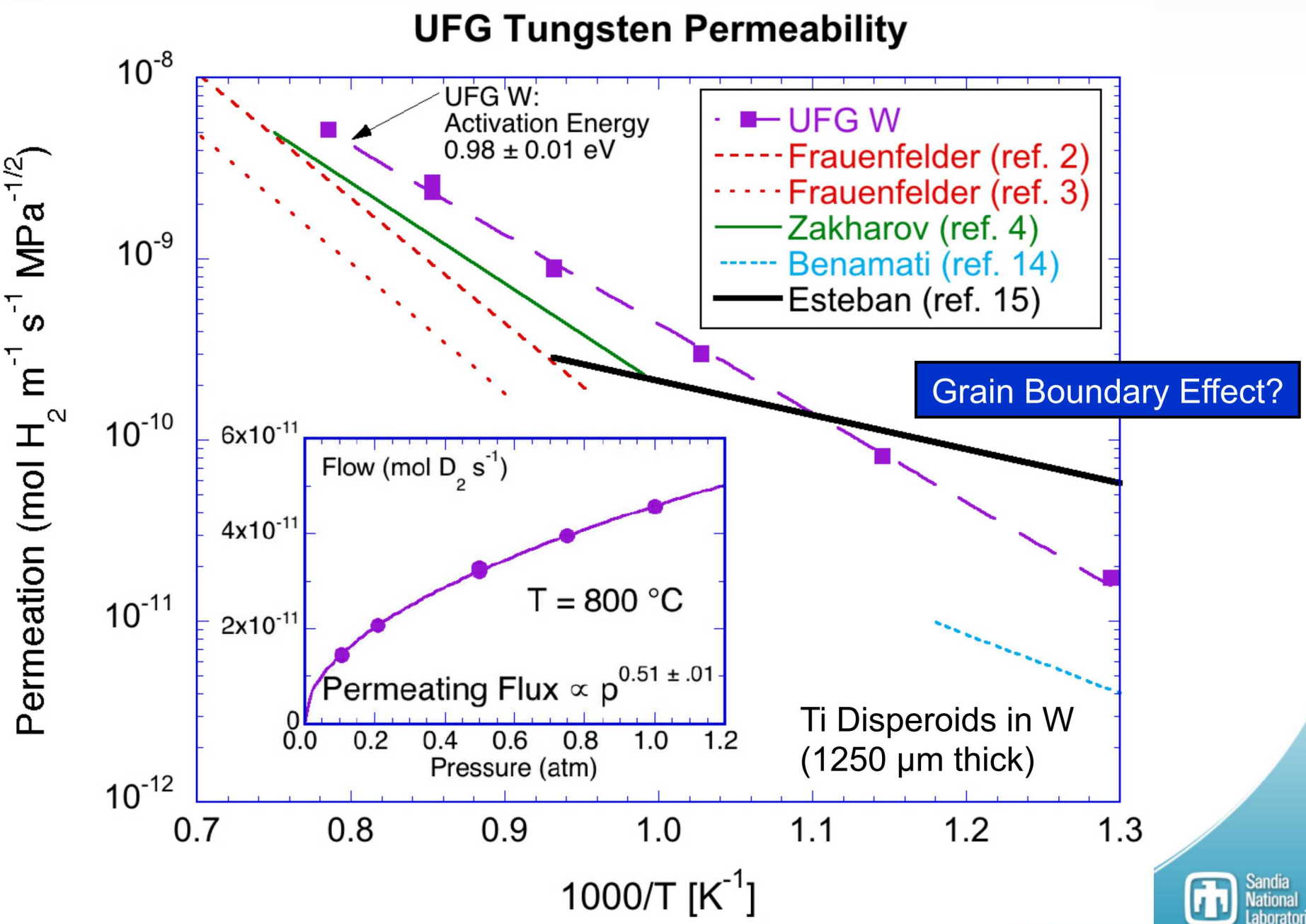
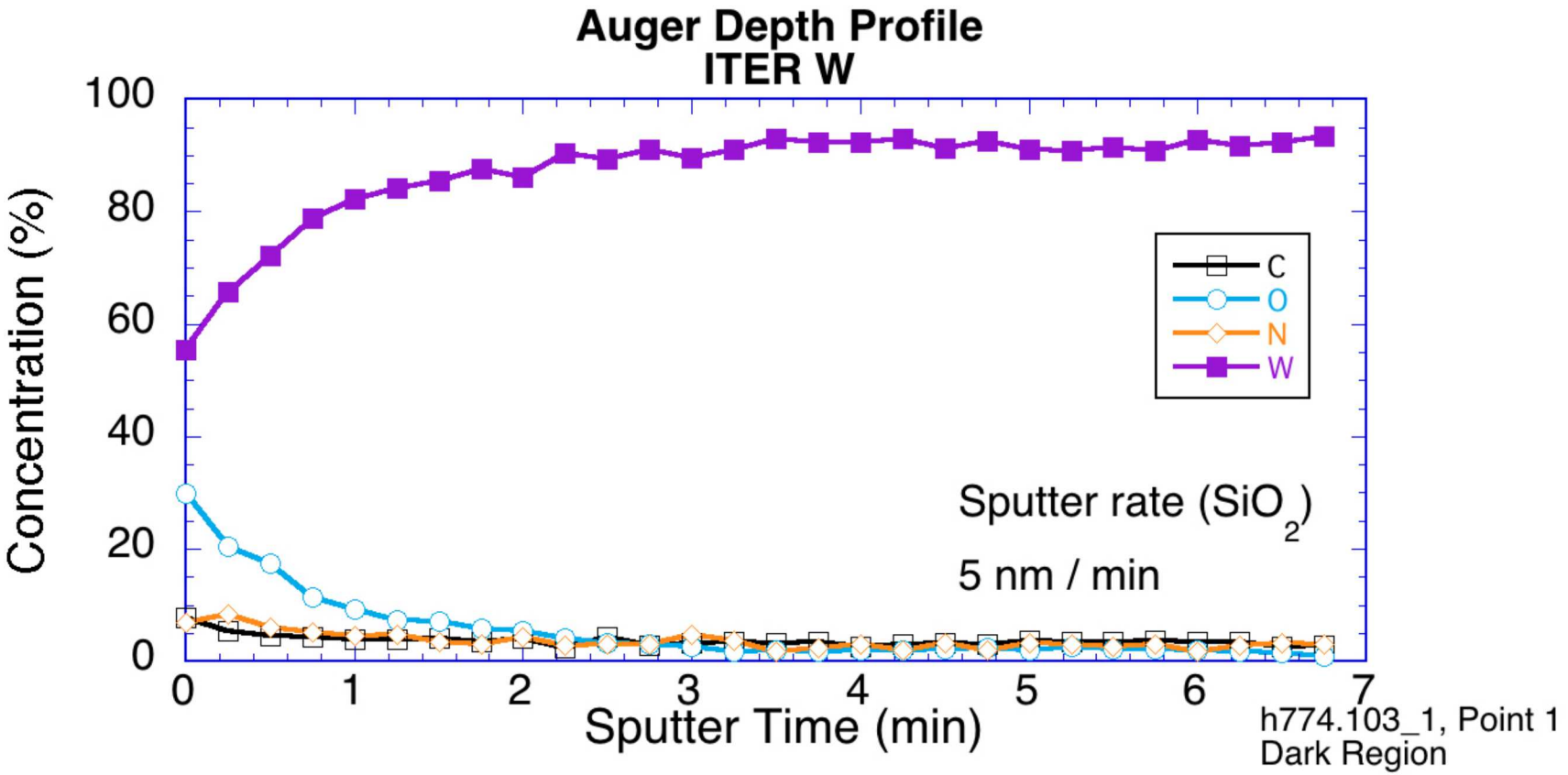
Higher Permeability Observed in Dispersoid-Strengthened Ultra Fine-Grained (UFG) Tungsten



Auger Electron Spectroscopy (AES) Depth Profiling



960 µm thick

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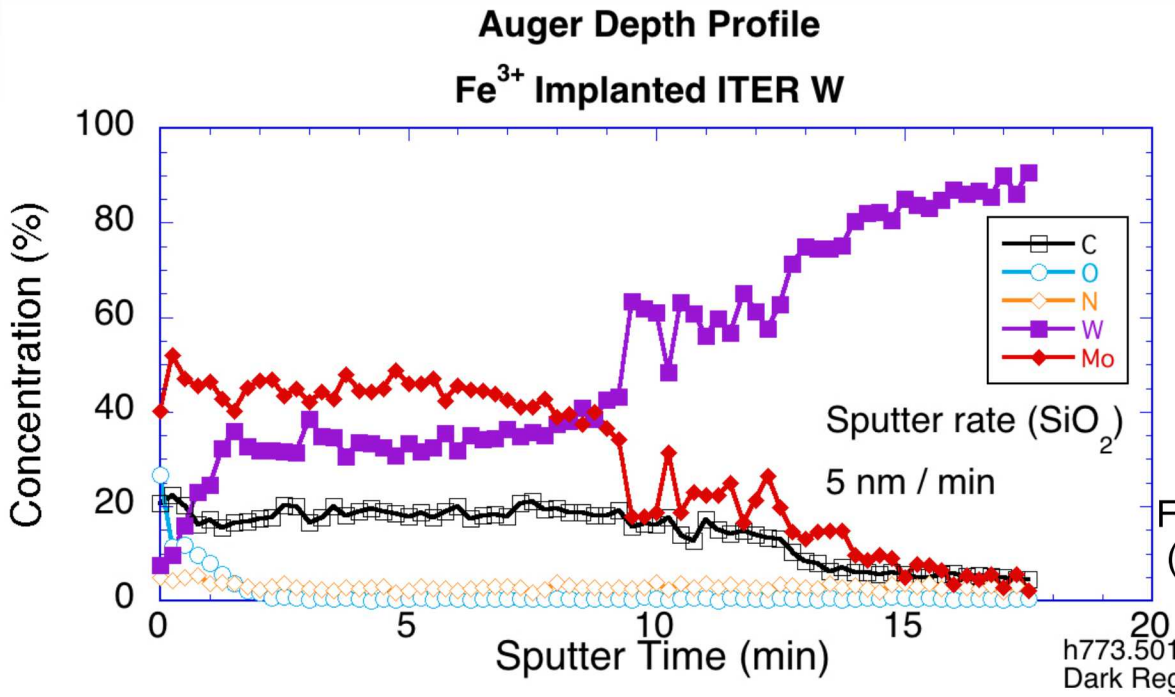
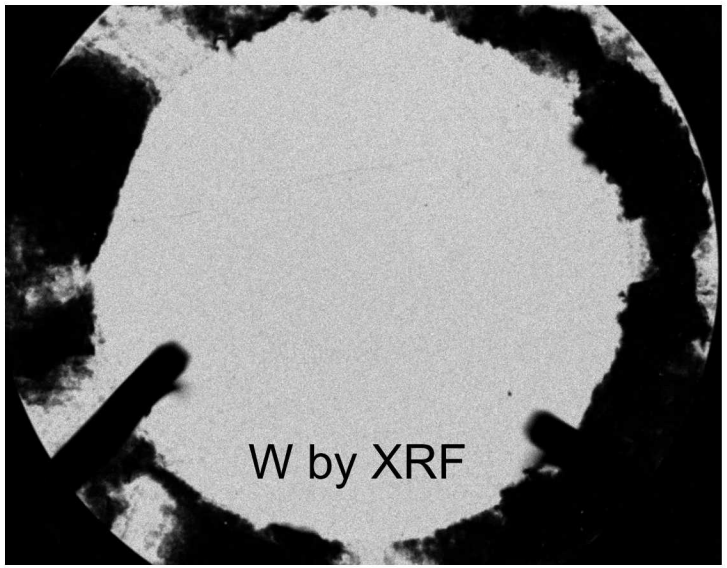
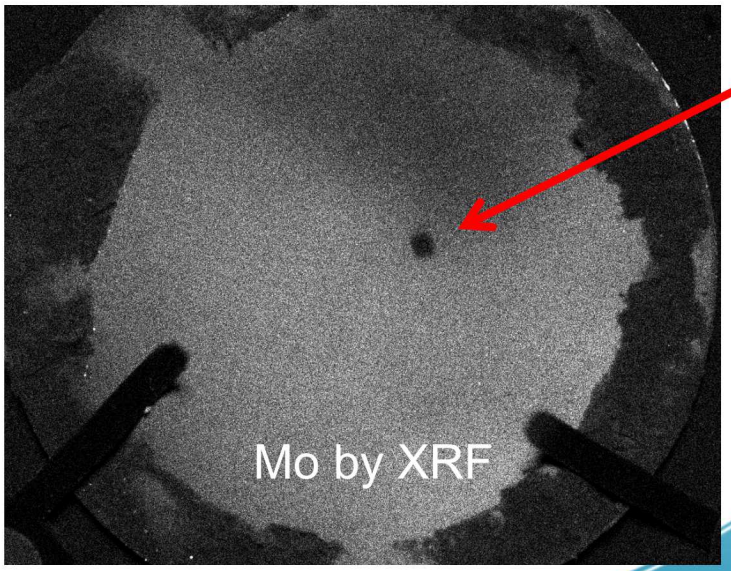
X-Ray Fluorescence Allows for Larger View of Surface Conditions



Fe^{3+} Irradiated ITER W
(1 dpa at 300 °C; 500 µm thick)





Auger Sputter Crater

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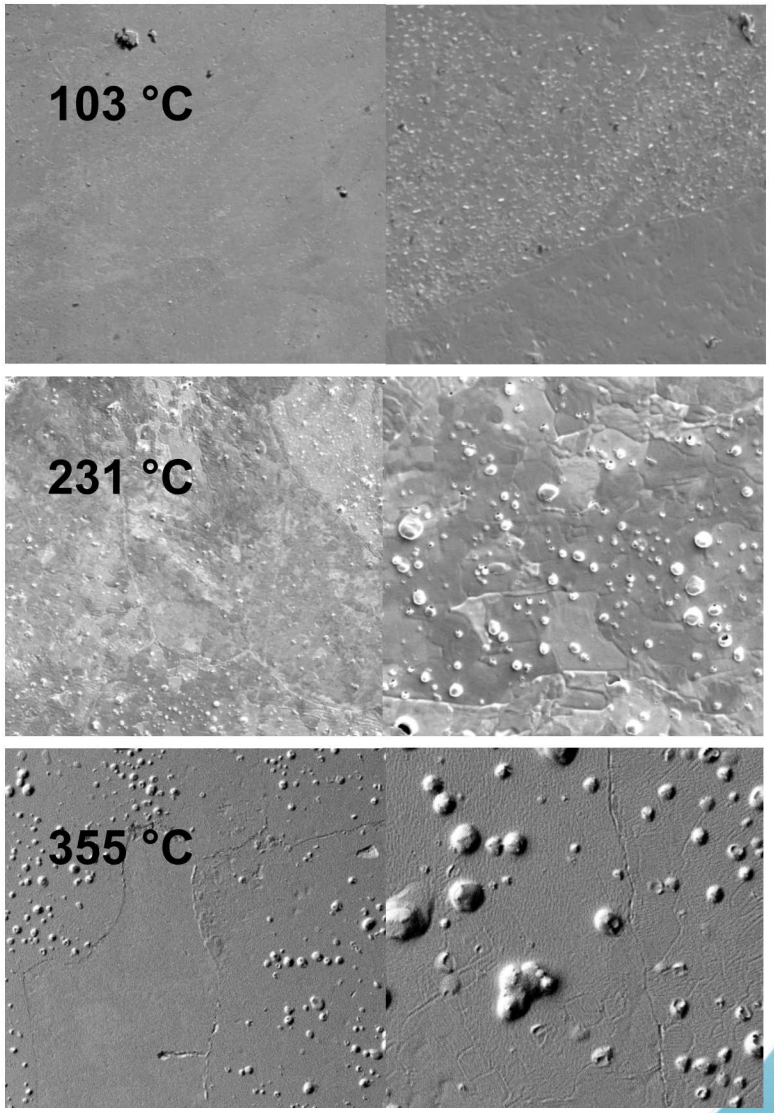
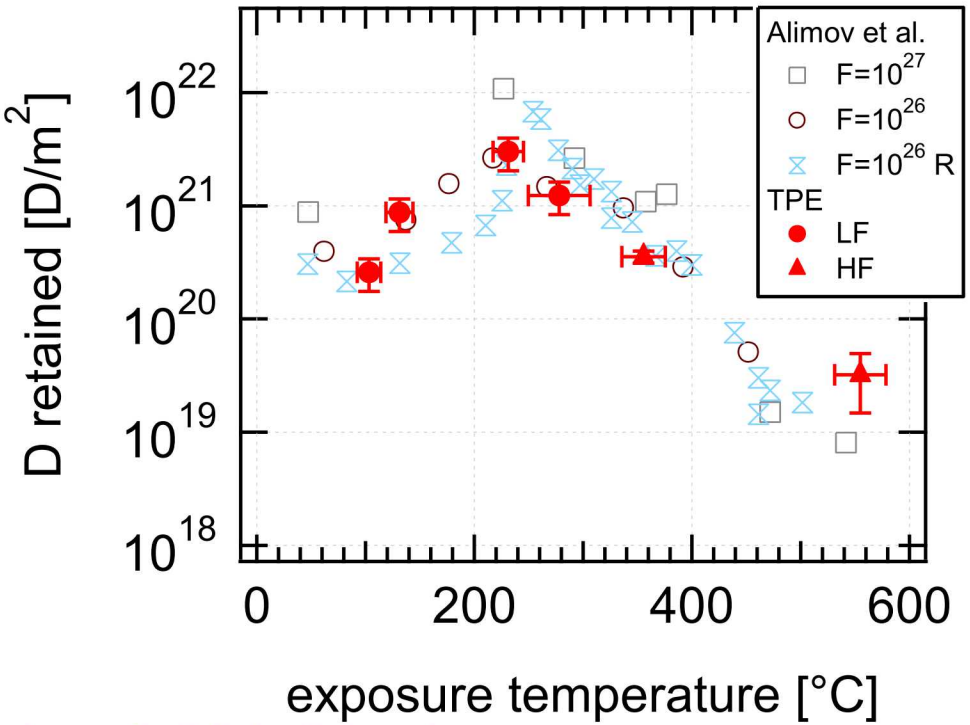
Multi-Technique Analysis of ITER W Exposed in TPE

- Surface analysis at Sandia and **Shizuoka University** (FY14 PHENIX exchange)
- Microscopy reveals near-surface H precipitates
- Density/size temp. dependence
- Growth determined by grain orientation





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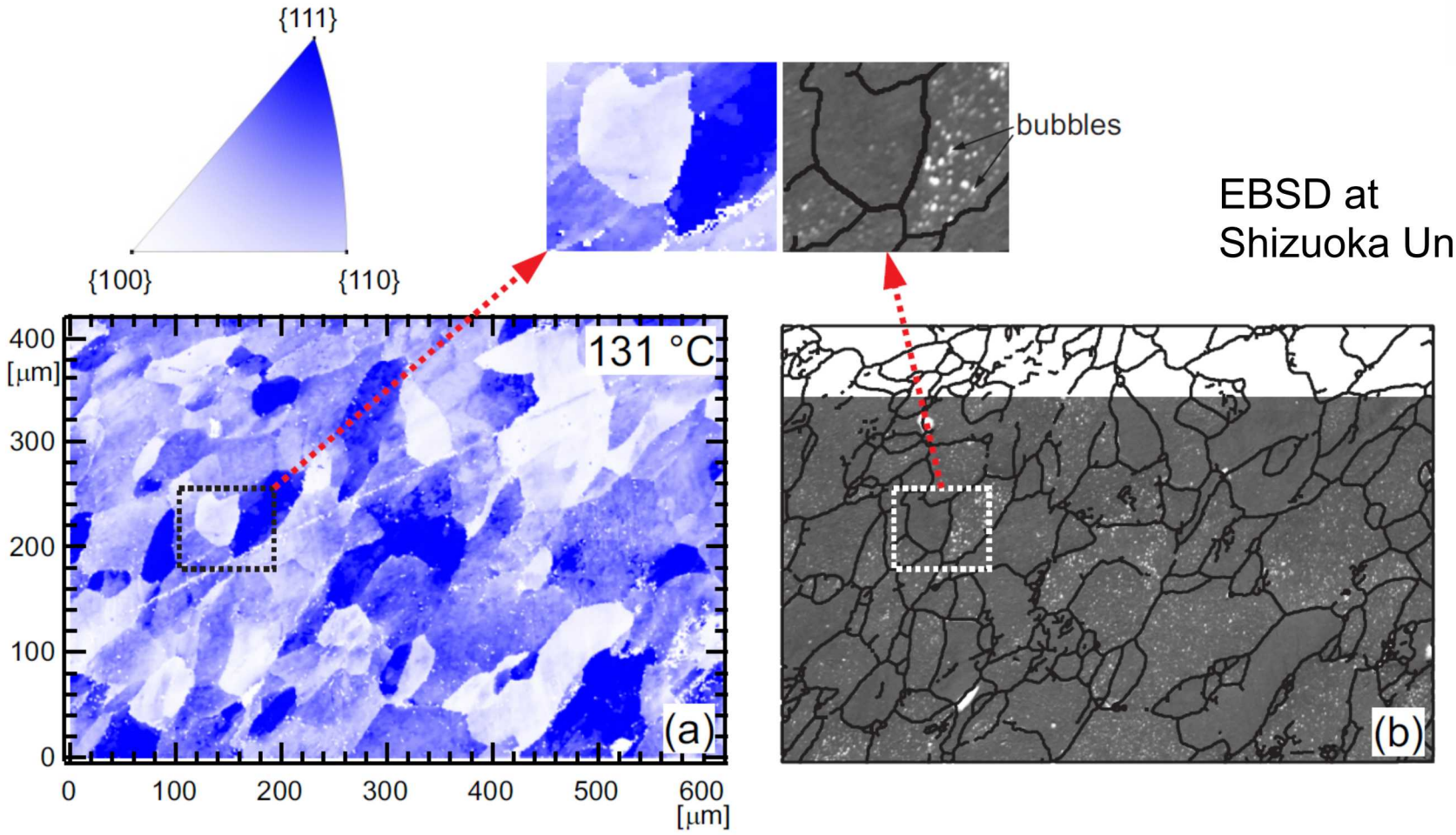
Electron Backscatter Diffraction Clarifies Orientation Dependence of Nucleation



EBSD at
Shizuoka University

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

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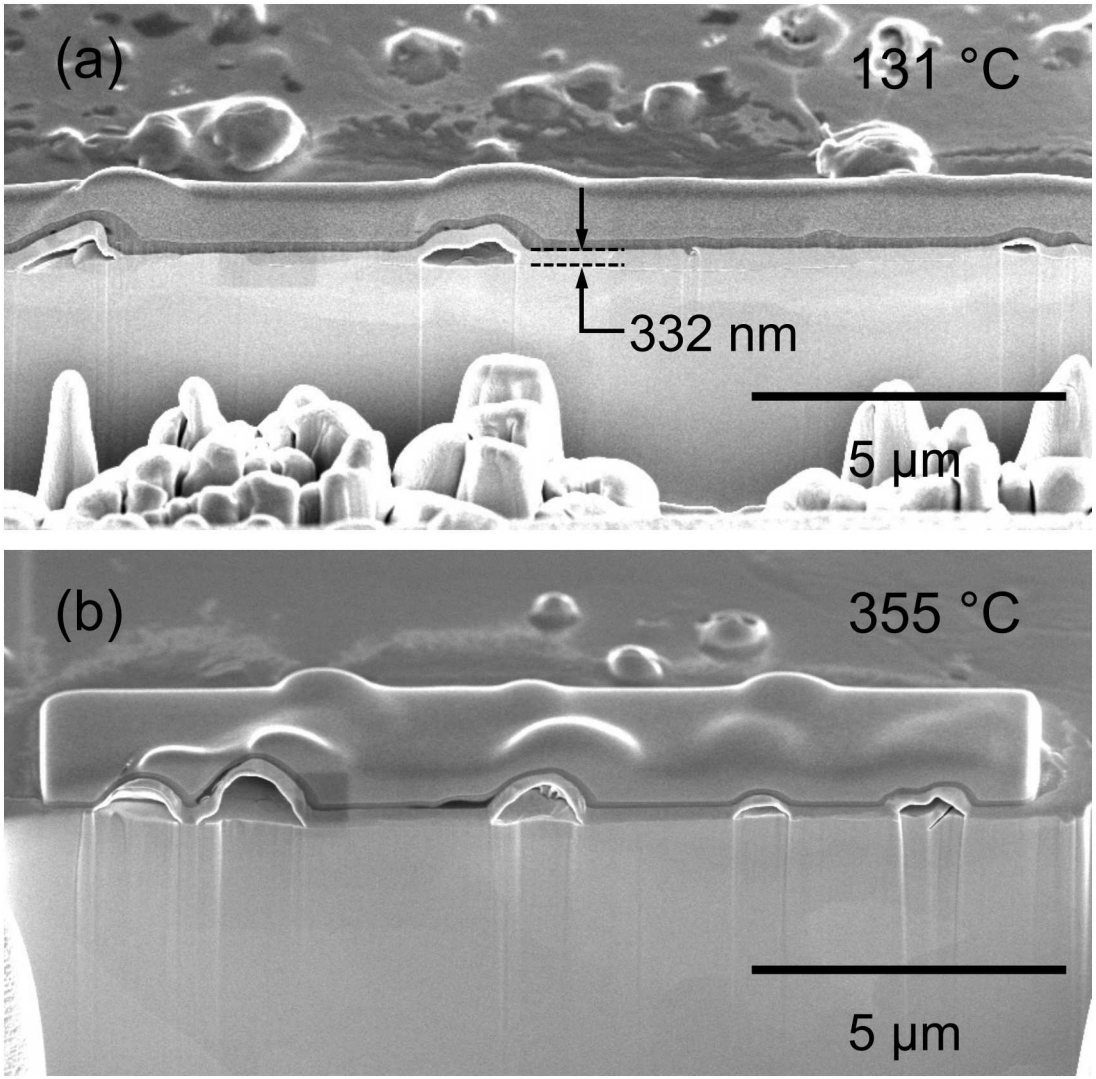
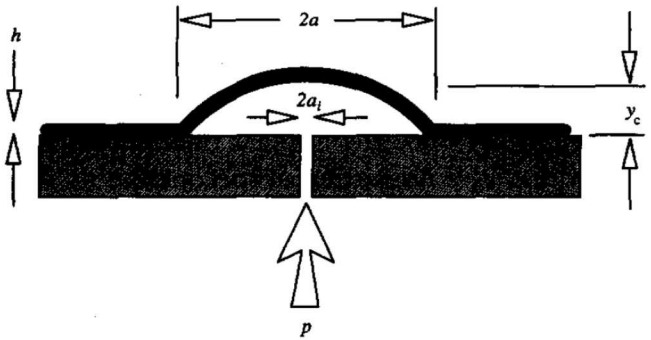
Focused Ion Beam Profiling Reveals Precipitates Expand Through Crack Extension



- Nucleation: platelet-shaped cracks
- Expansion due to internal gas pressure (> 1 GPa)



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

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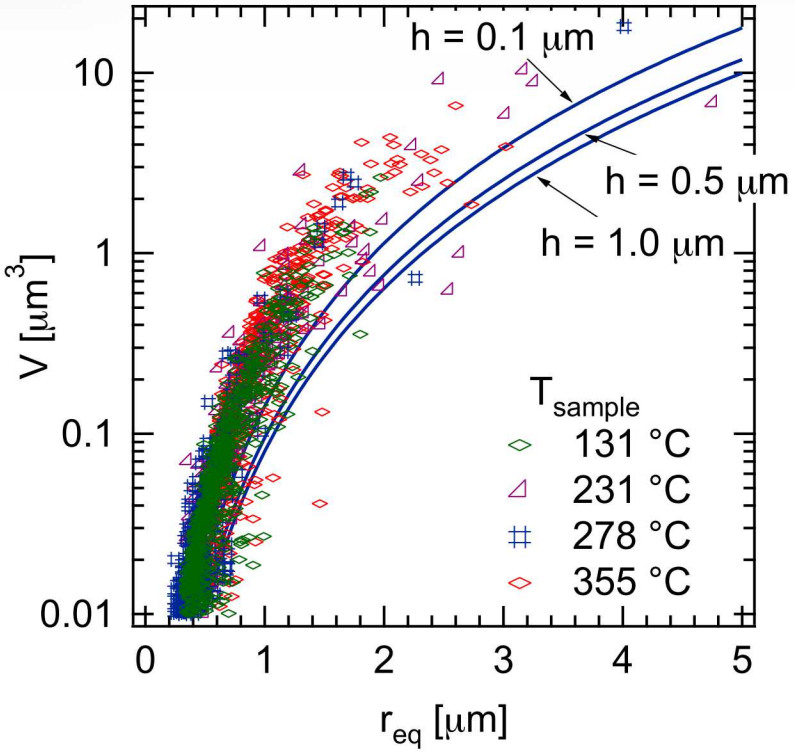
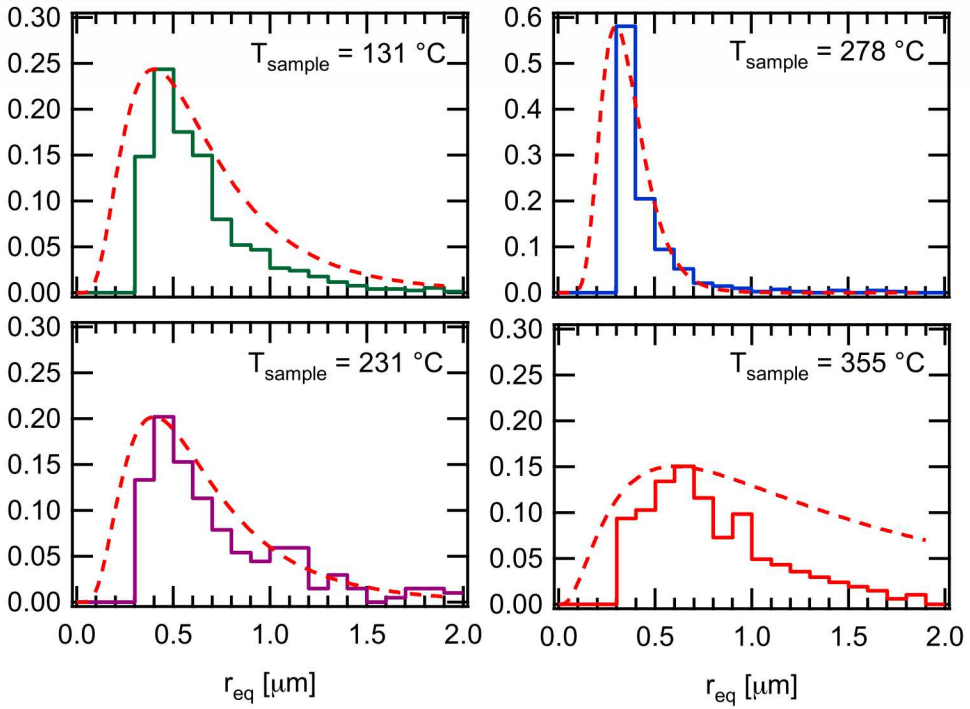
Atomic Force Microscopy Quantifies Bubble Size and Distribution of Deuterium Therein





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.*

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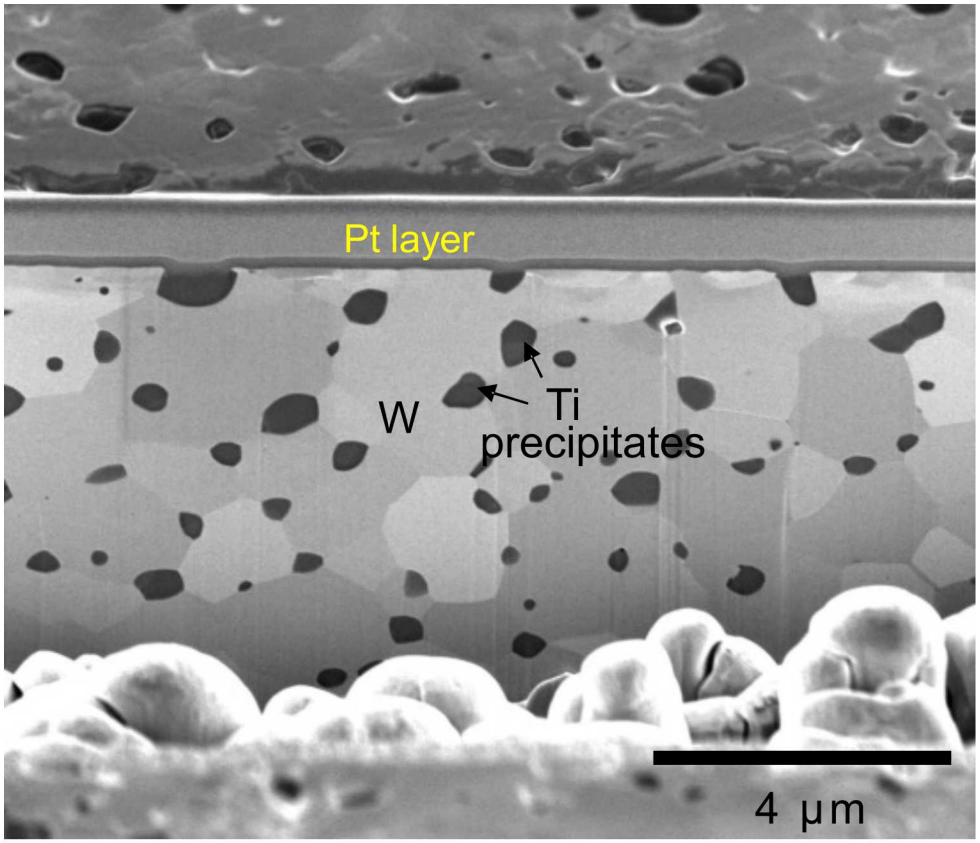
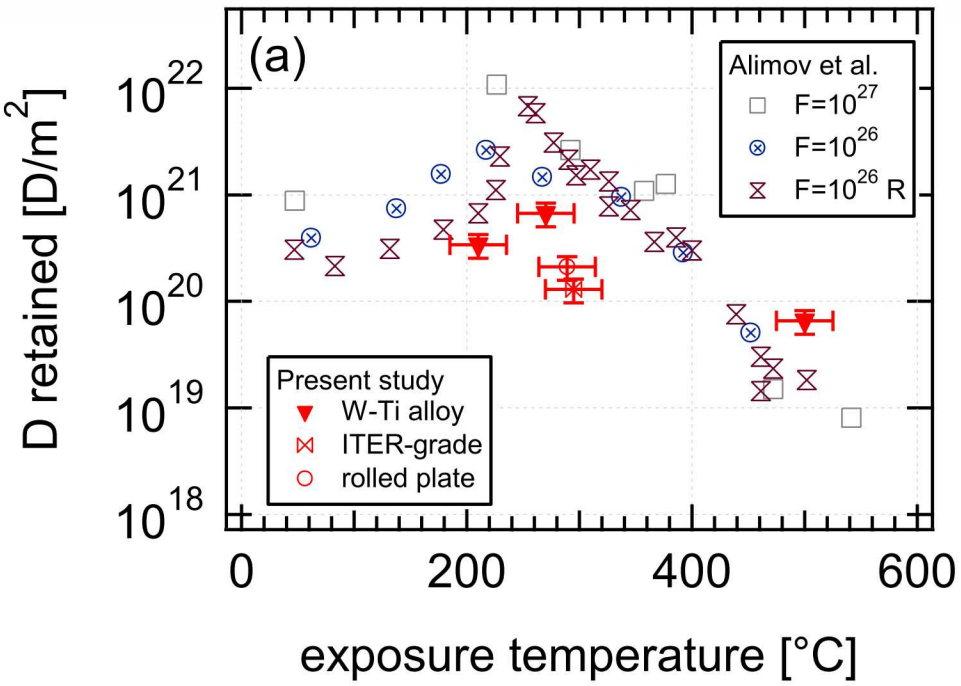
Plasma Retention Measurements Performed On Dispersoid Strengthened UFG Tungsten

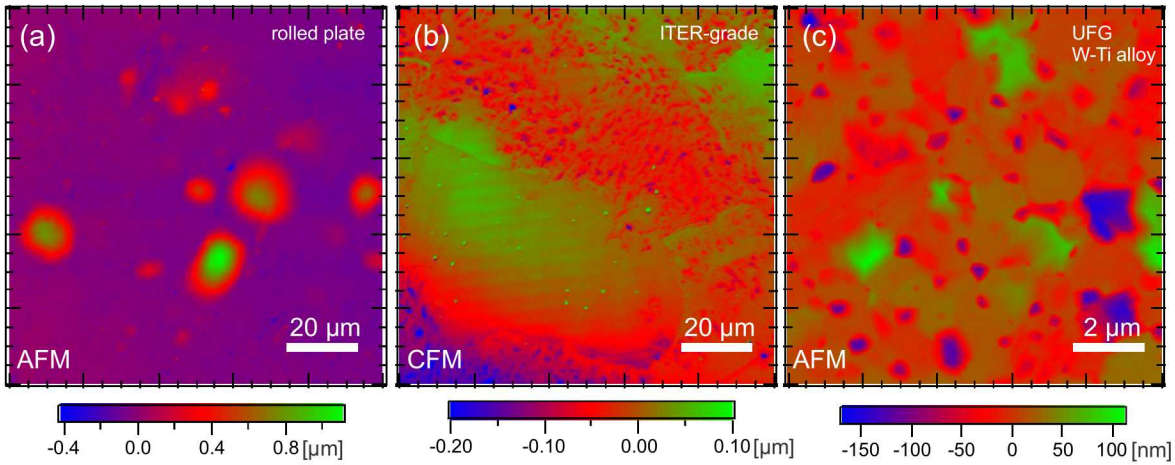


- W-Ti alloy developed at Univ. of Utah (Zak Fang)
- Microstructure offers possible improved strength and resistance to neutron damage
- **Response to plasma unknown**
- Collaboration with UCSD for high-flux deuterium plasma exposure

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. Kolasinski, D. A. Buchenauer, R. Doerner, et al., J. Mater. Res. (submitted).

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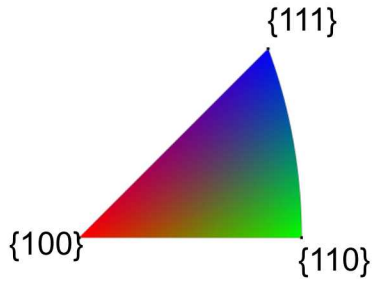
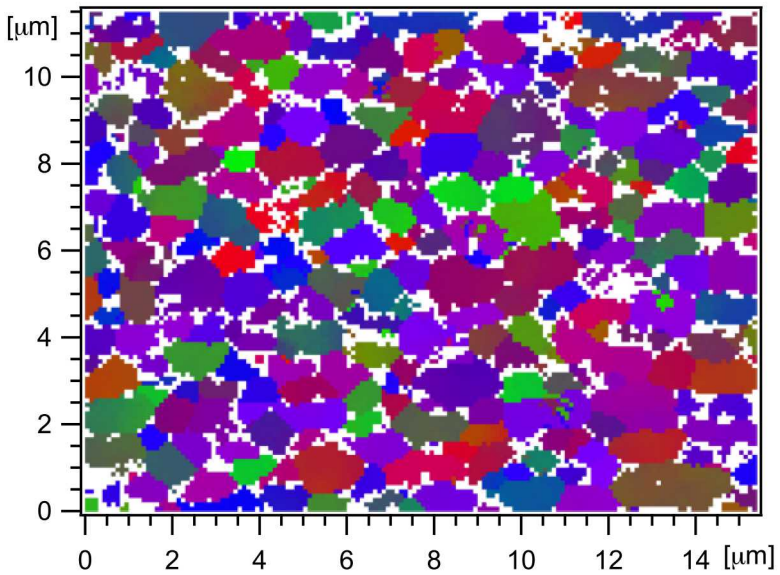
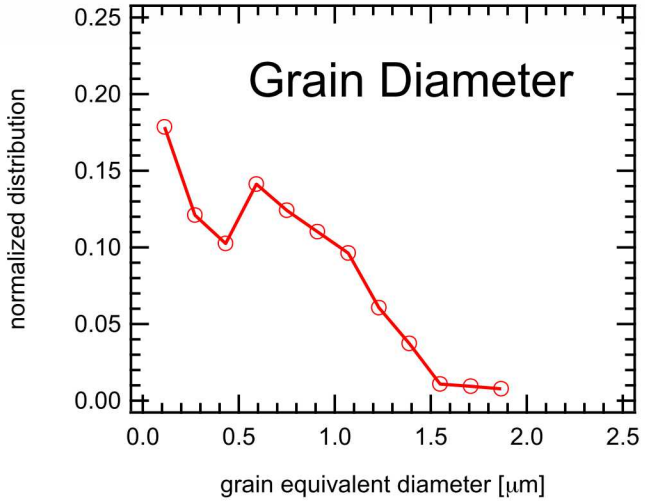
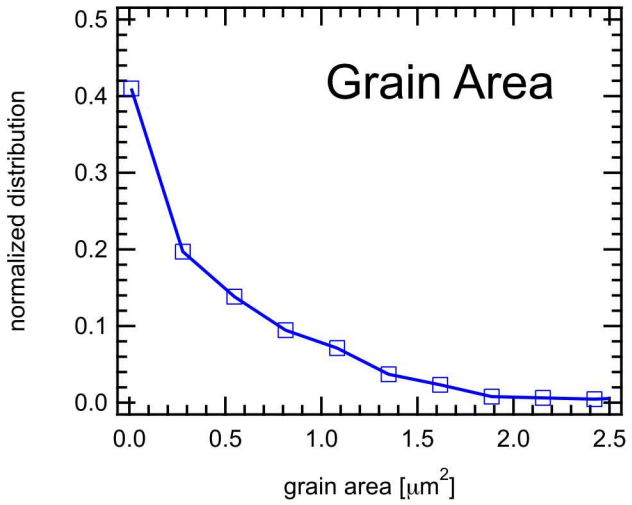
EBSD of UFG W Exhibits Random Grain Orientation With Grain Size Distribution Peaking << 1 μm









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

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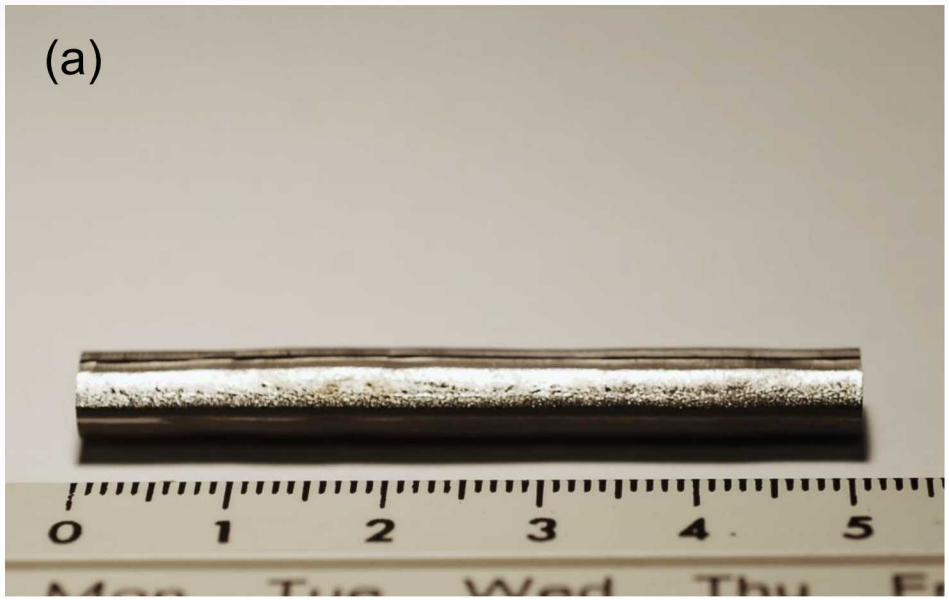
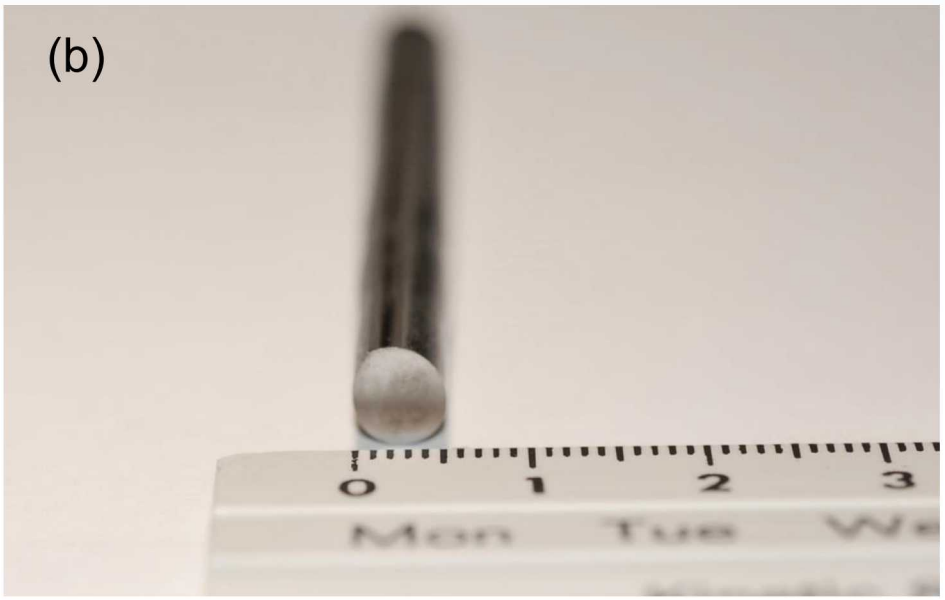
Single Crystal Tungsten Samples Prepared For HFIR Irradiation (RB19J Sub-Capsules)





W(110) single crystal samples:

- Images of (a) and (b) above are of the W(100) rod, and are included to show the non-uniform cross-section typical of single crystals.
- 30 specimens fabricated for insertion into PHENIX capsule (+ 6 reserve)
- Diameter: 5.97 mm
- Thickness 0.26-0.40 mm
- Laser marked and polished

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Summary

- By increasing the sealing temperature above the DBTT of tungsten, various grades of tungsten materials have been measured for D_2 gas driven permeation.
- The measured permeation of ITER tungsten compares well with commonly cited literature values, however UFG tungsten shows higher permeation and a lower activation energy. At the temperatures expected in a reactor divertor, however, the difference in negligible.
- 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.
- Single crystal tungsten samples for irradiation in HFIR have been prepared and shipped to ORNL. These will be used to study the role of neutron damage in tungsten absent of damage caused by processing.

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