SAND2014-17968PE

U.S. / Japan PHENIX Assignment Report

R. Kolasinski[(a)], C. Taylor[(b)], M. Shimada[(b)], and D. Donovan[(c)]

[(a)]*Sandia National Laboratories, Hydrogen & Metallurgy Science Dept., Livermore, CA*
[(b)]*Idaho National Laboratory, Fusion Safety Program, Idaho Falls, ID*
[(c)]*University of Tennessee, Dept. Of Nuclear Engineering, Knoxville, TN*

Y. Oya[(d)], T. Toyama[(e)], T. Chikada[(d)], K. Michibayashi[(f)], and Y. Hatano[(g)]

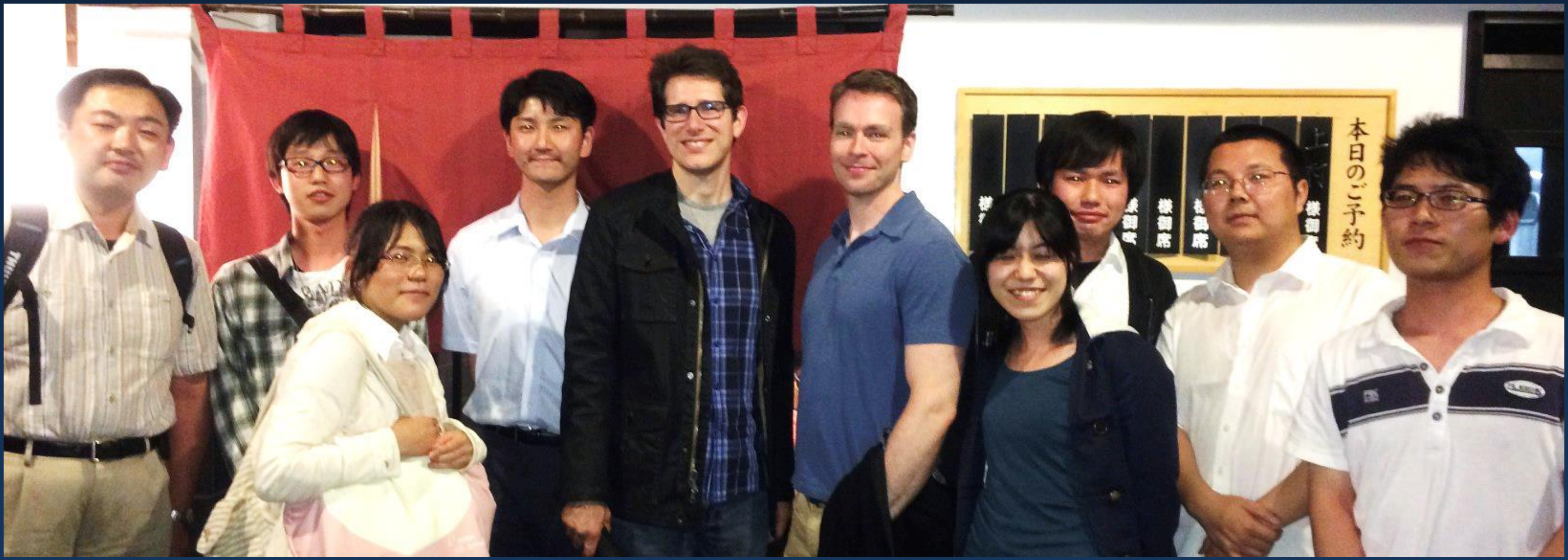
[(d)]*Shizuoka University, Department of Chemistry, Shizuoka, Japan*
[(e)]*Tohoku University, Institute of Materials Research, Narita-cho, Oarai, Ibaraki, Japan*
[(f)]*Shizuoka University, Department of Geosciences, Shizuoka, Japan*
[(g)]*Toyama University, Hydrogen Isotope Research Center, Toyama, Japan*

PHENIX Task 3 Planning Meeting *September 22, 2014*

Overview of U.S. Assignments

Four PHENIX assignments after the PSI Conference (June 9-13):

- Robert Kolasinski & David Donovan: Shizuoka University
- Chase Taylor: JAEA/Tohoku University
- Masa Shimada: Toyama University



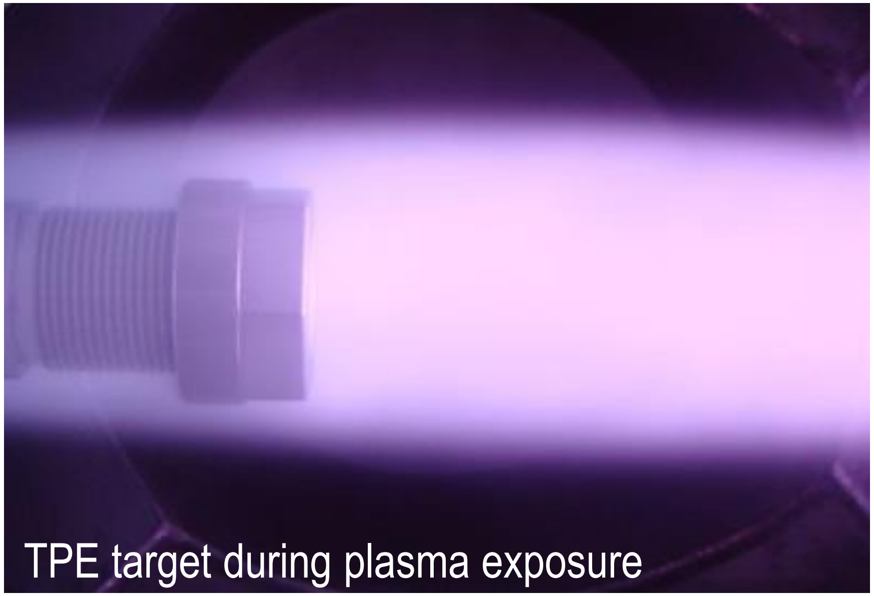
Outline

1 Analysis of bubble formation on ITER grade W surfaces exposed in TPE

2 Positron annihilation spectroscopy characterization of neutron-damaged materials

Motivation: Analysis of bubble growth in ITER-grade W samples exposed in TPE

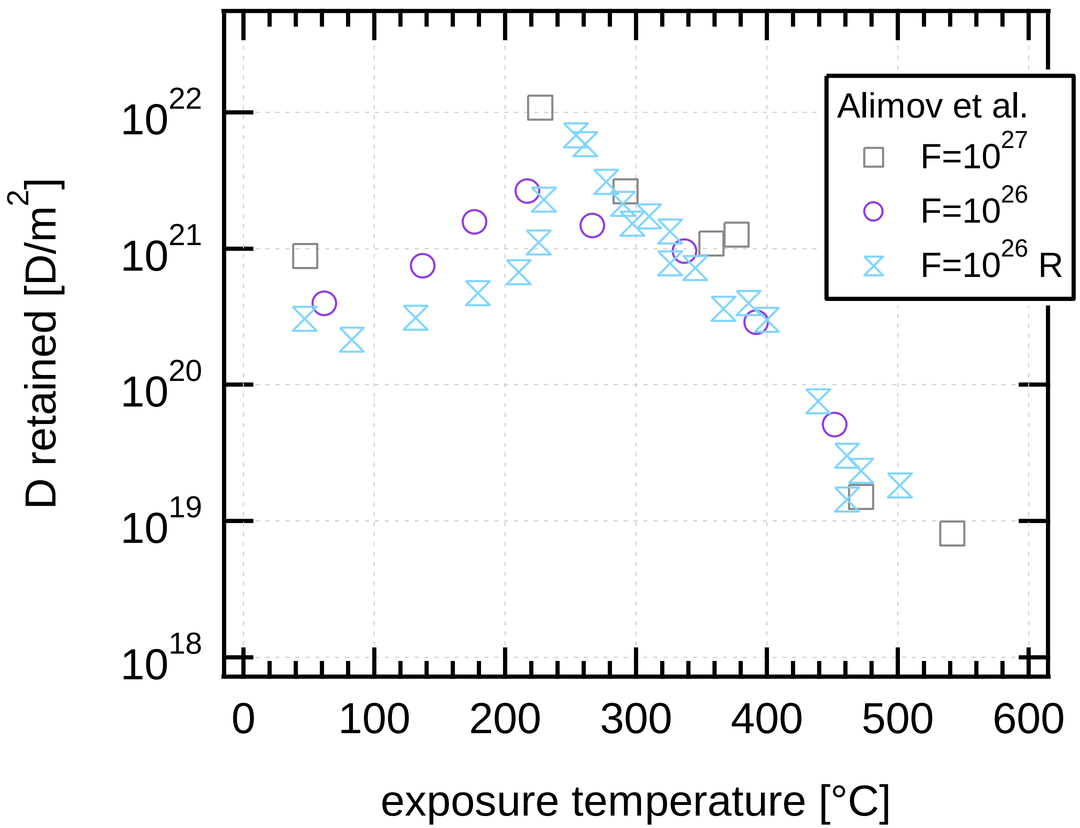
- Precipitation affects migration through material
- Bubble growth depends on microstructure
- Growth mechanisms critical to developing realistic models



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

Retention measurements correspond closely with those obtained in other laboratories



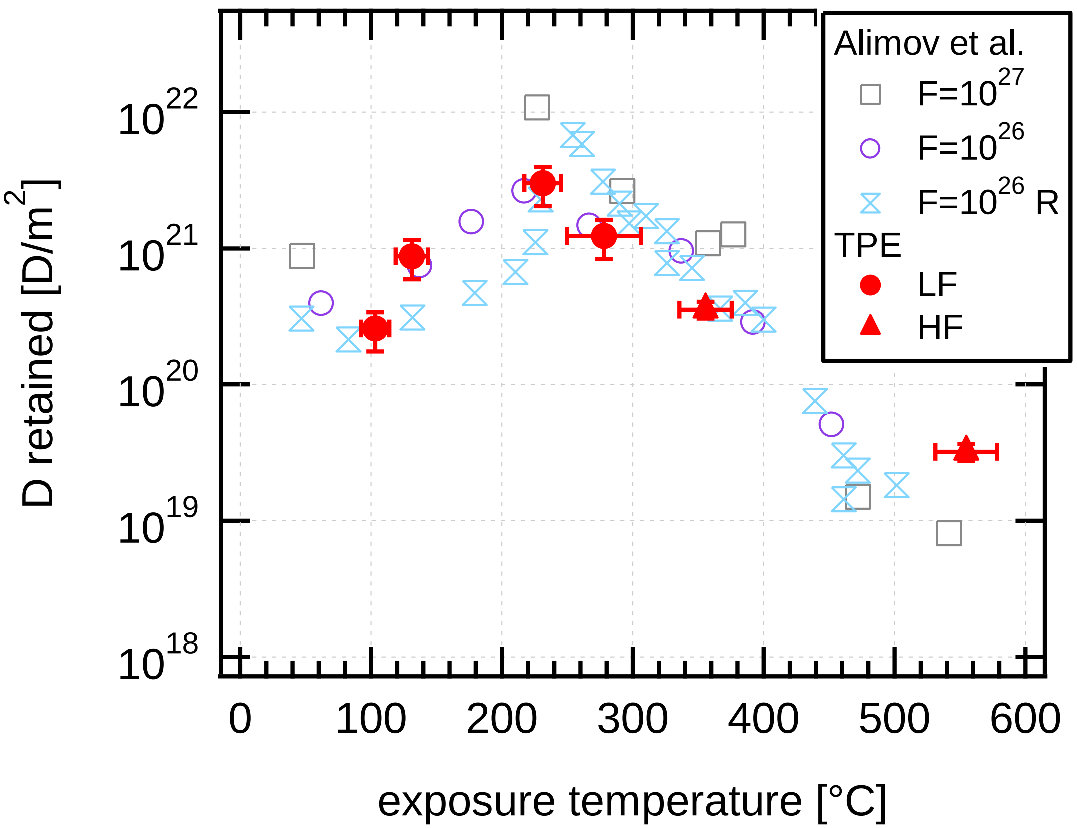
V. Kh. Alimov, et al. *J. Nucl. Mater.* **420** (2012) 519.

Previous work by Alimov et al:

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

Comparable exposure conditions

Retention measurements correspond closely with those obtained in other laboratories



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Comparable exposure conditions

TPE retention measurements:

- Correspond closely with Toyama/IPP meas.
- Confirm accepted retention temp. dependence.

XPS analysis shows that implanted C impurities in TPE have been reduced

Sputtering time / s	depth / nm	C	O	W
0	0.0	49.9	38.0	12.0
5	0.8	11.2	19.4	69.3
30	5.1	6.5	19.3	74.2
90	15.2	0.4	18.8	80.8
180	30.3	6.2	16.9	76.8

- Impurity
  Surface: C (C-C) and O (O-C, -OH, WO_3)
  Inner: O (WO_3)

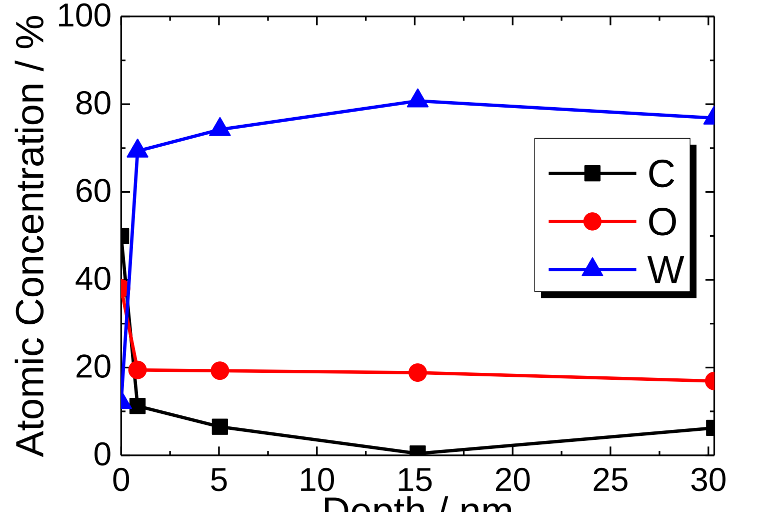
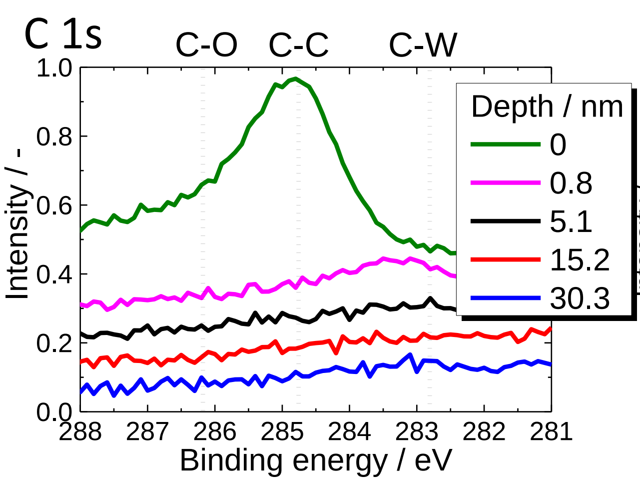
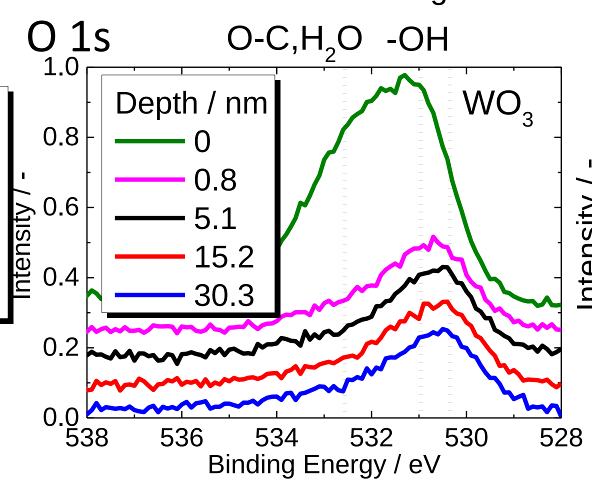
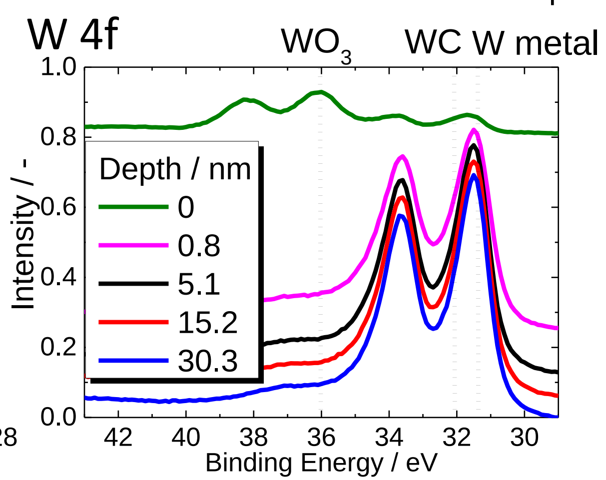


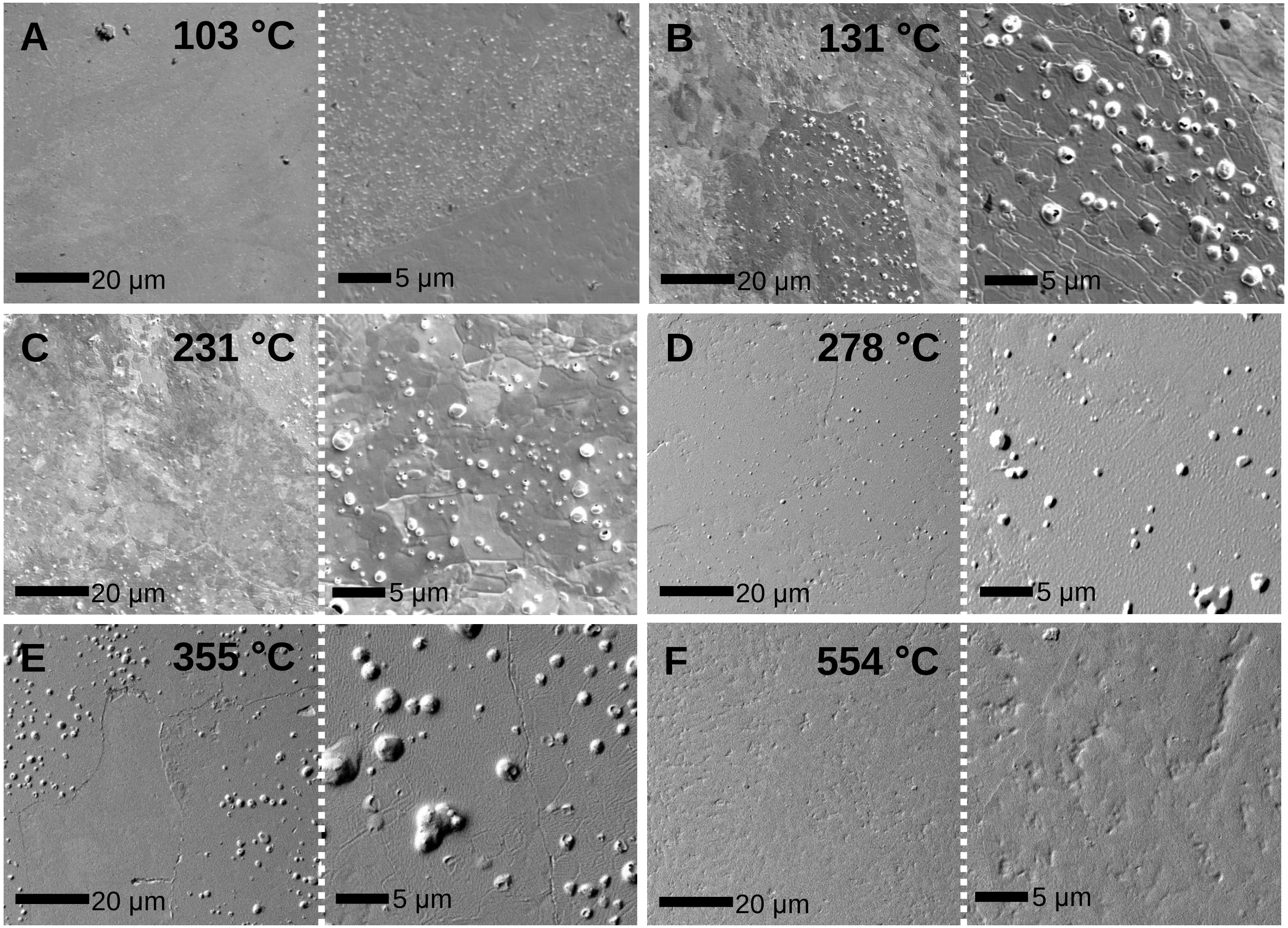
Fig. Atomic concentration for W12H annealed sample







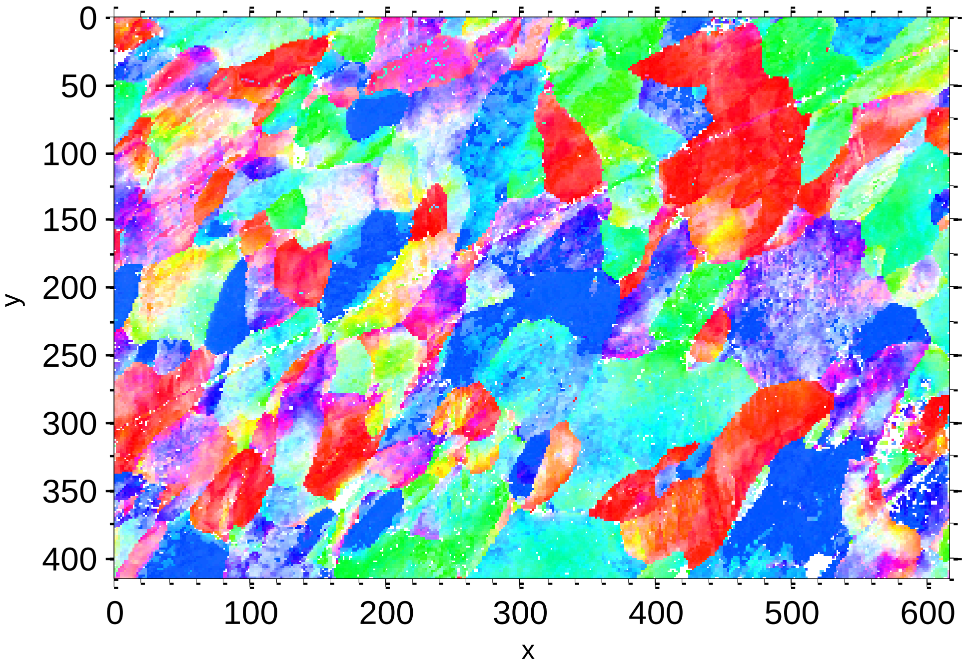
Surface morphology variation with temperature



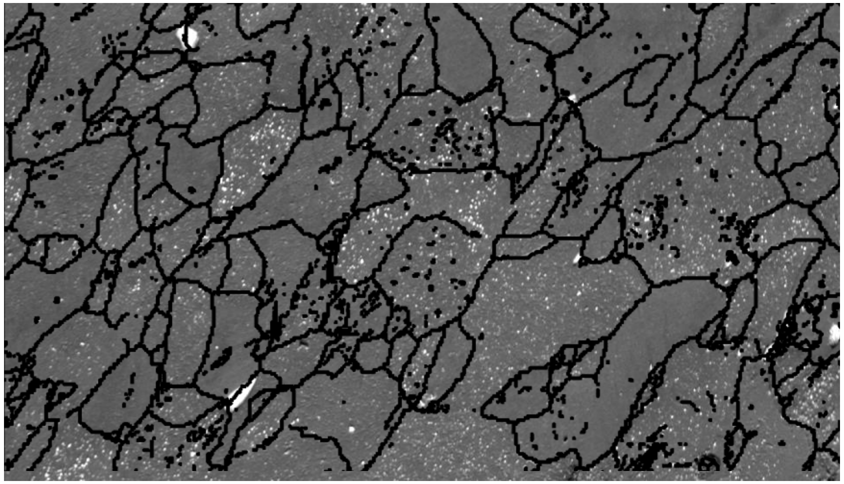
Key features:

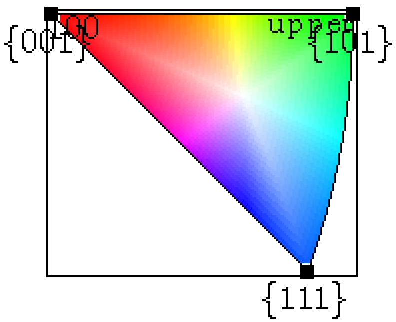
- Non-uniform coverage
- Bubbles are small (<10 μm dia.) compared with warm-rolled W material.
- Absent at temperature extrema.

EBSD measurements reveal dependence on grain orientation



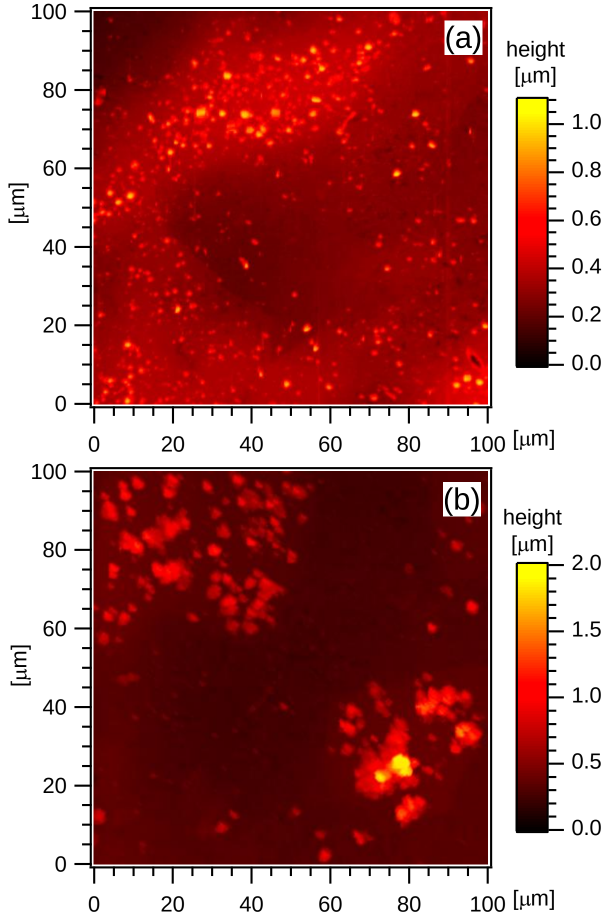
SEM image of the same area



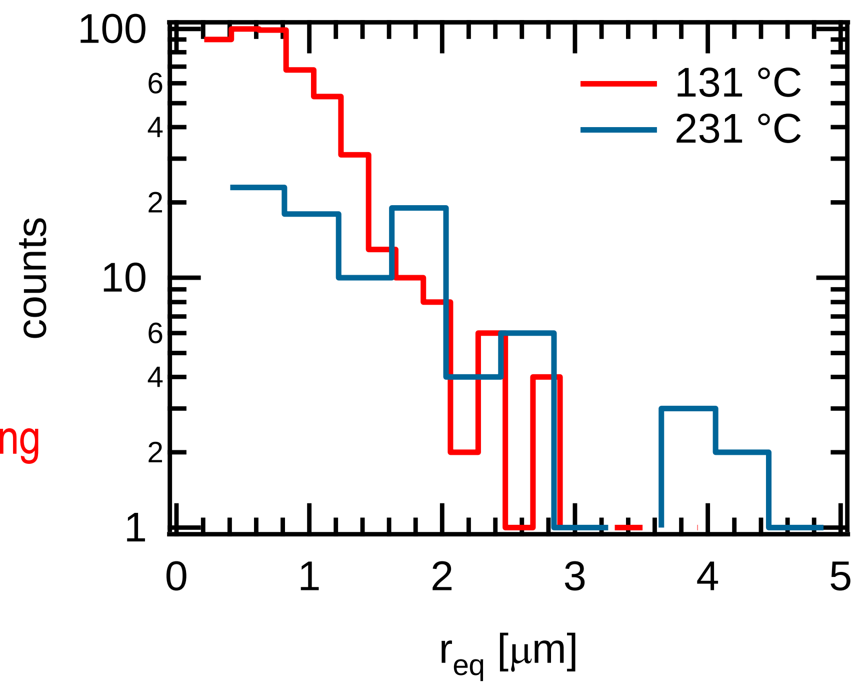


- Grain orientation indicated by inverse pole plot.
- Bubbles visible on grains with <111> and <110> directions aligned normal to surface
- Considerable distortion within individual grains
- Un-annealed sample showed increased distortion

Atomic force microscopy reveals details of surface structure



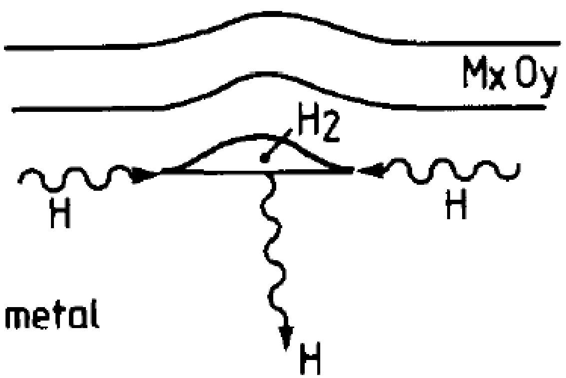
- Atomic force microscopy provides information on the shape of the deformed surface.
- Individual bubbles identified and analyzed automatically.



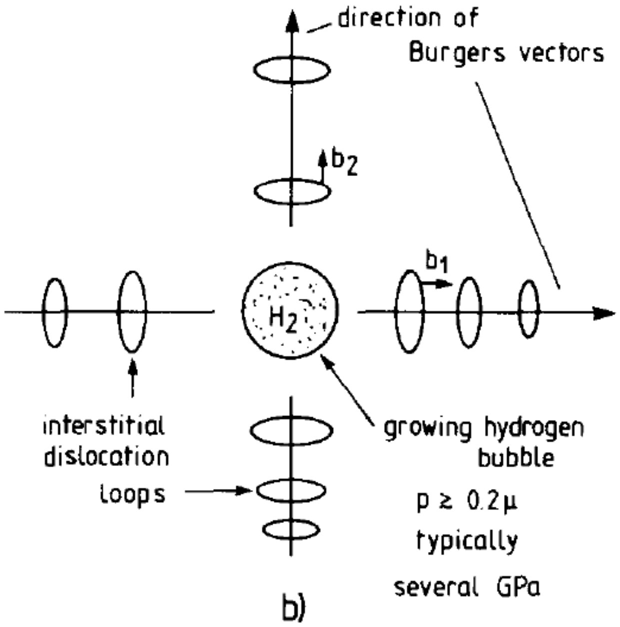
corresponding bubble size distributions

What bubble growth mechanisms are active in W during plasma exposure?

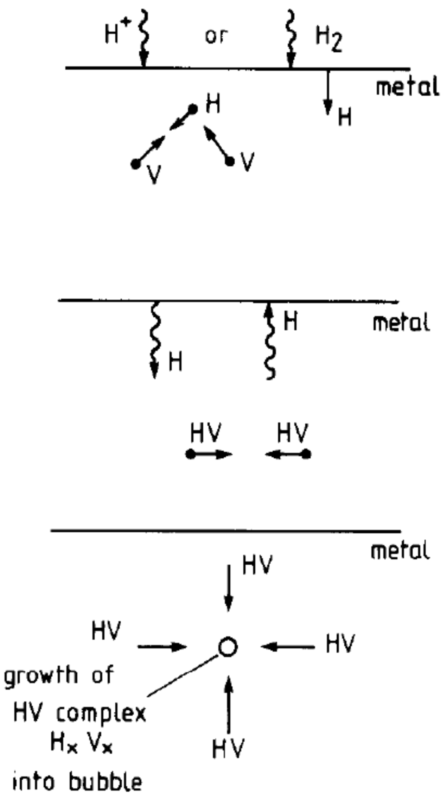
near-surface plastic deformation



dislocation loop punching



vacancy clustering



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

Far from the free surface, dislocation loop punching is favored

Three bulk precipitate growth mechanisms considered:

- Dislocation loop punching

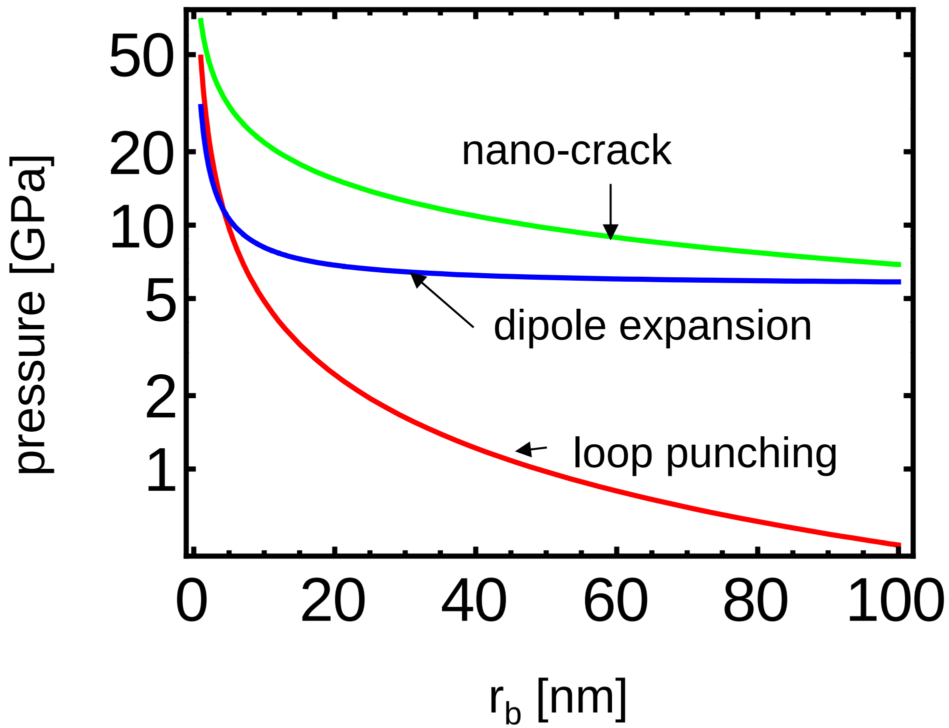
$$p_{LP} \geq \frac{2\gamma}{r} + \frac{\mu b}{r} \sim \frac{1}{r}$$

- Griffith nano-crack extension

$$p_{NC} \geq \sqrt{\frac{\pi\mu\gamma}{(1-\vartheta)r}} \sim \frac{1}{\sqrt{r}}$$

- Dislocation dipole expansion

$$p_{DE} \geq \frac{2\gamma}{s} + \frac{\mu d}{2r} \sim \frac{1}{r} + c$$



Based on methods developed in:
D. F. Cowgill, "Physics of He Platelets in Metal Tritides," in *Effects of Hydrogen on Materials* (2009).

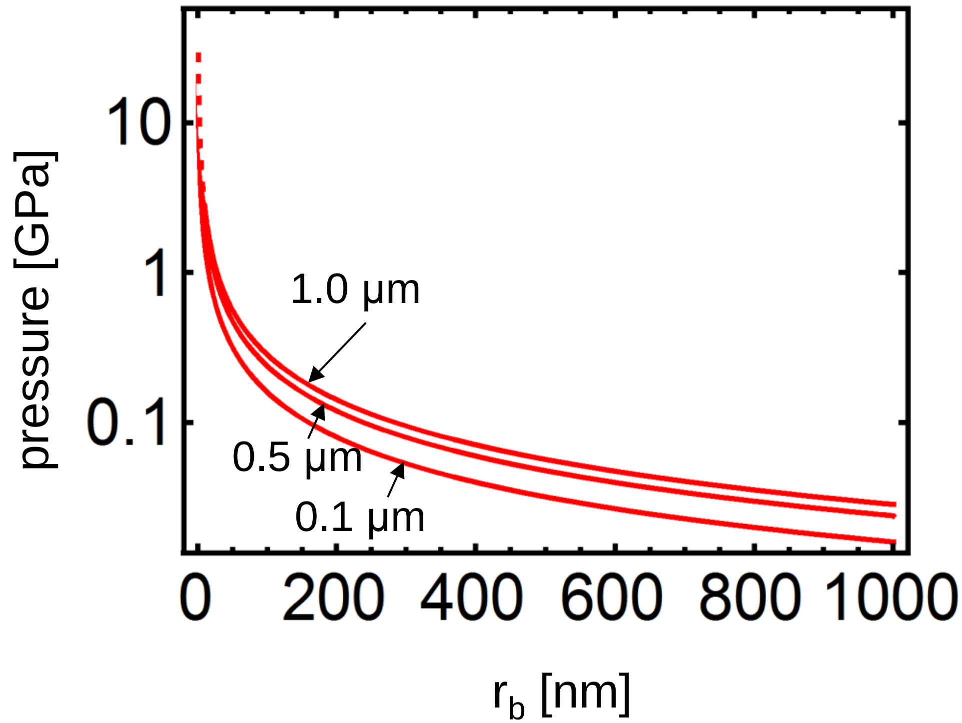
Near the free surface, bubbles may grow by crack extension

Crack extension competitive with loop punching near surface:

$$p_B \geq \frac{1}{r}\left(\frac{4\gamma(Eh)^{1/3}}{5C_1C_2}\right)^{3/4} \sim \frac{1}{r}$$

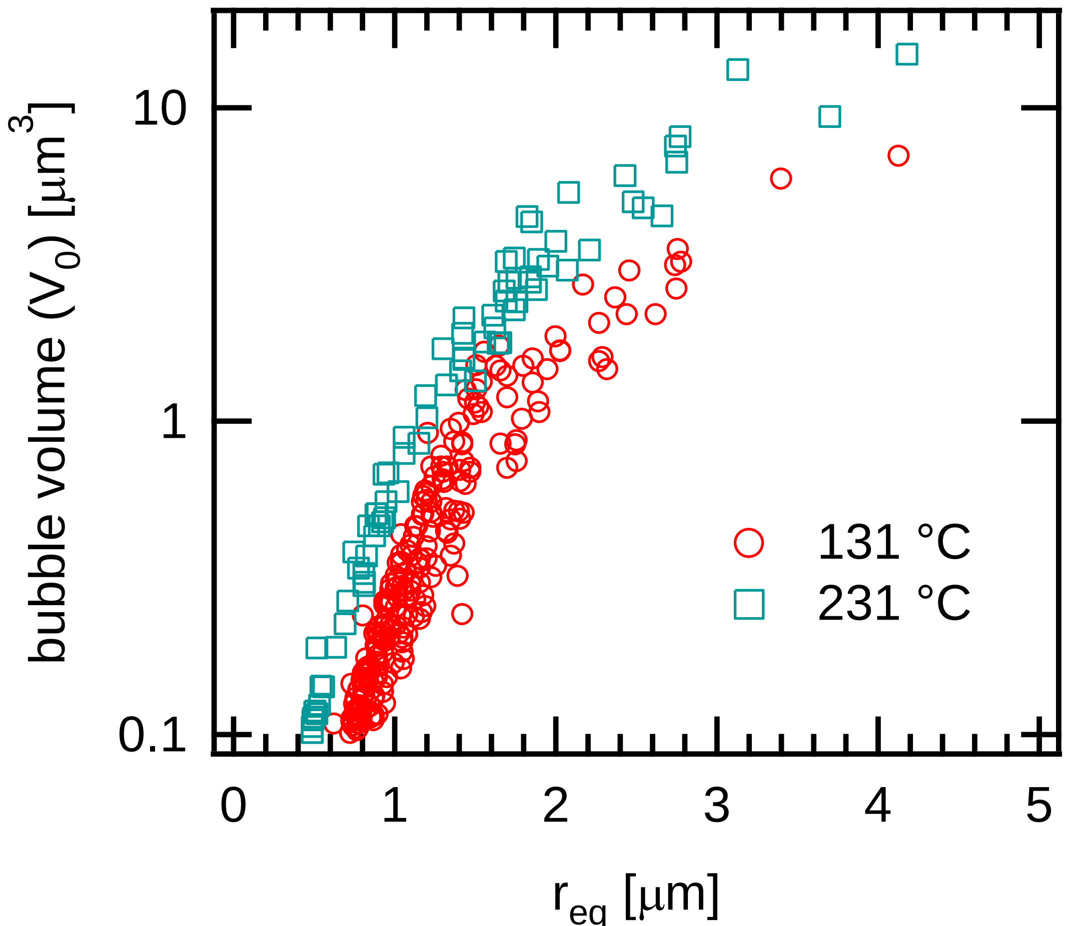
Limitations:

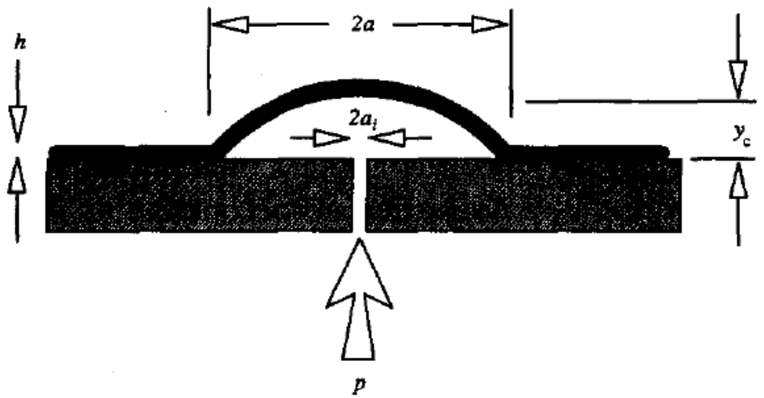
- Correction for thick blisters
- Effect of plasticity (blunting of crack tip)
- Hydrogen effects



Stress calculations based on calculations by K. Wan & Y. Mai, *Acta metall. mater.* **43** (1995) 4109.

Bubble volumes measured with AFM correlate well with blister model



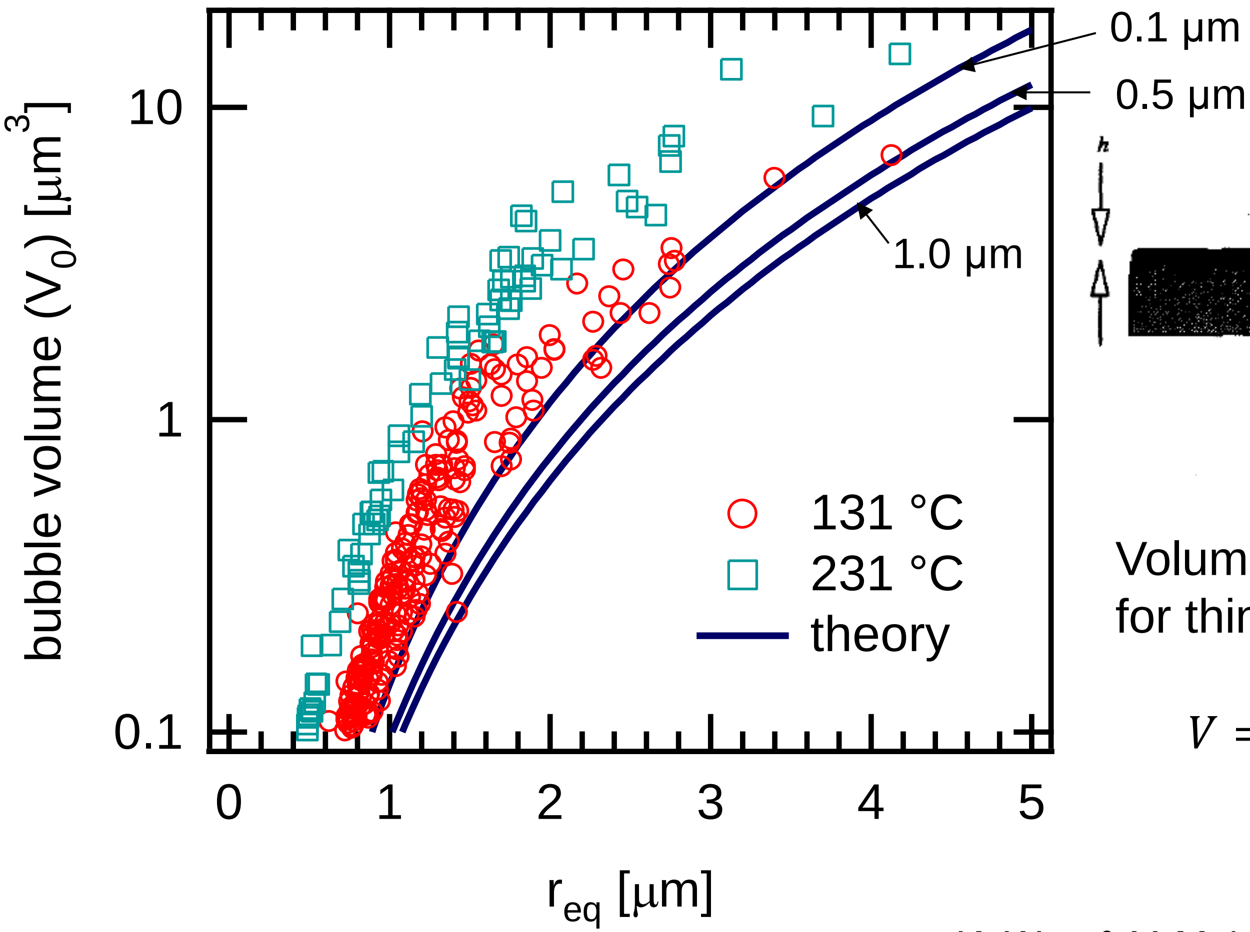


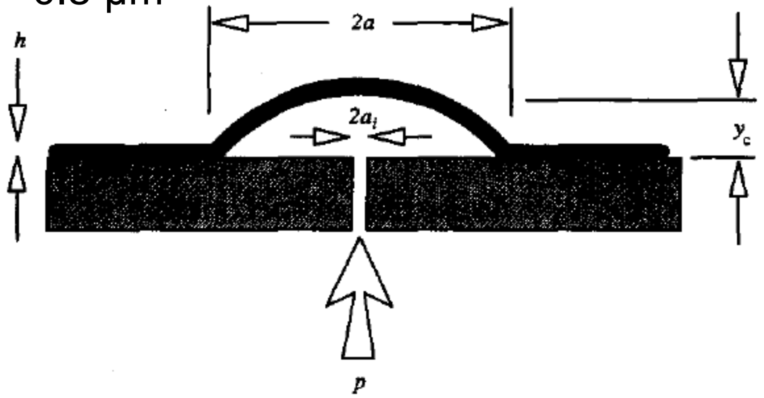
Volume modeled using blister test for thin film adhesion:

$$V = \int y(r)2\pi r dr = C_1 \pi a^2 y_c$$

K. Wan & Y. Mai, *Acta metall. mater.* **43** (1995) 4109.

Bubble volumes measured with AFM correlate well with deflection model





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Summary of surface morphology findings

- ITER-grade W sample exposed in TPE show similar retention to Toyama/IPP studies.
- Analysis performed at Shizuoka:
  - XPS shows implanted C reduced considerably
  - SEM/EBSD illustrate non-uniform bubble growth over surface
  - Bubble grow on (110) and (111) crystal planes
  - AFM analysis provide bubble volumes
- Modeling of bubbles:
  - Thin film adhesion model adapted to model blister grown on tungsten.
  - Model reproduces bubble sizes observed with AFM

Outline

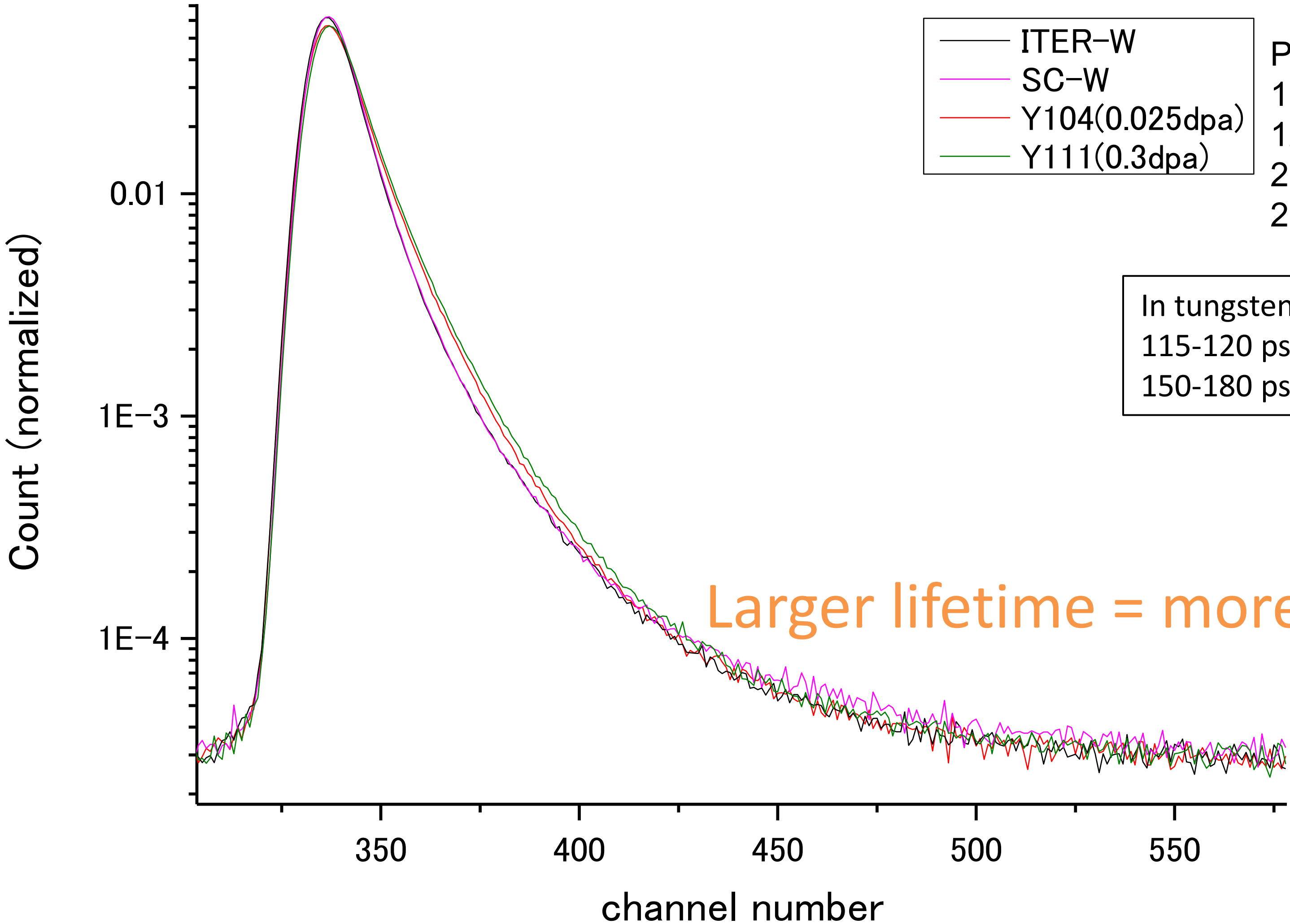
1 Analysis of bubble formation on ITER grade W surfaces exposed in TPE

2 Positron annihilation spectroscopy characterization of neutron-damaged materials

Results from JAEA/Tohoku University assignment

- Two positron annihilation spectroscopy techniques:
  - PALS: Positron annihilation lifetime spectroscopy.
  - CDB-PAS: Coincidence Doppler-broadening PAS.
- Samples
  - Single crystal W
  - ITER grade W
  - Y104, 0.025 dpa neutron damage
  - Y111, 0.3 dpa neutron damage
- Experiment details
  - PALS requires sandwich of two identical samples.
  - First sandwich: Cu-Na-W
    - Cu lifetime is similar to W
  - Second try: Si-Na-W

Positron Annihilation Lifetime Spectroscopy



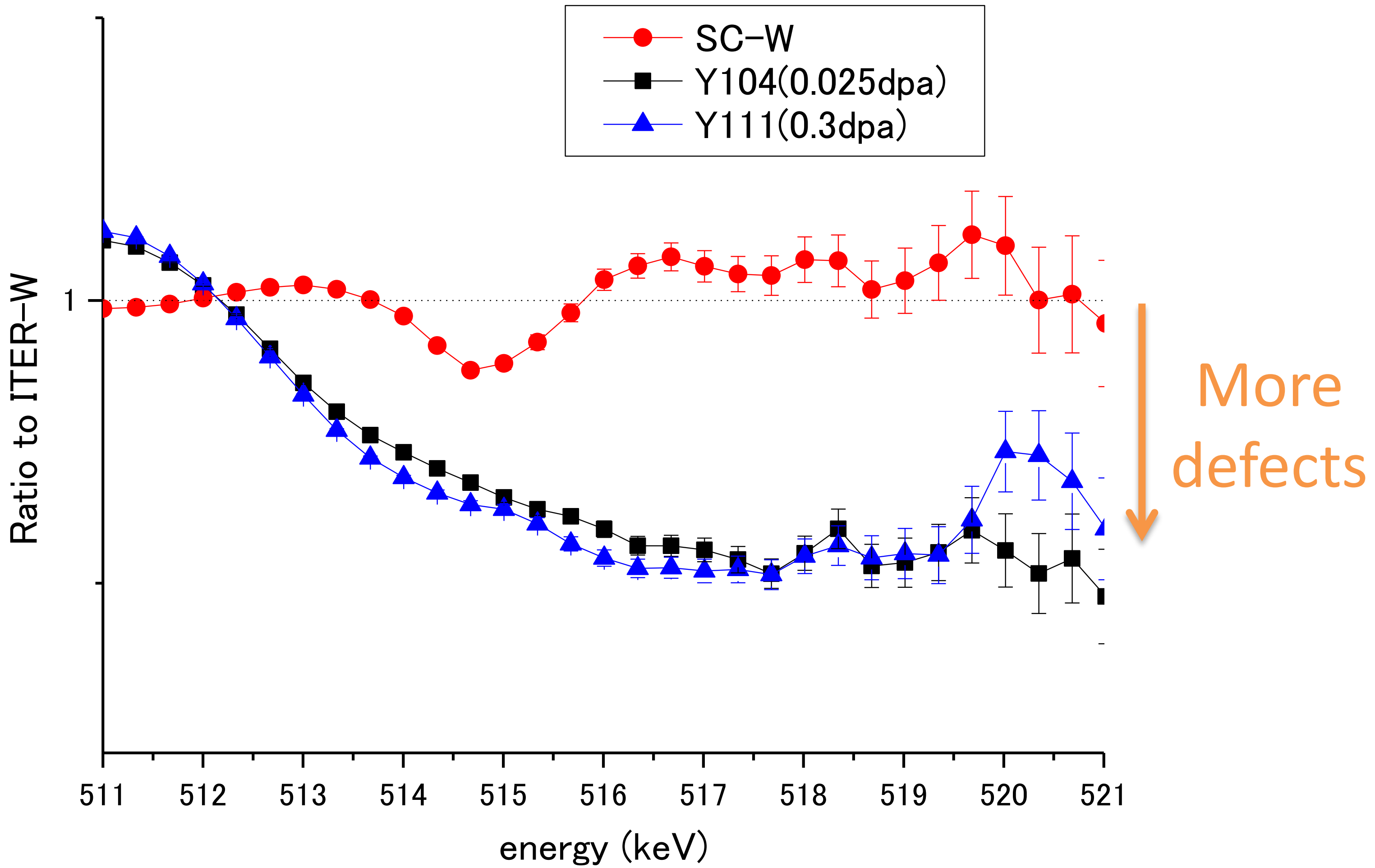
Positron lifetimes
133.5 ±0.5 ps
129.4 ±0.3 ps
202.1 ±0.9 ps
216.6 ±0.3 ps

In tungsten, positron lifetime is
115-120 ps (bulk)
150-180 ps (mono-vacancy)

Larger lifetime = more defects

Coincidence Doppler Broadening PAS

Ratio curves of coincidence Doppler broadening measurement



Preliminary Conclusions

Defect concentration →

Single crystal W	ITER W	Y104 (0.025 dpa)	Y111 (0.3 dpa)
129.4 ±0.3 ps	133.5 ±0.5 ps	202.1 ±0.9 ps	216.6 ±0.3 ps

In tungsten, positron lifetime is
115-120 ps (bulk)
150-180 ps (mono-vacancy)

- Anticipated relative defect concentrations verified.
- Defects in Y104 and Y111 are likely larger than mono-vacancies.

Acknowledgements

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- Our collaborators at INL and Sandia/CA:
  - Brad Merrill, Robert Pawelko, Lee Cadwallader
  - Dean Buchenauer, Donald Cowgill, Richard Nygren, Josh Whaley, Jon Watkins, Thomas Felter
- Our colleagues from Japan for their support of our visits.

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