

Thermo-mechanical properties of damaged tungsten surfaces

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Main thrust of the proposal

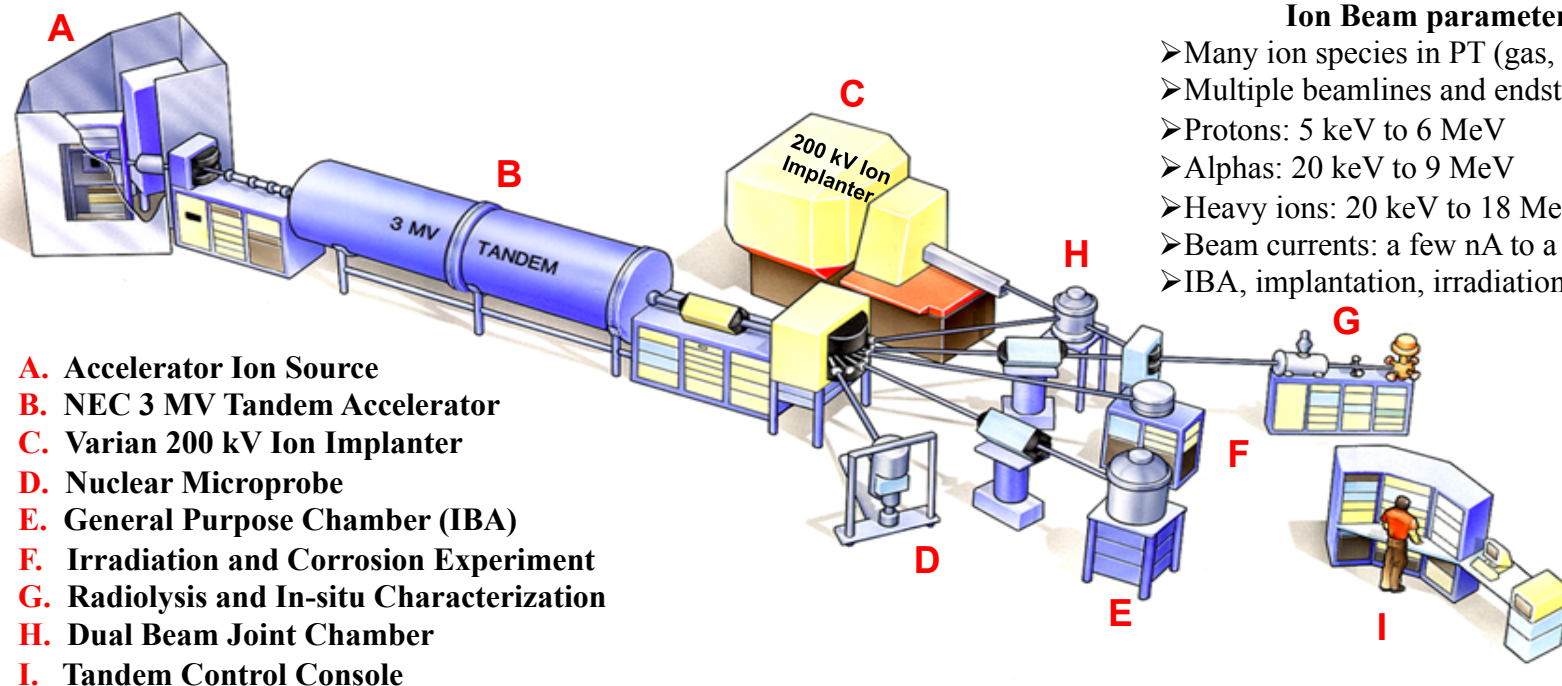
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- Utilize the unique capabilities of the LANL IBML (Ion Beam Materials Laboratory) and UCSD PISCES Facility to damage the near surface regions of W targets
- Employ novel nanotechnology diagnostic techniques to interrogate the near-surface region to determine the thermal and mechanical properties of the thin damaged layer

IBML uses energetic ions to simulate neutron damage of plasma-facing materials

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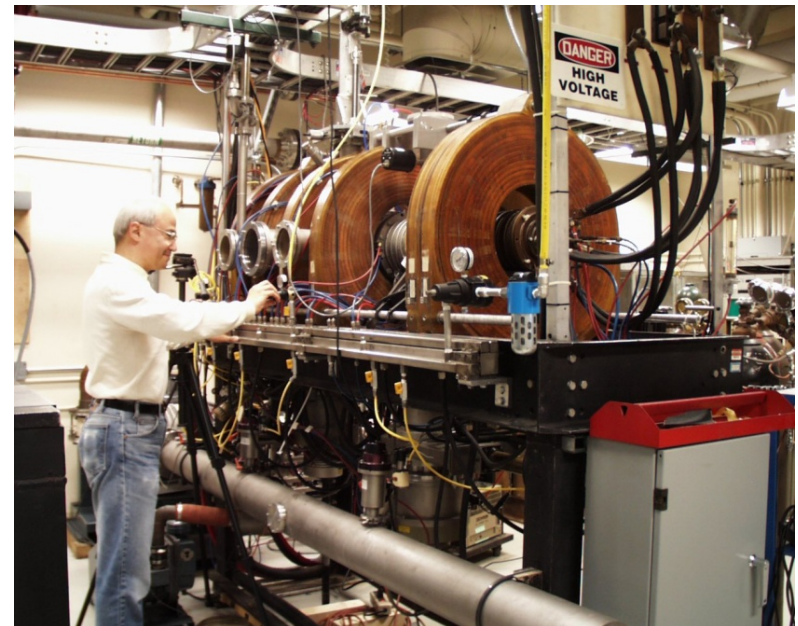
- A variety of heavy ions can impart surface damage to W targets
- Dual beam capability allows sequential and simultaneous He ion implantation
- Heated target holder allows damage annealing studies



High flux plasma exposure occurs in PISCES-A

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- PISCES-A device is used to facilitate sample sharing
- Simultaneous steady-state plasma exposure of damaged and undamaged targets to pure D, H, or He plasma, also quantified D/He mixed species plasma
- Γ_{ion} up to $5e22$ ions/m²s, $T_e \sim 5\text{-}20$ eV, $T_{\text{surf}} = \text{r.t. to } 900^\circ\text{C}$
- TEM analysis for determination of surface changes
- TDS for post-exposure retention studies

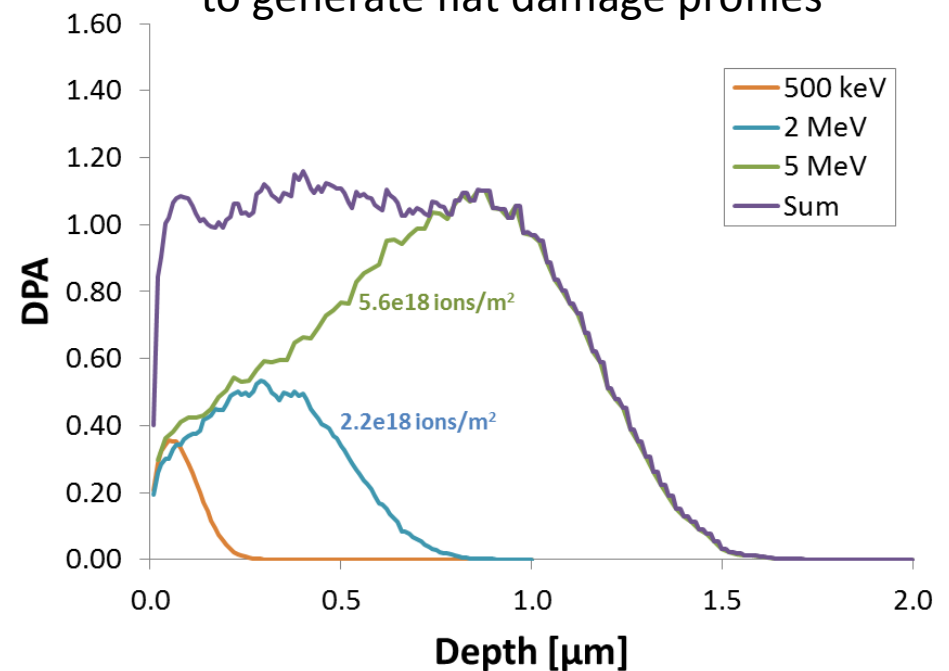


Nanotechnology diagnostics can probe only the damaged surface region of targets

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- Nanoindentation is used to measure changes in mechanical properties at the Center for Integrated Nanotechnologies (CINT) at LANL
- Nano3 Laboratory at UCSD provides clean room facilities for pattern deposition to measure thermal properties using a 3ω technique
- TEM analysis available at both locations

Multiple Cu^+ energies are overlaid to generate flat damage profiles



Research topics

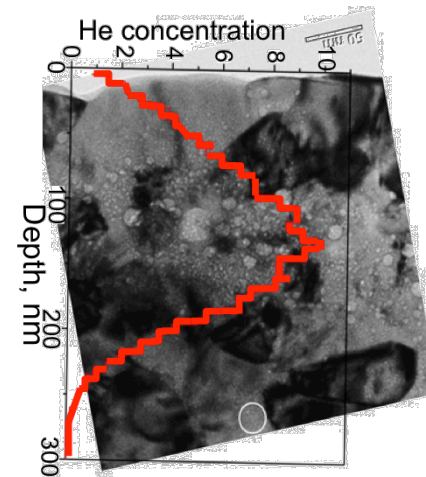
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- Effects of room temperature damage with varying dose/dpa
 - Develop diagnostic procedures
 - Validate techniques against literature
- Inclusion of He in damage (sequential exposures @ rt)
 - Compare He ion beam damage to He plasma effects
- Influence of damage w & w/o He on D migration
 - Compare fluence dependences of He ion beam and plasma
- Vary temperature during damage to examine annealing [YR2]
- Simultaneous damage and He irradiation at a variety of temperatures to investigate He impact on annealing damage [YR3]

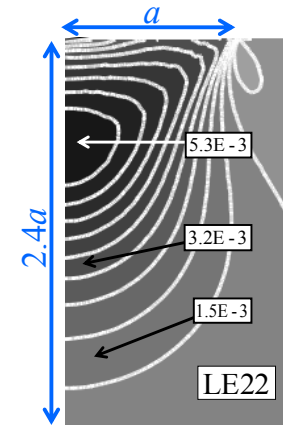
Using Indentation to Measure Radiation Damage

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- The length scales in indentation (~ microns) are very complimentary to the size of the irradiation affected zone
- This makes indentation an ideal technique for measuring changes in local mechanical properties caused by irradiation



He profile (atomic %) on TEM of irradiated Ag



Strain fields under a spherical indenter

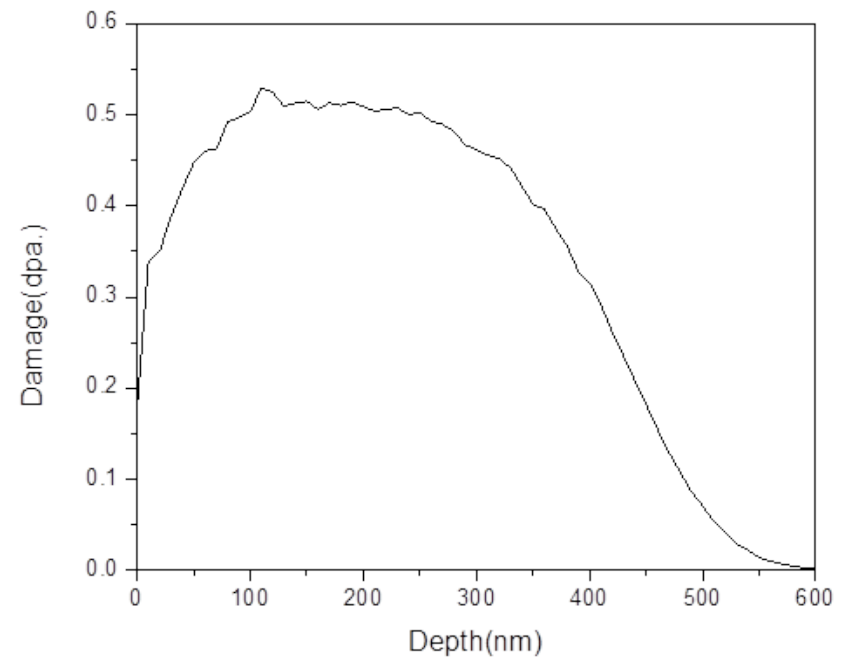
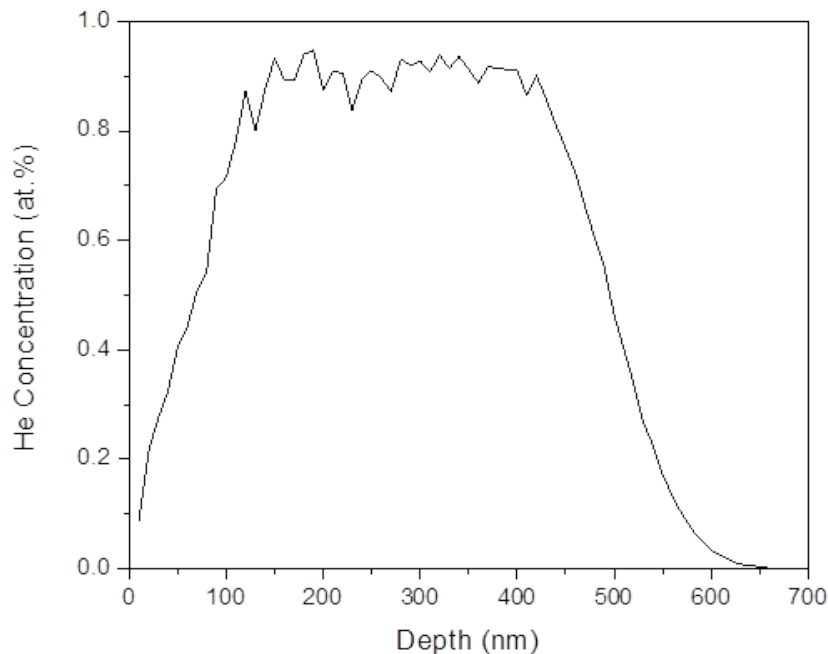
A series of differing indenter radii can be used to probe different depth scales

Indenter radius	Indentation depth (h)	Contact radius (a)	Indentation zone $\sim 2.4a$
1 μm	~ 10 nm	60 nm	144 nm
10 μm	~ 20 nm	250 nm	600 nm
100 μm	~ 40 nm	1,200 nm	2,880 nm
1000 μm *	> 200 nm	12,800 nm	30, 720 nm

Helium implanted W for nanomechanical testing (~0.51 dpa, ~0.92 at%, ~18000 appm/dpa, ~500 nm)

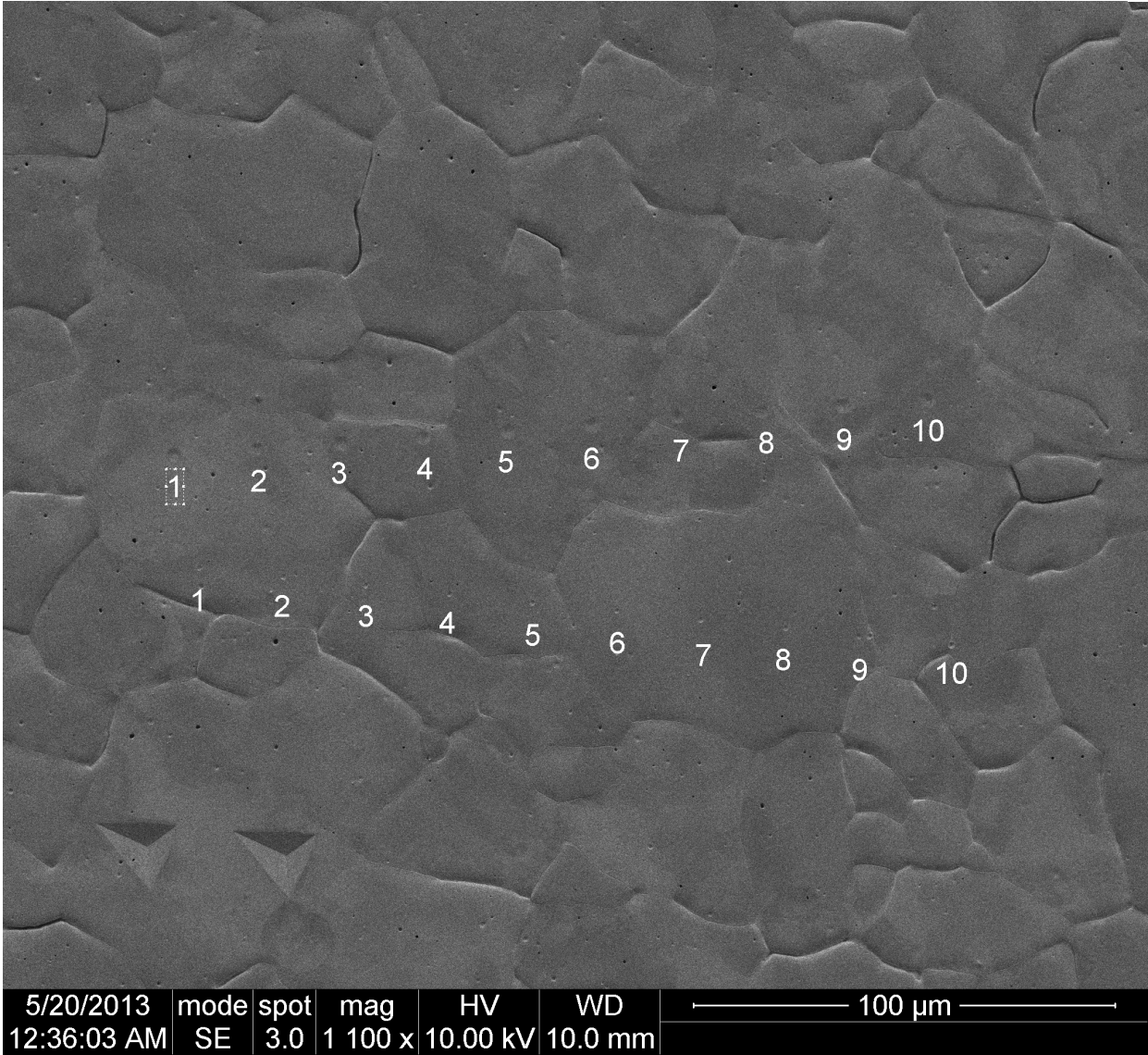
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Multiple energies were used to generate a box-like profile
50 keV @ 7.2×10^{15} ions/cm² 100 keV @ 8.0×10^{15} ions/cm²
150 keV @ 4.0×10^{15} ions/cm² 200 keV @ 2.0×10^{16} ions/cm²



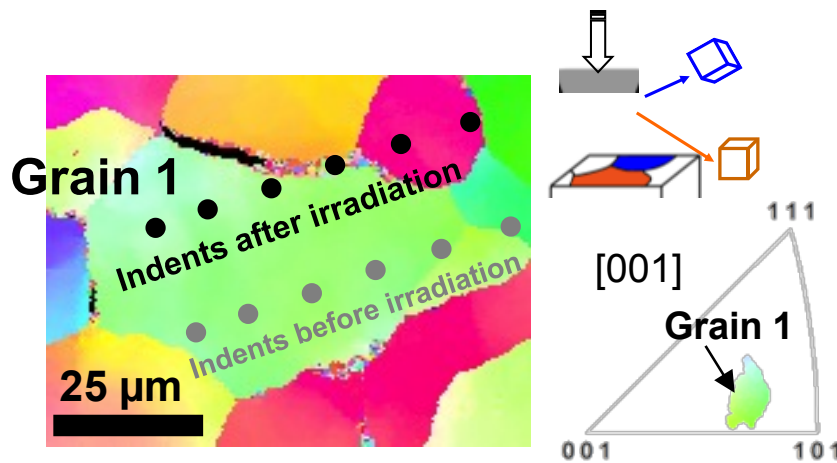
Indentation location on irradiated W sample

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Irradiated vs. Unirradiated Tungsten: Preliminary Indentation Results

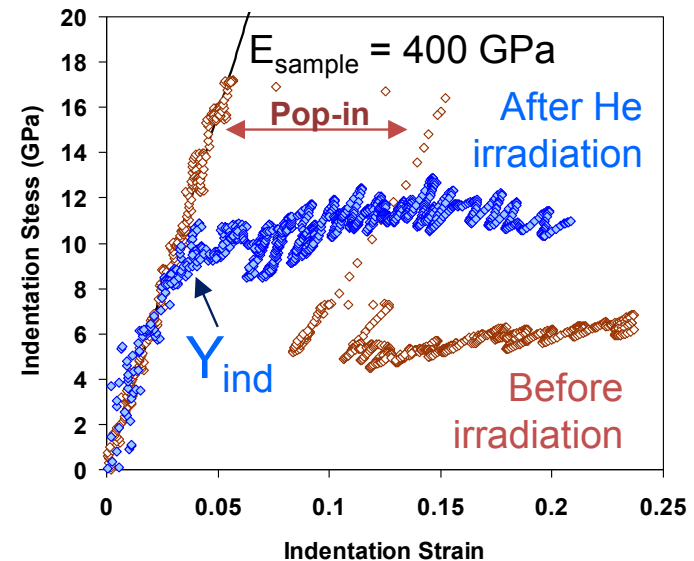
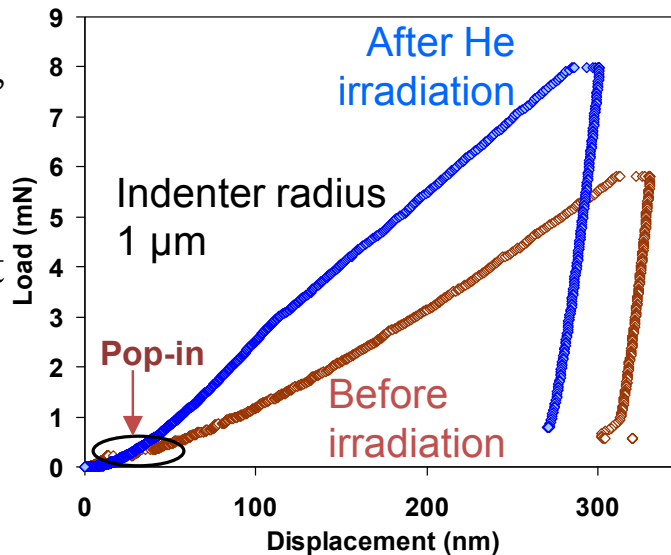
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Tungsten Grain 1	Modulus, GPa	Hardness GPa
Before irradiation	373 \pm 24	5.36 \pm 5.3
After He irradiation	376 \pm 44	8.82 \pm 1.4

Irradiation induced hardening is measured using Stress-Strain analysis

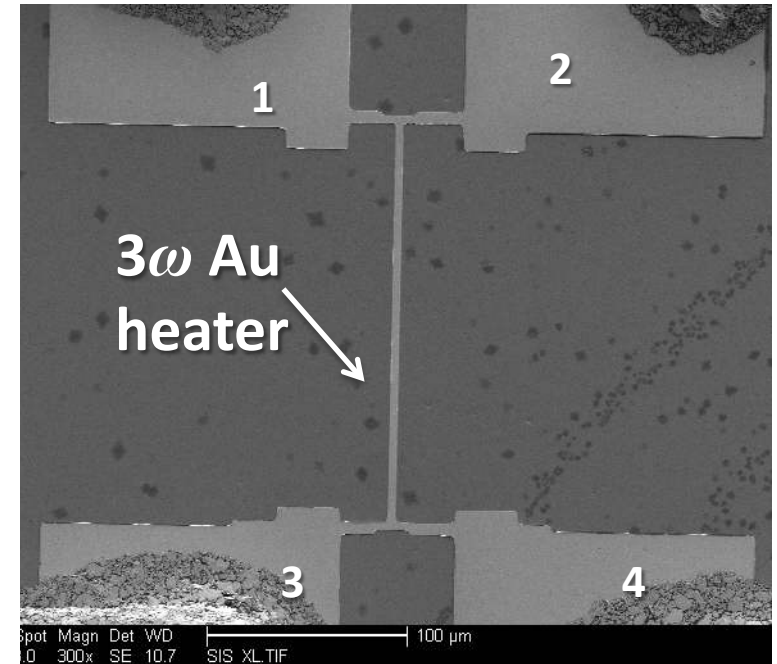
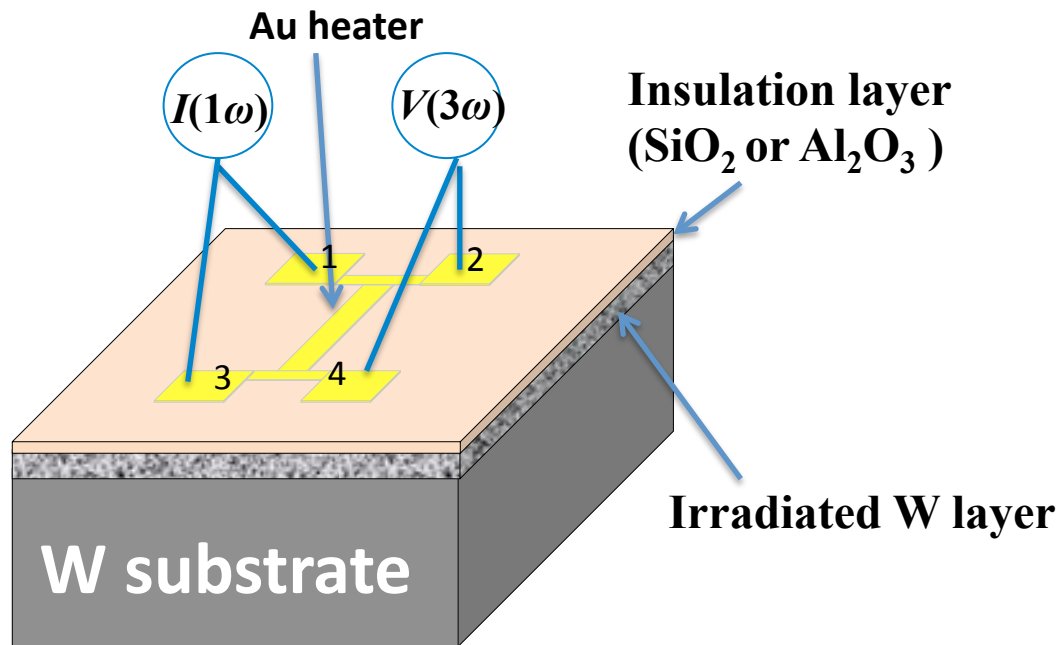
Lack of 'pop-ins' after irradiation indicates significant defect density in the indentation zone



Thin Film Thermal Conductivity Measurement

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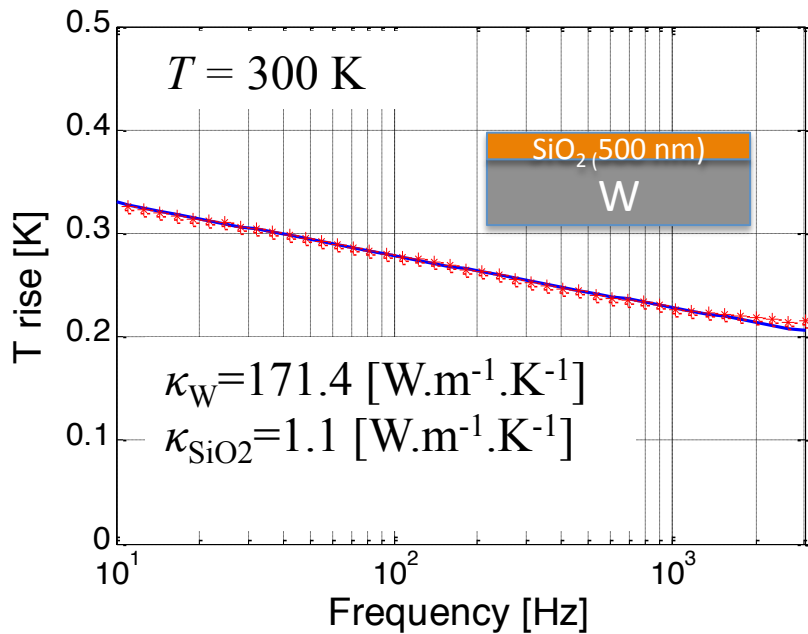
3ω method



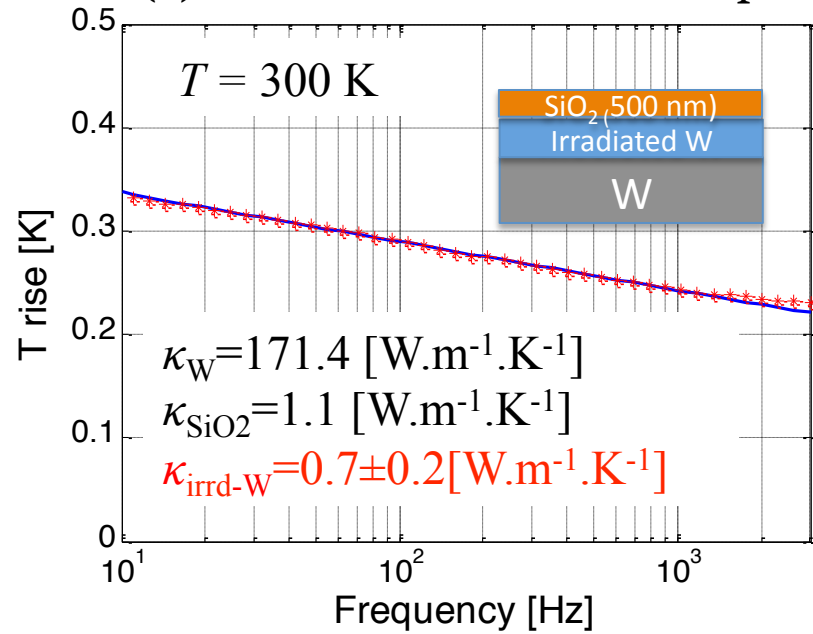
- Apply $I(\omega)$
- T oscillates at 2ω by Joule heating ($Q = I^2R$)
- R oscillates at 2ω ($R = R_0 + \alpha T$)
- Can measure T rise from $V(3\omega)$
 - $V_{3\omega} = I(\omega)R(2\omega)$

3 ω Data Reduction Method

(1) Reference sample



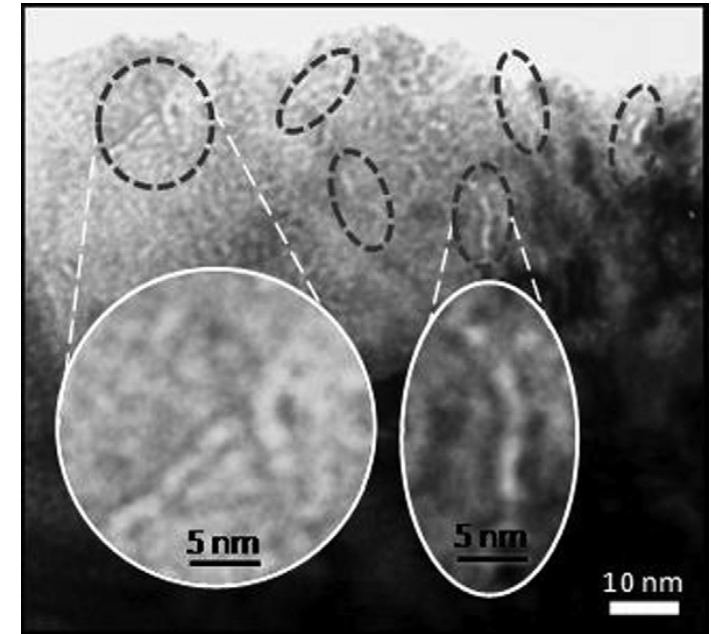
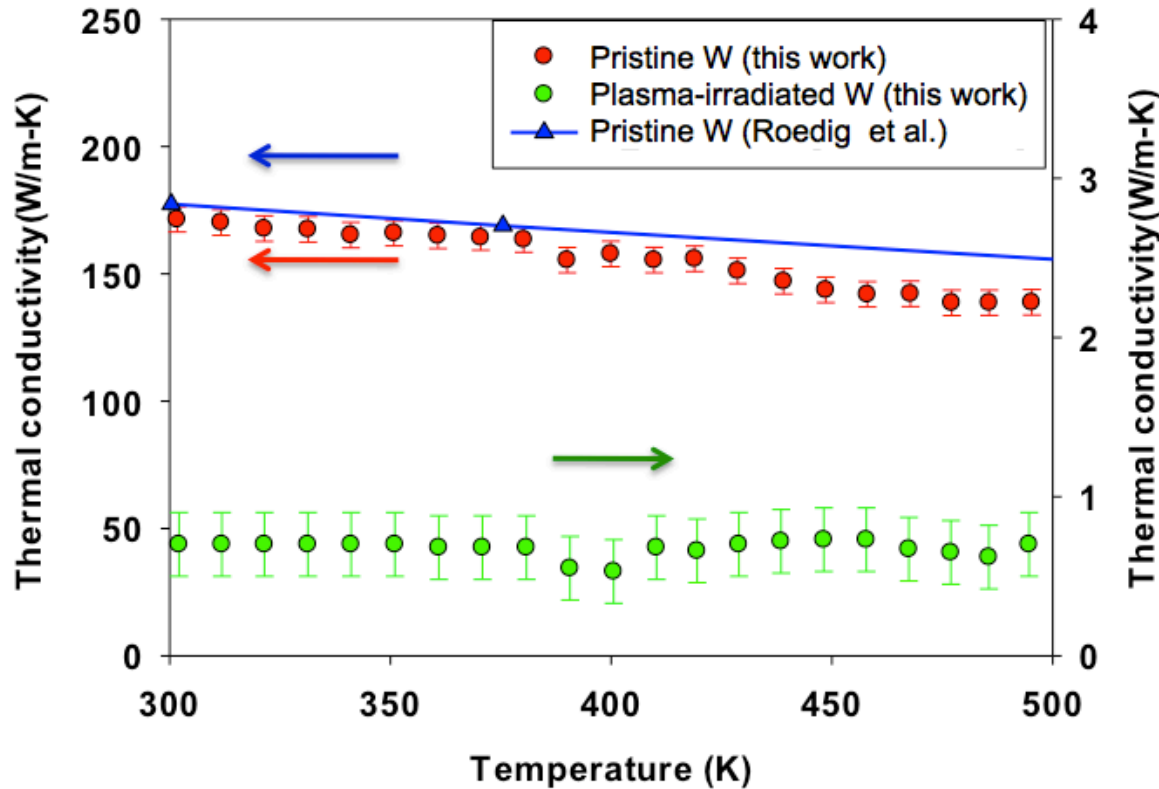
(2) He Plasma Irradiated sample



1. Fit the experimental data (triangle symbols) to a 2D heat transfer model (solid line)
2. Obtain parameters for the reference sample (κ of W substrate & SiO₂ layer)
 - The obtain κ values agree well with the literature values. ($\kappa_{\text{W}} = 174 \text{ W/m-K}$, $\kappa_{\text{SiO}_2} = 1.1\text{-}1.2 \text{ W/m-K}$, see J. Appl. Phys. **81** (6) 2590 (1997))
3. Apply the parameters from the reference sample to the fitting of the irradiate sample to obtain the only unknown parameter, namely, κ of the irradiated layer.

Thermal Conductivity of Plasma(He)-Irradiated Tungsten

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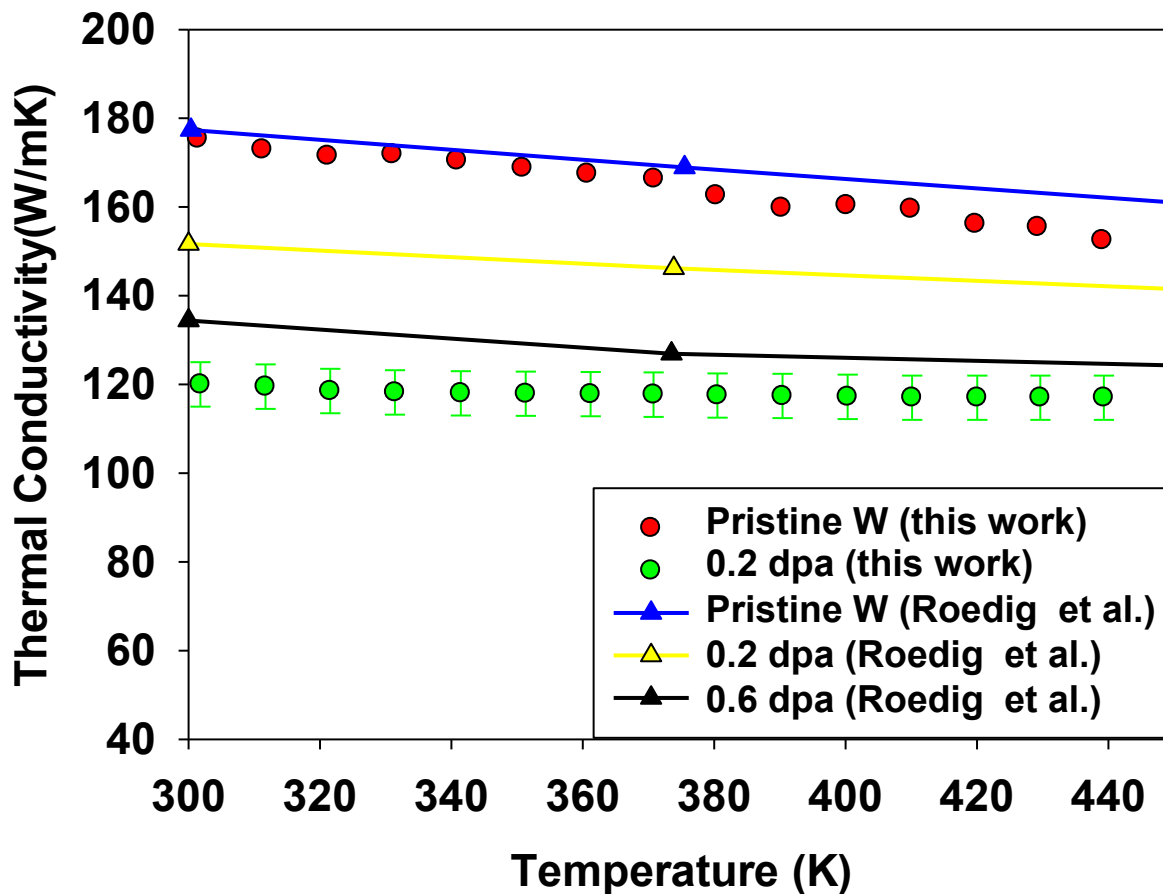
M. Miyamoto et al., JNM 415 (2011) S657

- κ of plasma-irradiated W ($0.7 \pm 0.2 \text{ W}\cdot\text{m}^{-1}\text{K}^{-1}$) is much lower than that of pristine W, presumably due to the defects formed during the irradiation.
- Between 300 and 500 K, κ of the plasma-irradiated W is independent of the temperature, also indicating that the electron scattering is dominated by the defects rather than phonon.

Ref: M. Roedig et. al , J. of Nucl. Mater. **329–333** (2004) 766-770

Thermal Conductivity of Ion-Irradiated Tungsten

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$\kappa_{\text{irrd}_0.2\text{dpa}} = 120 \pm 5$ [W.m⁻¹K⁻¹]
(~30% lower than pristine W)
and independent of temperature,
indicating defect formation after
ion irradiation of 0.2 dpa dose.

This value is somewhat lower
than that what was reported in
Roedig *et al.* (~150 W/m-K for
0.2 dpa irradiation), which used
the laser flash method.

Ref: M. Roedig et. al , J. of Nucl. Mater. **329–333** (2004) 766-770

Plasma exposure is used to measure impact of damage on D migration

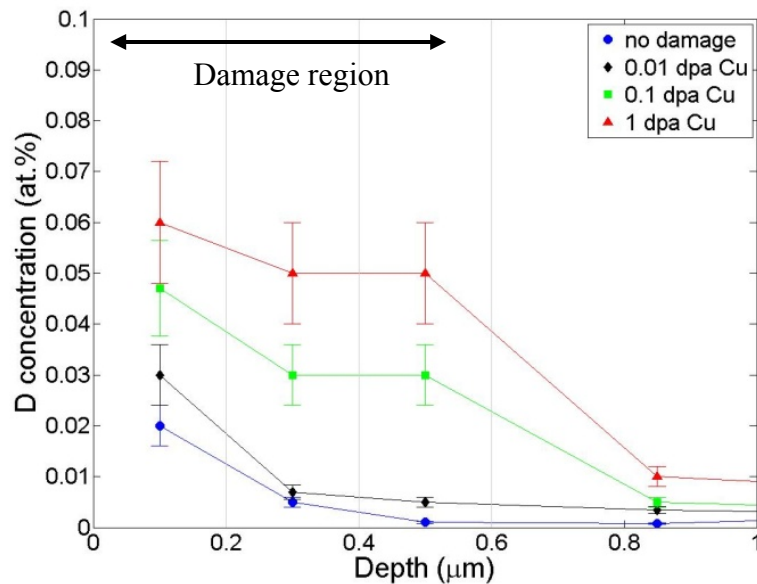
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- Targets damaged with ions at LANL are brought to PISCES for plasma exposure
- After plasma, targets are returned to LANL for D depth profiling using NRA
- Finally, targets are returned to UCSD for TDS determination of total D content in the sample bulk

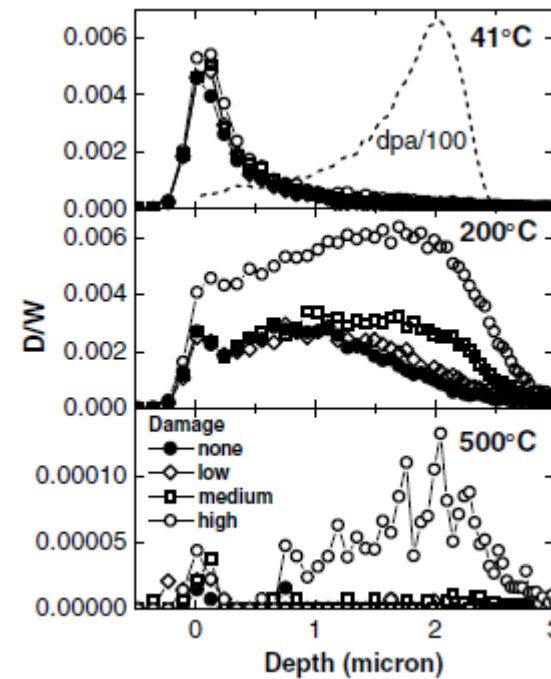
Plasma implanted D accumulates in damaged regions

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Retention increases with increasing damage level



Retention behavior of self-damaged W and W damaged by Cu ions begins to deviate at around 1 dpa.



12 MeV Si⁺

Low = 0.006 dpa

Medium = 0.06 dpa

High = 0.6 dpa

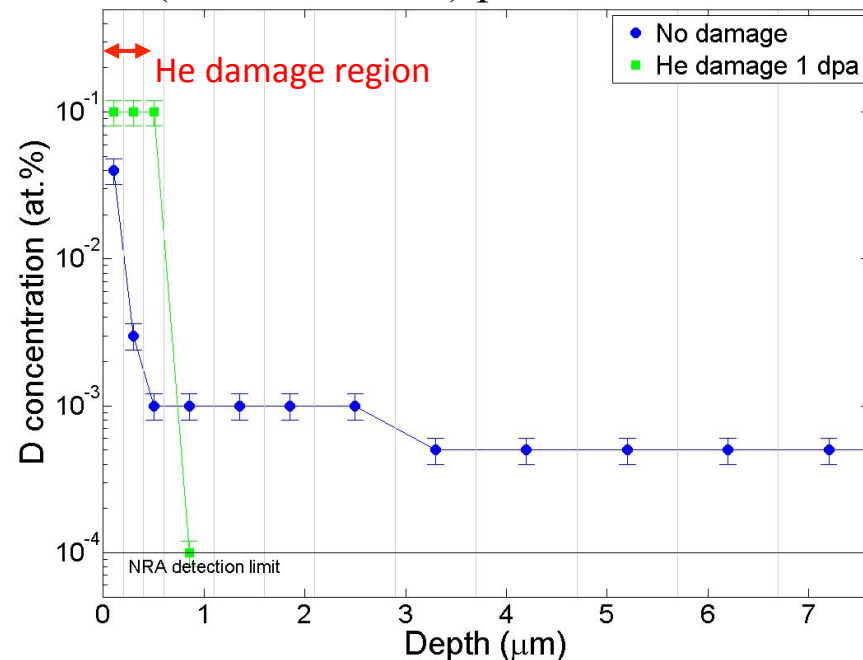
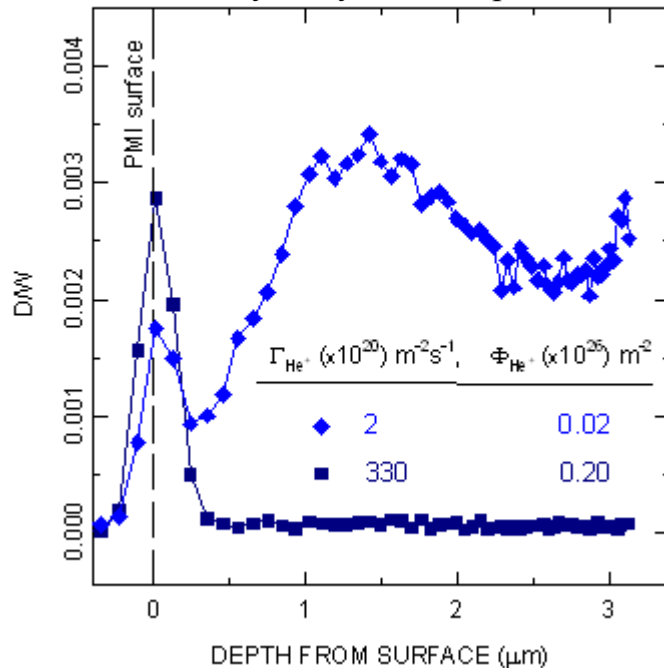
From: W. Wampler and R. Doerner, NF 49(2009)115023

He plasma pretreatment requires large He^+ flux to inhibit D migration into the bulk, while high energy He^+ does not

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200 keV He^+ to a peak 1 dpa
($5 \times 10^{20} \text{ He}^+/\text{m}^2$) pretreatment

NRA analysis by W. Wampler, SNLA



- Plasma ion energy was below W damage threshold, so helium concentration in W needs to be large enough to favor agglomeration and bubble growth (i.e. high flux necessary).
- 200 keV He ions can create and populate damage sites resulting in similar effects at much lower flux

Status and summary

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- 3 year project, year 1 recently completed
- Staffing, sample preparation and setup are all completed
- Initial results from nano-indentation and 3ω thermal measurements verify the capability of measuring the thermo-mechanical properties of thin damaged layers
- Preliminary results incorporating He into damaged regions seem to support basic principles of D/He interactions in tungsten and highlight the importance of helium production due to neutron bombardment