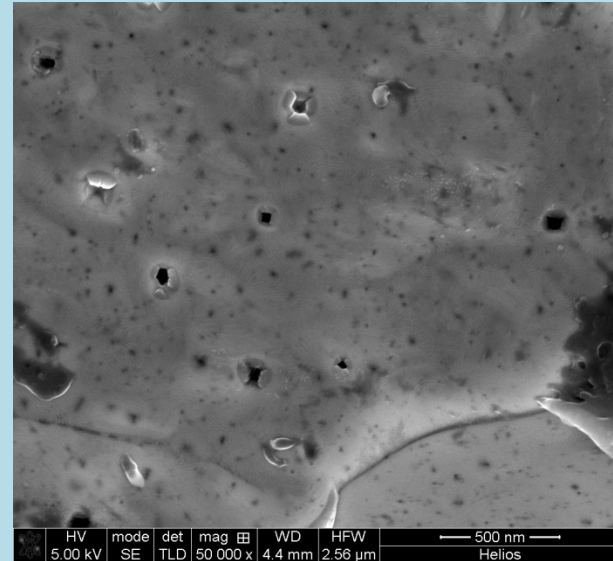
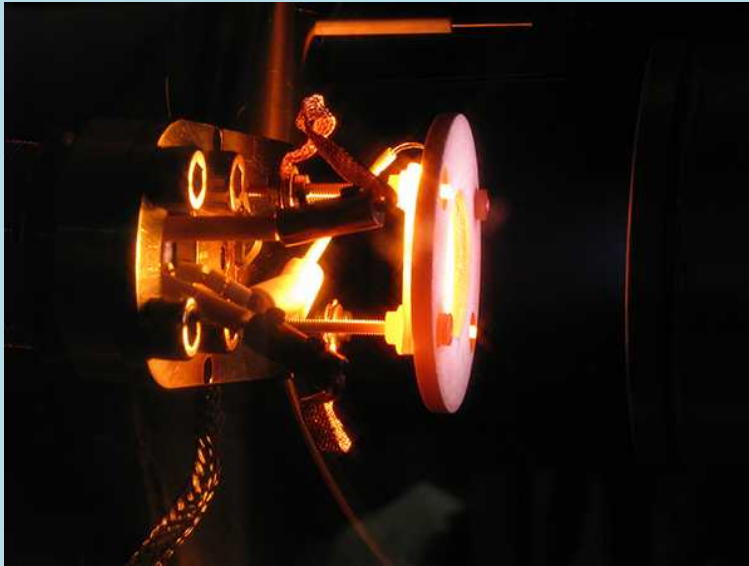


Characterization of helium bubble growth in tungsten exposed to low-flux plasmas

SAND2014-4352C



David C. Donovan

Japan-US Workshop on Heat Removal and Plasma-Materials
Interactions for Fusion – Kyoto University – June 4, 2014

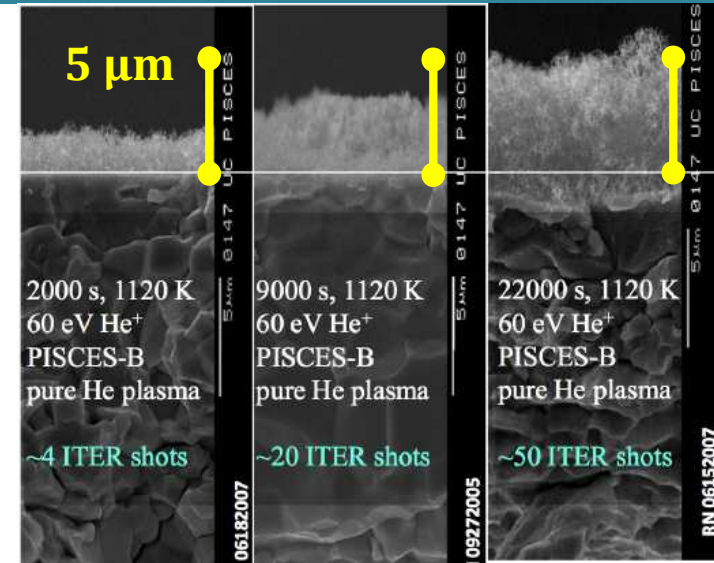


**Sandia
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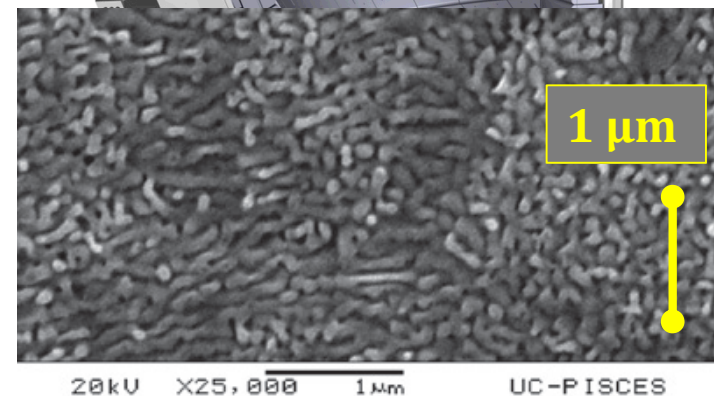


Tungsten will be the divertor material for ITER

- Tungsten chosen for high melting point, thermal conductivity, low erosion.
- He ion damage to tungsten can cause tendril growth.
- Tendrils are 10-50 nm in diameter and can grow to at least 5 microns in length.
- **Problem**: Erosion of tendrils will create prohibitively large amounts of tungsten contaminants in the fusion plasma.

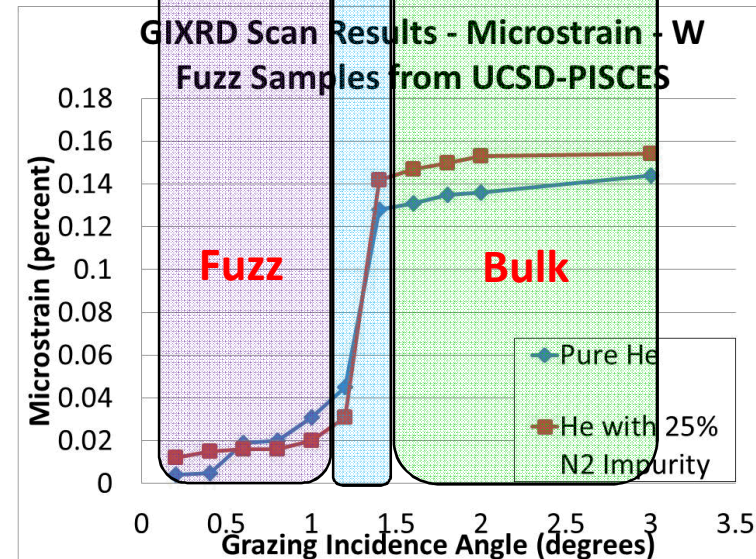
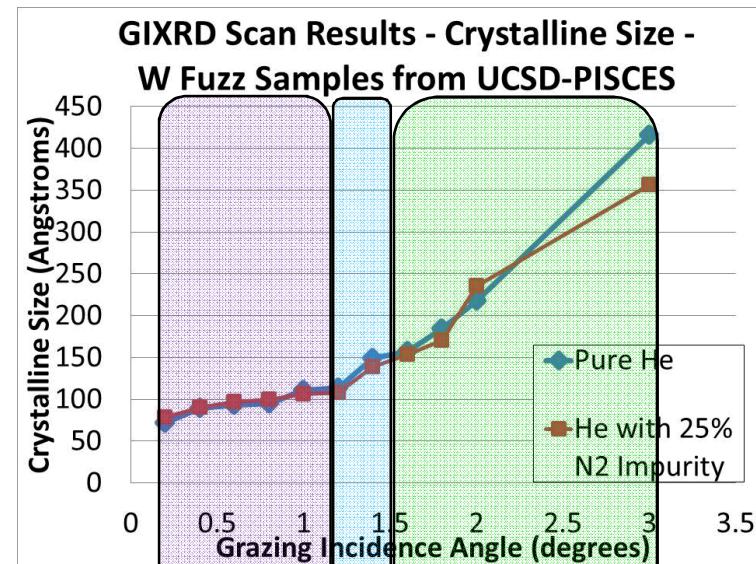
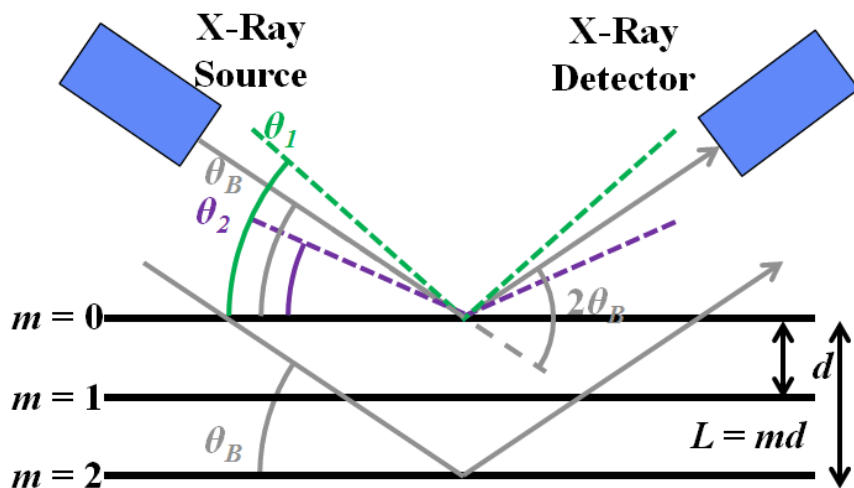


W sample exposed to high flux linear He ion beam at UCSD.



X-Ray Diffraction performed on W samples exposed to high flux He ion beam at UCSD

- Grazing Incidence XRD is used to measure near surface ($\sim 1\ \mu\text{m}$) conditions including microstrain and crystalline size.
- Previous theories had predicted high strain in the tendrils due to He implantation.
- GIXRD measurements showed little to no strain in fuzz layer.
- Tendril growth may be a mechanism to escape the strain in the bulk.



Low flux He ion beam used to bombard W samples at Sandia

Experimental Setup

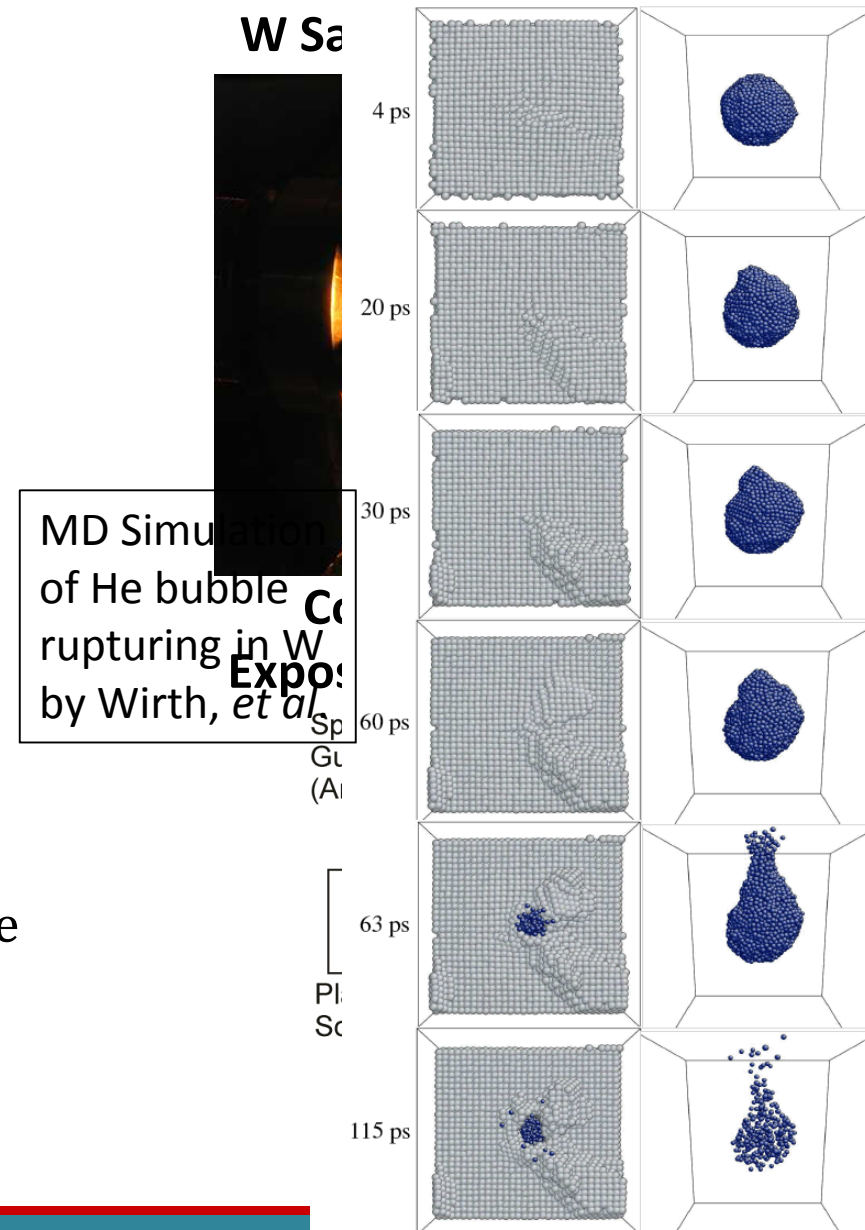
- An Electron Cyclotron Resonance (ECR) Source is used to ionize and accelerate He atoms.
- The low flux ion beam bombards a W target on a heater stage capable of reaching 1000 °C.

Next Step

- Install characterized ECR source on Scanning Tunneling Microscope (STM) vacuum vessel.
- Study early stages of tendril growth.

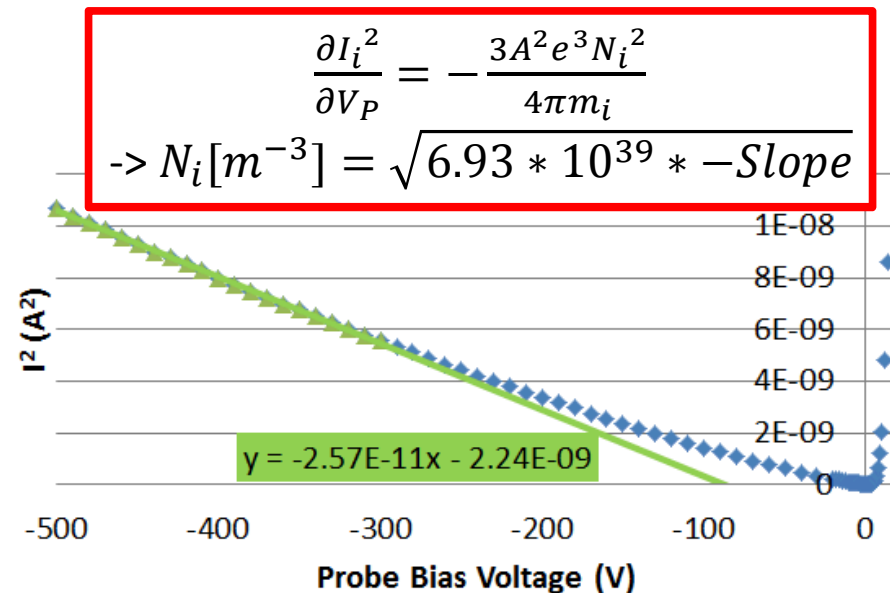
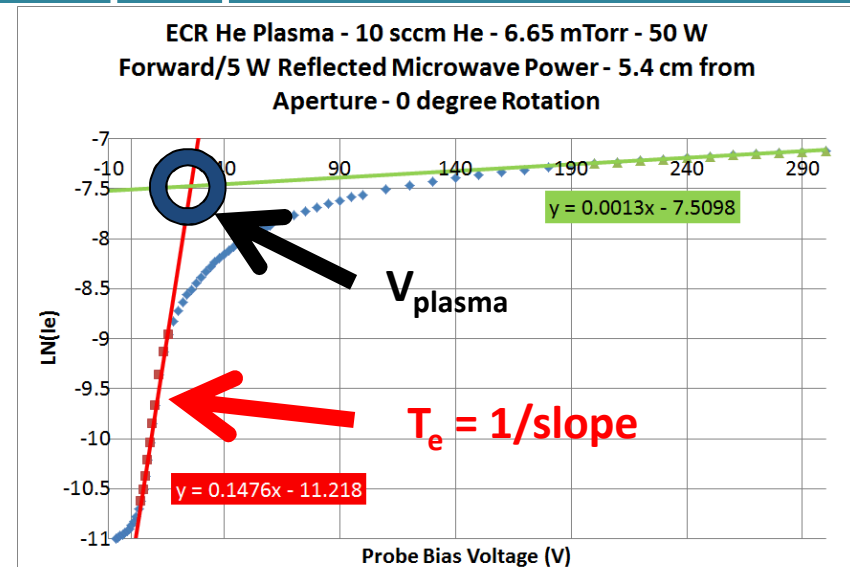
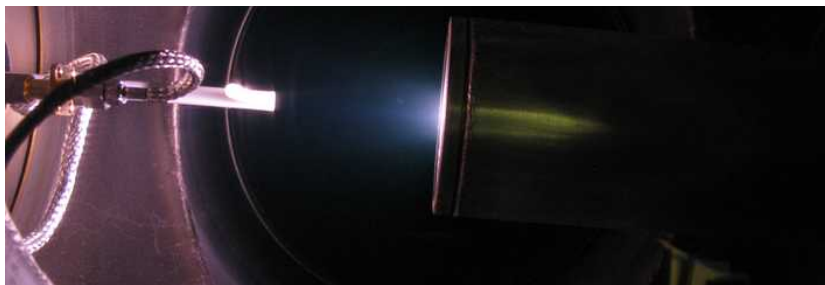
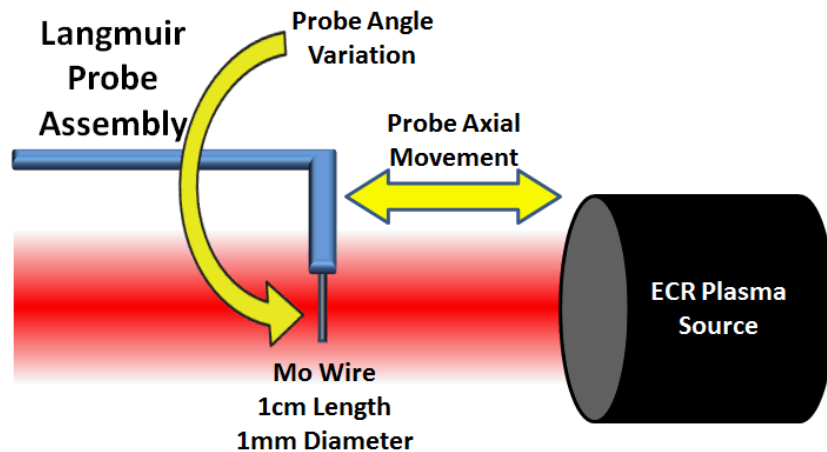
Goal

- Provide experimental analysis to contribute to MD simulations of He bubble and tendril growth in W.
 - Brian Wirth (UT-Knoxville)
 - Sergei Krashennnikov (UCSD)



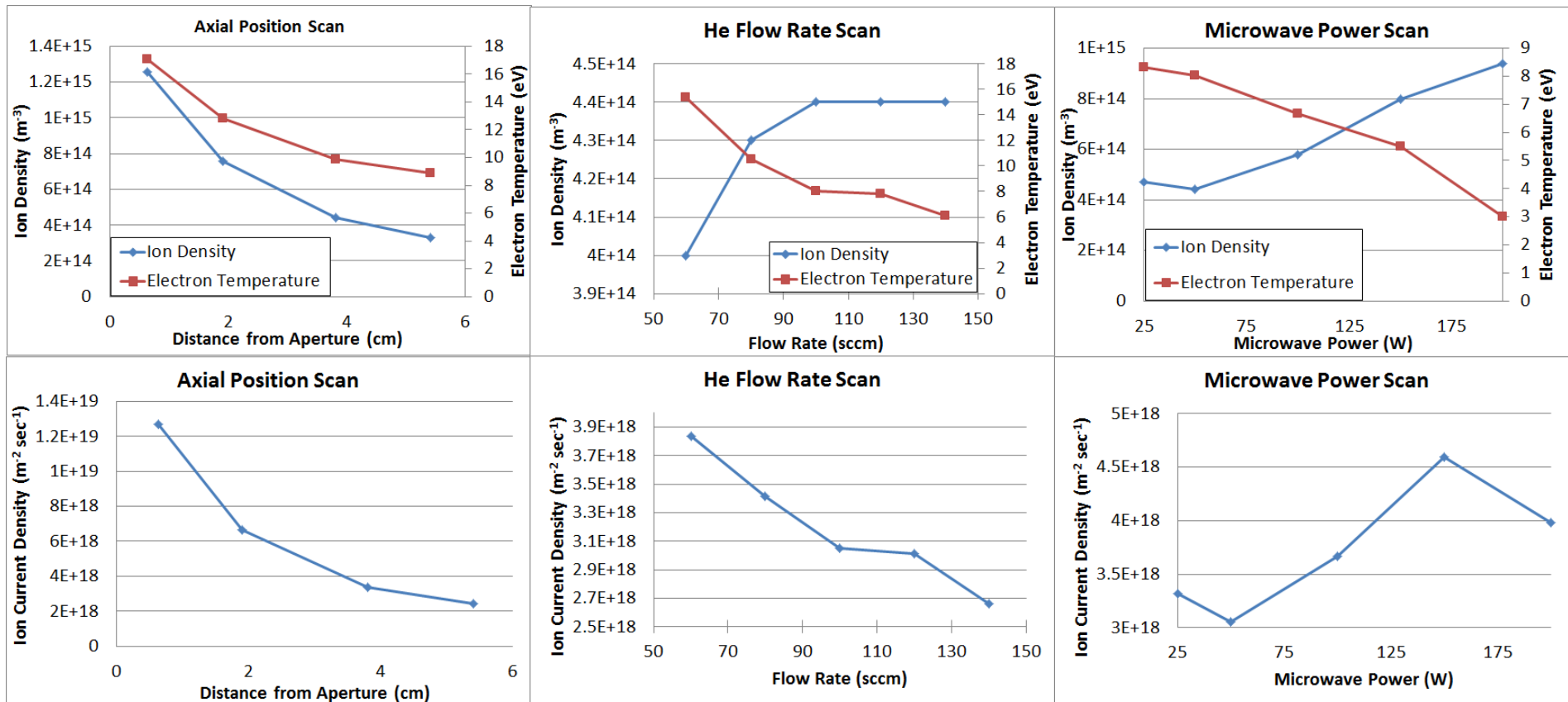
Langmuir probe was used to create I-V traces to determine plasma properties

- **Langmuir Probe (LP)** used to determine plasma density, electron temperature, and ion flux.
 - Measurements taken while varying pressure, microwave power, magnetic field strength, distance from source.



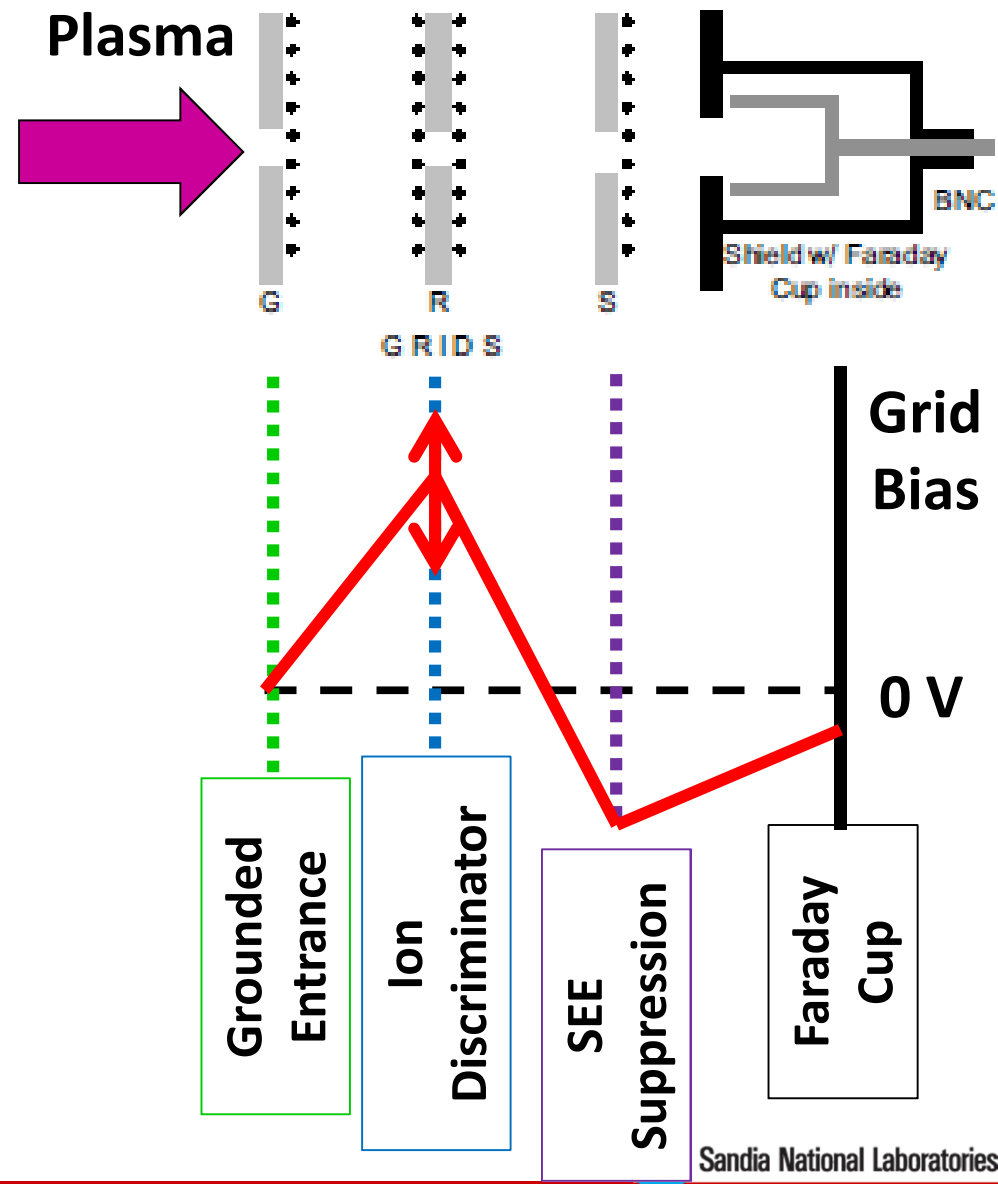
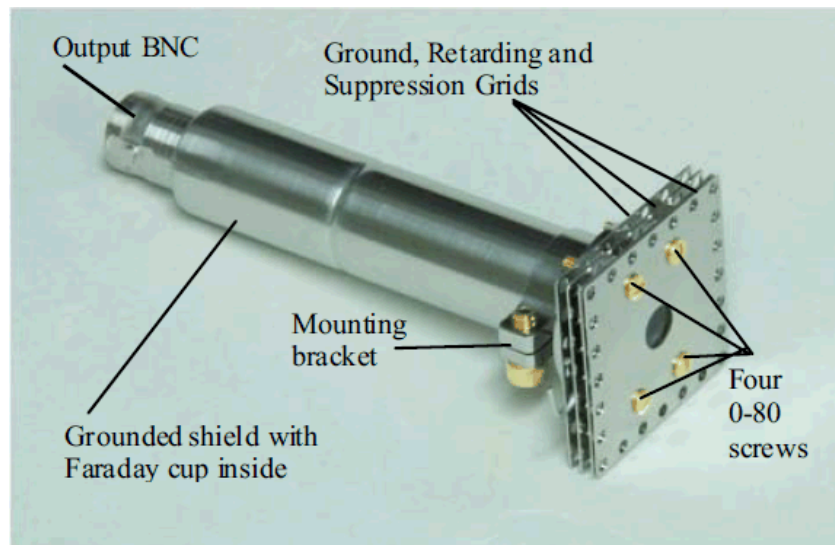
Langmuir probe studies used to determine plasma properties of the ECR plasma source

- Initial exposures are able to be placed close to the ECR source aperture.
- Future exposures on in-situ diagnostic platforms will involve greater separation between sample and source, requiring an understanding of plasma parameters as a function of distance from the source.



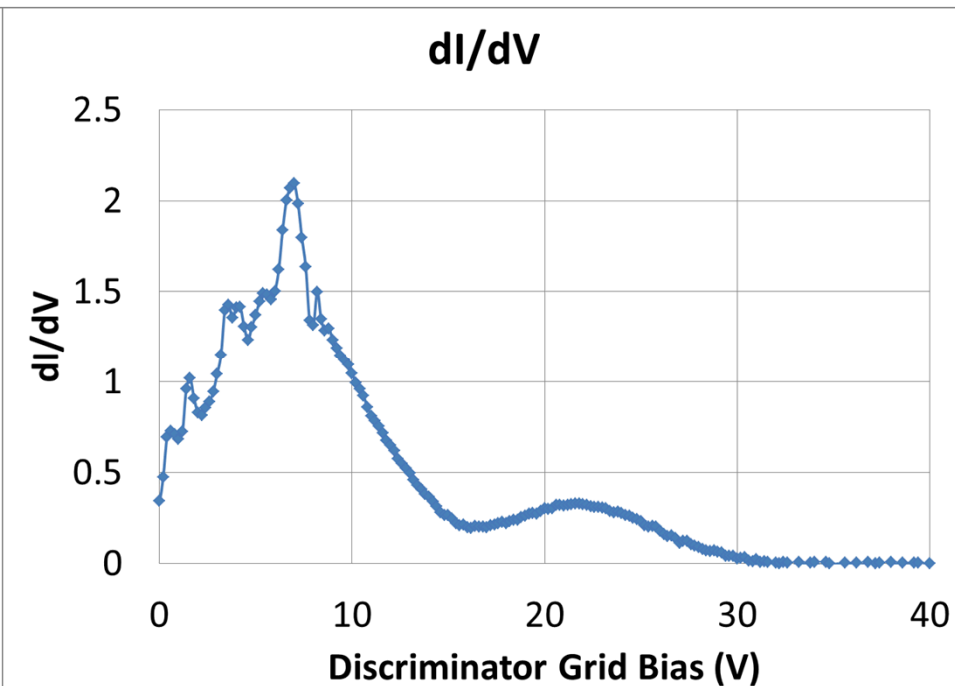
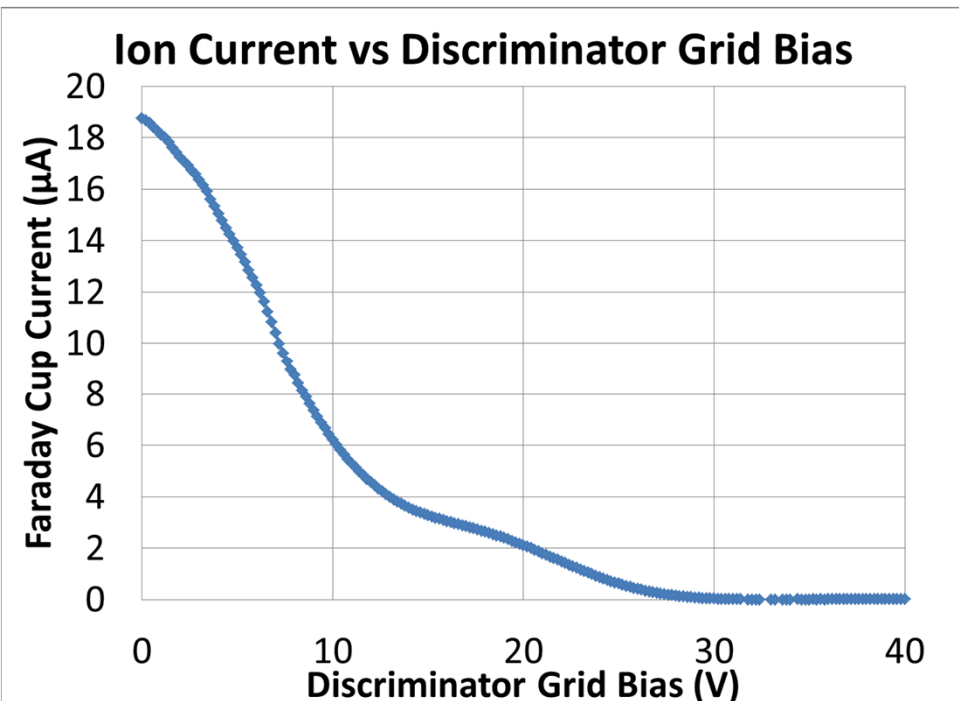
Ion energy spectra obtained using Retarding Field Analyzer (RFA)

- RFA uses biased grids to repel ions below an applied potential threshold and collects ions above the threshold.
- By varying the potential of the threshold, the ion energy spectrum can be discerned.



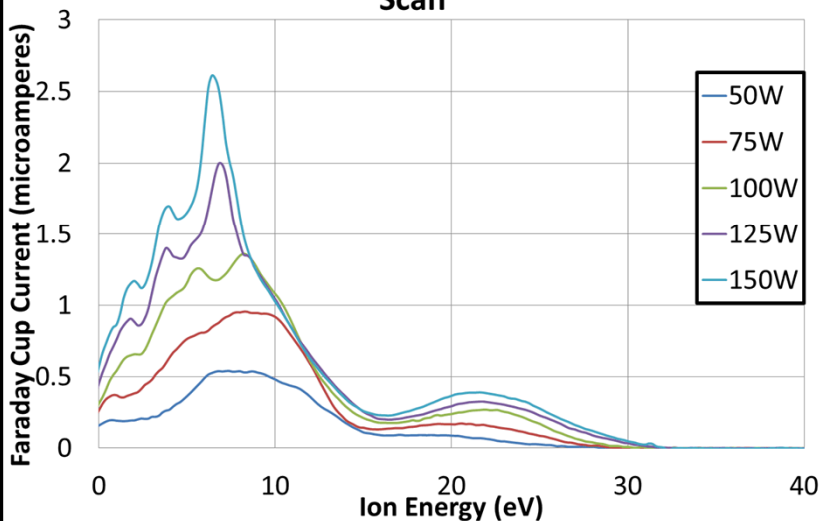
Ion energy spectra obtained by differentiating current collected on Faraday cup

- Secondary Electron Emission Grid is set to a sufficiently negative potential to block all electrons from reaching the Faraday cup and reflect SEE.
- All Faraday cup current is therefore ion current (I_{ion})
- Discriminator Grid bias (V_D) is increased until current on Faraday cup current reaches zero.
- Differentiating Faraday cup current as a function of V_d provides the incoming ion energy spectra.

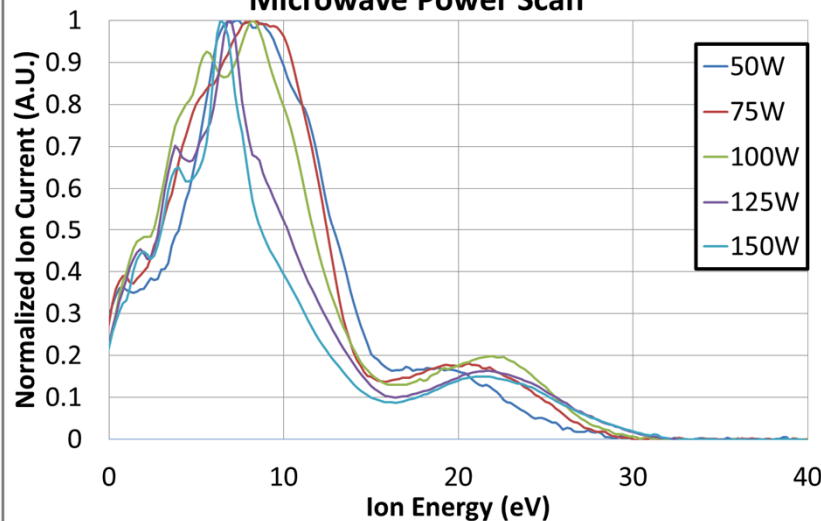


RFA Ion Energy Spectra collected while varying Microwave Power and Distance from Source

RFA Ion Energy Spectra - Microwave Power Scan

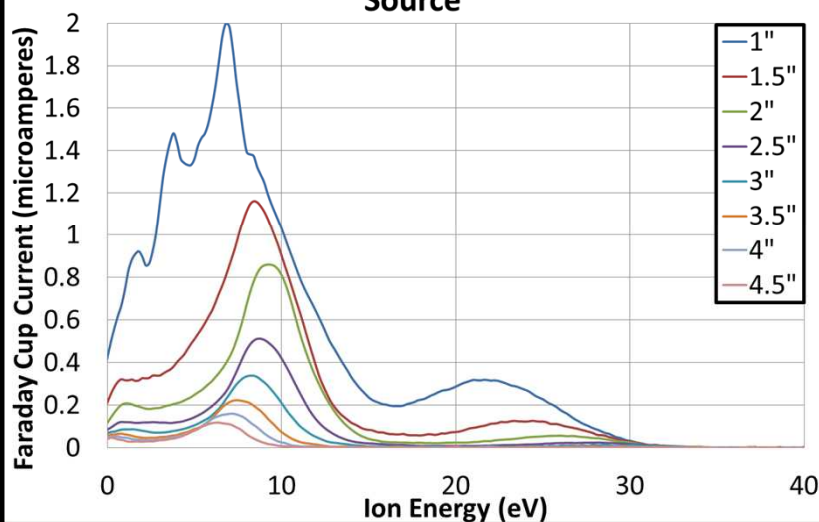


RFA Normalized Ion Energy Spectra - Microwave Power Scan

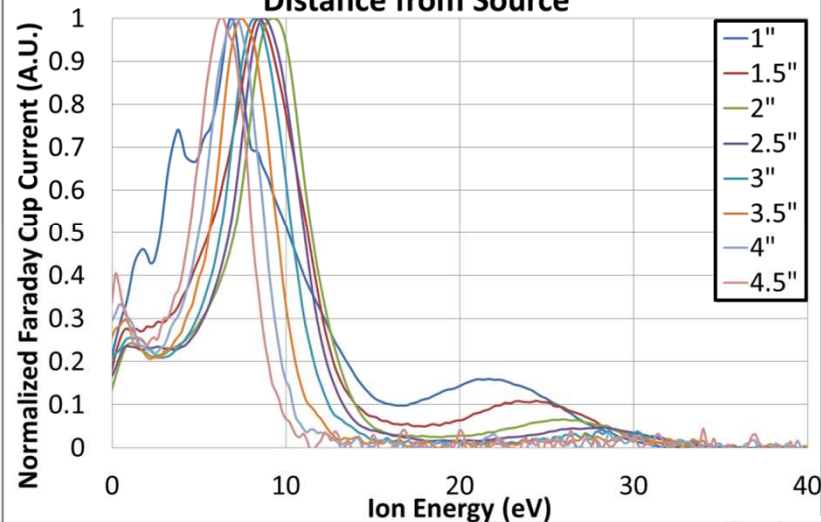


Ion energy decreases slightly with increasing microwave power

RFA Ion Energy Spectra - Distance from Source



RFA Normalized Ion Energy Spectra - Distance from Source

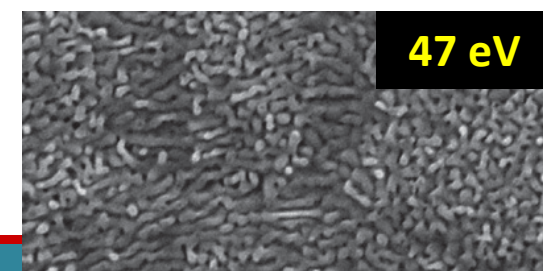
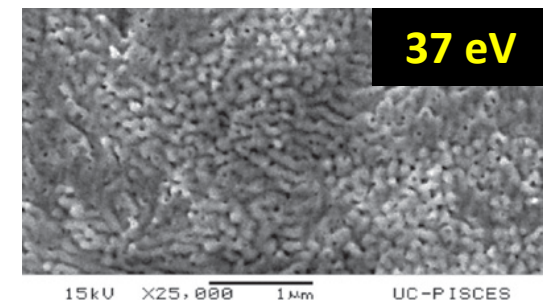
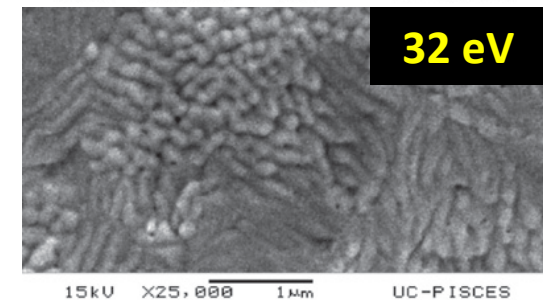
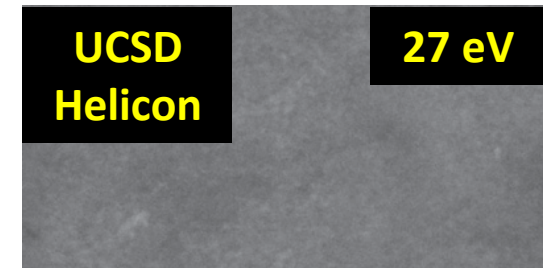


Ion energy decreases slightly with distance from source

Operating conditions for ECR plasma source were based on previous studies at UCSD

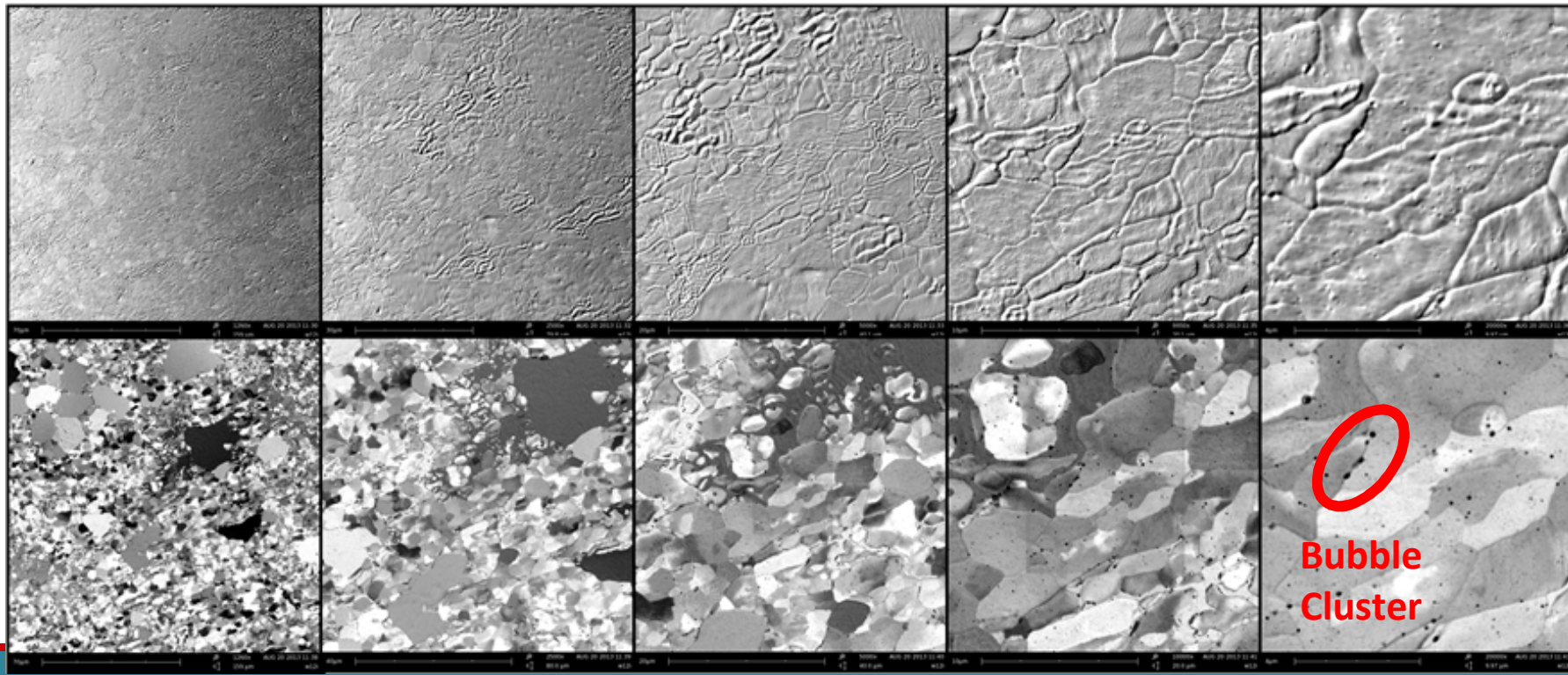
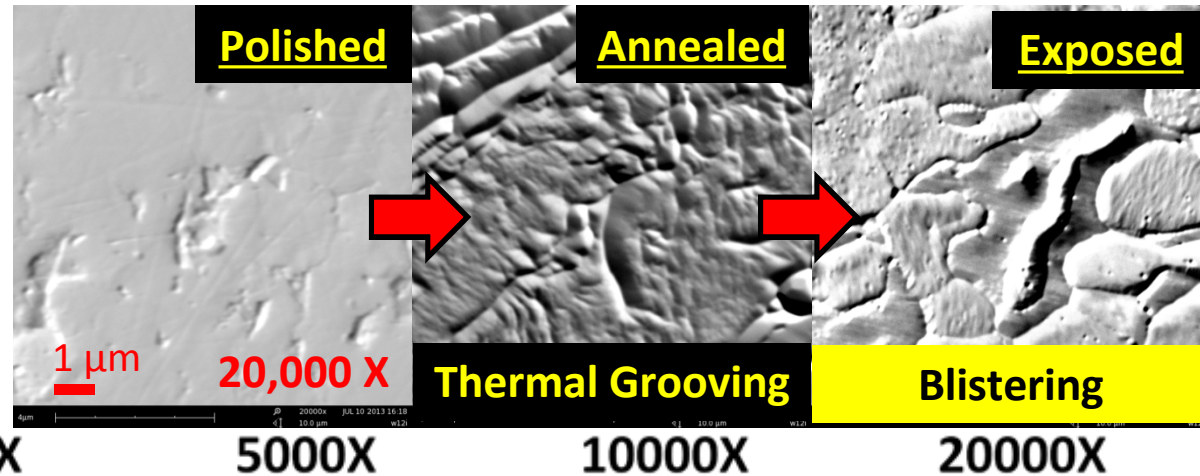
- Baldwin, et al. at UCSD used a low pressure rf Helicon source to grow W fuzz.
 - Minimum conditions for surface morphology change were $T_{sample} > 900\text{K}$ and $T_{ion} > 27\text{ eV}$
 - Baldwin, et al., JNM, 2010
- SNL chose an ECR plasma source to offer a lower flux to study early stages of morphology change.
 - ECR source also offers much lower T_e than traditional keV magnitude ion beam sources.

	UCSD Helicon	SNL ECR Plasma
Feed Gas	He	He
Temperature (K)	1120	1270
Sample Bias (V)	0 to -33	-20
Ion Flux ($\text{m}^{-2} \text{sec}^{-1}$)	4.0×10^{20}	2.5×10^{19} (~6%)
Total Fluence (m^{-2})	8.6×10^{24}	3.2×10^{24} (~40%)
Exposure Time (hrs)	6	40 (non-consecutive)
Ion Temp (eV)	27 to 57	28

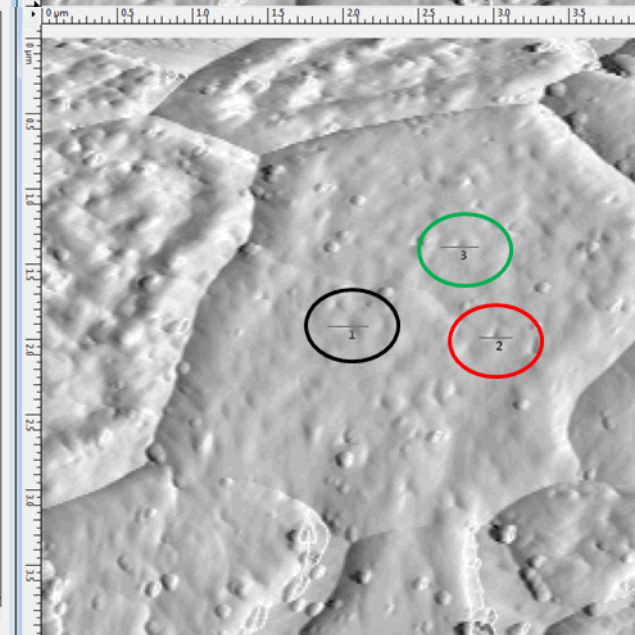
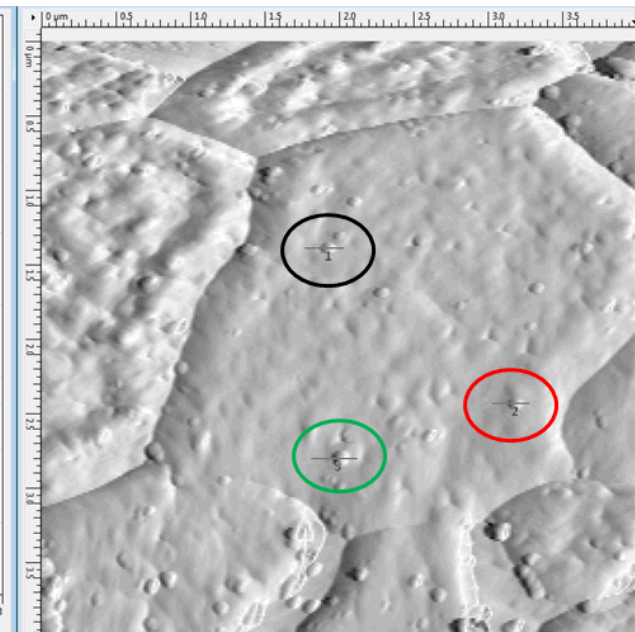
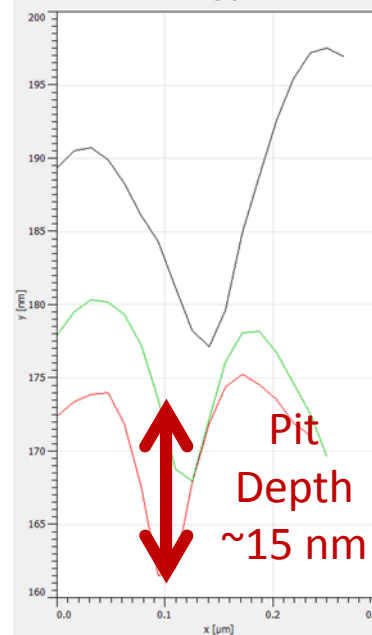
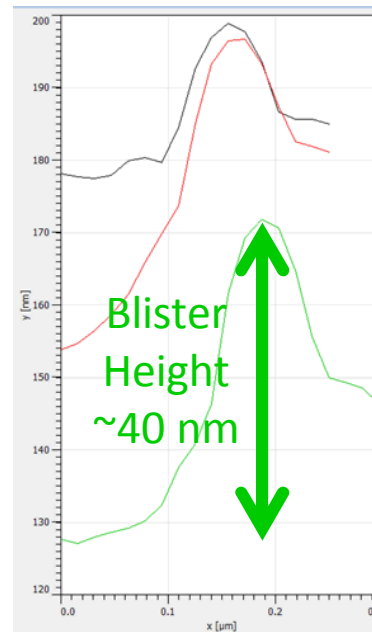


Plansee W disc at 1000°C exposed to low flux He ion beam from ECR source for 40 hours at SNL

- SEM shows no tendrils, but did create bubbles.
- Bubble clusters found near grain boundaries and single blisters found within grains.

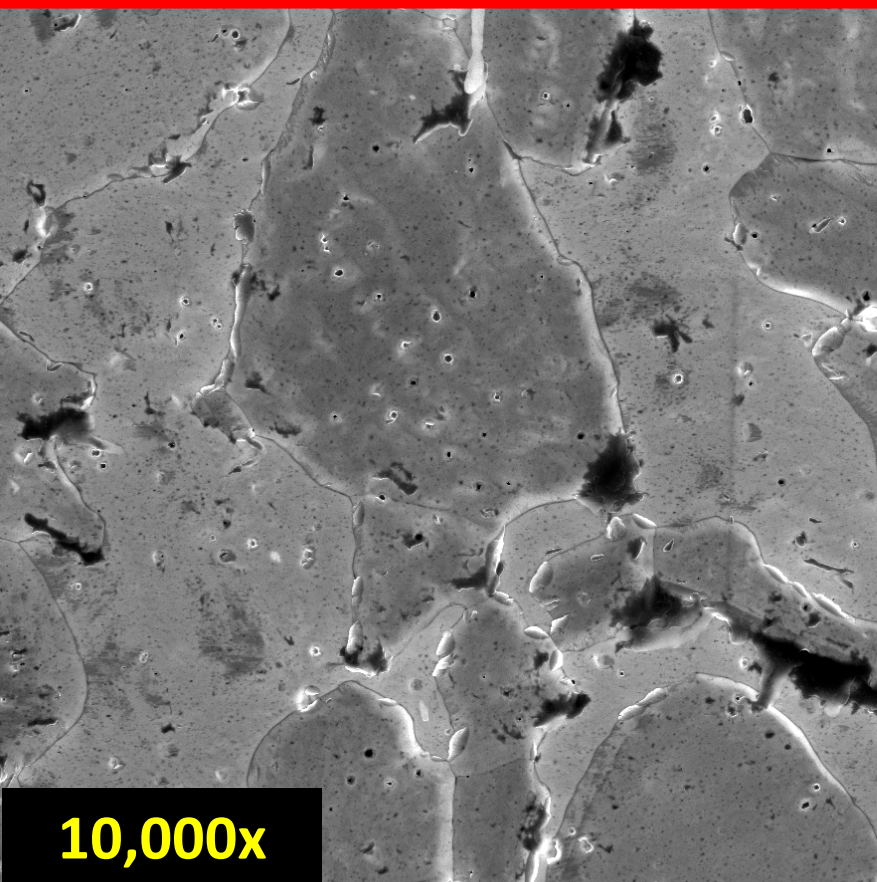


**Atomic Force
Microscopy (AFM)**
scan shows bubbles
are up to 150 nm in
diameter, 40 nm high
and the pits are up to
15 nm in depth

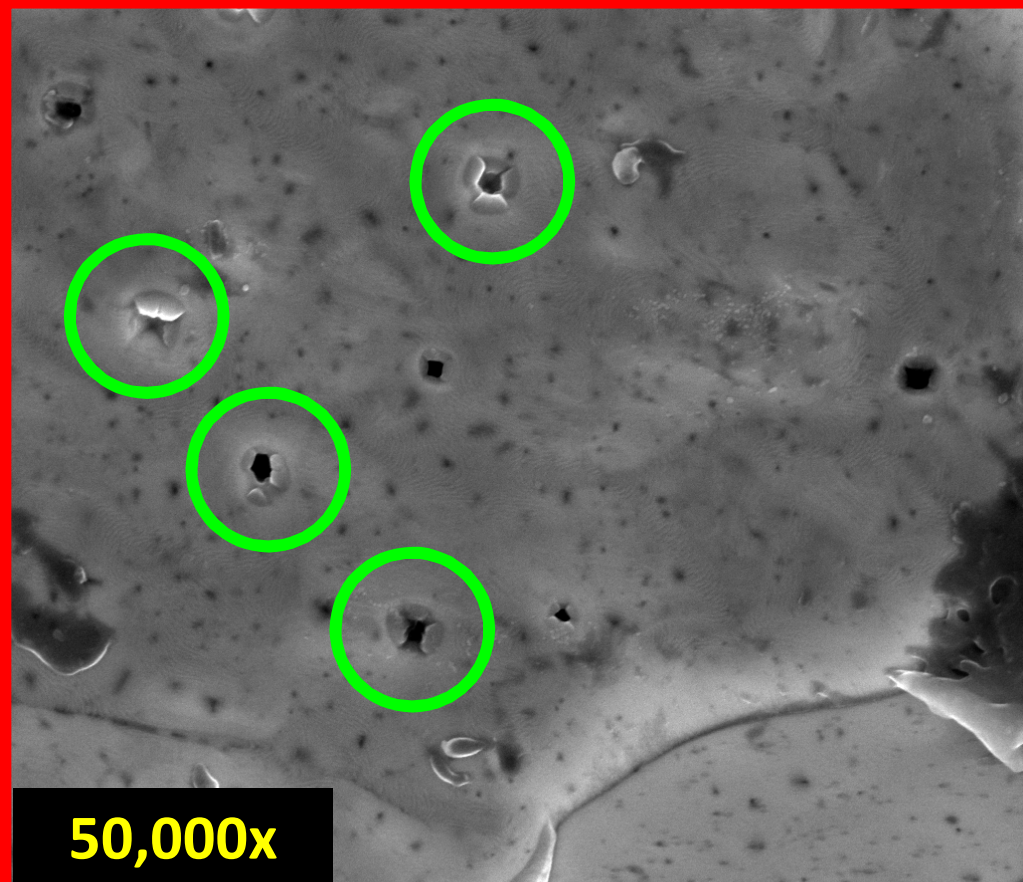


High resolution SEM images of He bombarded Plansee W shows remnants of burst blisters

- Images taken by Graham Wright at MIT.
- Blisters are ~ 150 nm in diameter.
- Blisters are found within varying amount of their caps fractured or blown off.



	HV	mode	det	mag	WD	HFW	
	5.00 kV	SE	TLD	10 000 x	4.4 mm	12.8 μ m	4 μ m Helios



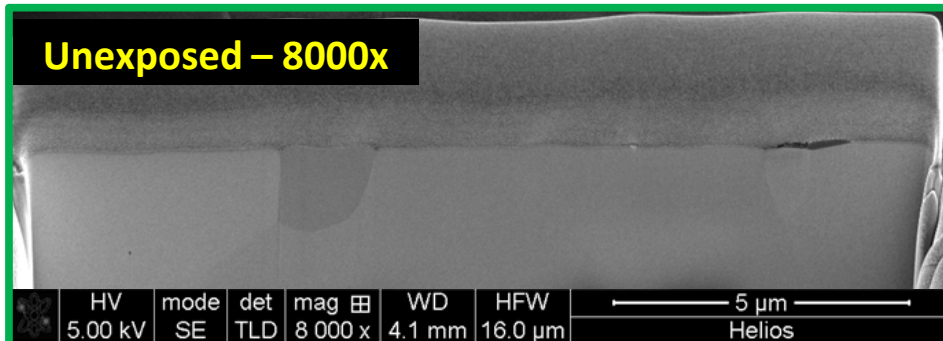
	HV	mode	det	mag	WD	HFW	
	5.00 kV	SE	TLD	50 000 x	4.4 mm	2.56 μ m	500 nm Helios

Focused Ion Beam (FIB) cross sections show bubbles forming below the surface

- FIB images taken by Graham Wright at MIT.
- Comparisons made between Plansee W and ITER-Grade W.
 - ITER-Grade W has grains aligned perpendicular to the surface to maximize heat conduction.
- Sub surface bubbles grow up to 100 nm in diameter.

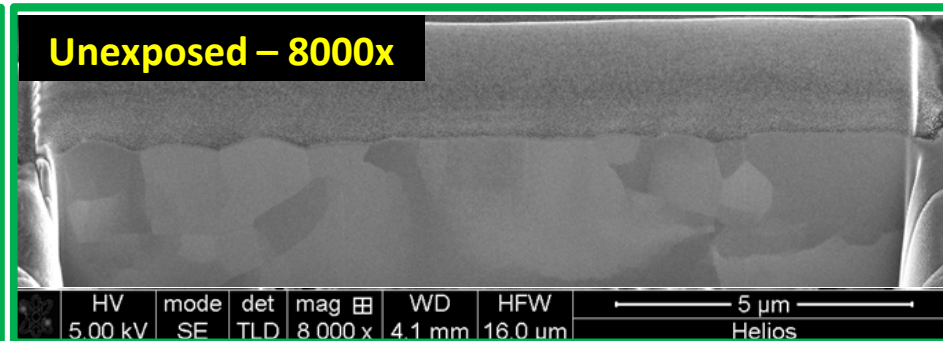
Plansee W

Unexposed – 8000x

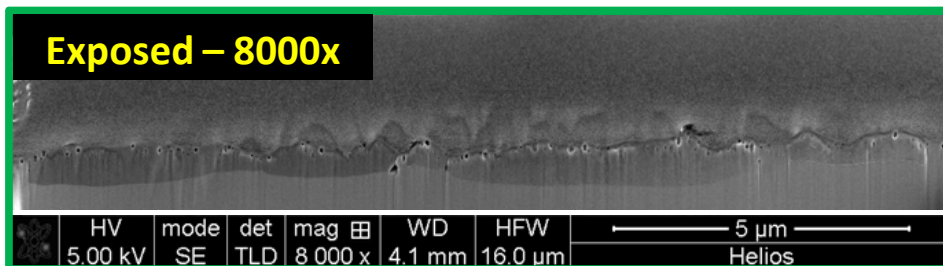


ITER-Grade W

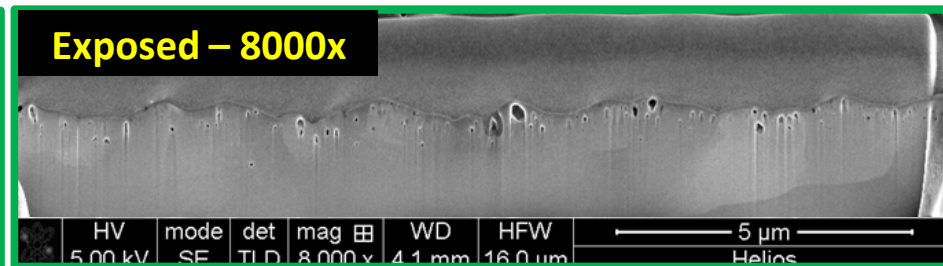
Unexposed – 8000x



Exposed – 8000x



Exposed – 8000x

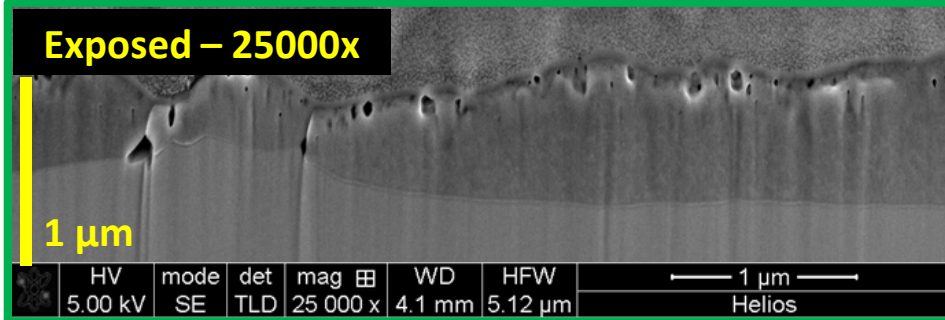


Bubbles form at greater depths in ITER-Grade W as opposed to Plansee

Plansee W

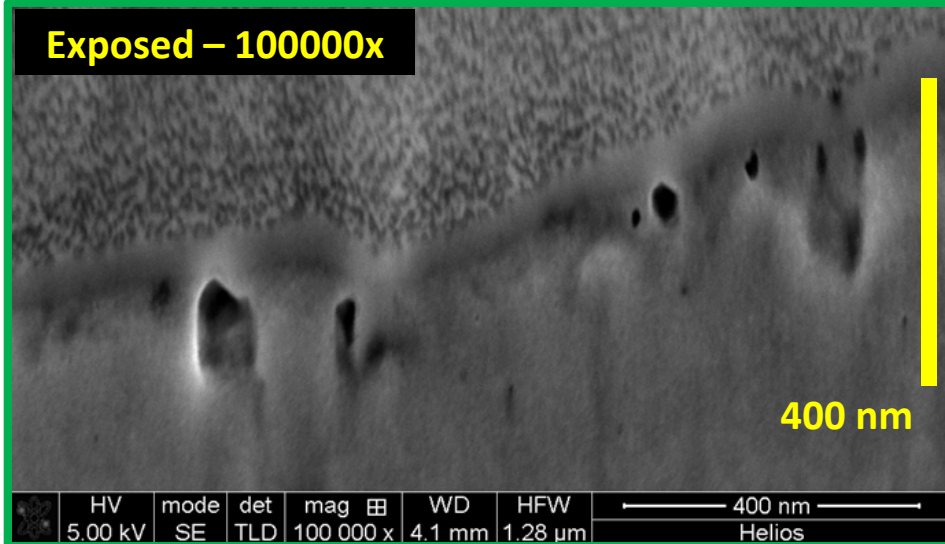
Exposed – 25000x

1 μm



Exposed – 100000x

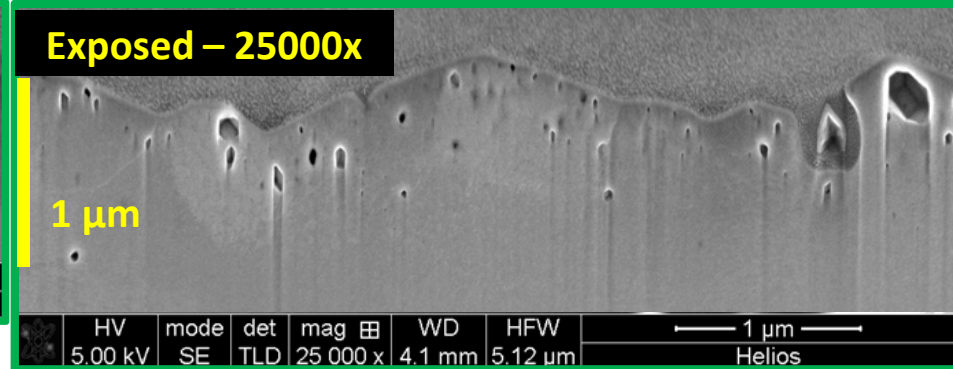
400 nm



ITER-Grade W

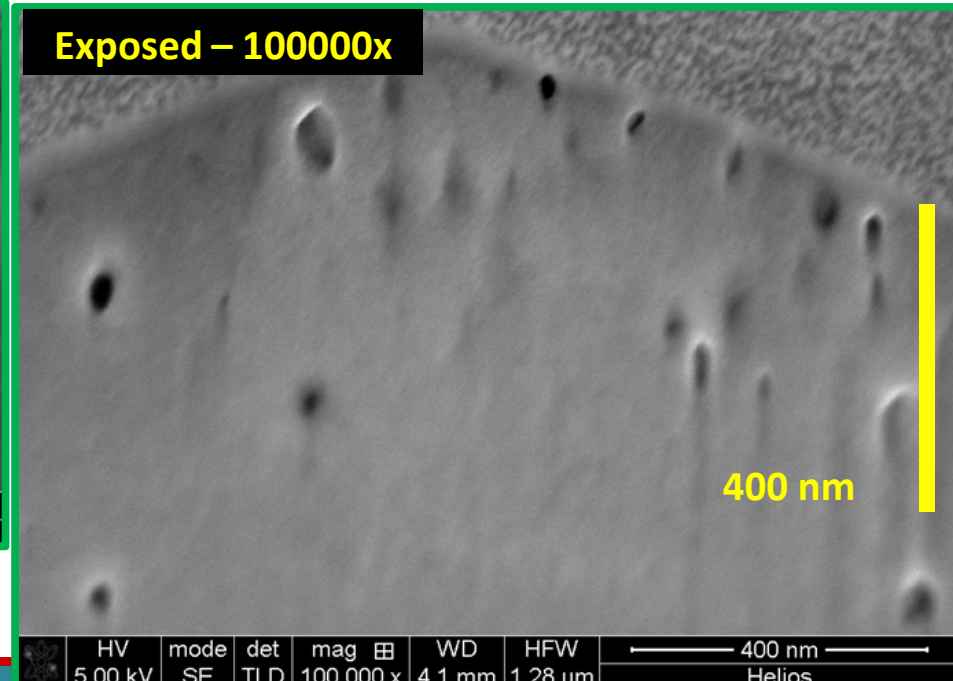
Exposed – 25000x

1 μm



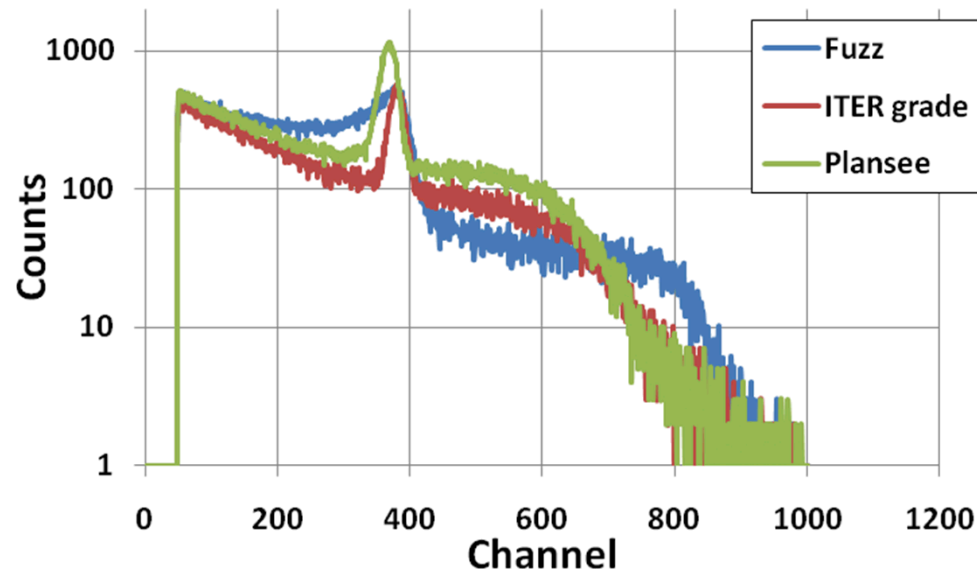
Exposed – 100000x

400 nm



Elastic Recoil Detection (ERD) performed at MIT to determine concentration of He as a function of depth

- ERD analysis performed on Plansee and ITER-Grade W samples from SNL and a fuzz sample from MIT.
- First 10-15 nm of bubble samples is the “deficit” layer with only 0.5 at% He.
 - Fuzz samples typically show higher He concentration in this region.
- Beyond 15 nm, He concentration in bubble samples quickly increases to 5-7 at%.
- Low He concentration in deficit layer may be an indication that the caps for bubbles within the first 15 nm are too thin to withstand the pressure in the bubbles and burst, releasing the trapped He.



Accomplishments on He Implantation Studies

- Performed GIXRD measurements of W fuzz samples from PISCES that have provided new insights into microstrain in the fuzz tendrils.
- Characterized and utilized a low flux He ion source for exposing heated samples.
 - Majority of ITER divertor will receive low flux of He, requiring a better understanding of various stages of surface damage.
- Performed SEM, AFM, FIB, ERD material analysis of exposed W samples and identified large He bubbles that form below the surface and create pits on the surface.
- Collaborated with material modeling groups to aid in determining a mechanism for these surface morphology changes to W.

Conclusions and Future Work

- Low flux He implantation of heated W samples results in the formation of surface blistering and sub-surface bubbles with diameters up to 150 nm.
- ITER-Grade W with planes oriented perpendicular to the surface form bubbles at greater depth than standard Plansee W.
- He concentration in the near surface (10-15 nm) is significantly lower than at greater depths, possibly due to burst blisters releasing high pressure He bubbles.
- **Future plans** involve exposing additional samples at lower sample temperatures, higher total fluence, higher ion energy to monitor various stages of bubble growth and determine threshold at which tendrils begin to grow.
- ECR plasma source will later be placed on an existing STM experimental stage at SNL to perform in-situ exposure and analysis of W samples to observe angstrom scale surface damage.

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- Richard Nygren
- Bill Wampler
- Jon Watkins

Sandia Sample Analysis

- Ray Friddle
- Vitalie Stavila

