
# Steady-State $He^+$ Implantation In Engineered First Wall Armor Materials

S. J. Zenobia, B. Egle, T. Knowles, R. Raffray, J. Shea, S. O'Dell, G. Kulcinski and the IEC Group

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Fusion Technology Institute
University of Wisconsin-Madison

THE UNIVERSITY of WISCONSIN MADISON

# Outline



- Fine- (FGW) and Nano-grain (NGW) tungsten
  - Surface morphology change
  - Helium retention
  - Preliminary observations
- W-needles
  - Experimental setup
  - Surface morphology change
  - Summary
- Conclusions, Future Work & Questions

# Progress Since Last Meeting (FGW & NGW)

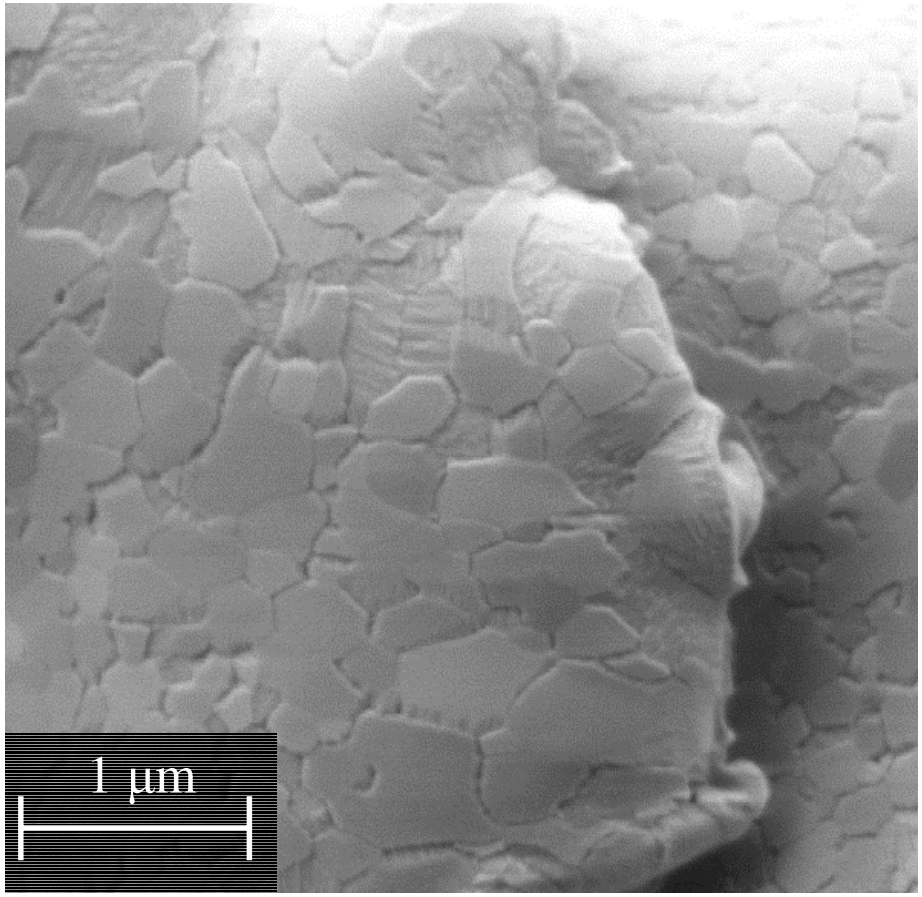


| Irradiation conditions using 30 keV $^3He^+$ | | | | |
|---|---|---|---|---|
| FGW | φ<br>T | $3x10^{17}$ $cm^{-2}$<br>700±50 ºC | $9x10^{17}$ $cm^{-2}$<br>850±50 ºC | $10^{19}$ $cm^{-2}$<br>1050±50 ºC |
| NGW | φ<br>T | $10^{17}$ $cm^{-2}$<br>1150±50 ºC | $10^{18}$ $cm^{-2}$<br>1050±50 ºC | $10^{19}$ $cm^{-2}$<br>1000±50 ºC |

- Scanning electron microscopy (SEM) & focused ion beam (FIB) milling analyses were performed to diagnose surface morphology change
- Pre- & post- irradiation masses were measured to determine the extent of mass loss
- Retained helium fluence was measured using $^3He(d,p)^4He$ nuclear reaction analysis (NRA) at the UW 1.7 MV tandem accelerator facility

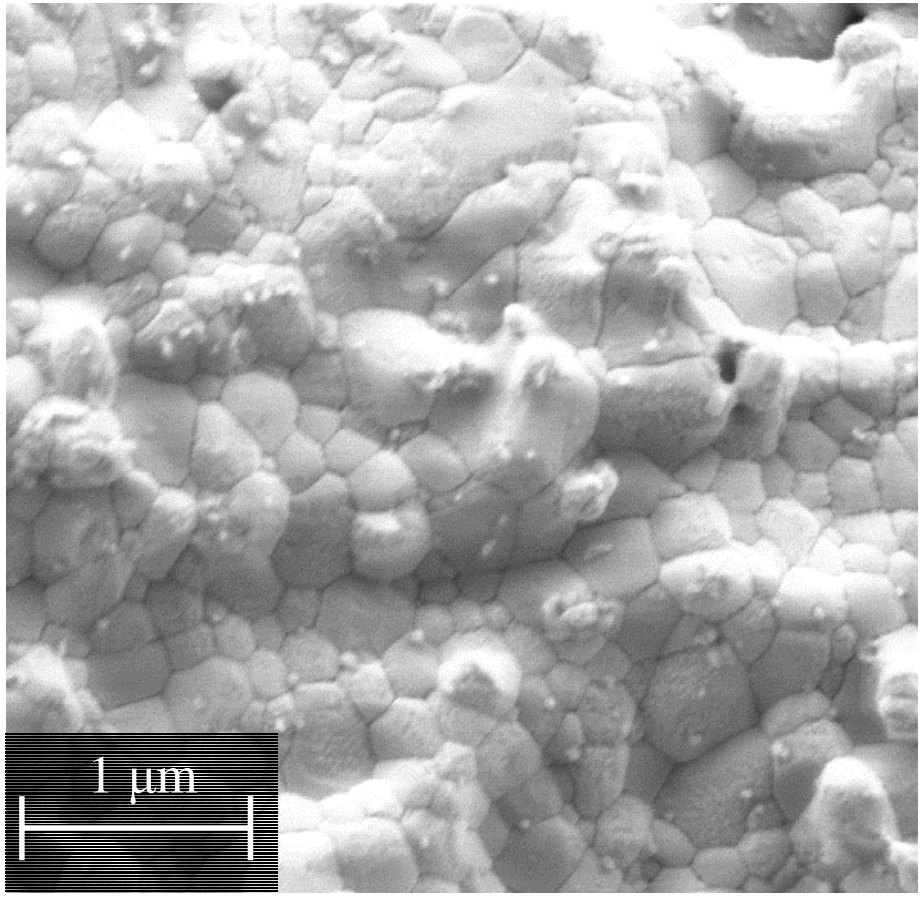
# Unirradiated Fine- & Nano-Grain Tungsten






## *Fine-Grain Tungsten*

- ~87% dense
- Grain size < 5 μm

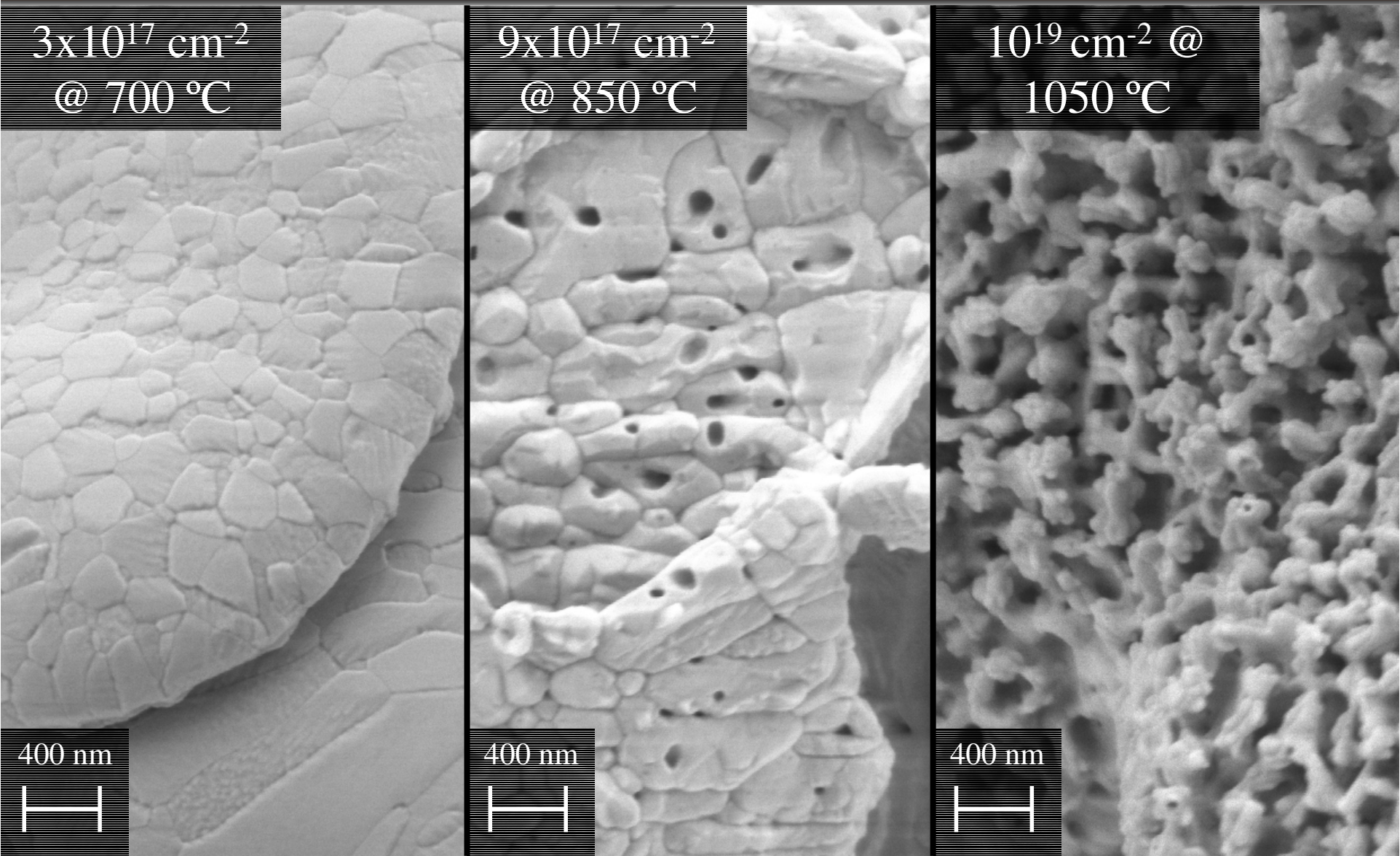



## *Nano-Grain Tungsten*

- 80-90% dense
- Grain size ~ 240 nm

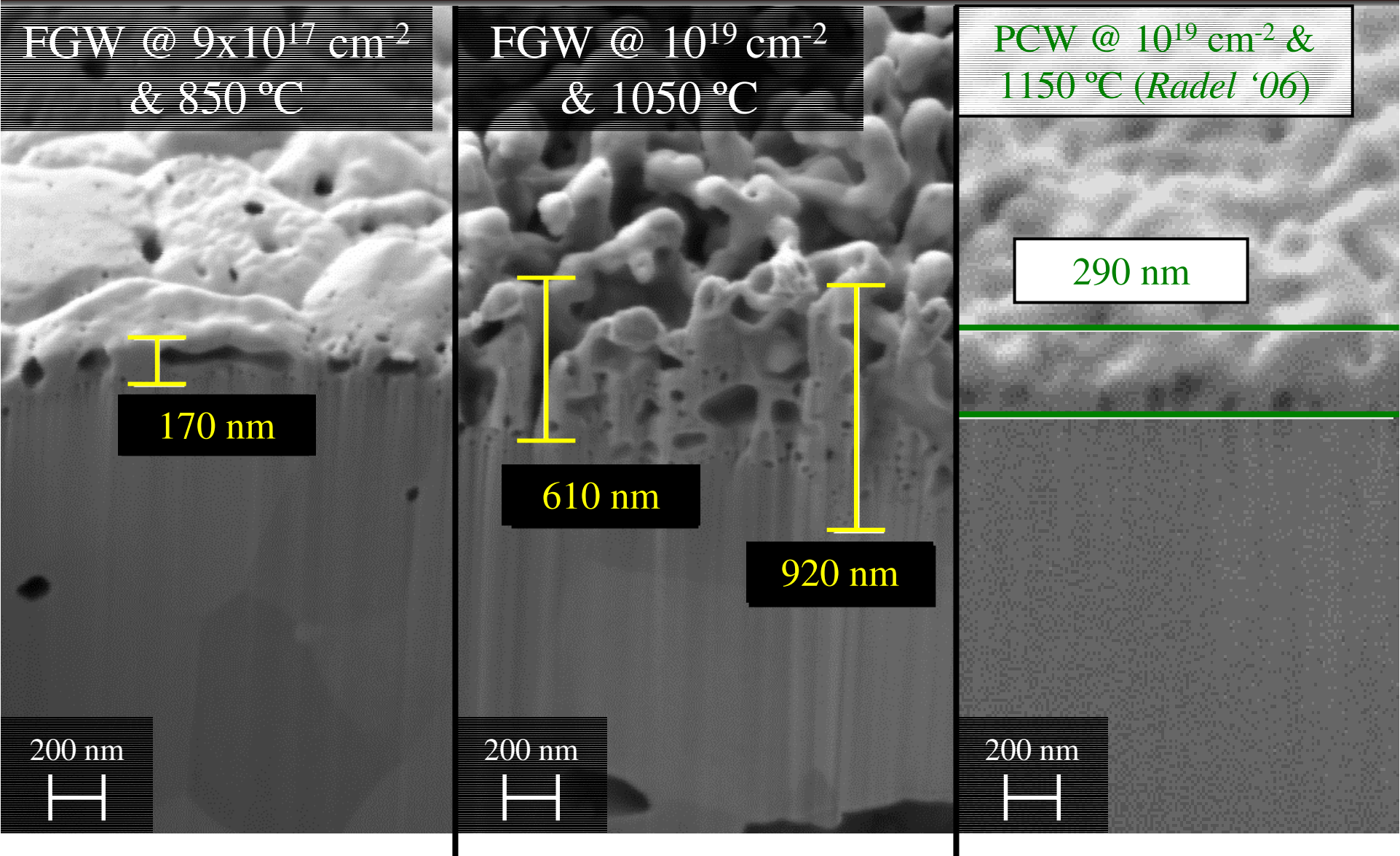
# SEM Analysis Reveals Pore Formation Threshold In *FGW* Is Near ~$10^{18}$ $He^{+}/cm^{2}$





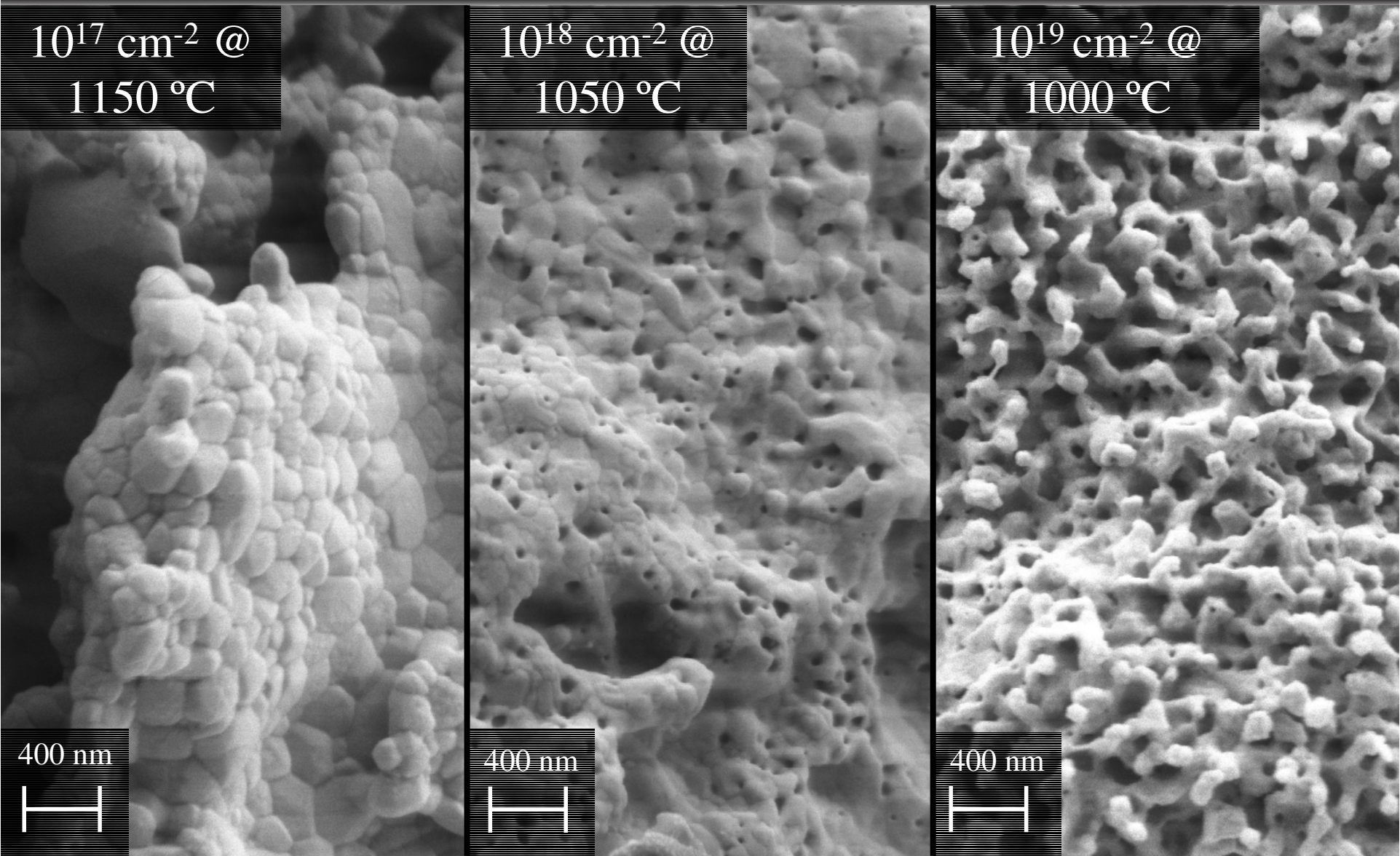
# FIB Reveals Penetration Depth Of Helium Bubbles Up To ~900 nm Below *FGW* Surface





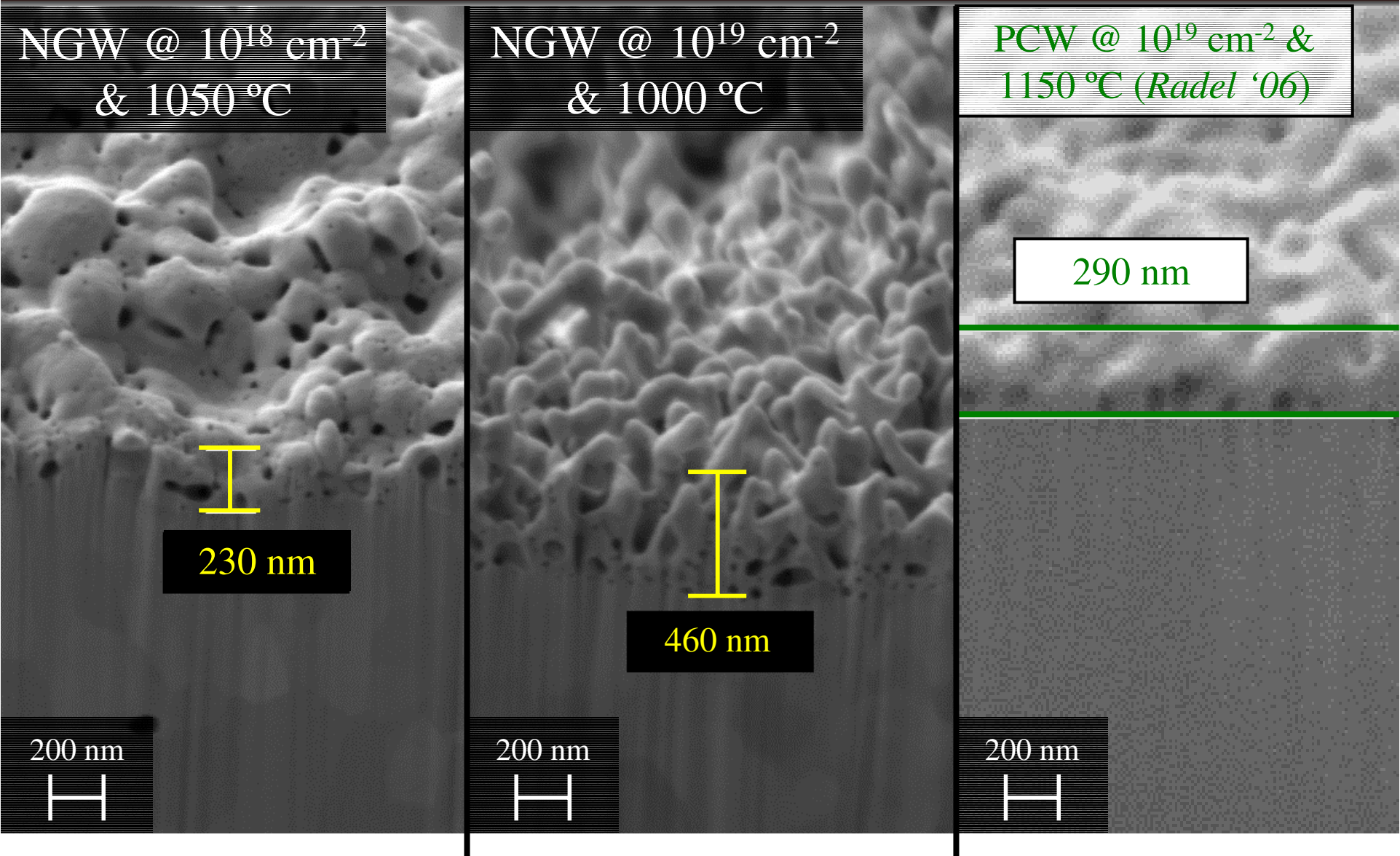
# SEM Analysis Reveals Pore Formation Threshold In *NGW* Is Near ~$10^{18}$ $He^{+}/cm^{2}$





# FIB Reveals Substantial Penetration Depth Of Helium Bubbles Up To ~ 500 nm Below *NGW* Surface





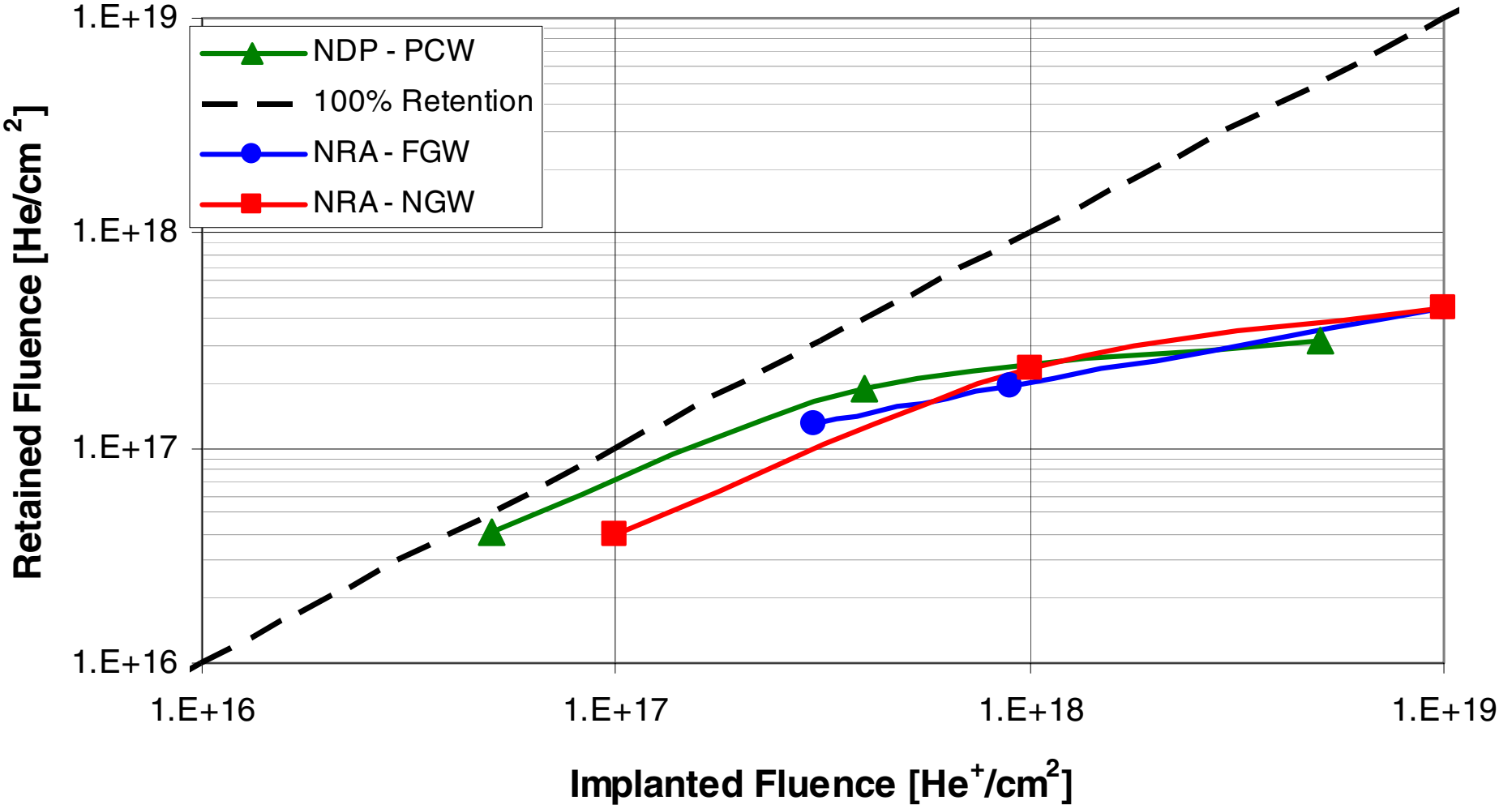
# Mass Loss Per Full Power Year [kg/FPY] Operation In The Reference HAPL Chamber – *w/o Ion Abatement*



| Fluence [$He^+/cm^2$]: *FGW* | Time [FPD] @ reference $He^+$ spectrum (10-90 keV) | Waste [kg/FPY] | Fluence [$He^+/cm^2$]: *NGW* | Time [FPD] @ reference $He^+$ spectrum (10-90 keV) | Waste [kg/FPY] |
|---|---|---|---|---|---|
| $3x10^{17}$ | 0.3 | 500±200 | $10^{17}$ | 0.1 | 3700±2600 |
| $9x10^{17}$ | 4 | 510±80 | $10^{18}$ | 4.4 | 550±250 |
| $10^{19}$ | 44.2 | 120±10 | $10^{19}$ | 44.2 | 60±20 |

# NRA* & NDP* Gives Retained Helium Fluence In *PCW, FGW* & *NGW* (700 – 1150 °C)






*NRA = Nuclear Reaction Analysis

*NDP = Neutron Depth Profiling

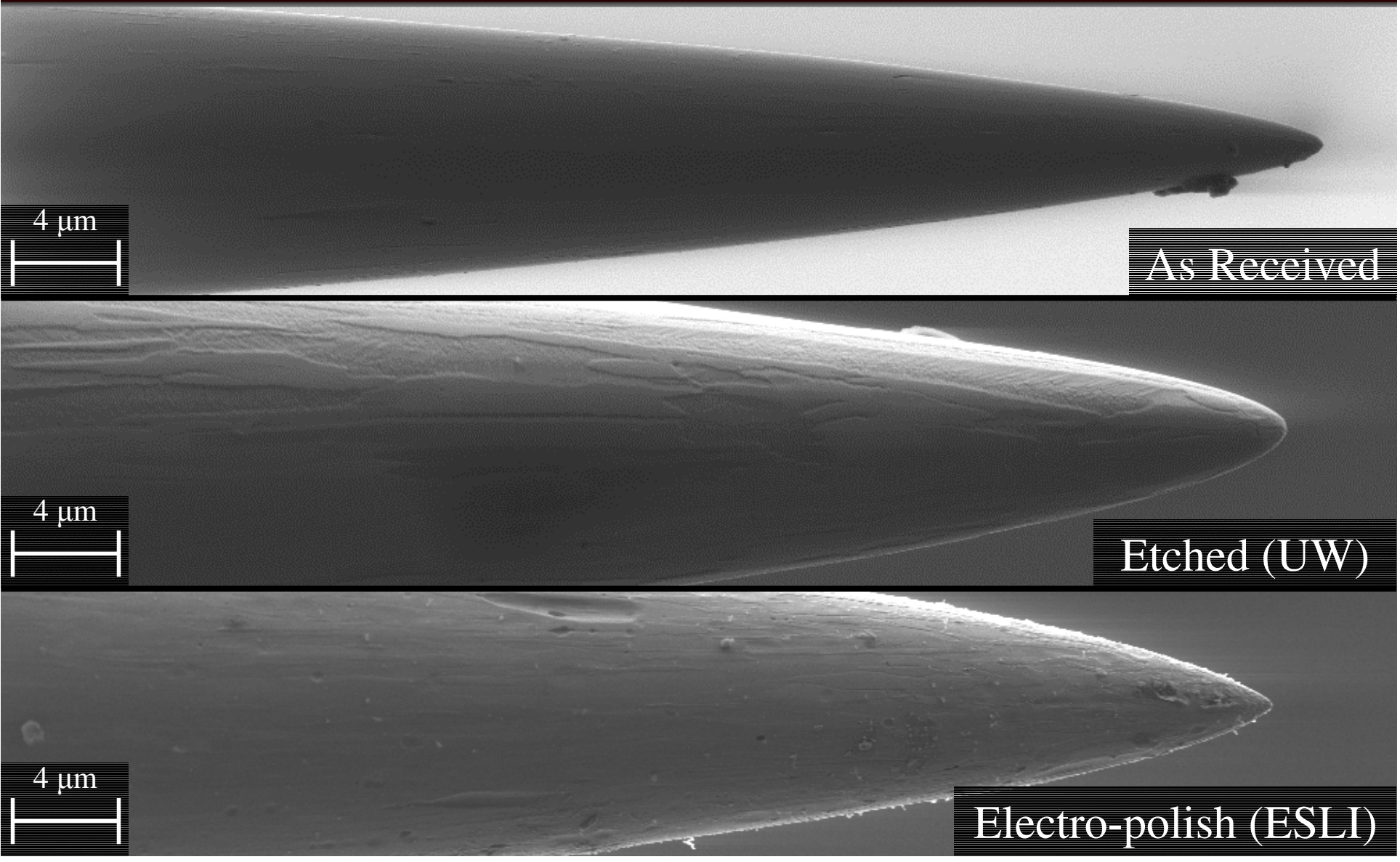
# Preliminary Observations (FGW & NGW)



- The threshold for pore formation in FGW lies between $3x10^{17}$ – $9x10^{17}$ $He^+/cm^2$ and between $10^{17}$ – $10^{18}$ $cm^{-2}$ in NGW, becoming extensive at $10^{19}$ $cm^{-2}$
- Sub-surface pores penetrate much deeper than the range of 30 keV $He^+$ (84 nm), up to ~900 nm in FGW and ~500 nm in NGW
- Both FGW & NGW specimens sustained measurable mass loss at all irradiation fluences and temperatures
- NRA reveals that the retention characteristics of standard PCW, FGW & NGW are very similar at fluences above ~$10^{17}$ $He^+/cm^2$ over the examined temperature range

# ESLI Tungsten Needles



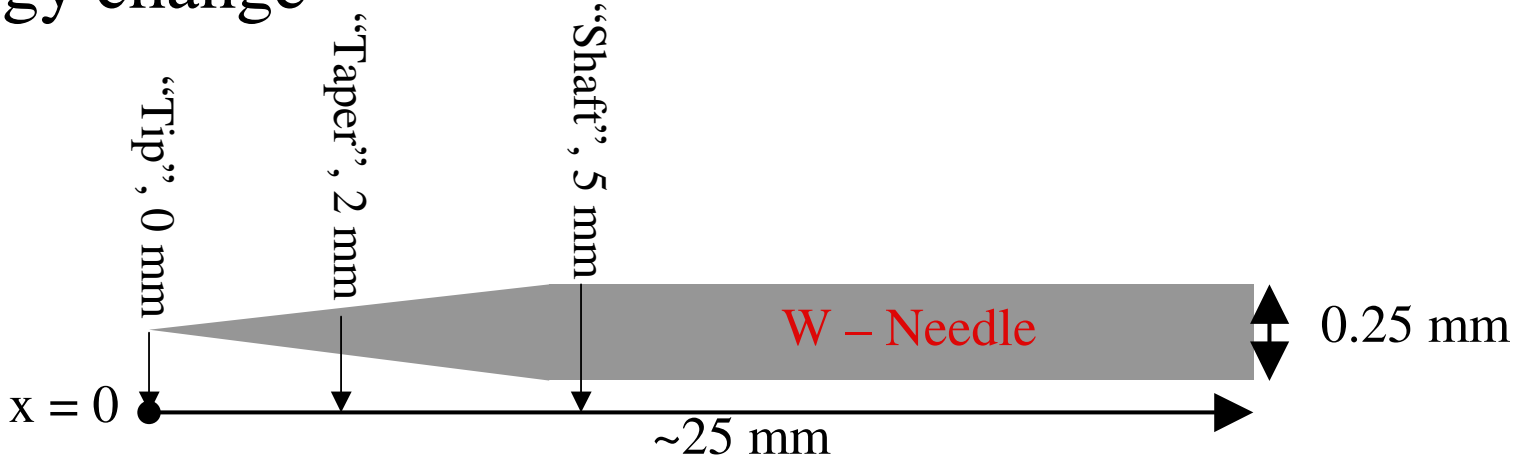


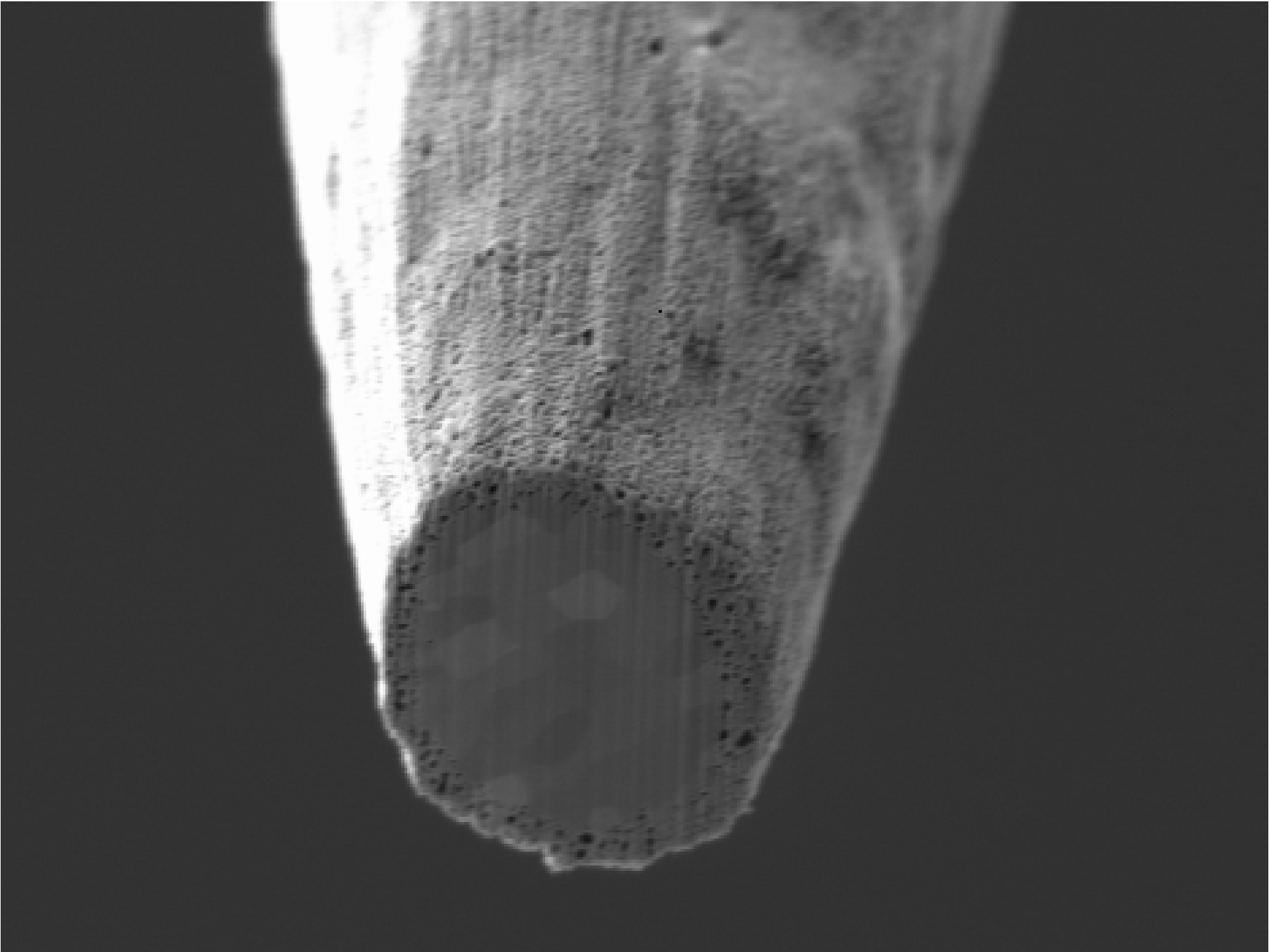
# $He^+$ Implantation Of W-Needles In An *Electric Field-Free Environment*



| Irradiation conditions using 100 keV $^4He^+$ ions | |
|---|---|
| UW Etch | ESLI Electro-polish |
| φ - $3x10^{18}$ $cm^{-2}$<br>T - 700±50 ºC | φ - $1.3x10^{19}$ $cm^{-2}$<br>T - 1000±50 ºC |

- SEM & FIB analyses were performed to diagnose surface morphology change

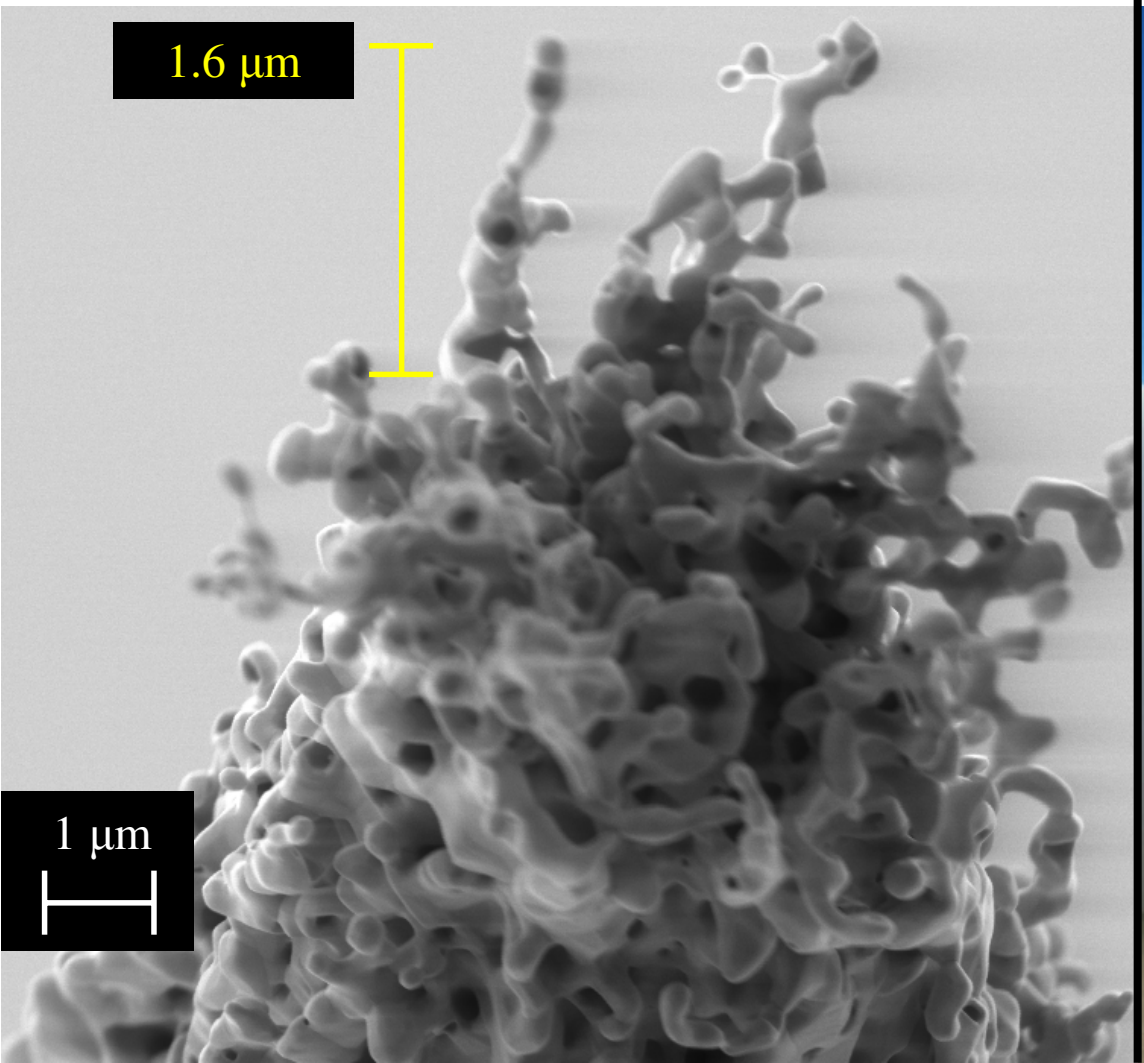




# *'Chihuly Effect'* Observed On W-Needle Tips After High Fluence Helium Ion Implantation



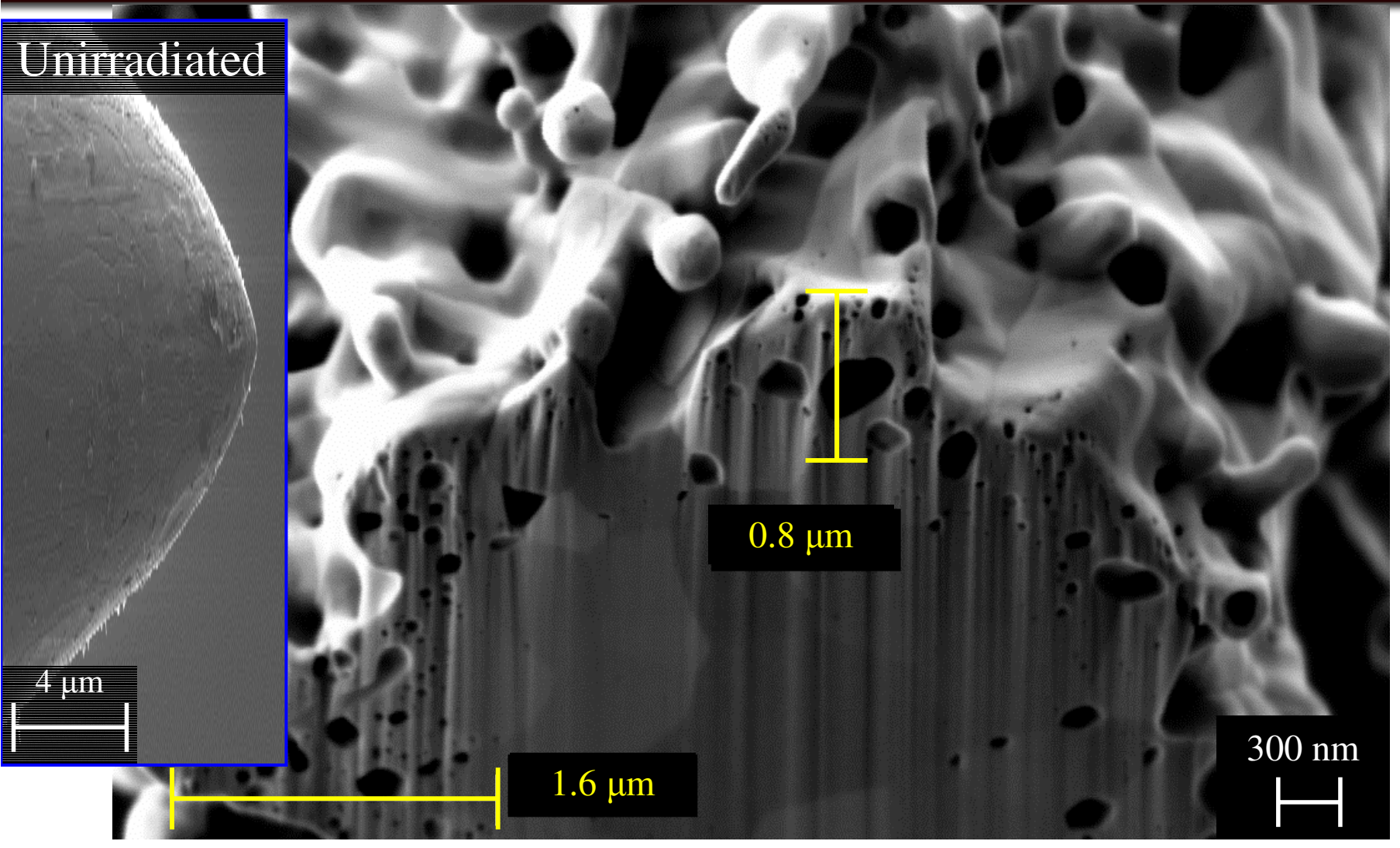
Fluence ~ $1.3 \times 10^{19}$ $He^+/cm^2$, Temperature ~ 1000 °C




Dale Chihuly Glass Art
www.chihuly.com®

# FIB Analysis Of 'Chihuly' W-Needle Shows μ-Scale Surface Modification & Pore Penetration






Fluence ~ $1.3 \times 10^{19}$ $He^{+}/cm^{2}$, Temperature ~ 1000 °C

# Summary



- Implantation to $3x10^{18}$ $cm^{-2}$ at ~700 °C showed pore formation on all parts of the W-needle
- Implantation to $1.3x10^{19}$ $cm^{-2}$ at ~1000 °C resulted in the micron-scale 'Chihuly effect' on all parts of the W-needle
- FIB analysis confirmed uniform flux distribution of implanted helium ions
- Pore penetration depths at the needle tips ranged from ~300 – 1600 nm, much greater than the predicted SRIM range of ~90 nm (100 keV $He^+$)

# Conclusions



- Thresholds for pore formation in FGW & NGW are increased from standard PCW by about a factor of 10
- The surface morphology response of FGW & NGW does not appear to be better than standard PCW at intermediate ($10^{18}$ $cm^{-2}$) and high ($10^{19}$ $cm^{-2}$) implanted $He^+$ doses
- At fluences above $10^{17}$ $cm^{-2}$, both FGW & NGW show comparable helium retention to that of standard PCW
- *Single* W-needles do not show increased robustness to $He^+$ implantation to that of flat PCW
- *Single* W-needles do not show a noticeable increase in the threshold fluence necessary for pore formation

# Future Work



- Nano-grain tungsten
  - Ten (10) additional specimens are in preparation at PPI
  - Fluence & temperature scans using Egle's ion gun technology (E-field free environment)
  - High temperature anneals between incremented implant doses
- W-Needle
  - Implantation of needle clusters
  - Implantation with deuterium ions
  - Chemical analysis using Energy Dispersive Spectrometry (EDS)

# Questions?



**Samuel Zenobia**
**University of Wisconsin-Madison**
**1500 Engineering Drive**
**Madison, WI 53706**
**(608) 265-8699**
**zenobia@wisc.edu**

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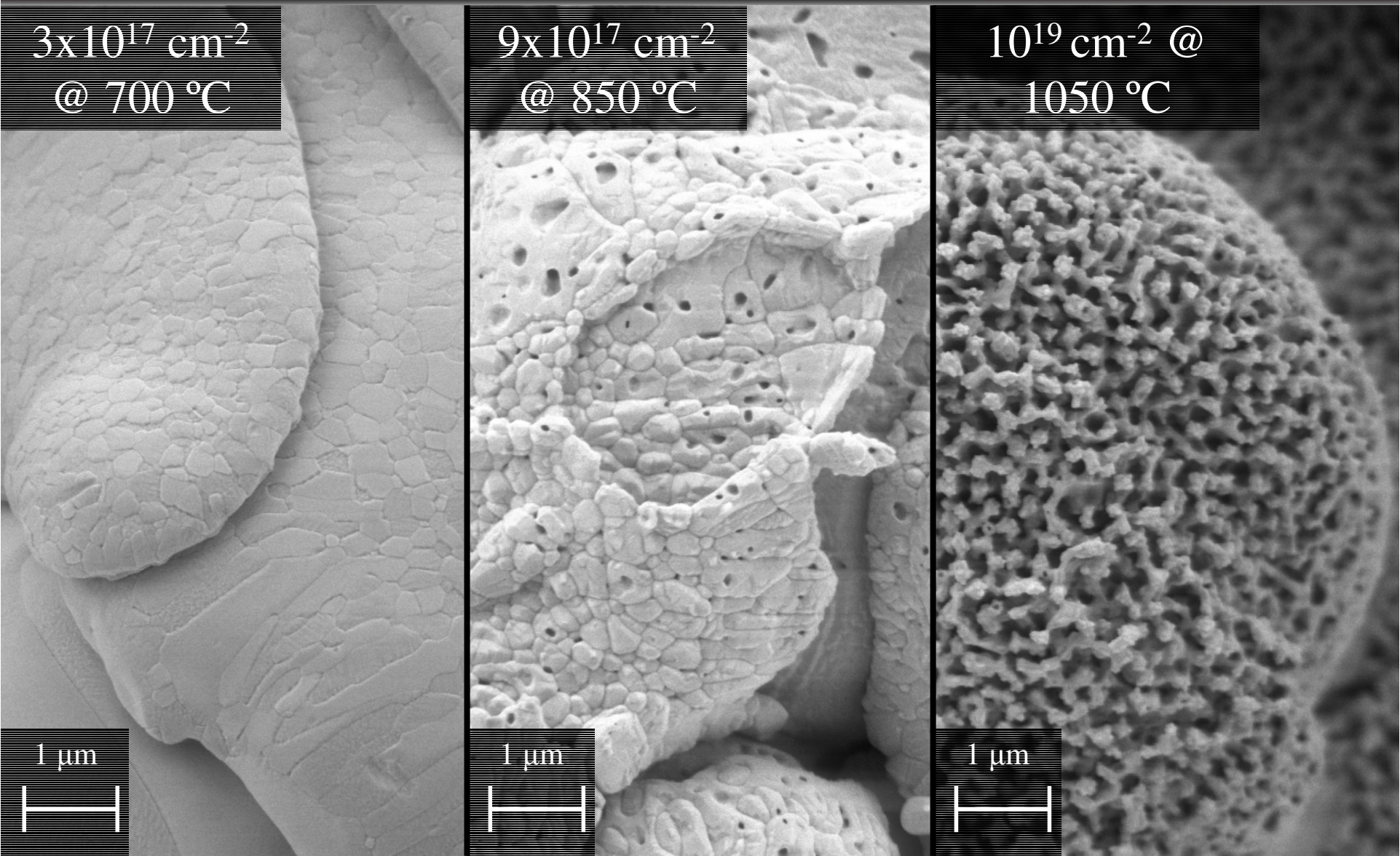

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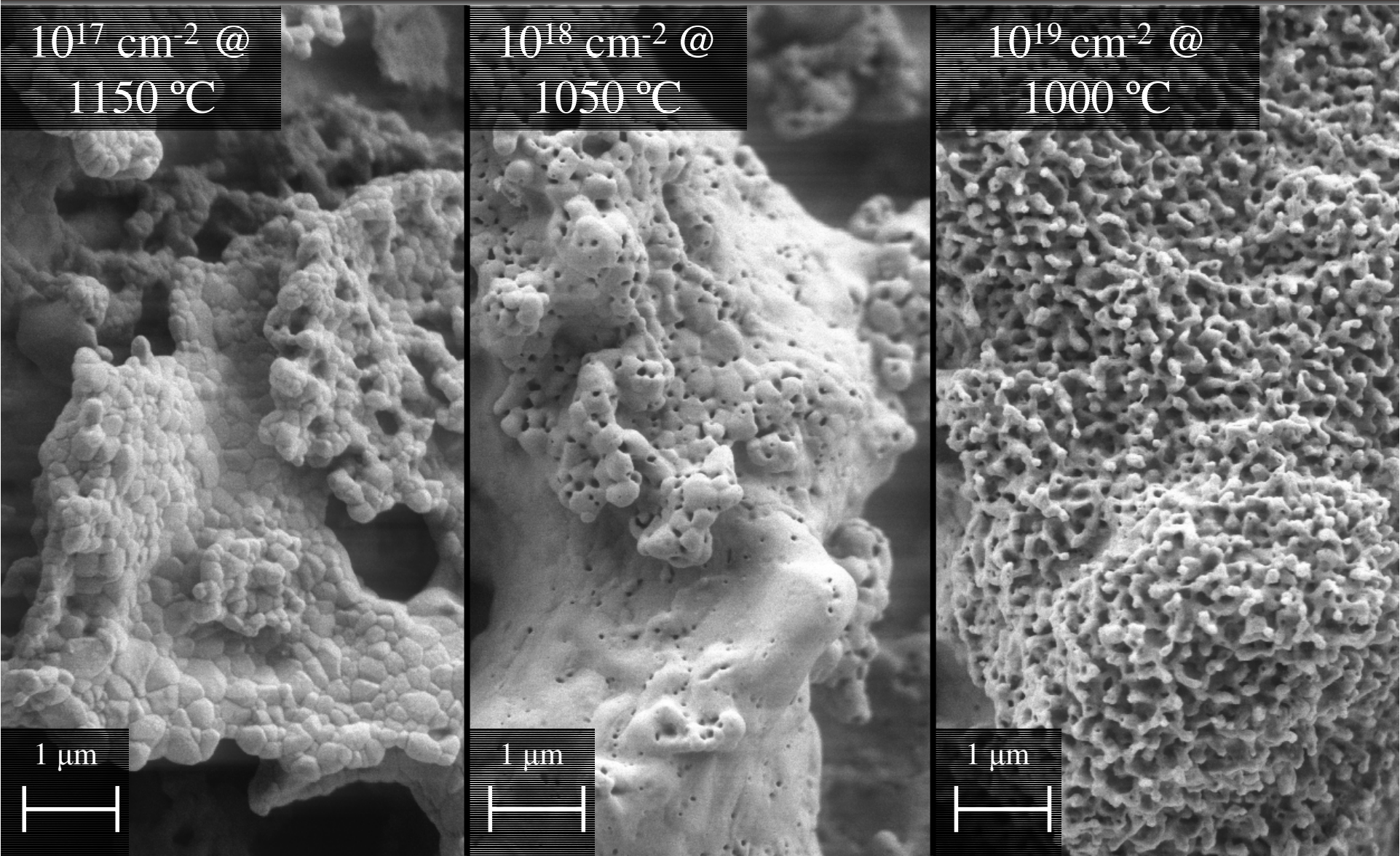
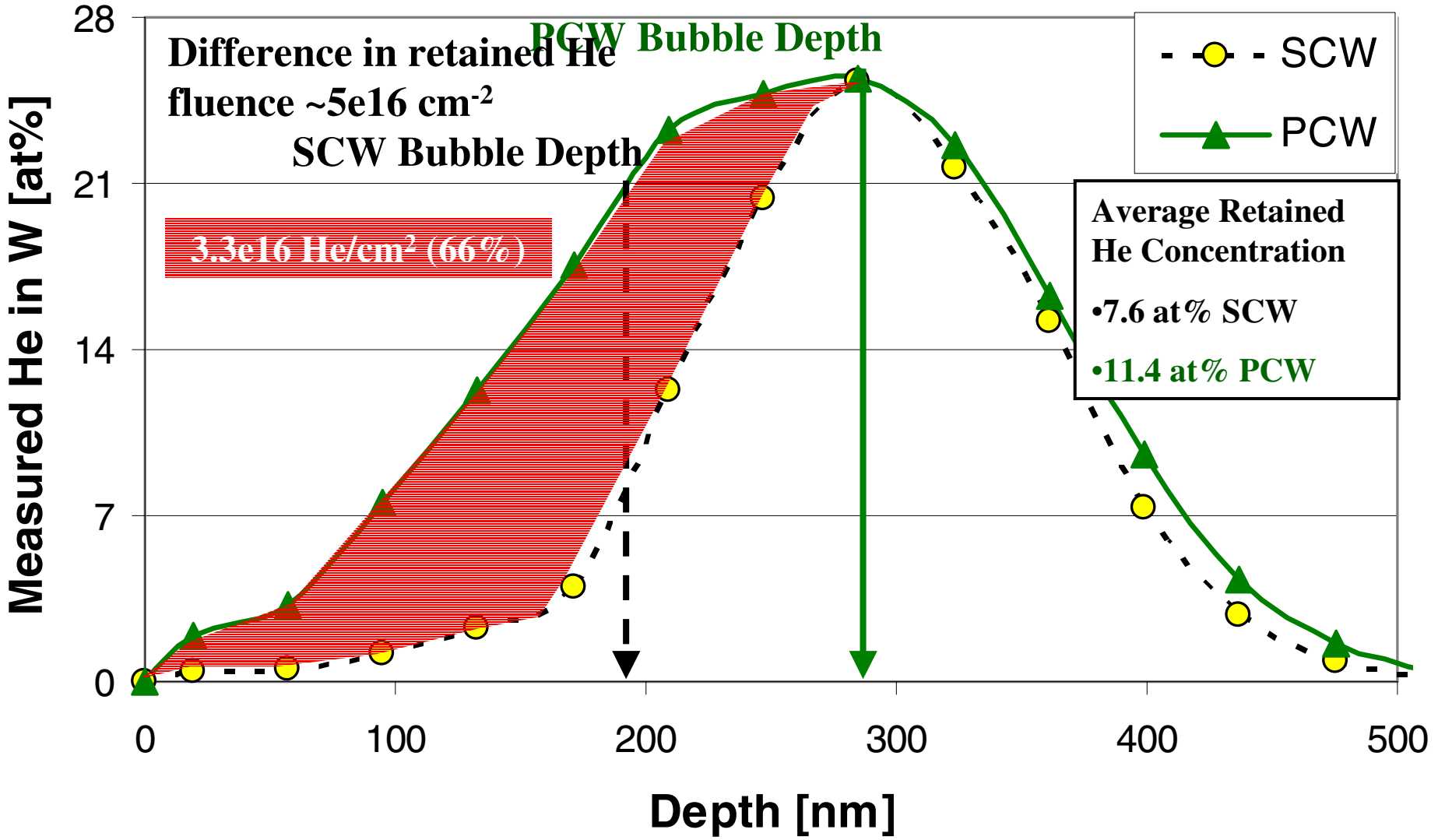


# Adjusted Densities of Porous Layers Show Increased Range of Helium Ions

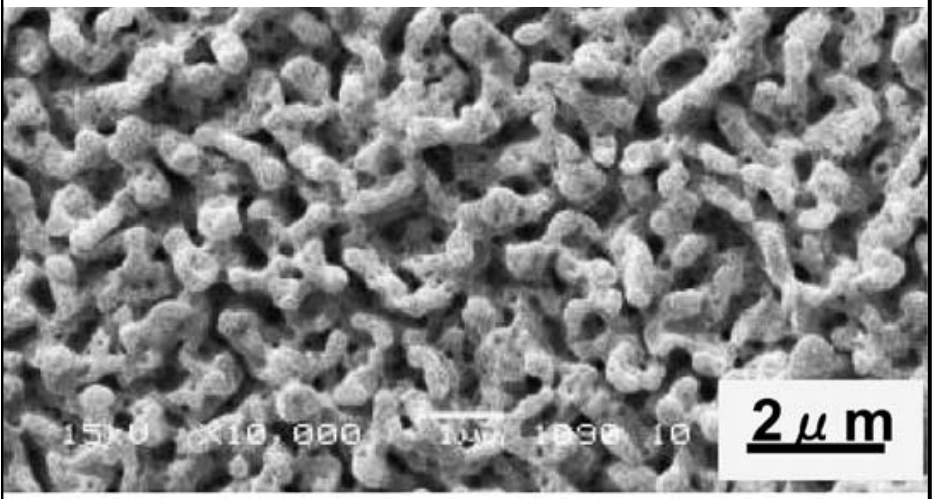


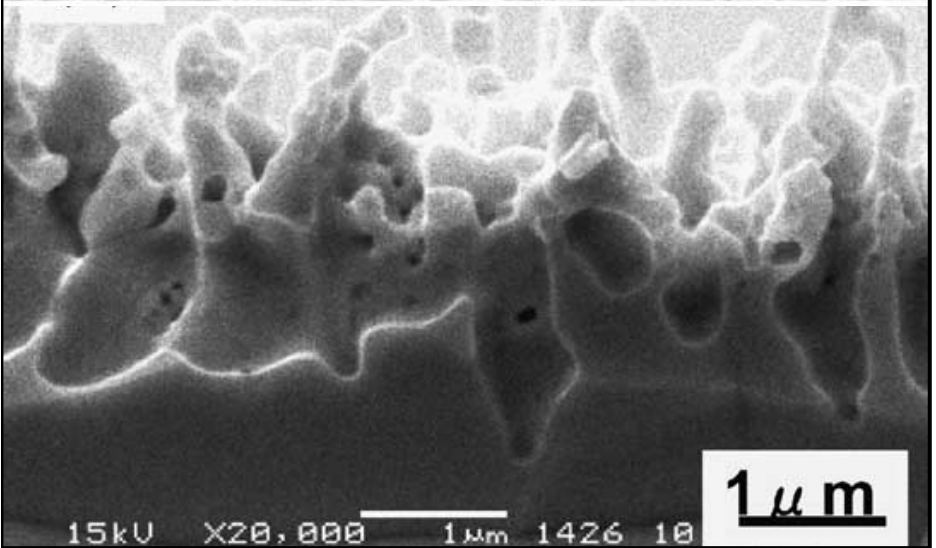


# Coral Spires & Extended Porous Layers in FGW & NGW Consistent w/ Literature Findings

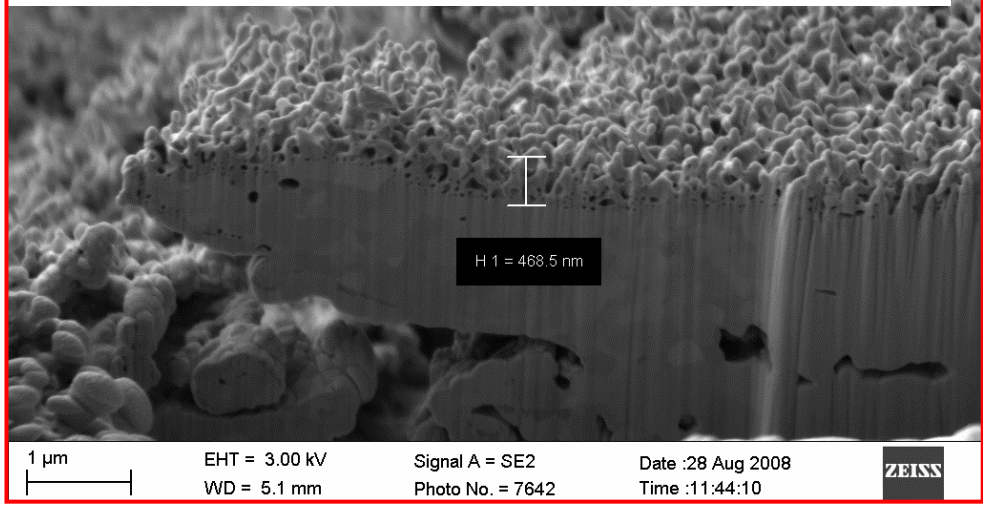


*Tokunaga, et al. (2004)* PCW – 15 keV $He^+$ @ 2600 ºC and $3.23x10^{19}$ $cm^{-2}$

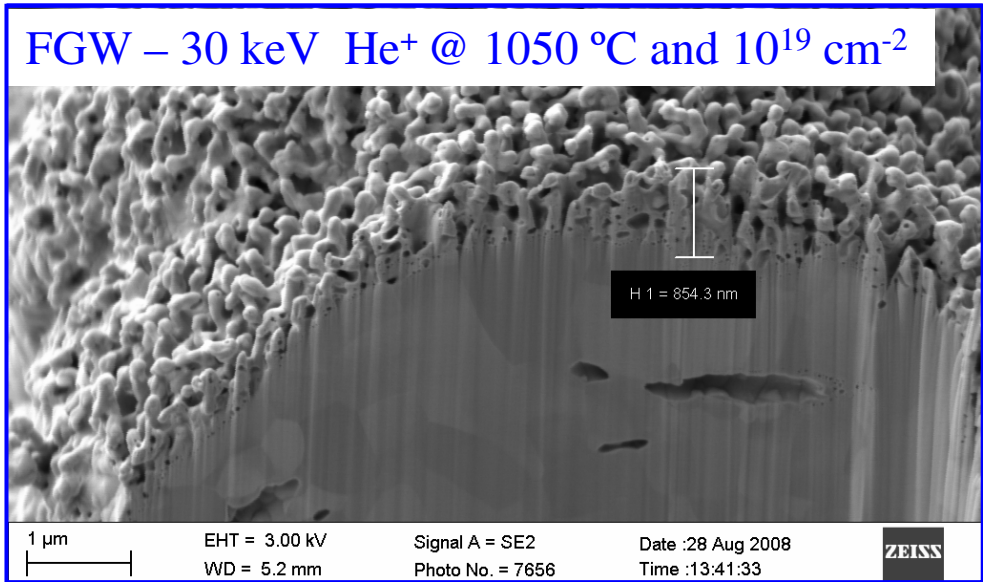



NGW – 30 keV $He^+$ @ 1010 ºC and $10^{19}$ $cm^{-2}$

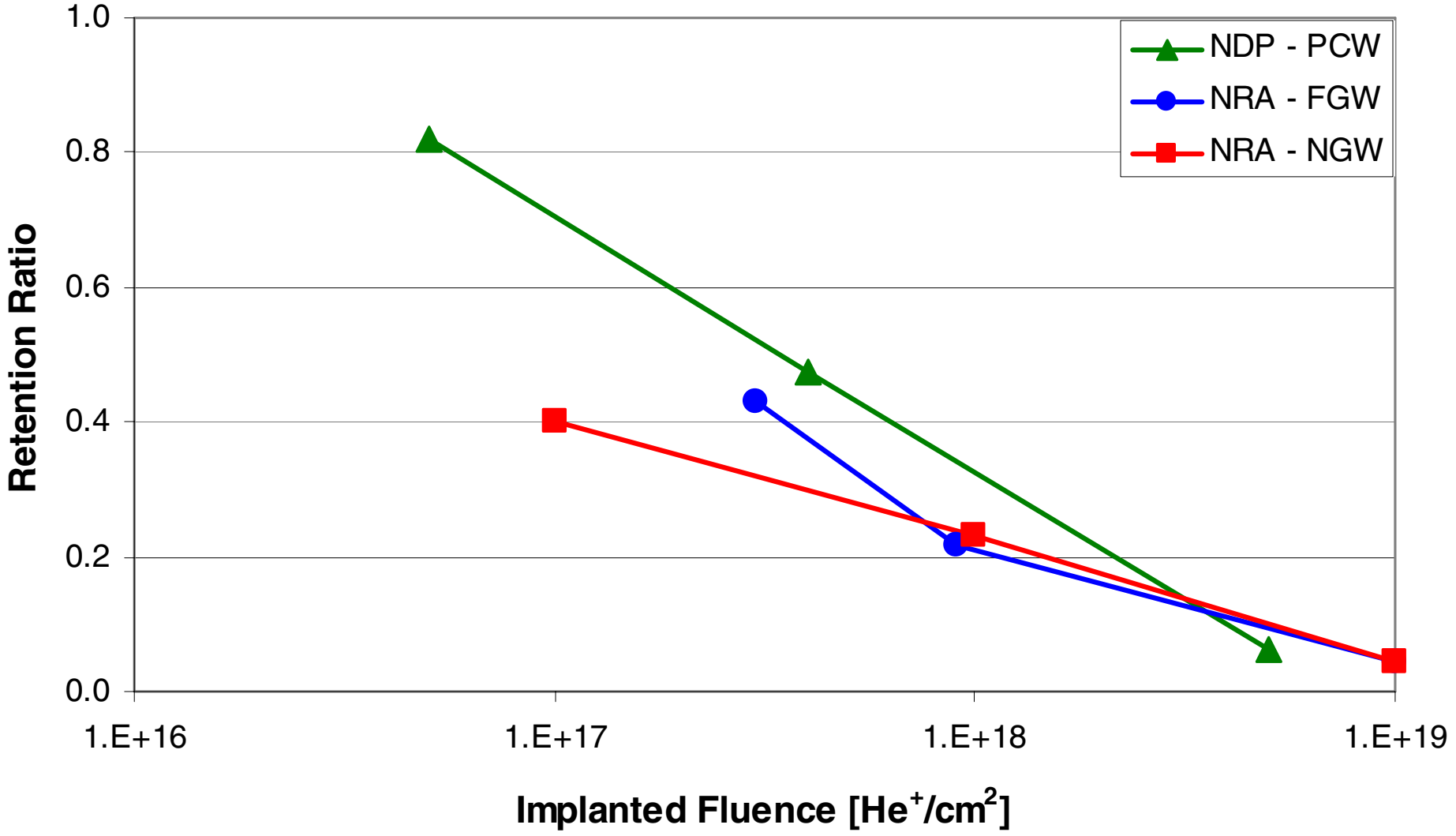



FGW – 30 keV $He^+$ @ 1050 ºC and $10^{19}$ $cm^{-2}$



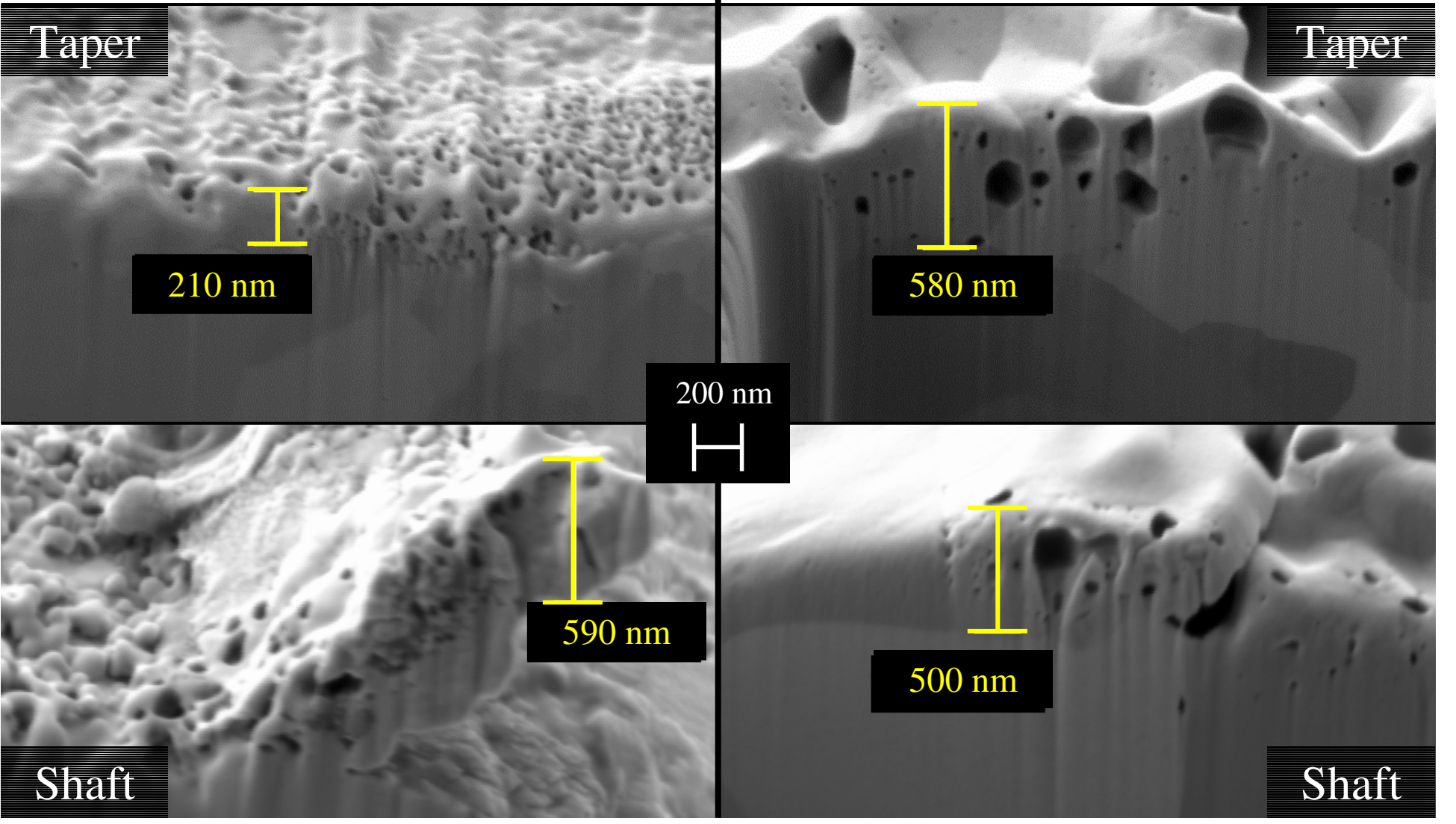
# PCW, FGW & NGW All Show Increased Helium Trapping Efficiency At Lower Fluences





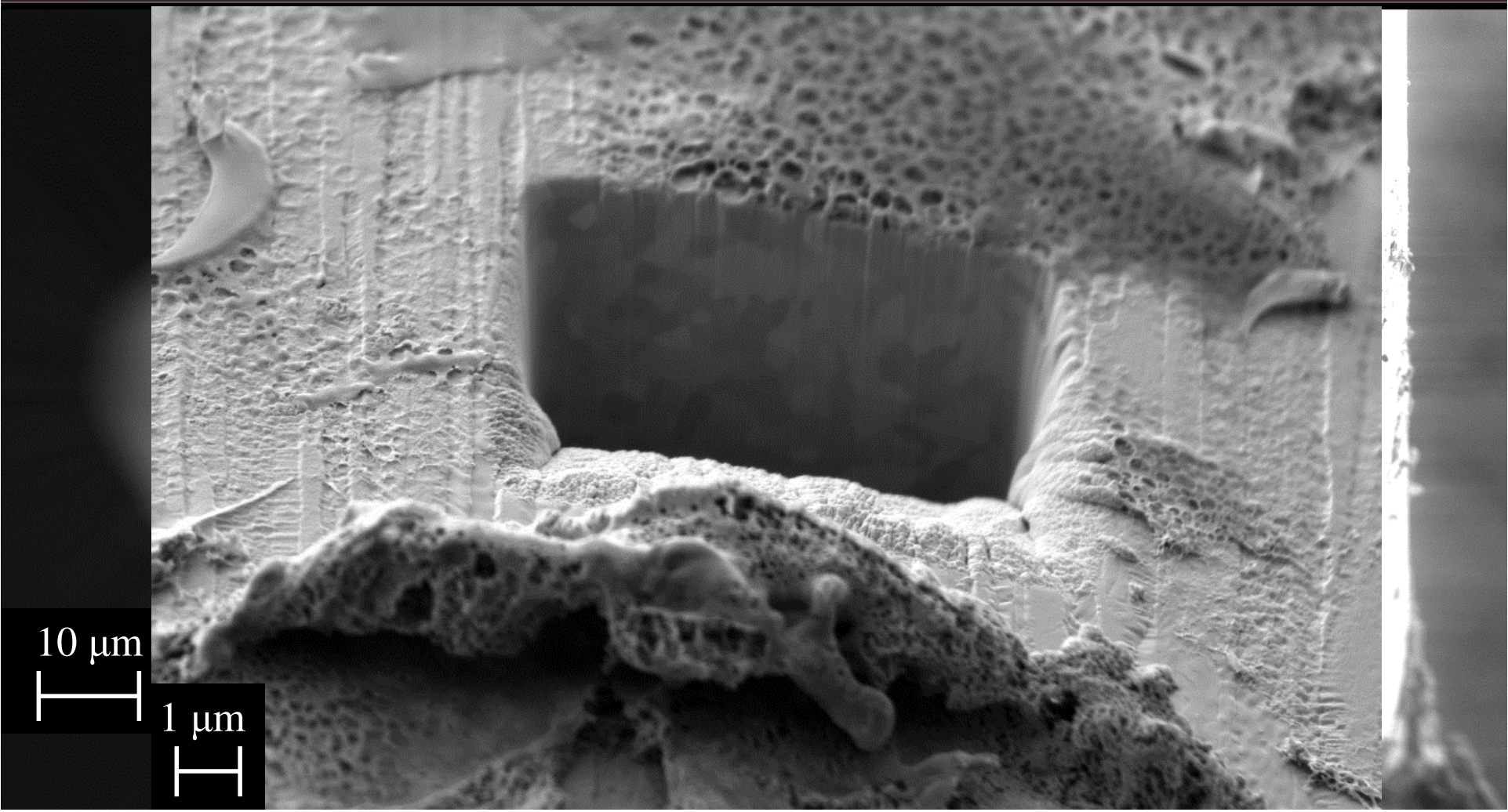
# Surface Morphology Change Is Also Observed On Tapers & Shafts of W-Needles Implanted w/ $He^+$





# Blistering & Exfoliation Is Also Observed On W-Needles Implanted w/ *Helium Ions*

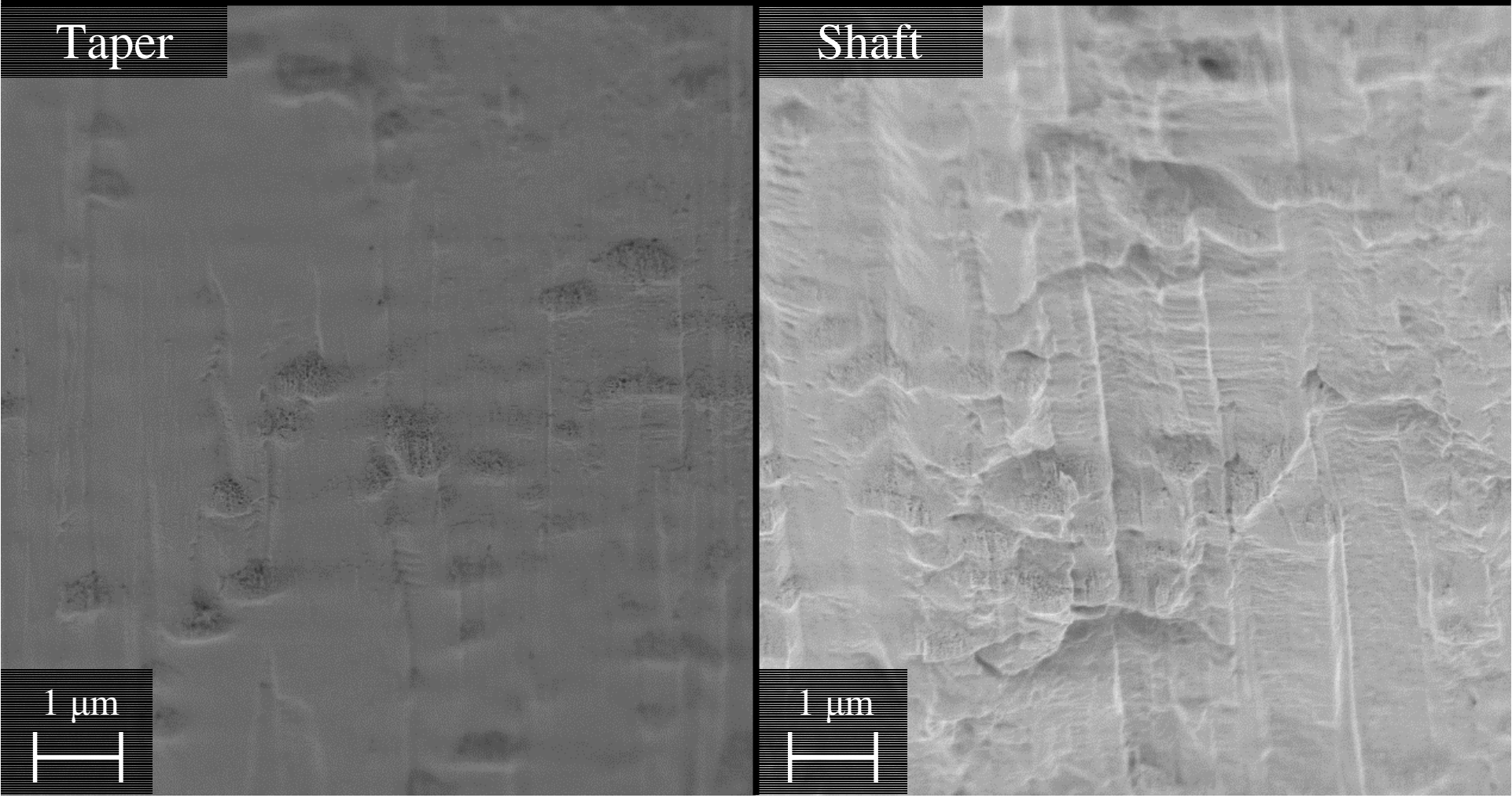





Fluence ~ $3 \times 10^{18}$ $He^{+}/cm^{2}$, Temperature ~ 700 °C

# Pore Formation Occurs At Each Part of the W-Needle Irradiated w/ $He^+$

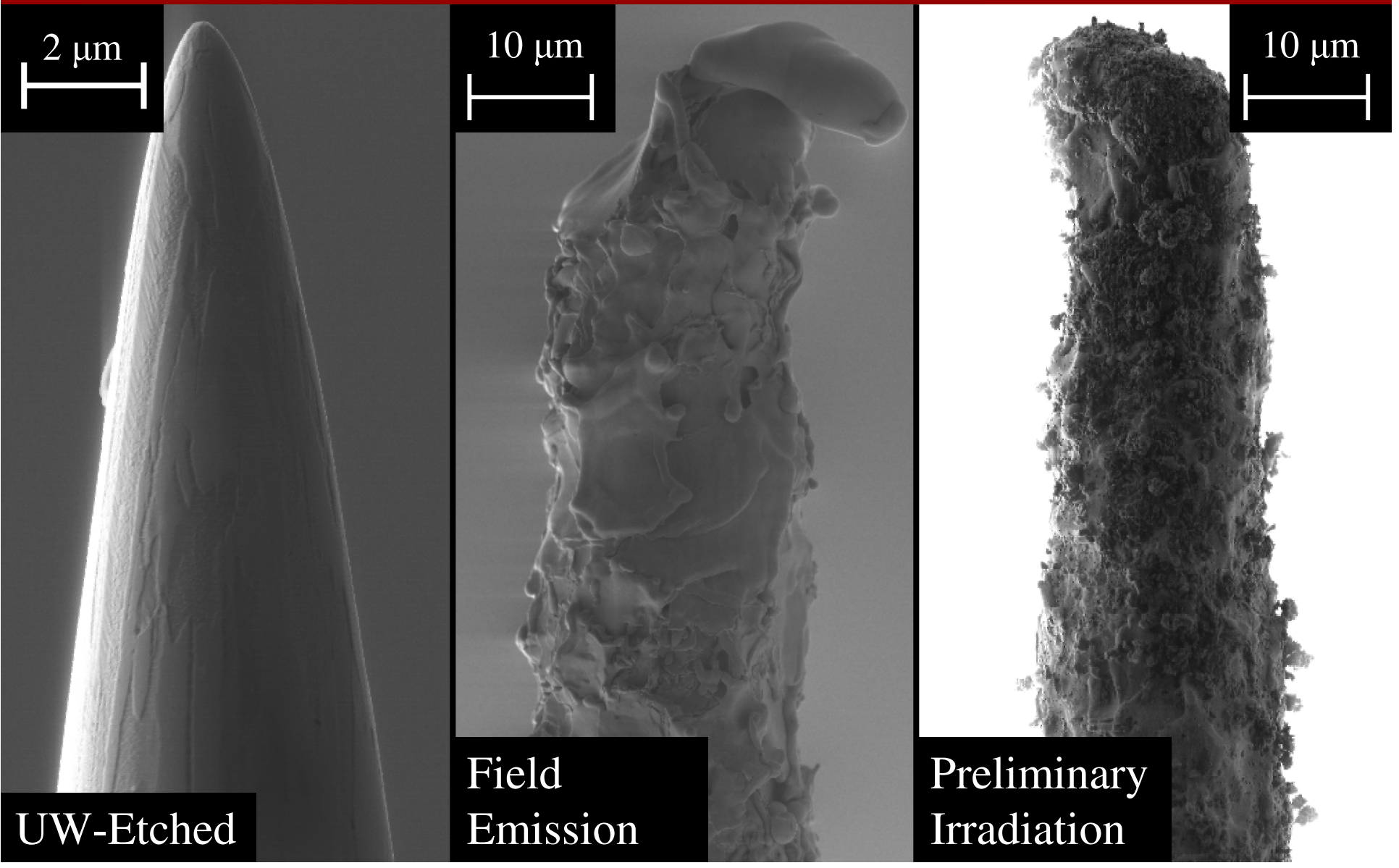





Fluence ~ $3 \times 10^{18}$ $He^+/cm^2$, Temperature ~ 700 °C

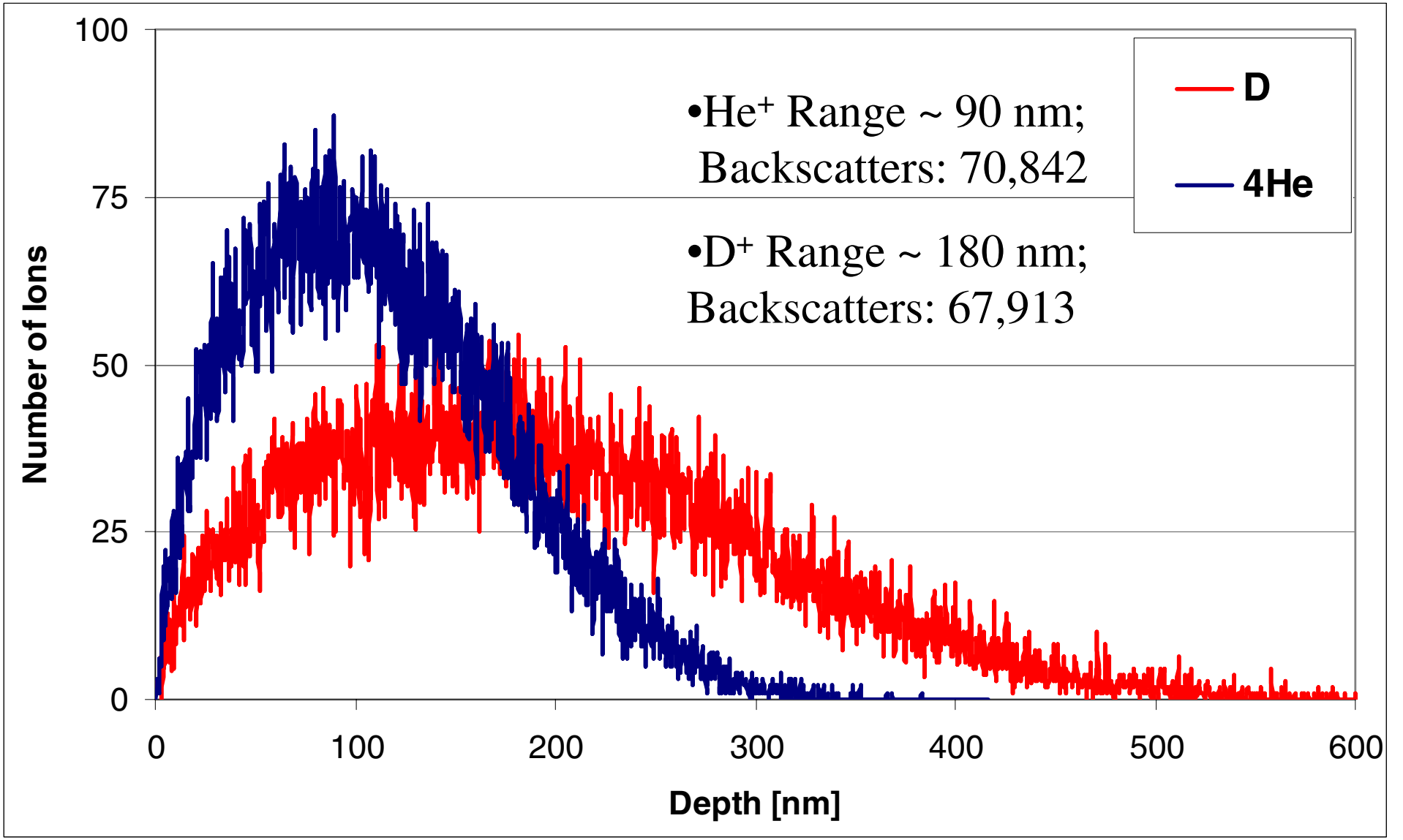
# Tip of Irradiated W-Needle Became Dulled from Arcing or Handling





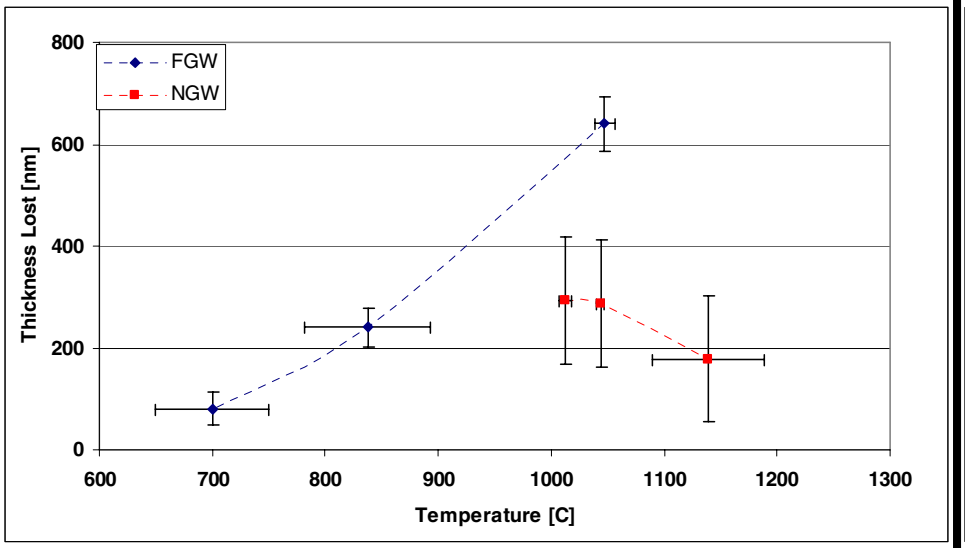
# SRIM Calculations of $He^+$ & $D^+$ Range In WNeedles @ 87° Angle of Incidence

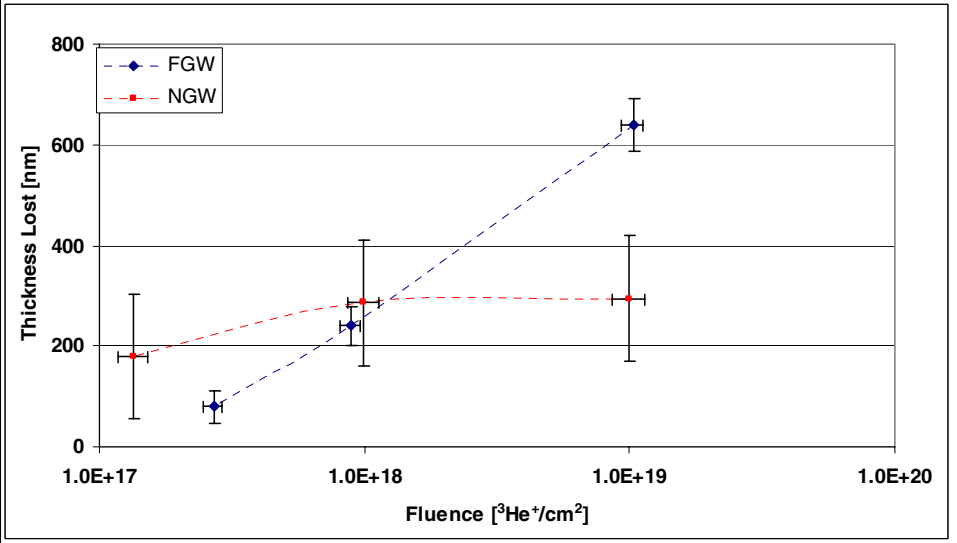




# Calculations From Mass Loss Correspond To Severe Erosion From FGW & NGW Surfaces





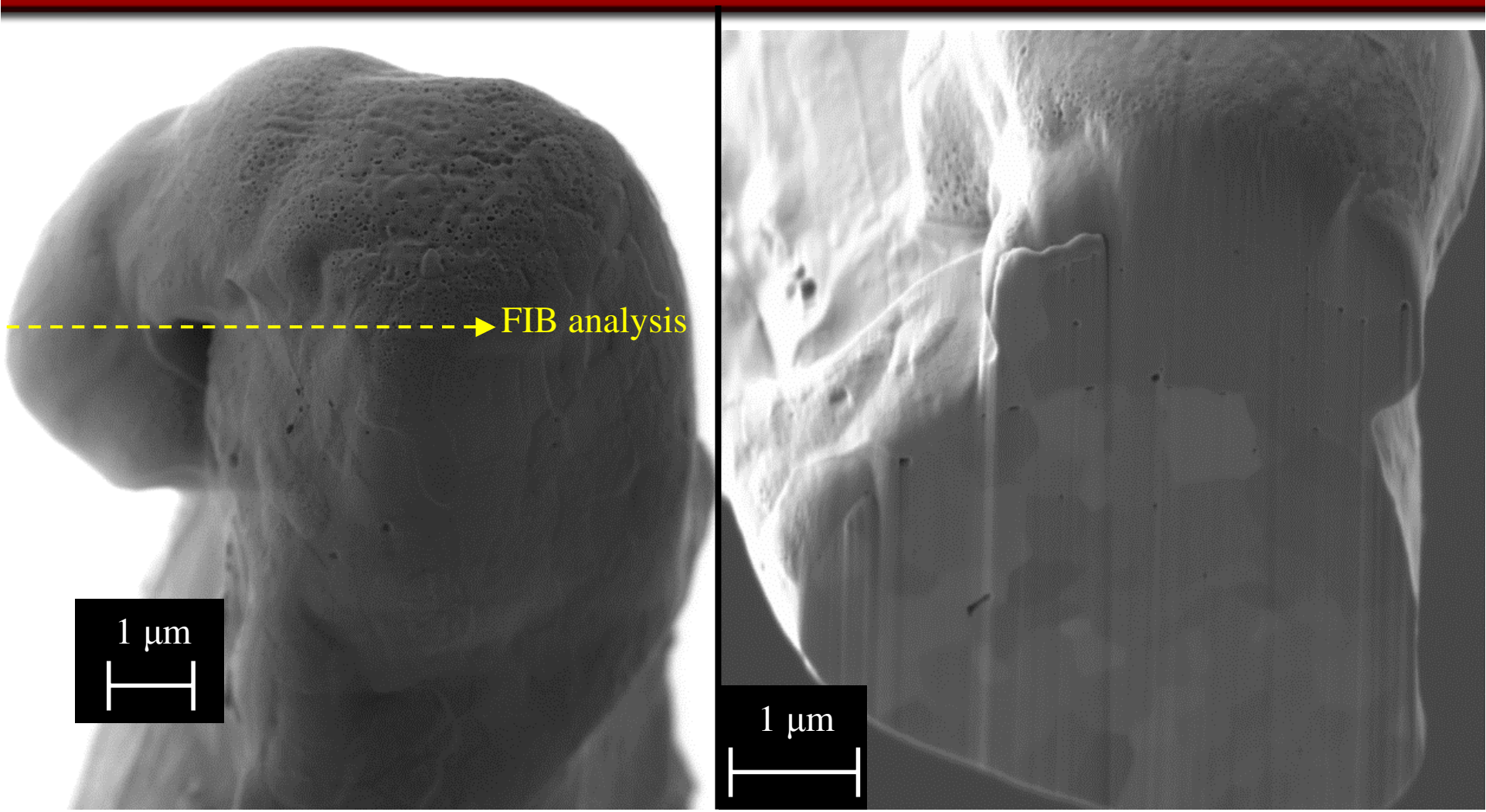




- At similar temperatures FGW is eroded more severely than NGW
- Within error, surface erosion of NGW is constant for the examined temperature range
- Mass loss of FGW increases with irradiation temperature

- The maximum thickness lost in FGW was ~650 nm and ~300 nm in NGW
- Thickness lost in FGW appears to scale logarithmically with implant fluence

# Deuterium Irradiated W-Needle Sustained Minor Pore Formation On Blunted Tip



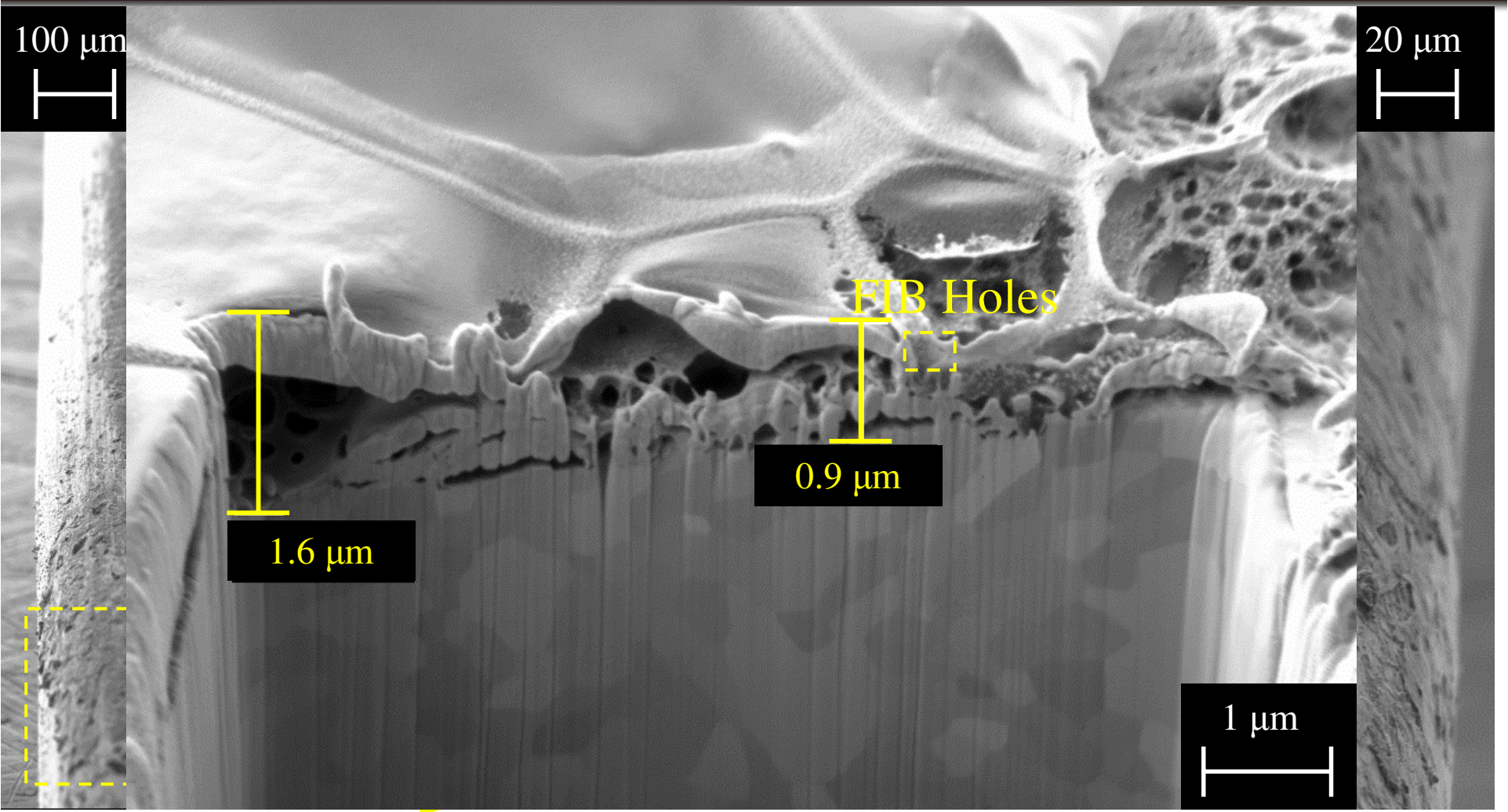



Fluence ~ $4 \times 10^{18}$ $D^+/cm^2$, Temperature ~ 750 °C

~0.5 FPD in Reference HAPL Chamber (< 90 keV)

# Taper And Shaft Of $D^+$ Irradiated Needle Show Extensive Blistering





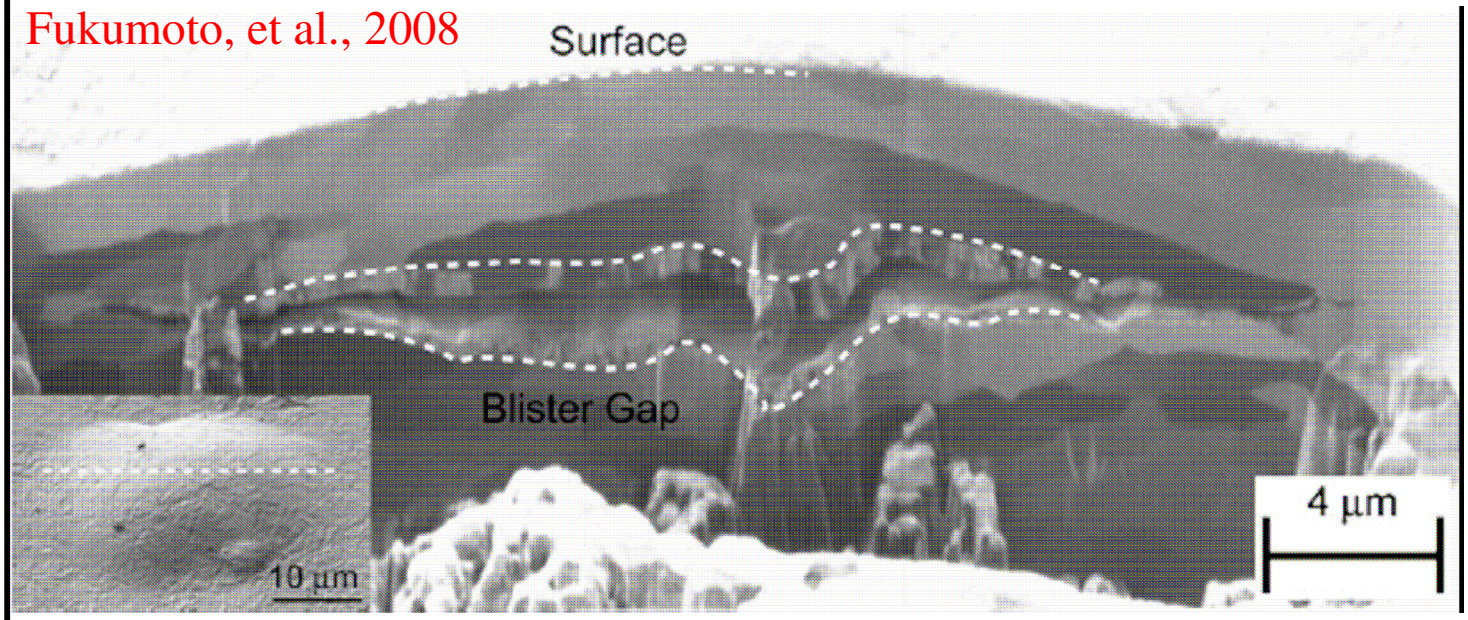

Fluence ~ $4 \times 10^{18}$ $D^+/cm^2$, Temperature ~ 750 °C

~0.5 FPD in Reference HAPL Chamber (< 90 keV)

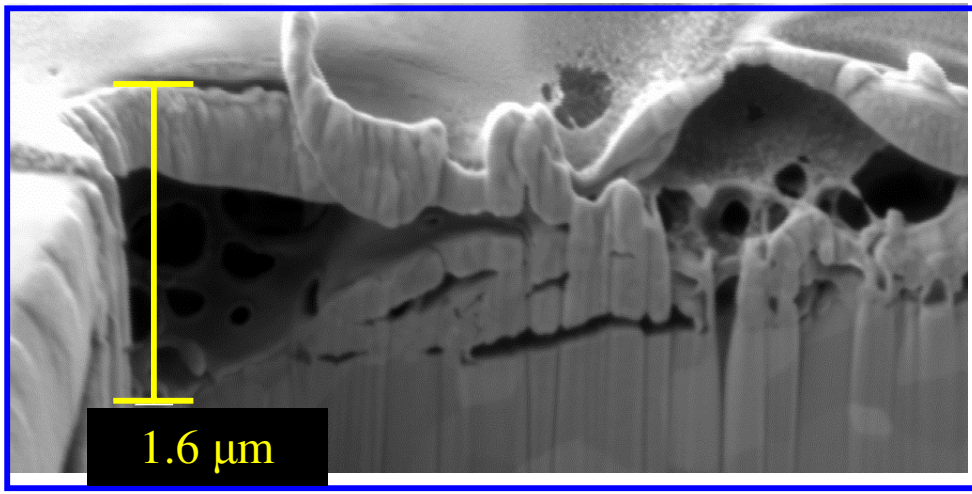
# Blistering Of Tungsten After Hydrogen Species Ion Implantation Consistent w/ Literature Findings



Fukumoto, et al., 2008




- flat PCW
- (1) 700 keV $H^-$
  - $1.6 \times 10^{18}$ $cm^{-2}$
  - T < 200 °C
- (2) 1 keV $H^+$ & $C^+$
  - 0.8% $C^+$
  - $8 \times 10^{20}$ ions/$cm^{-2}$
  - T~380 °C




- UW-IEC Results, 2008
- W-Needle
- 100 keV $D^+$ (4e18 $cm^{-2}$)
- 750 °C