Surface Near Helium Damage in Materials Studied with a High Throughput Implantation Method,

Peter Hosemann;

S. Stevenson, D. Frazer; Y. Yang; Yujun Xie; Xie Huang, Y. Yang, M. Balooch; F. Allen

University of California, Berkeley;

U.S. DEPARTMENT OF ENERGY

Office of Science

future

NSF

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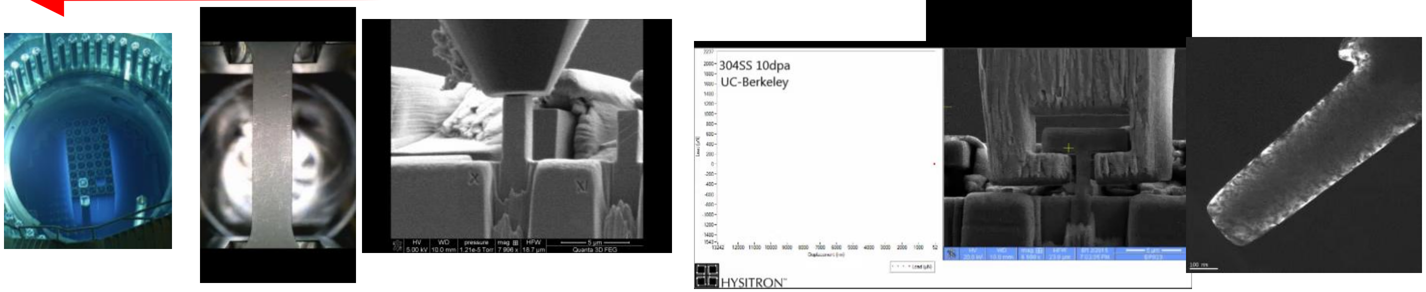
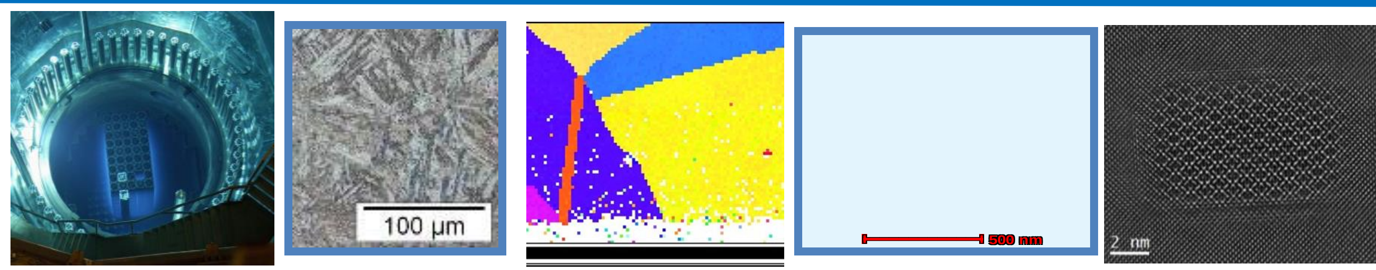
4/18/2024

NSUF user facility at UCB:

m-mm mm-µm µm-nm nm



Mechanical testing



Microstructure

Laser processing/ Laser analytics

Helium Ion Beam Microscopy

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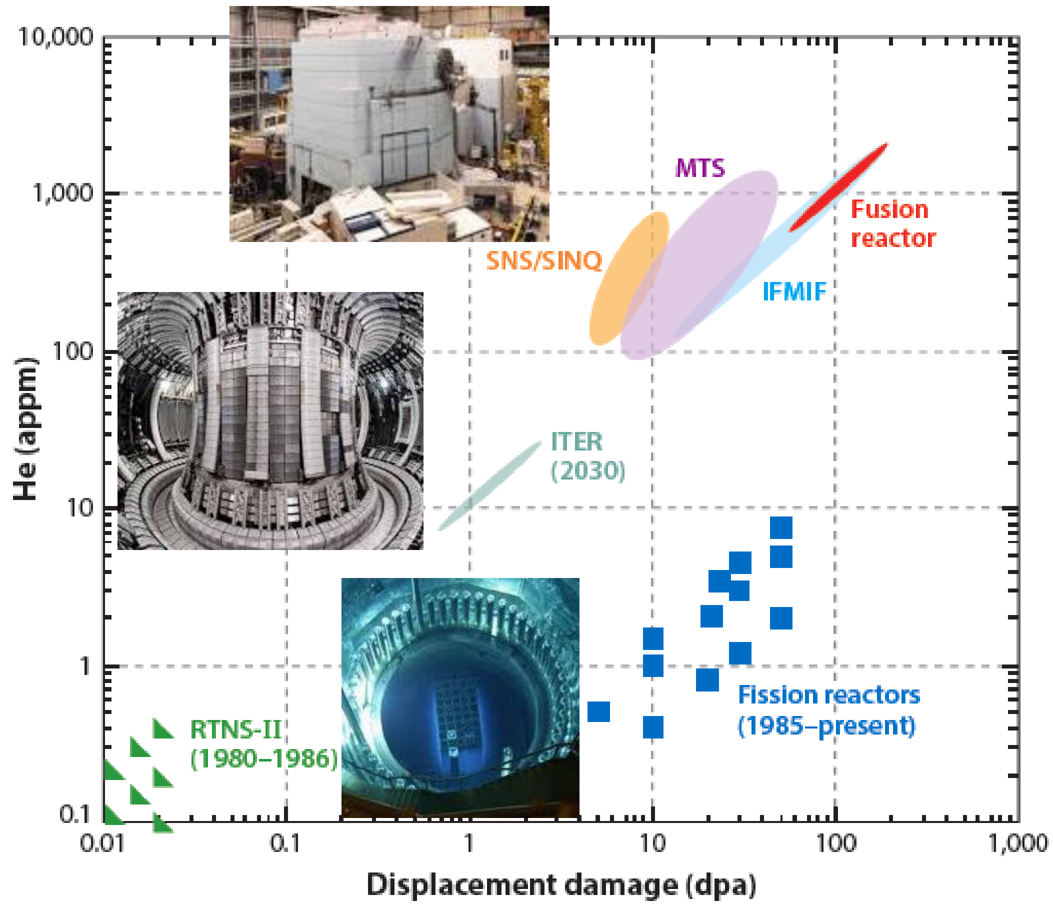
Helium damage in nuclear applications

Helium can be created due to (n,α) nuclear reactions which leads to the formation of He bubbles in the material.

T and He content define the formation of He bubbles.

Can a novel materials testing approach lead to new insight?



S.J. Zinkle and L.L. Snead
Annu. Rev. Mater. Res. 2014. 44:8.1–8.27

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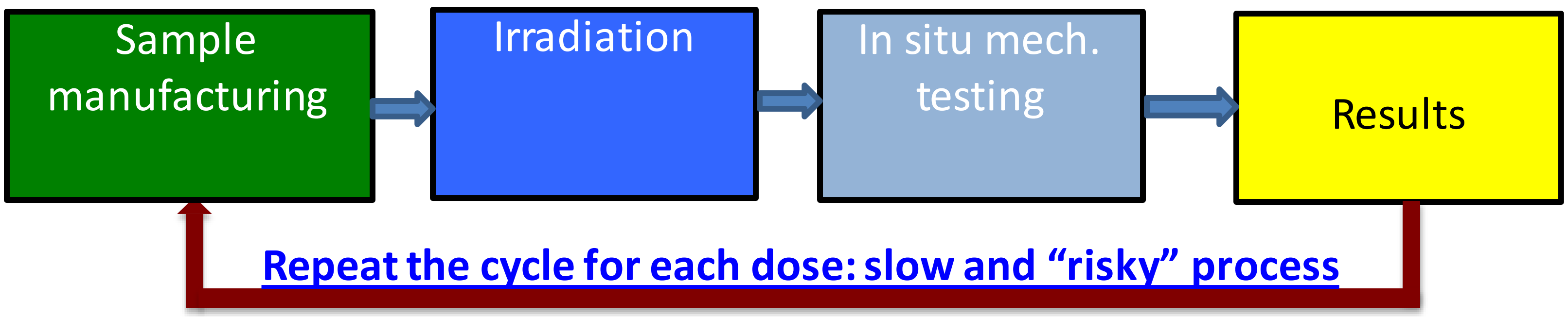
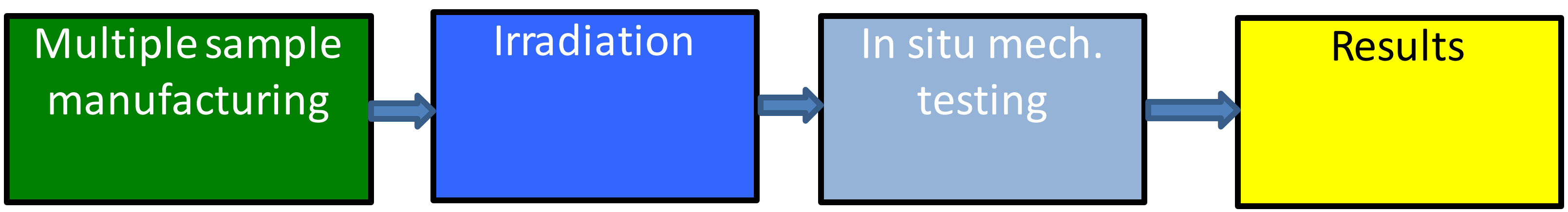
Workflow on implantation and mechanical property studies



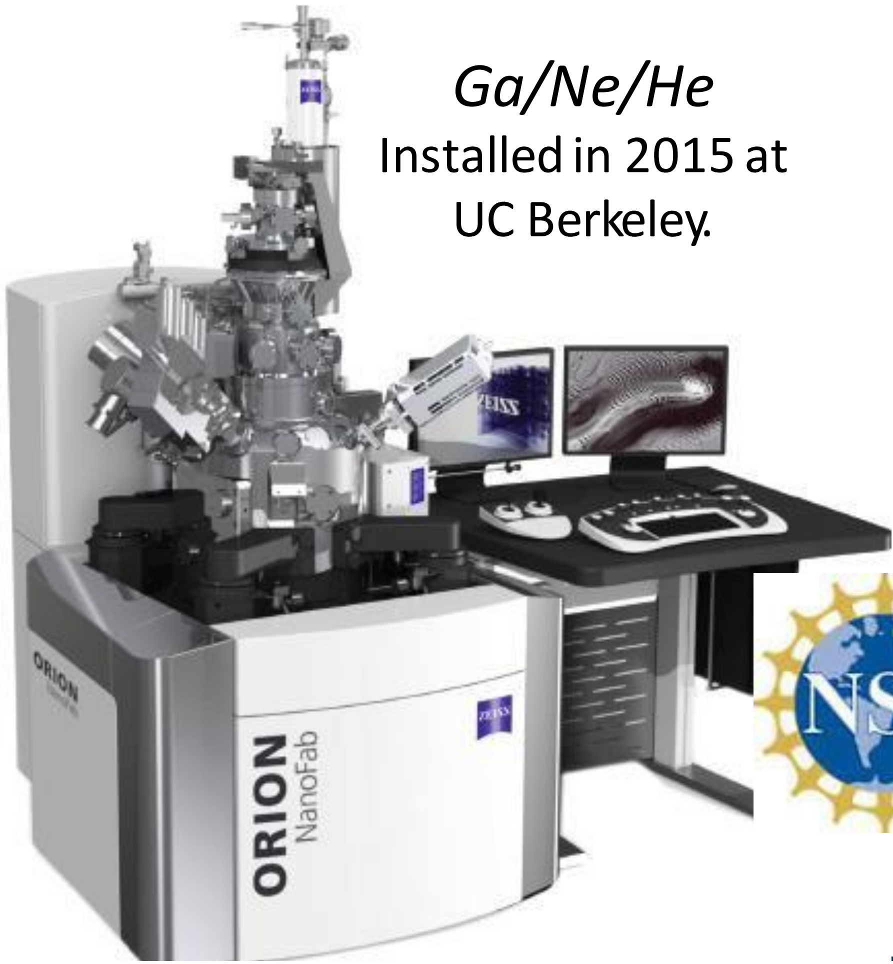
2) Many samples should see multiple doses/irradiation parameters



The same area/grain cannot be followed

The approach presented here, allows to follow the same grain/area to multiple doses within one irradiation

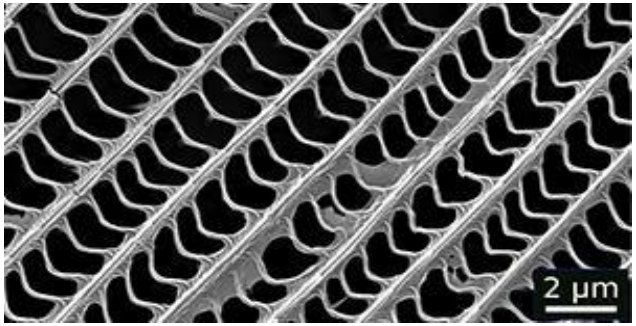
Micro area implantation using the ORION Nanofab

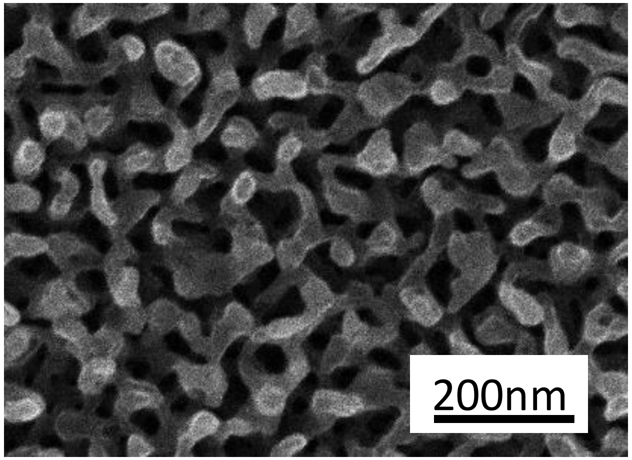


Ga/Ne/He
Installed in 2015 at UC Berkeley.

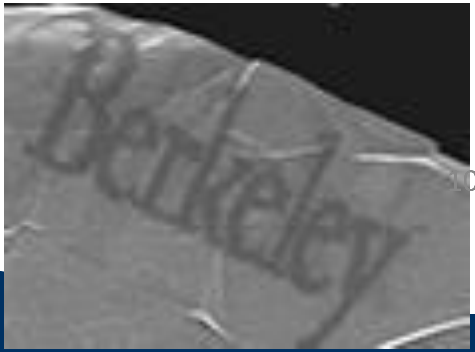
Imaging:

Non conductive samples without coating





Patterning

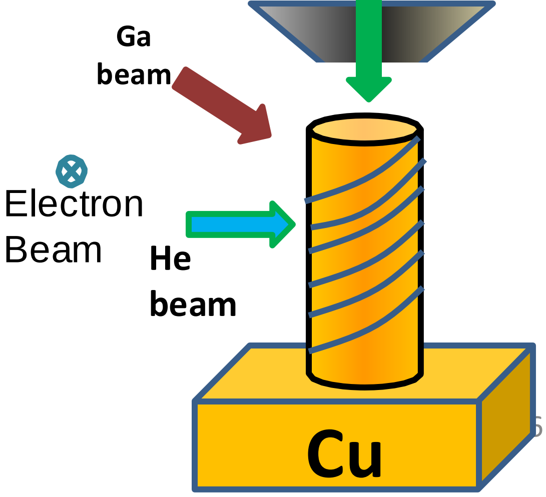


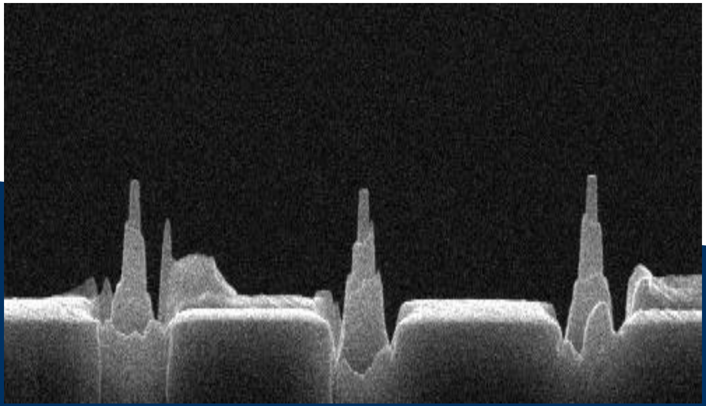
Using the He beam for Implantation in scanning mode

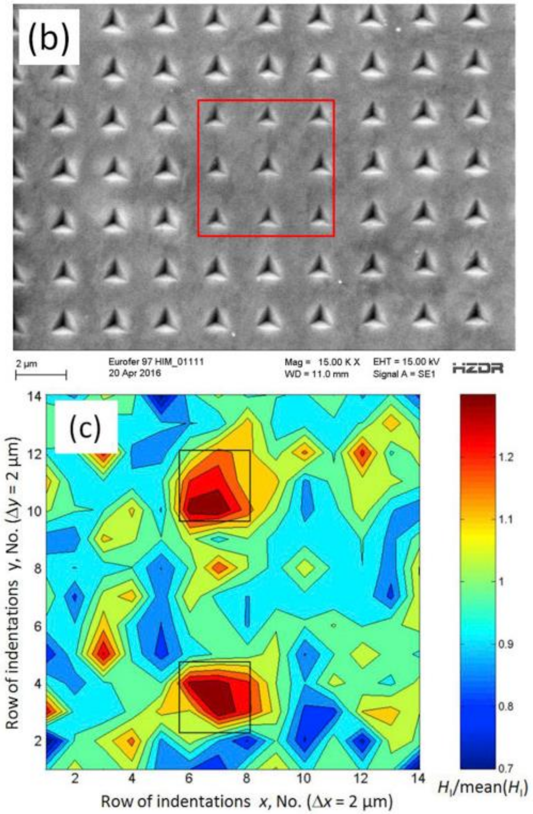
Previous work using similar methodology

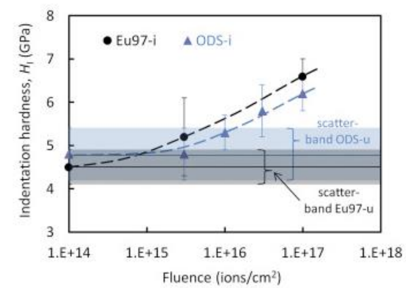
Previous work Z. Wang et al
Acta Mat 121 (2017) 78-84

F. Bergner, G. Hlawacek, C. Heintze,
J. Nucl. Mat, 505 (2018) 267-275









Materials studied at UCB's tool to date

171 citations and H-factor of 11.4

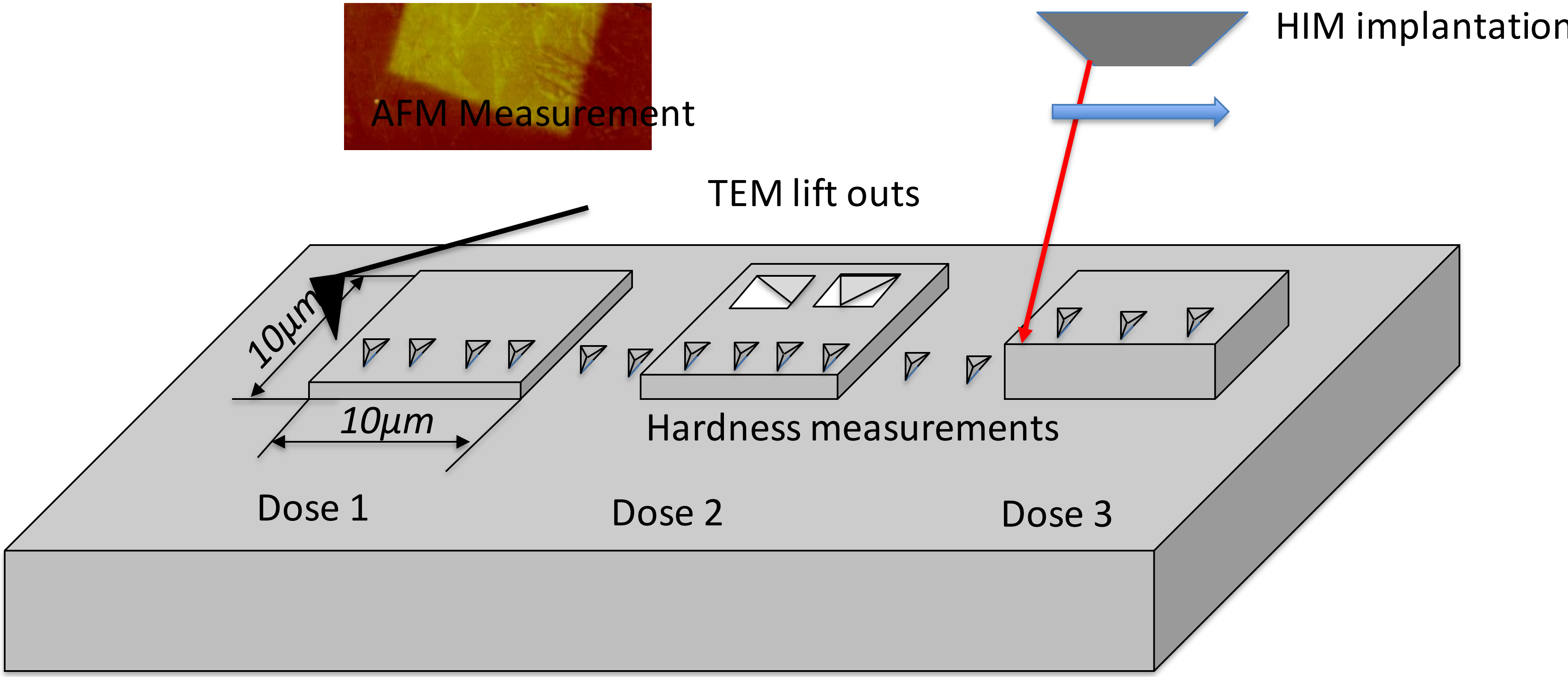
Pure elements

- Vanadium (P. Hosemann JMR 2021)
- Titanium (in preparation)
- SiC (Ambat JOM 2020
- Si (Huang 2023 JAP
- W (Balooch J Nucl Mat 2022
- W (Scripta Allen 2020)
- Copper (Winter J. Nucl. Mat. 2018)
- Copper (Yang J. Nucl. Mat. 2018)
- Copper (Wang Acta Mat. 2016)

Alloys and composites

- 316l –oxidized (Hong JAP 2022
- Zr-BMG –(Huang J. Nucl. mat 2024
- W-Cu composite (Wurmshuber Scripta 2022
- Cu-Fe-Ag composite (Wurmshuber Mat Char. 2022)
- 304 (Schoell JOM 2020
- YsO3-Fe layer (Mairov Scripta 2019)
- F82H (Kooknoh unpublished)
- HEA (Kooknoh unpublished)

Experimental setup for rapid survey of material under Helium implantation



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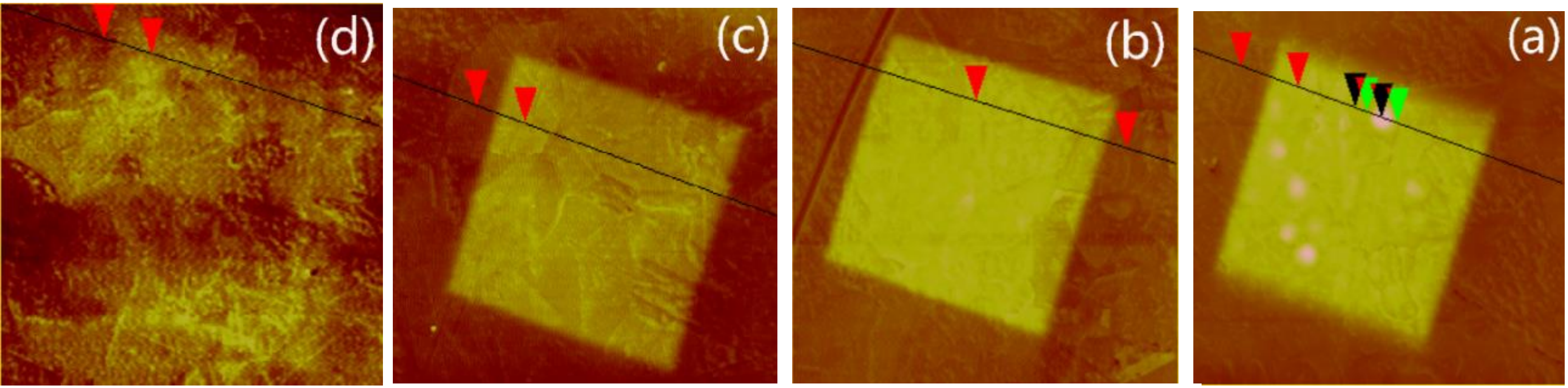
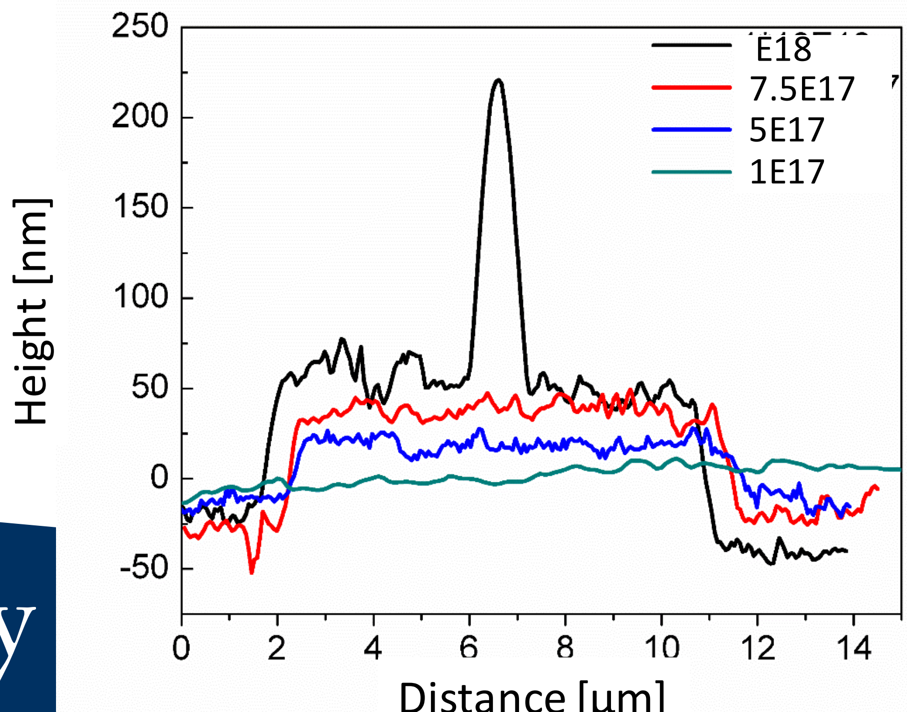
FCC material (Cu)

Scanning probe results (AFM)



1E17 He/cm^2 | 5E17 He/cm^2 | 7.5E17 He/cm^2 | 1E18 He/cm^2

Y. Yang
JNM 2018



Increased volume with higher dose.

At 1E18 He/cm^2 blisters occur

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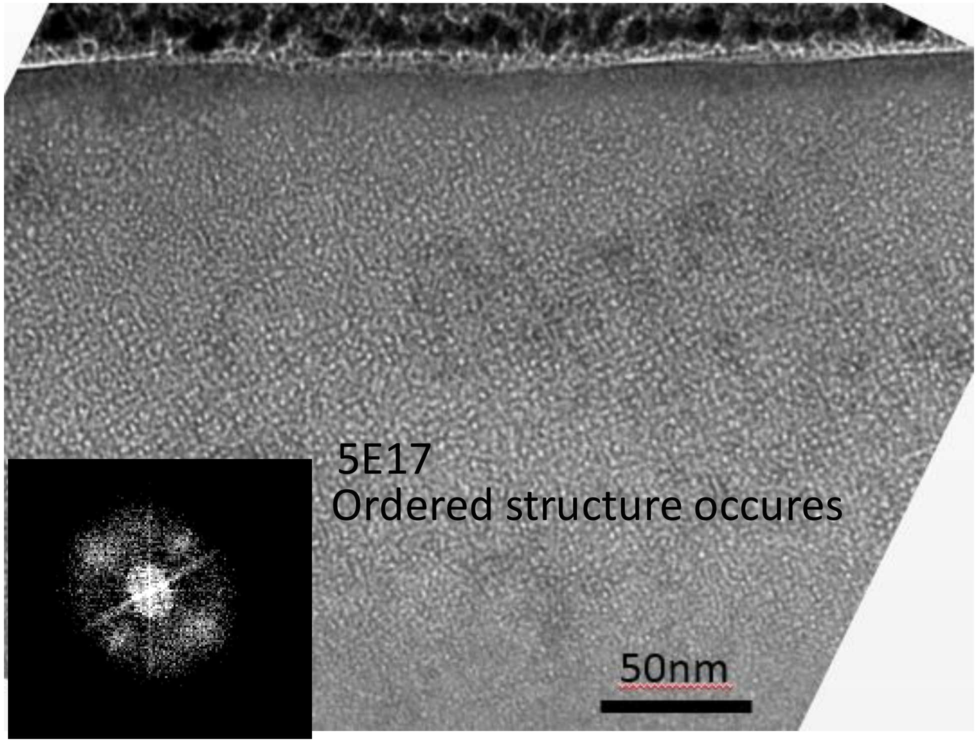
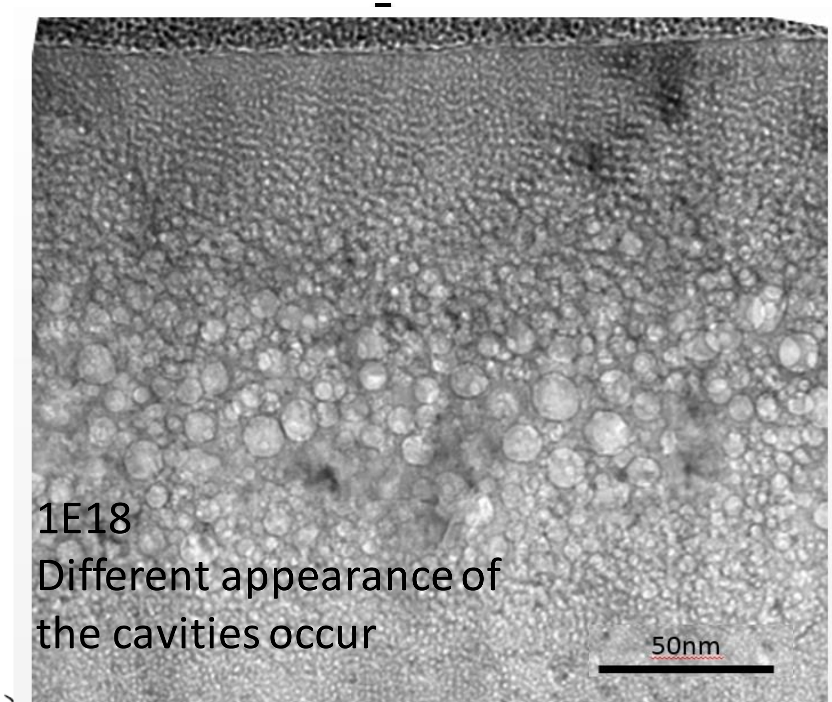
TEM of the implanted samples





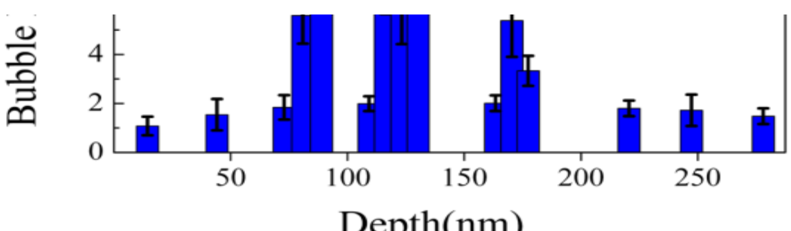
(b)

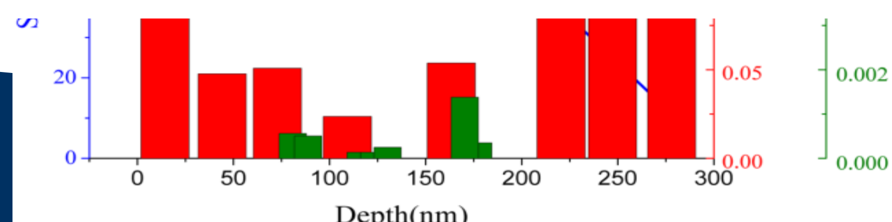
bubbles (bubbles/nm^3)

bubbles (bubbles/nm^3)

<u>Good agreement between TEM and AFM (sputter yield 0.1 atom/He ion)</u>

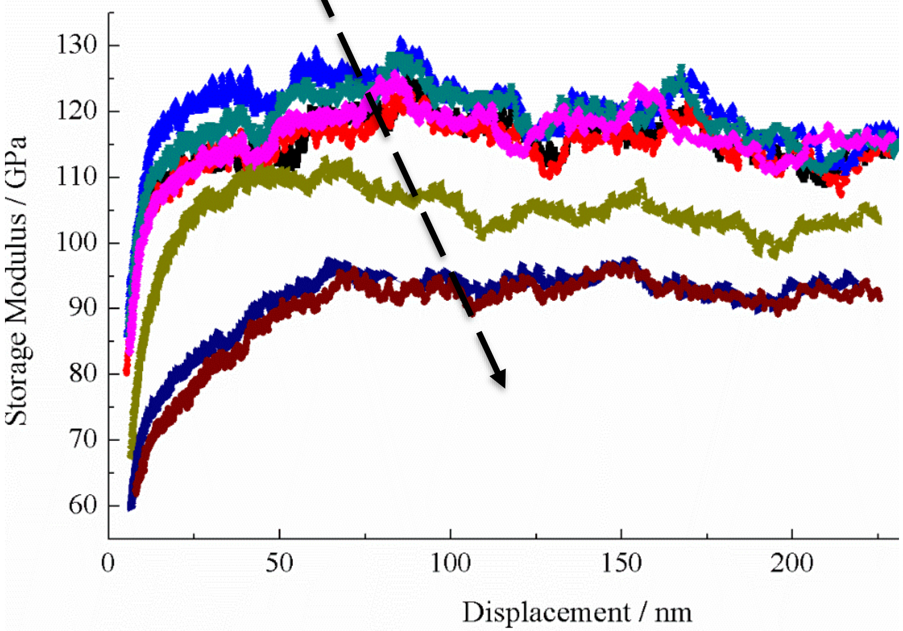
<u>Van der waals consideration leads to the conclusion that the bubbles are under pressurized</u>



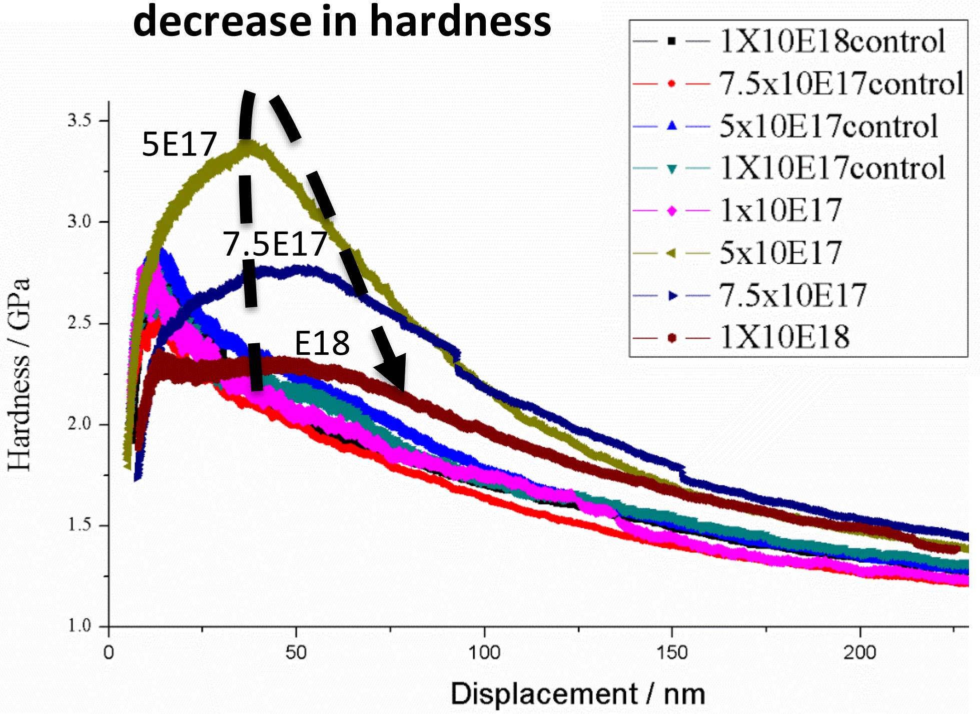


Nanoindentation results

Reduction in Elastic properties with more He.



Increase and then decrease in hardness



Elastic properties decrease with helium content
Hardness increases and decreases with helium content

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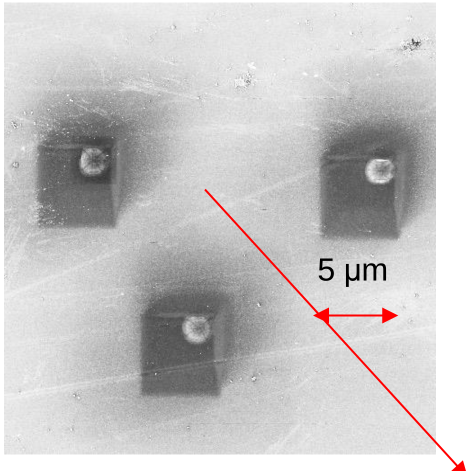
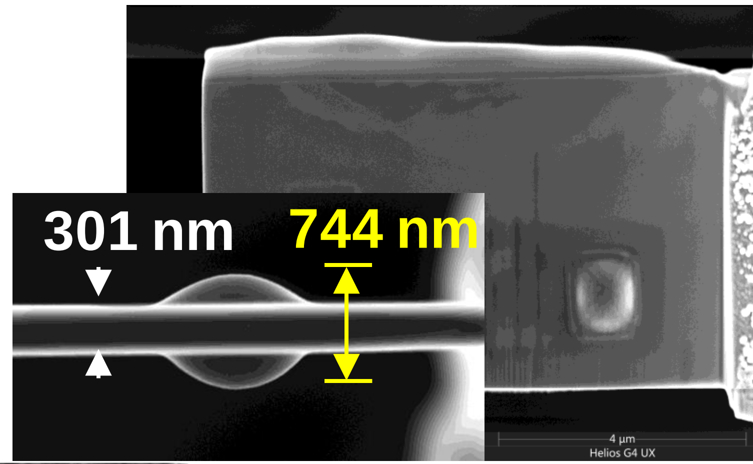
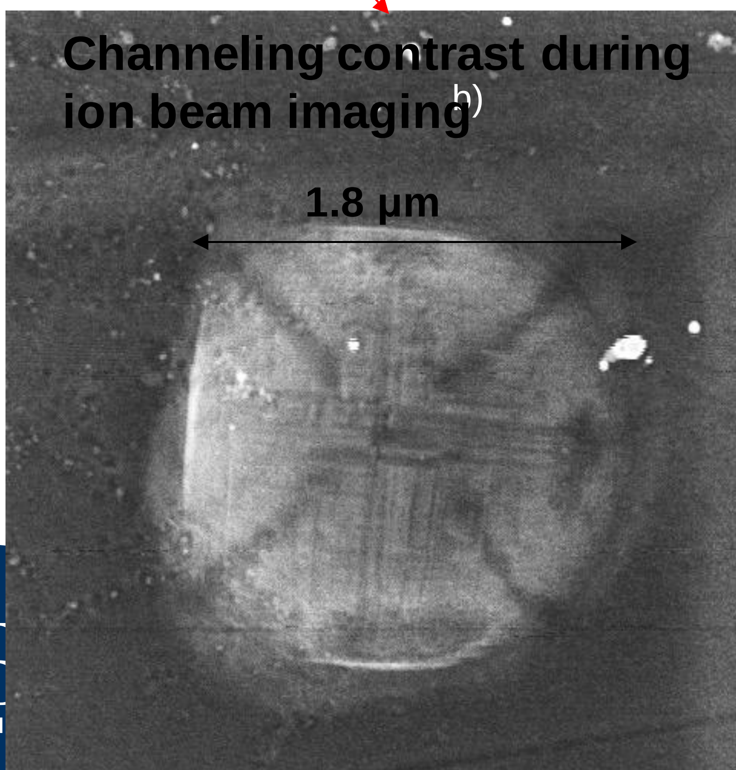
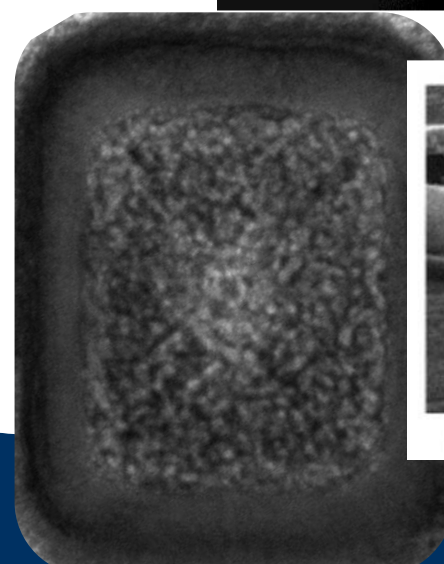
Blistering and Channeling

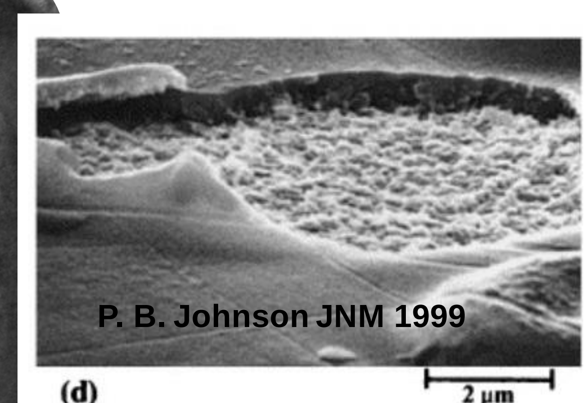


Blistering starts to occur around 7×10^{17} ions/cm^2 dose, at a rate of 3×10^{15} ions/cm^{2}s for **both Cu(100) and polycrystalline Cu**

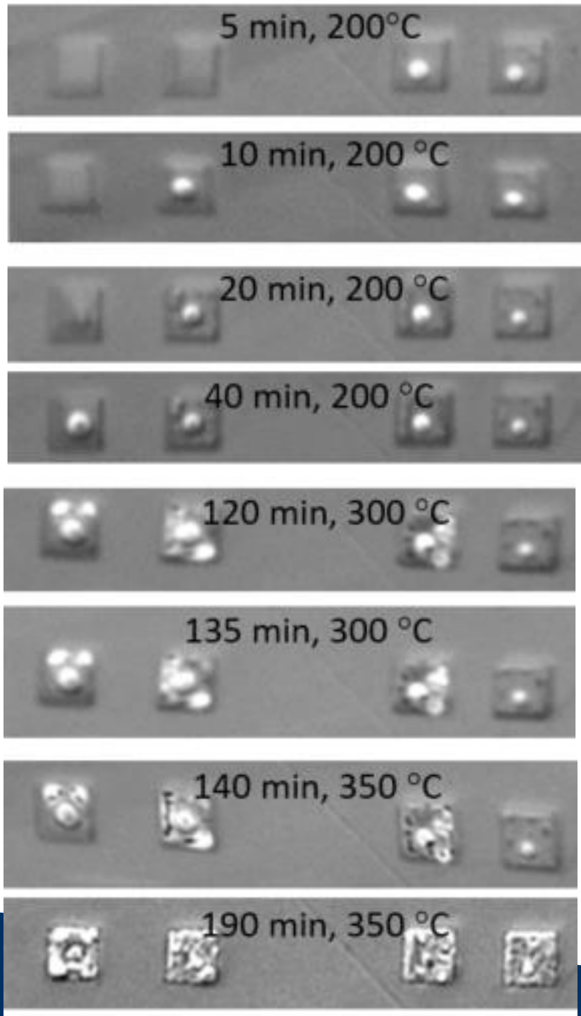


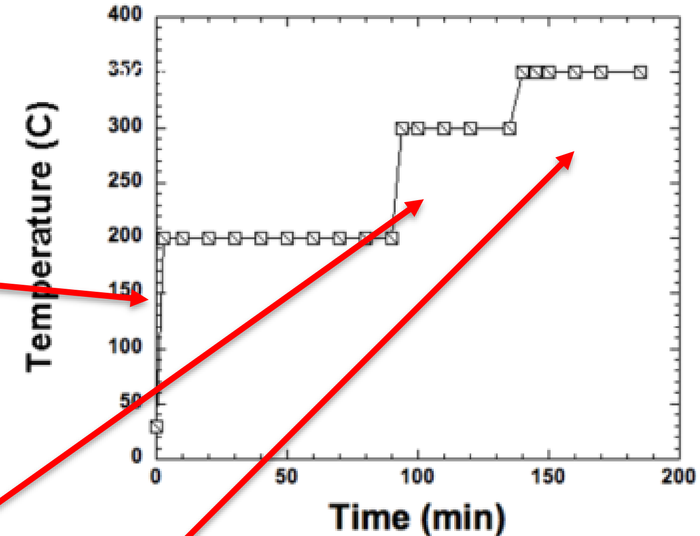




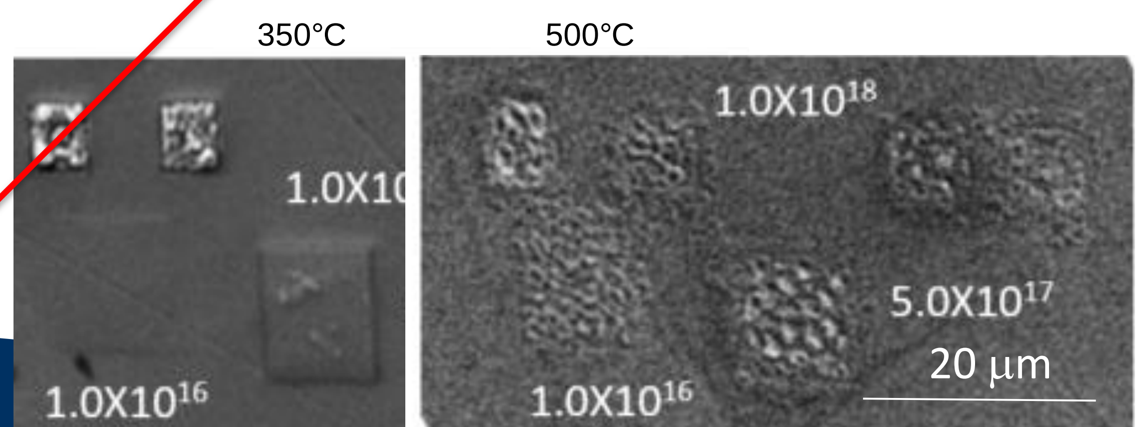


Annealing after irradiation inside the SEM





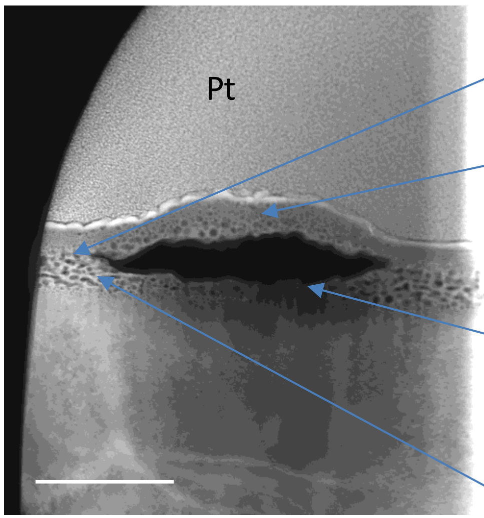
Migration of Helium bubbles to the surface

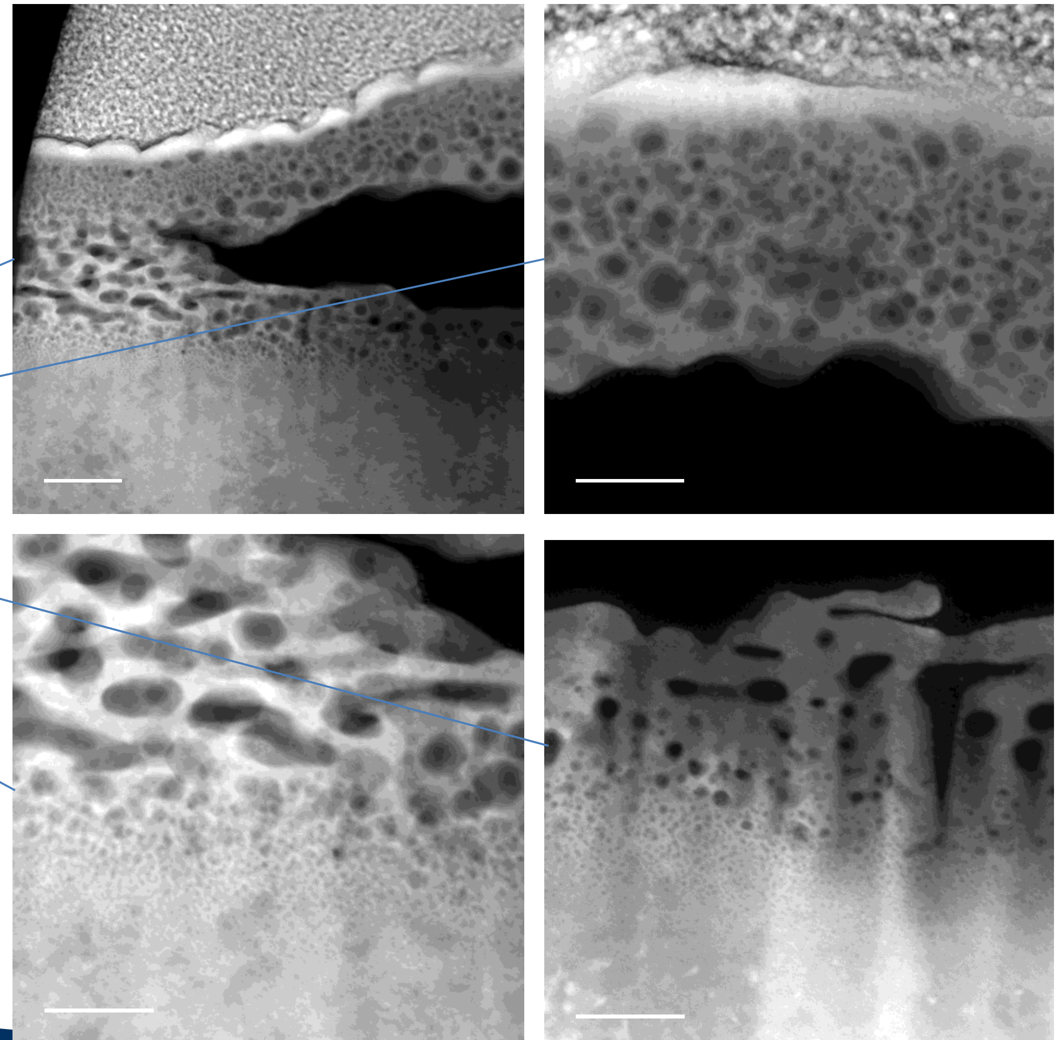


Unpublished data

TEM of the Blisters

HAADF-STEM

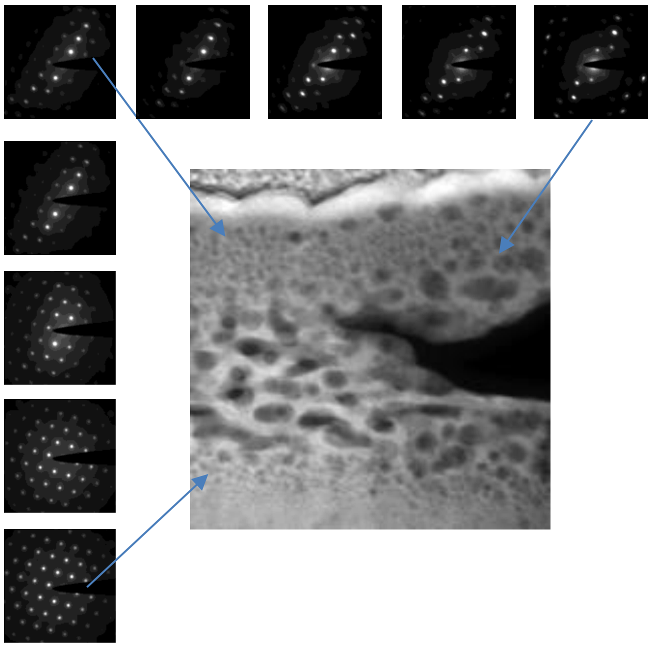


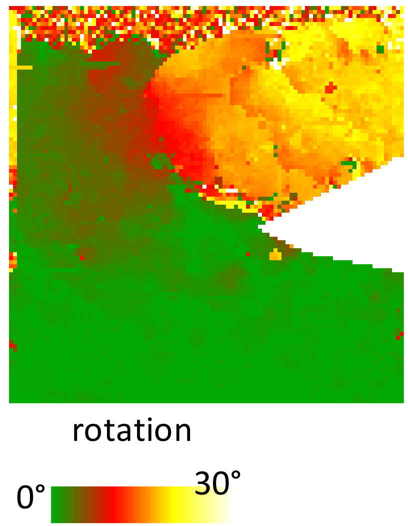


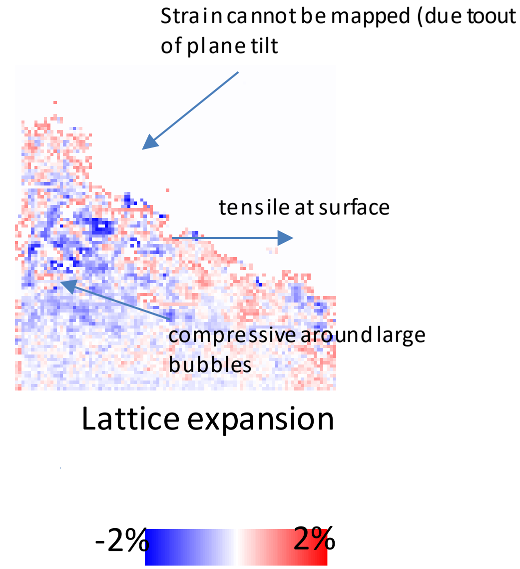
Bubble size gradient:
Bottom: 1 nm – 5 nm clear gradient
Middle large bubble up to 20 nm
mixture of bubble radii

4D STEM of the Blisters

Sample remains fully single crystalline







Continous rotation between areas, discontinous roatation wheere delamination occurs, see also roation map

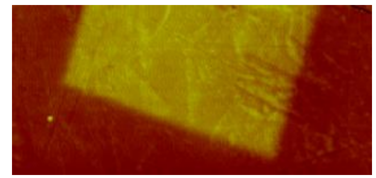
FCC material (316l and oxidation)

Does Helium irradiation accelerator oxidation?

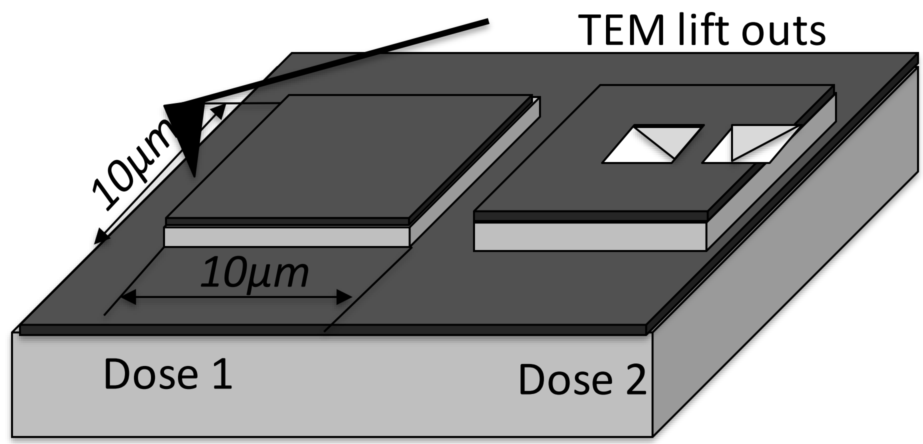
Oxidation of pre-implanted samples (316l)

Research Question: What will happen to helium bubbles when the material is oxidized?

AFM Measurement



M. Hong et al.
J. Apl. Phys Nov. 2022



Oxidized at 400C for 5h and 10h post implantation
Question: Dose the pre-implanted area oxidize faster?

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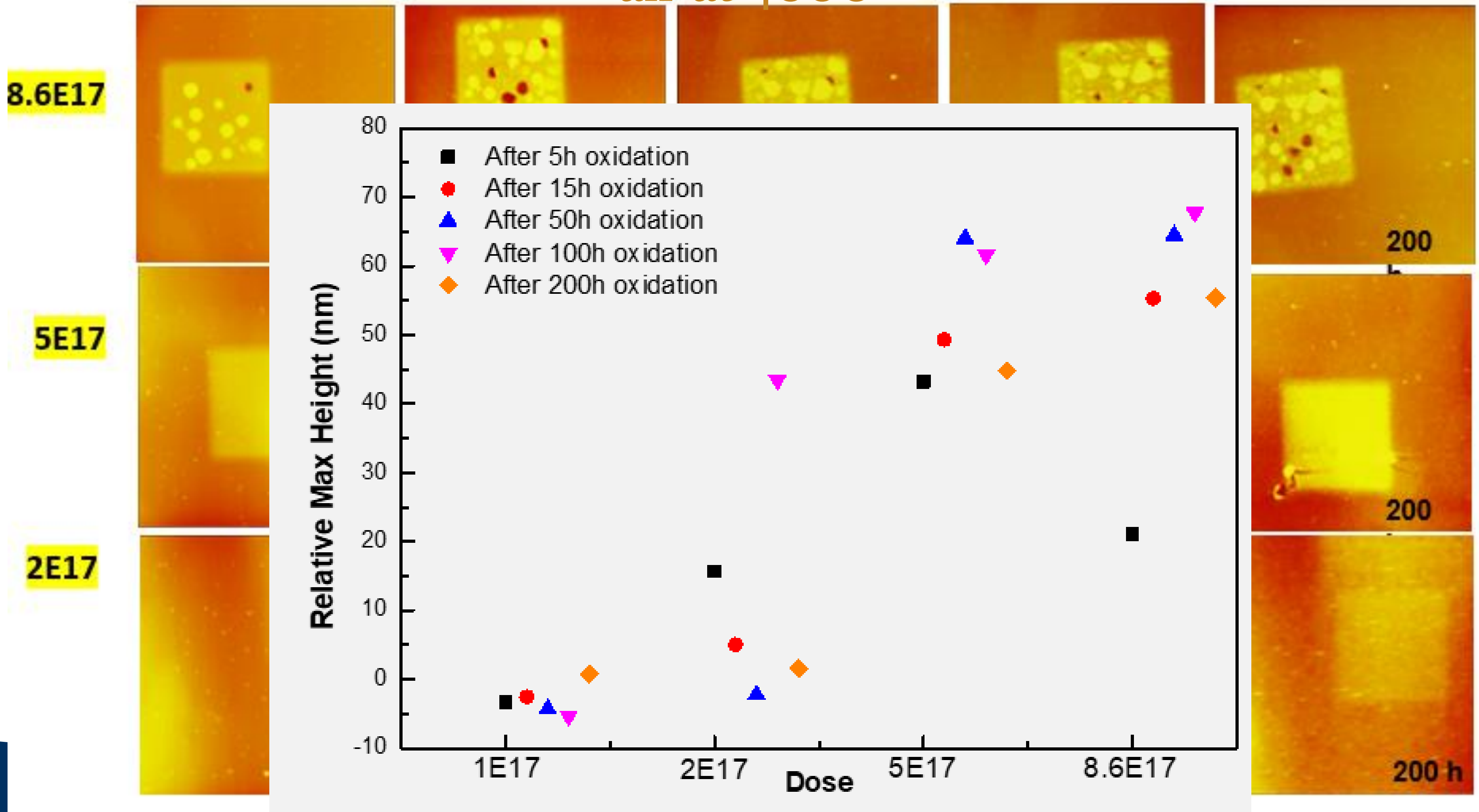
Surface changes after exposure

air at 400C



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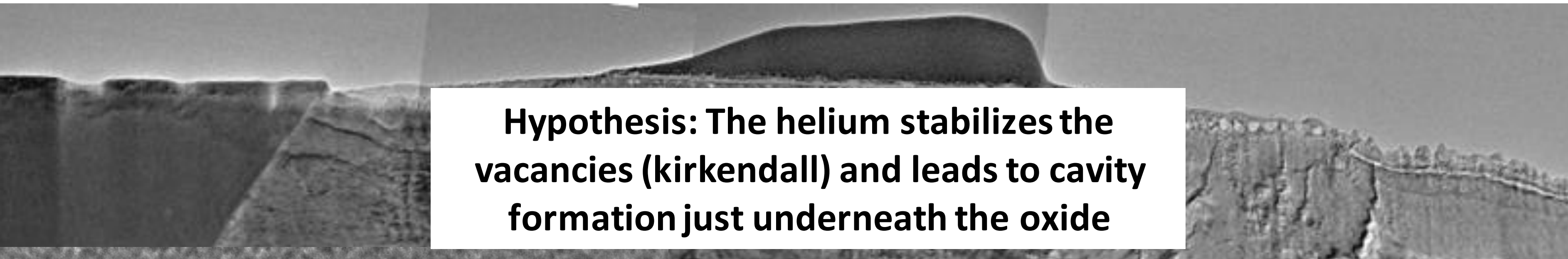
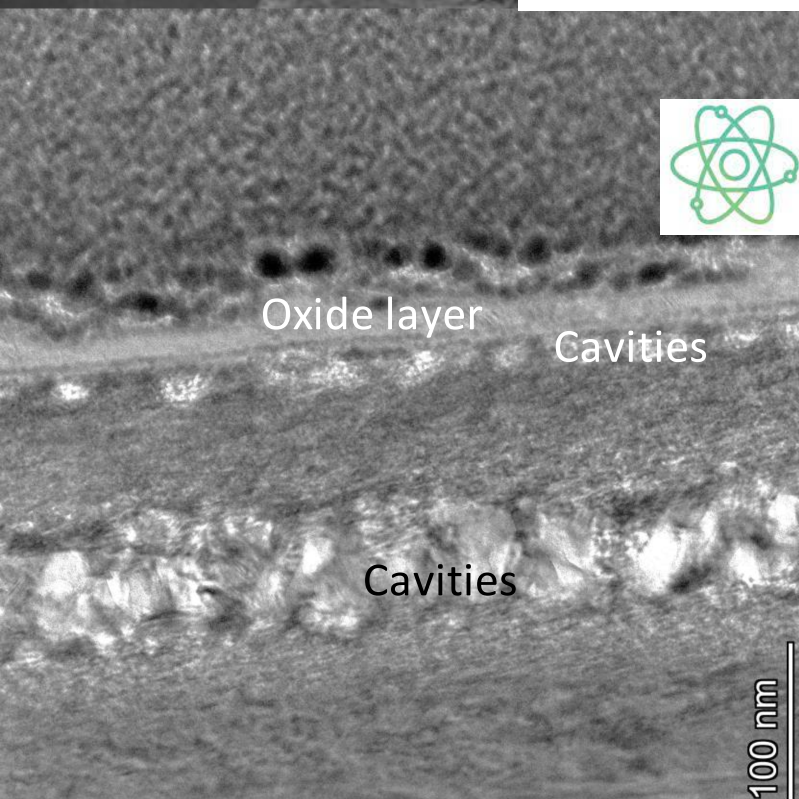
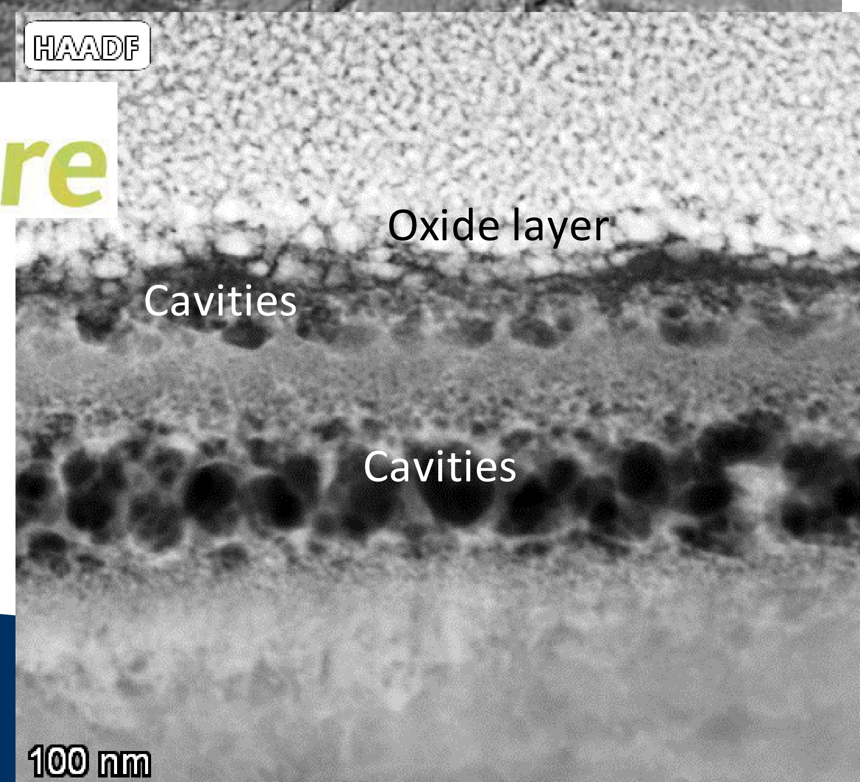
Observation of pre-implanted oxidation 400C 5h 5E17

M. Hong, J. Appl. Phys. 2022



Hypothesis: The helium stabilizes the vacancies (kirkendall) and leads to cavity formation just underneath the oxide





HIM implantation in Si

X. Huang, Y.J. Xie, M. Balooch, S. Lubner and P. Hosemann

Published J. Appl. Phys. 2022

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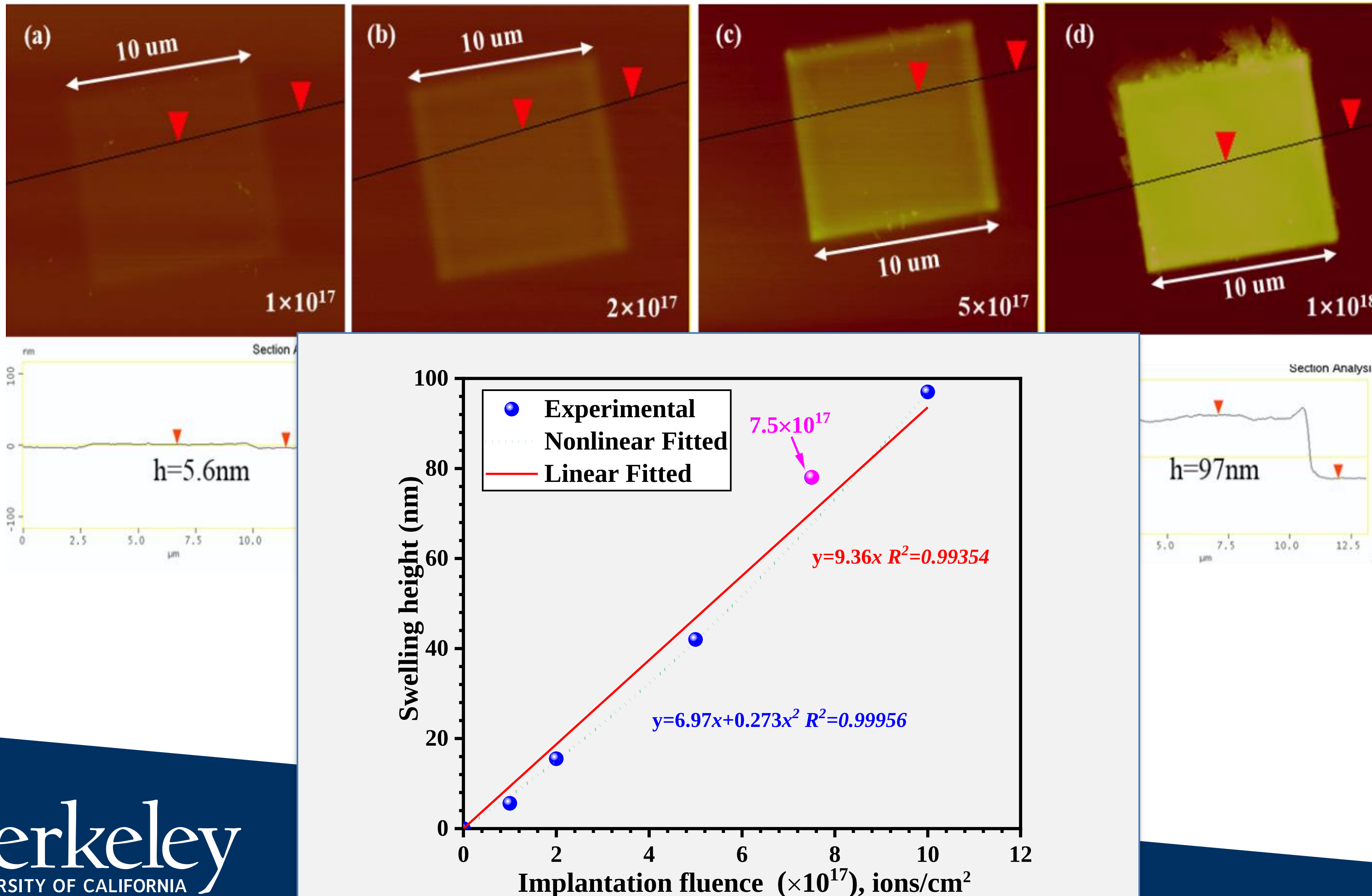
Swelling as a function of dose in Silicon

M. Balooch & P. Hosemann
Unpublished data 2021



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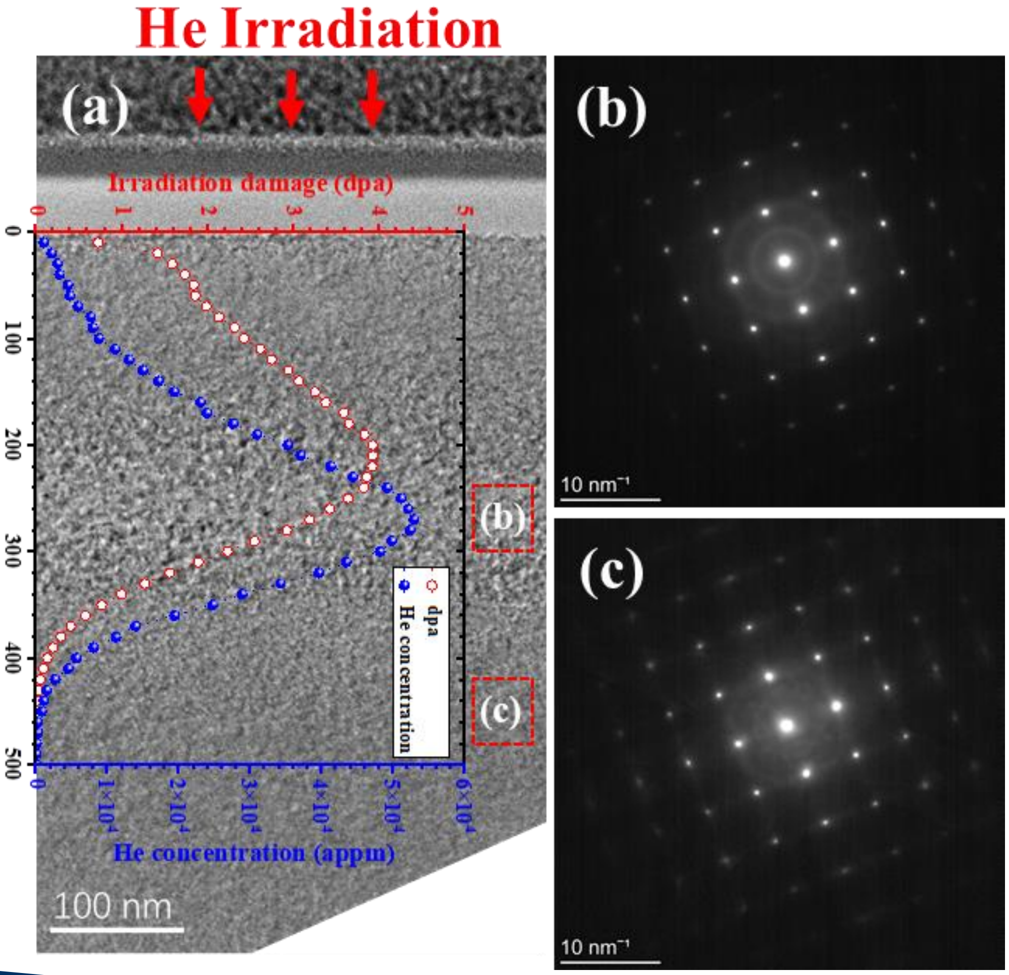
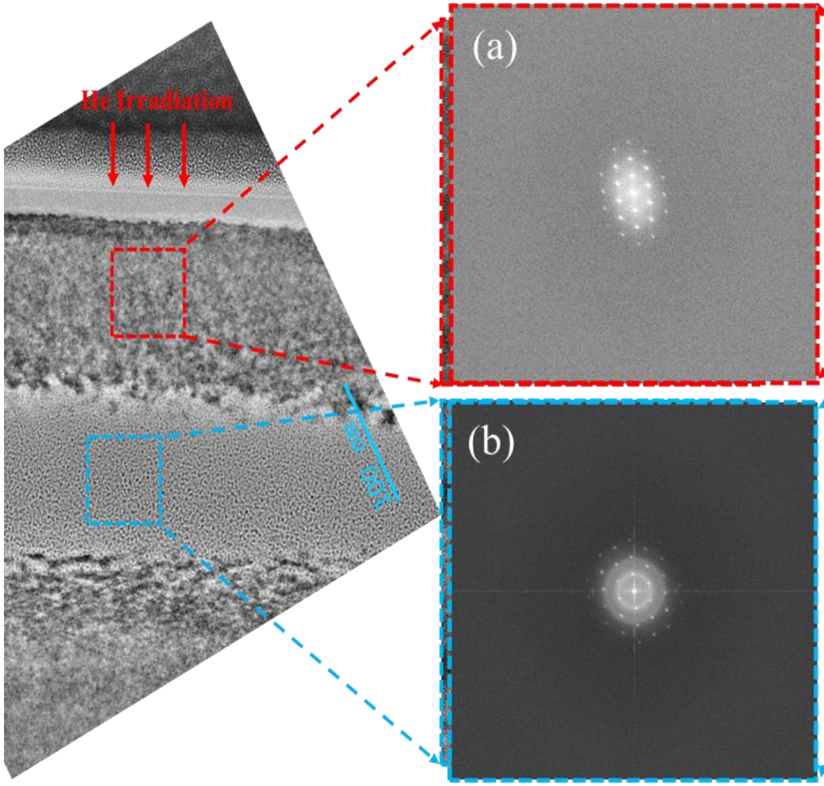
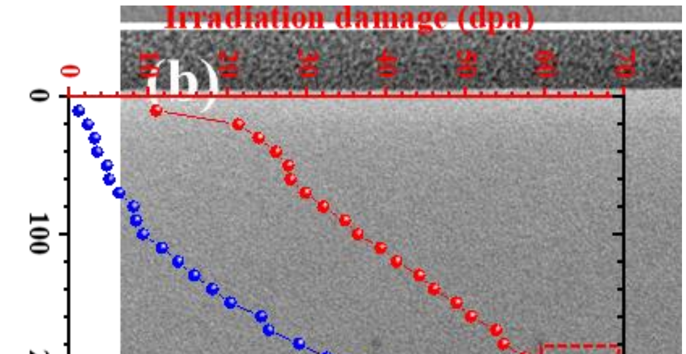
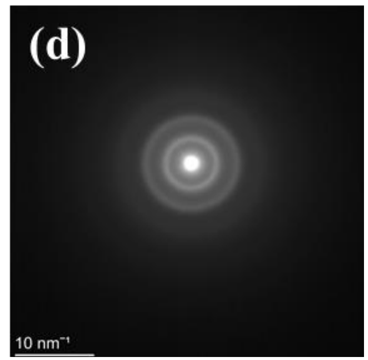
Dose $5x10^{16}$ He ions/cm^2 and 1E17ions/cm^2

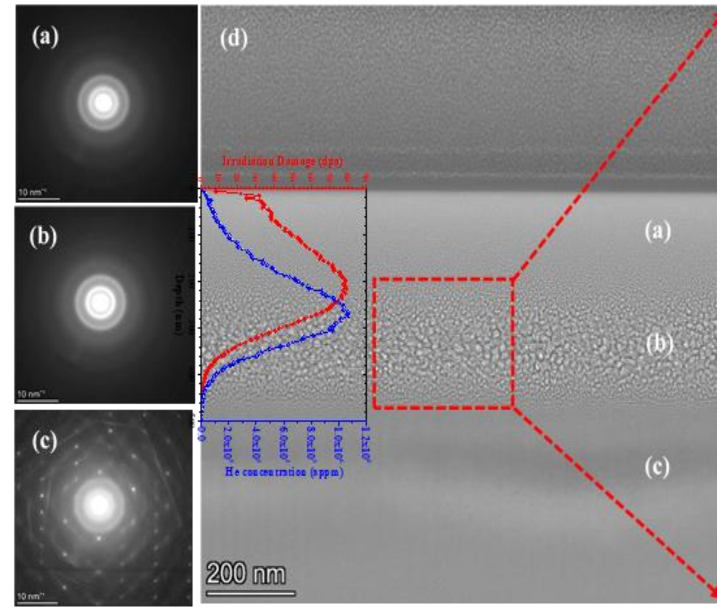


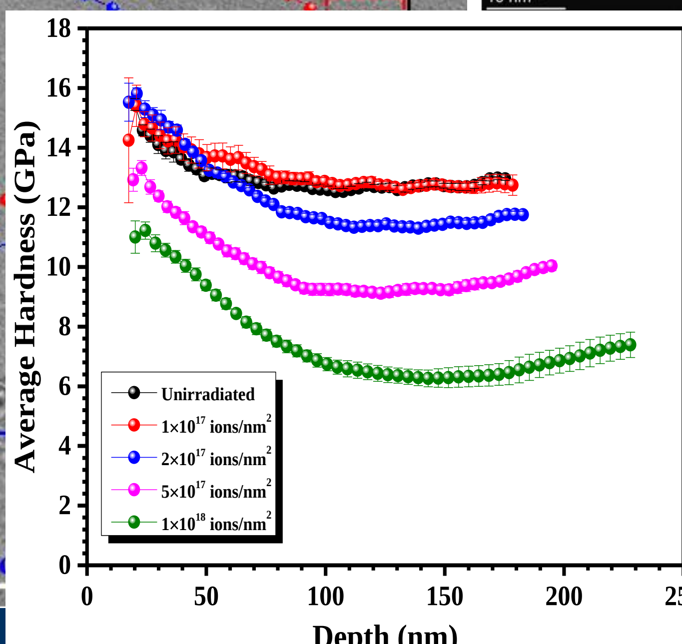


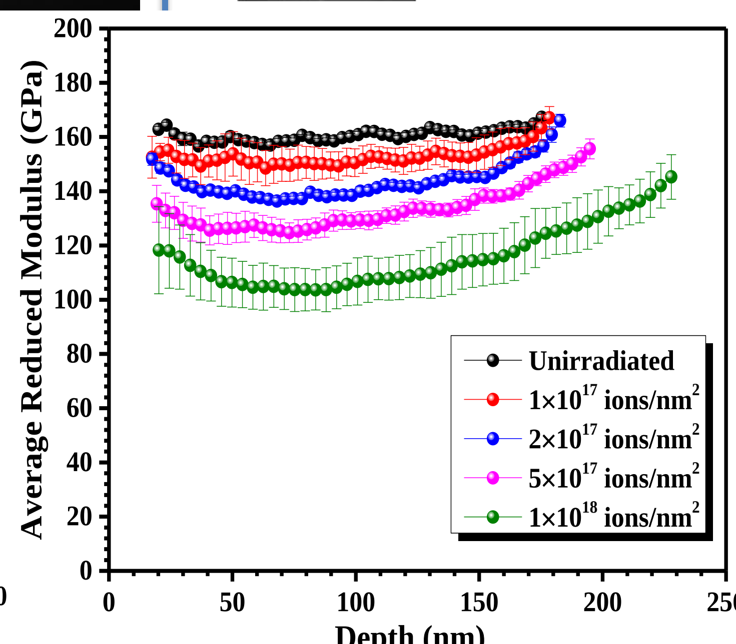
Dose $7.5x10^{17}$ ions/cm² 1 $x10^{18}$ ions/cm²

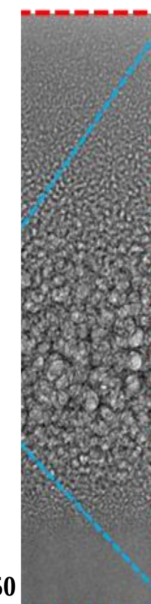






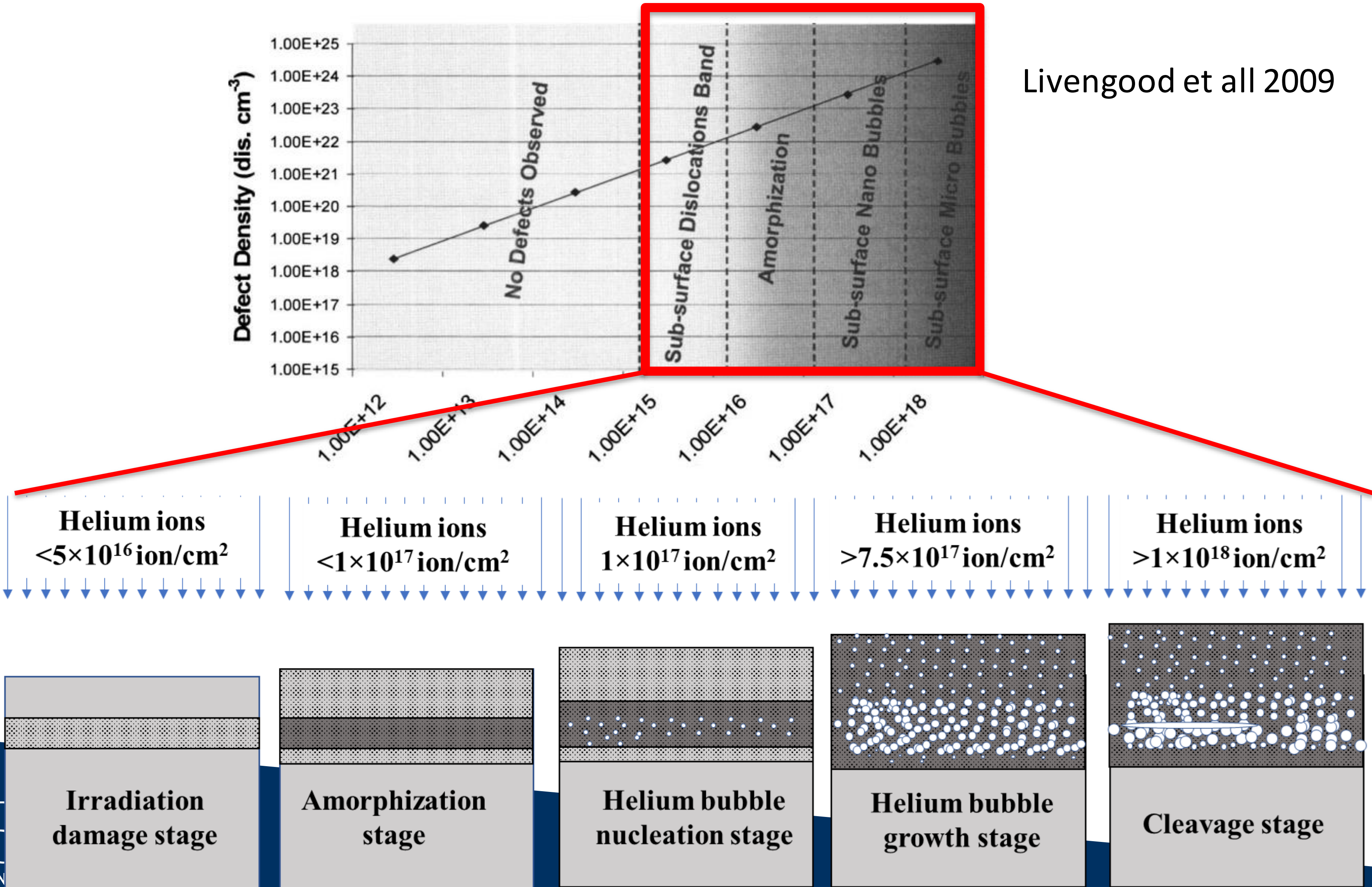






Changes in Silicon with Helium dose

Livengood et all 2009



Summary

- Helium implantation with HIM is a rapid and high throughput method to evaluate materials evolution under Helium implantation
- Highly localized and precise method to target specific regions of interest
- Large library of data has been established
- International community started to use similar methods

THANK YOU FOR YOUR ATTENTION!

Questions ?

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Summary

- Introduction of Helium implantation using HIM and surface near techniques for rapid screening studies and detailed property examinations.
- Examination of Helium swelling and blistering using TEM and AFM in Cu yields good agreement between different characterization techniques.
- Blisters start to develop by linking up Helium bubbles and developing critical cracks.
- Find the change of Silicon upon Helium ion beam irradiation as a function of dose.
- Ion beam and electron beam channeling occurs on the blisters.
- Residual stress measurements reveal that blistered material exceeds the flow stress at similar length scales.

THANK YOU FOR YOUR ATTENTION!

Questions ?

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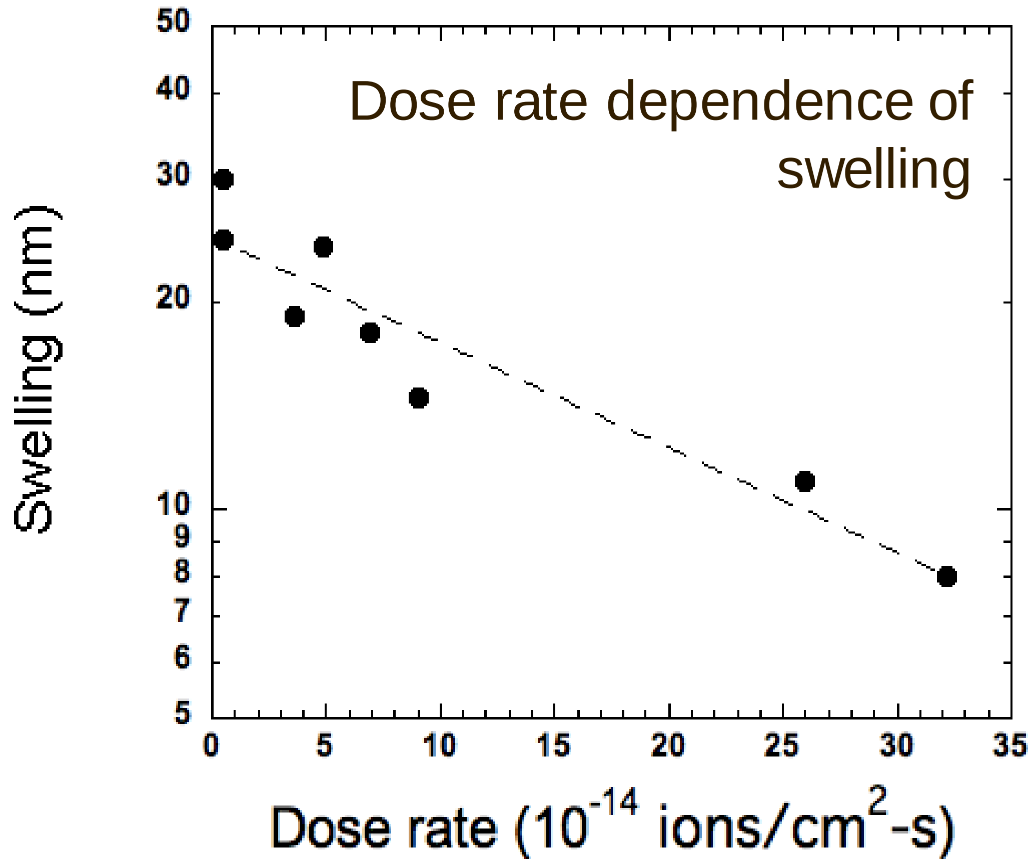
Weak effect of different dose rates

25 keV He ions
2×10^{17} ions/cm^2
implantation

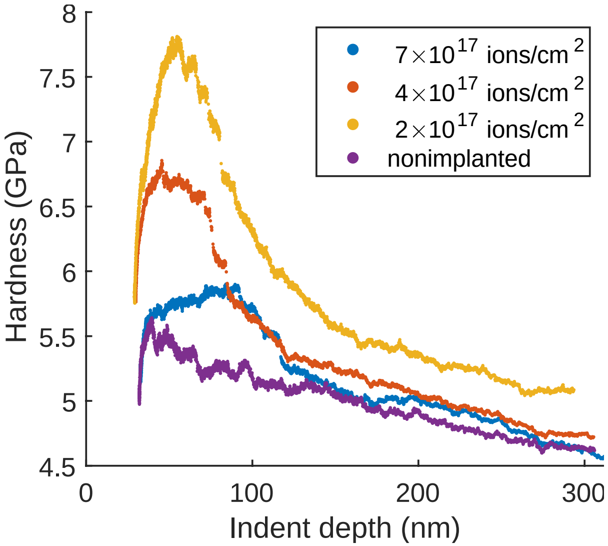


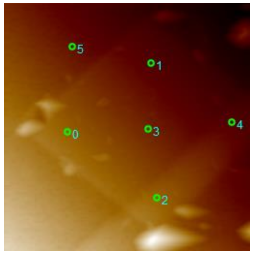
TEM will show how the bubble structure changes with dose rate

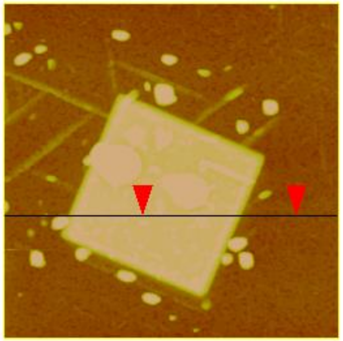
Helium implantation in hcp

Titanium

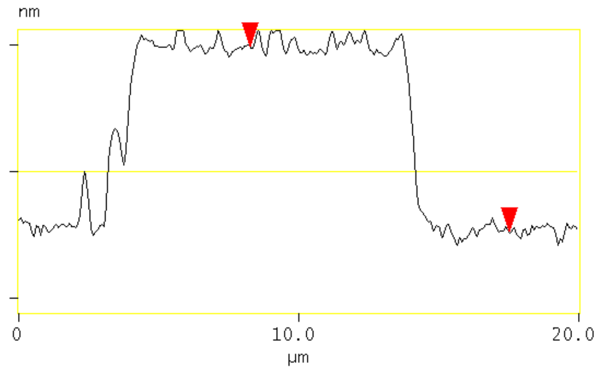
Volumetric swelling (AFM) and indentation

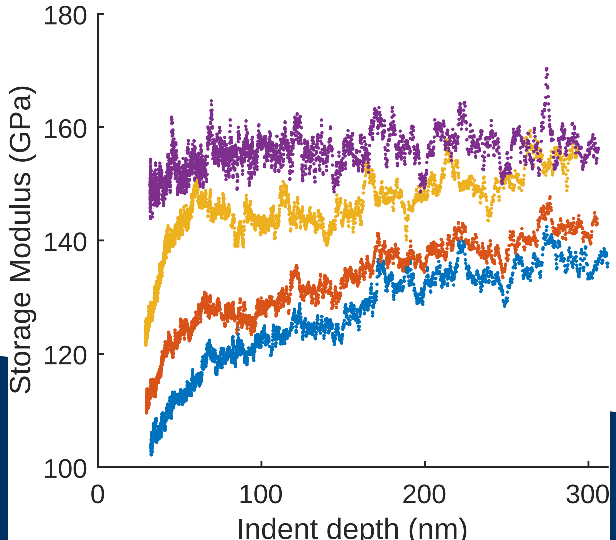


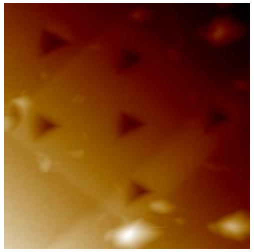




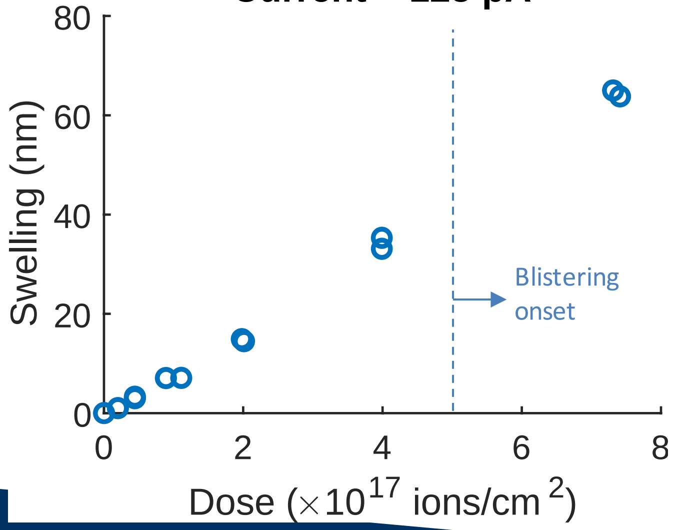
7×10^17 ions/cm^2 implant



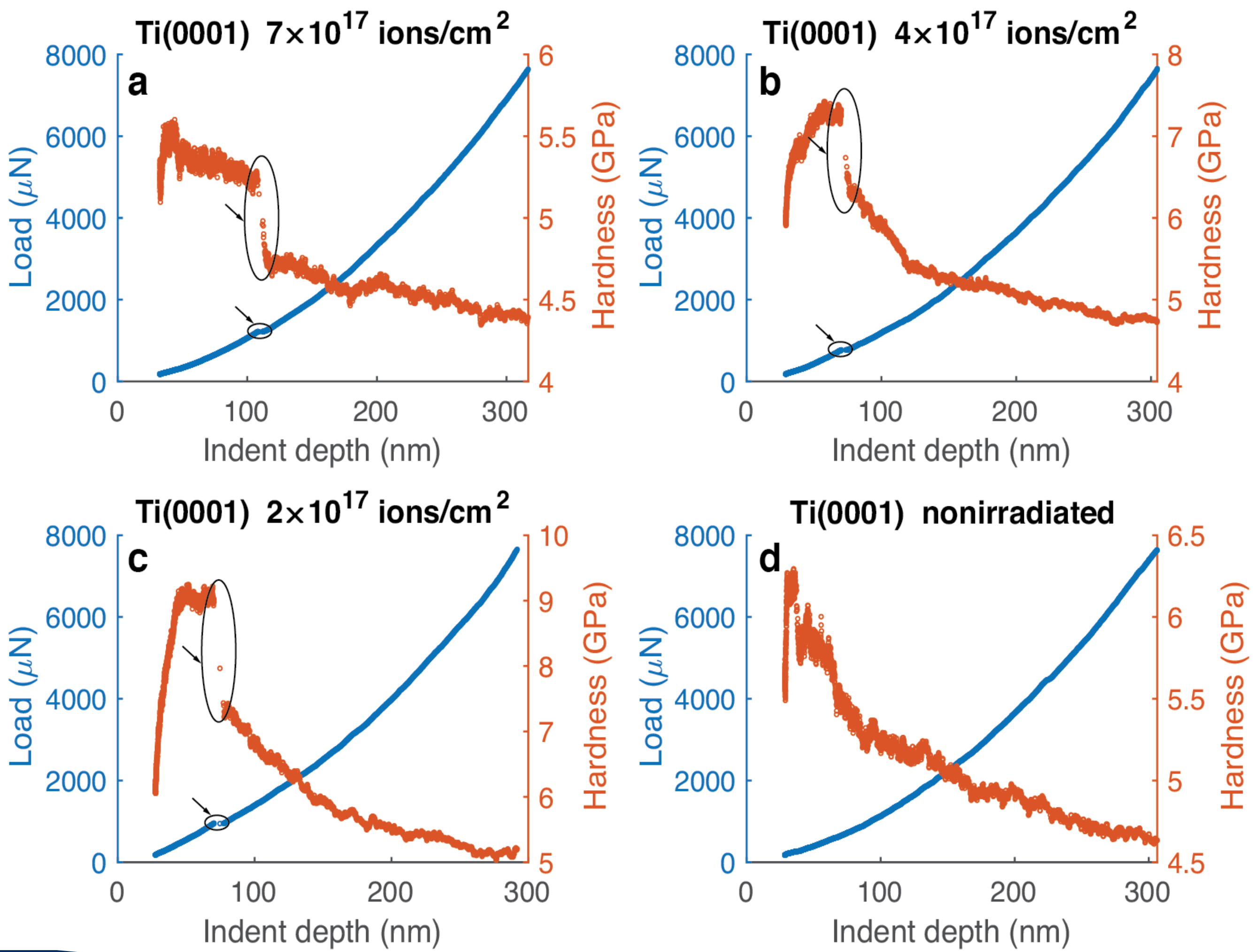




Dose dependence of swelling
Current = 125 pA



Pop-in behavior (Nanoindentation)



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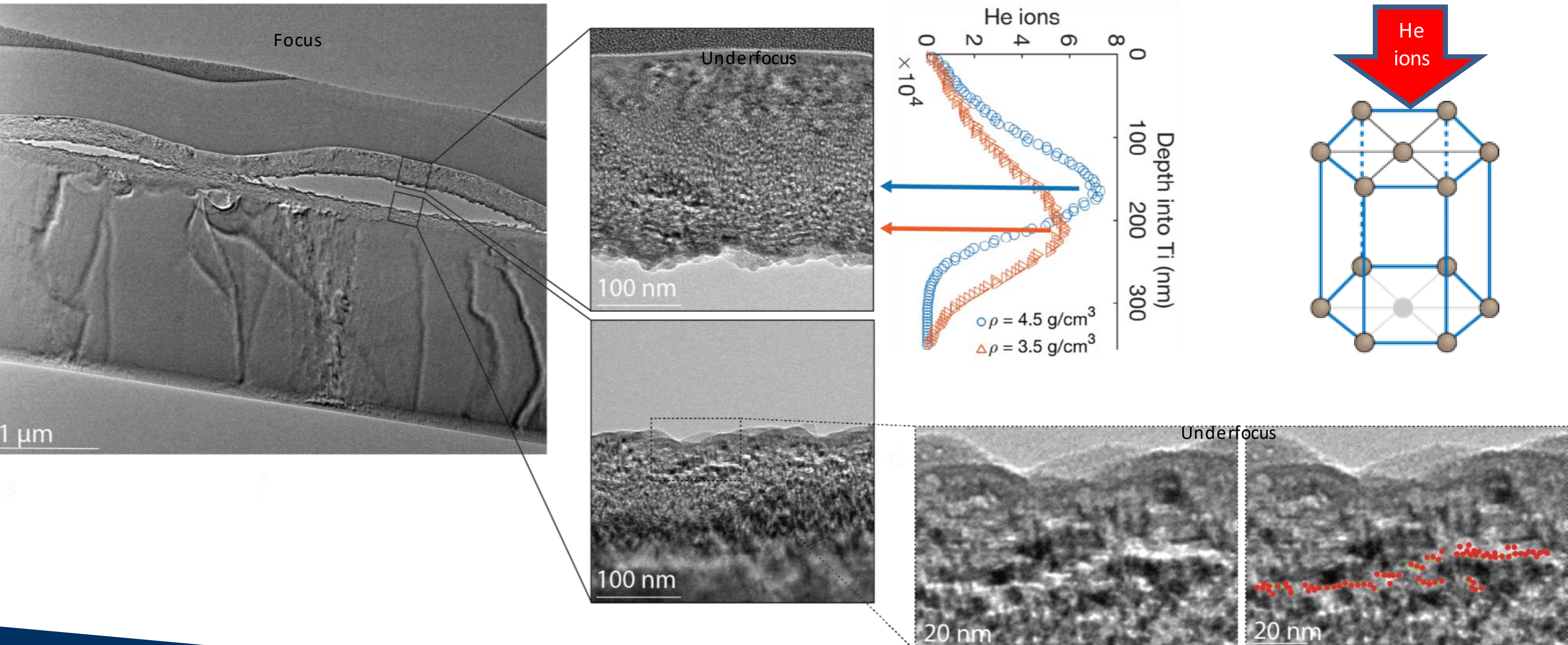
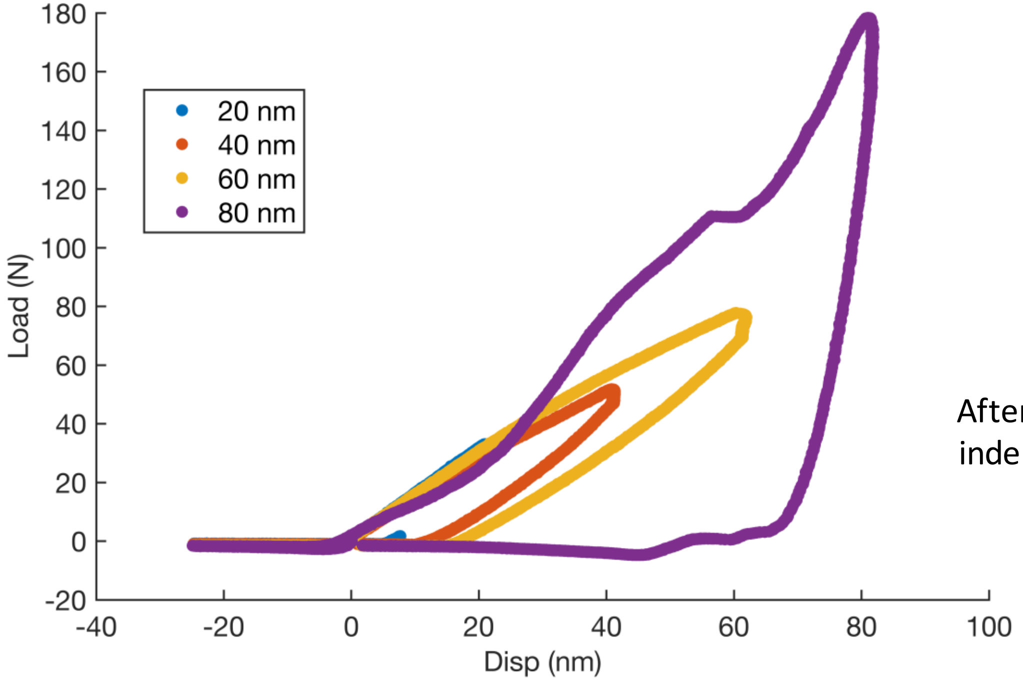
Bubble distribution (TEM)

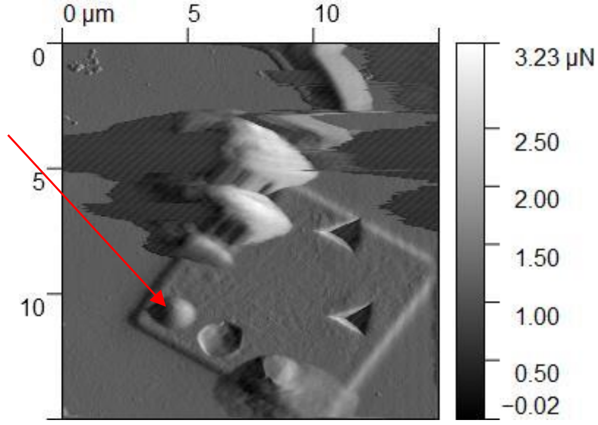
$7{\times}10^{17}$ ions/cm^2 implant



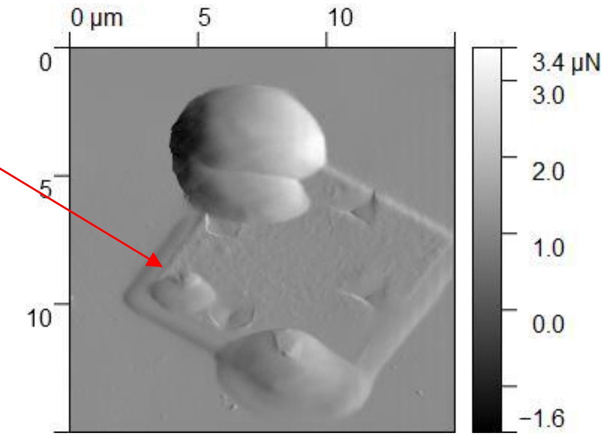
Ti(0001) 7E17 ions/cm2: 20 nm – 80 nm



Before blister indentation



After blister indentation



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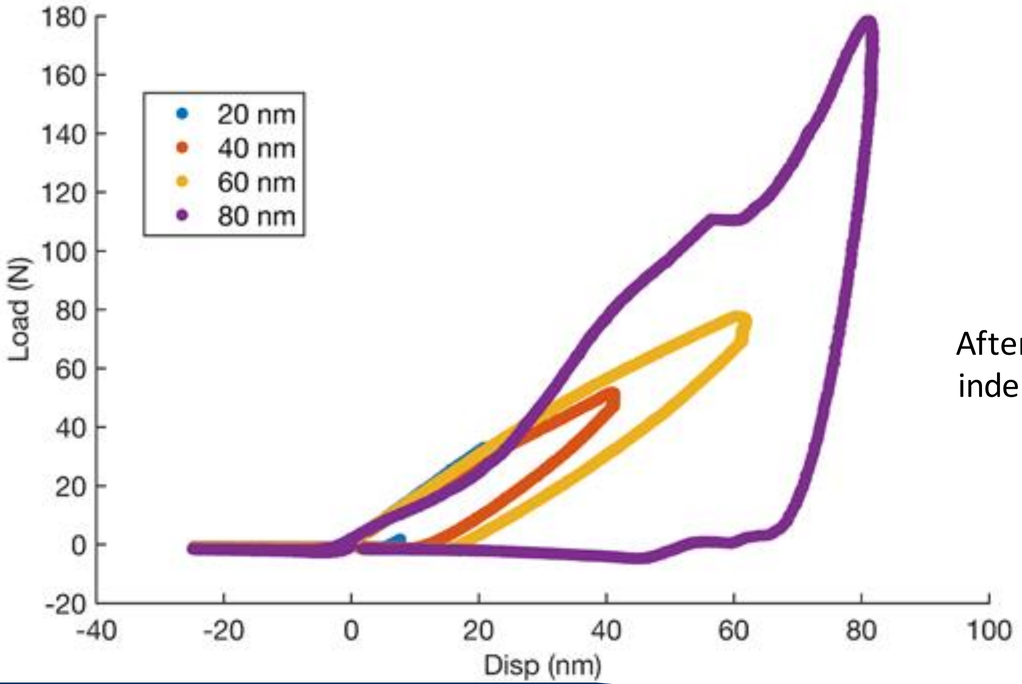
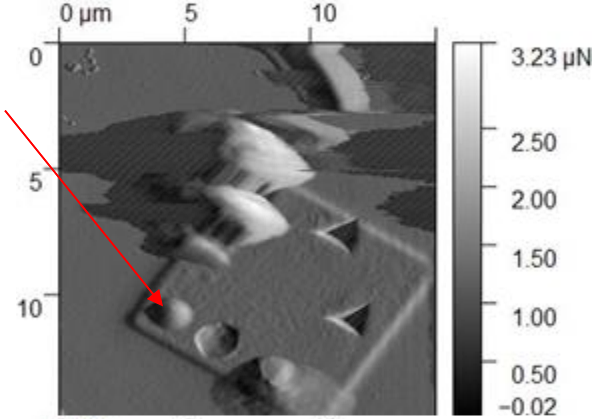
Ti(0001): 20 nm – 80 nm

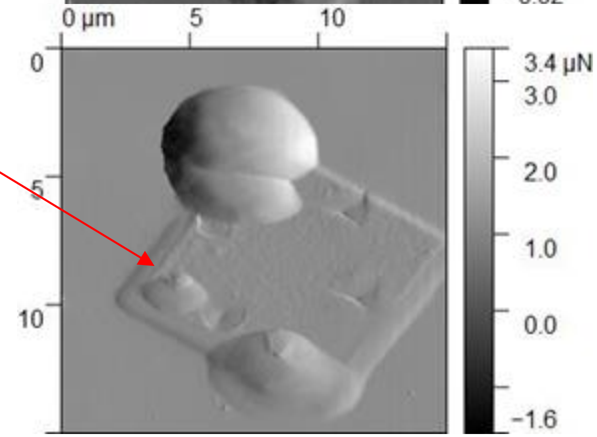


7×10^{17} ions/cm^2 implant

Before blister indentation



After blister indentation



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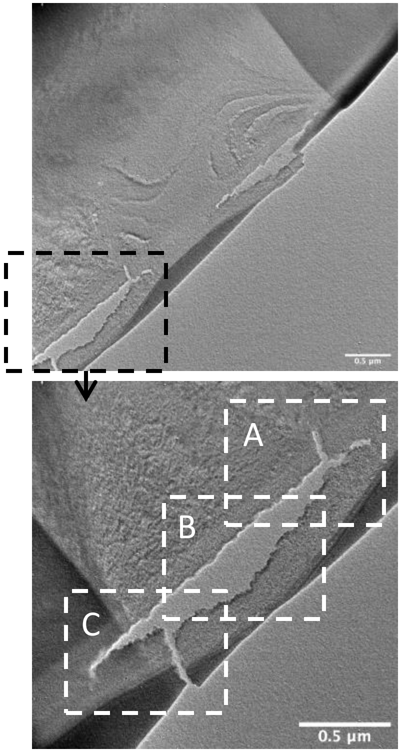
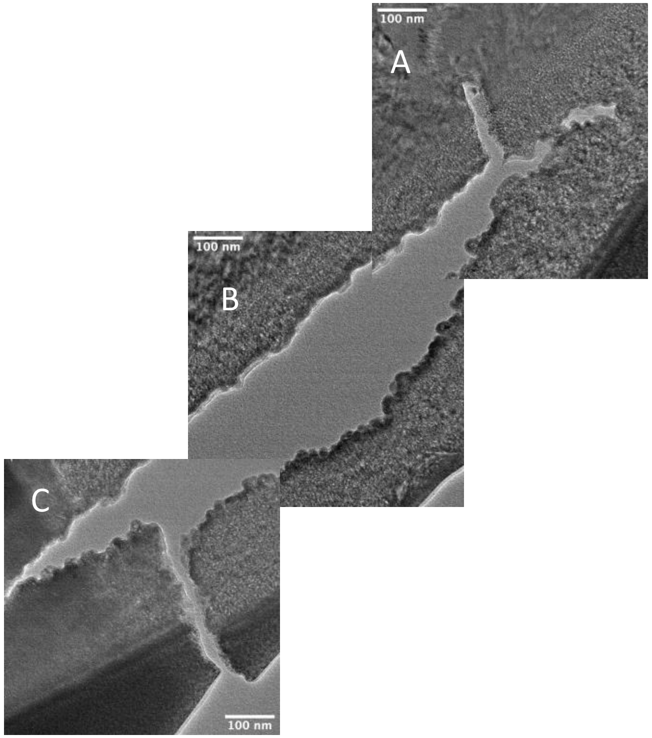
Ti(101bar0) in situ indentation





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Relaxation strain and average residual stress

- Relaxation strain was calculated with the help of a customized MATLAB based DIC code:
- http://it.mathworks.com/matlabcentral/fileexchange/50994-digital-image-correlation-and-tracking
- A uniform distribution of residual stresses was assumed over the region of interest and calculations were made according to method described in the following papers:
- https://doi.org/10.1016/j.jmps.2019.01.007
- https://doi.org/10.1016/j.matdes.2018.02.044
- Other calculation parameters are as follows;
  - Modulus of Elasticity (E) → 127.60 (literature)
  - Poisson's ration (ν) → 0.37 (literature)

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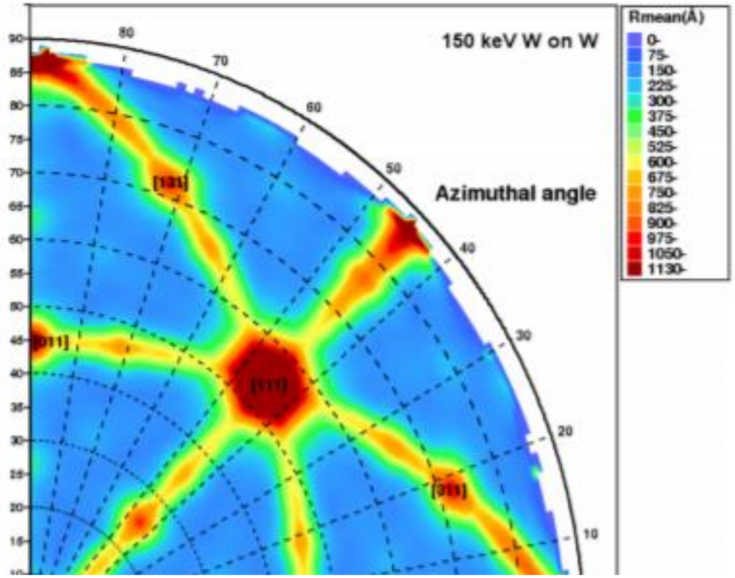
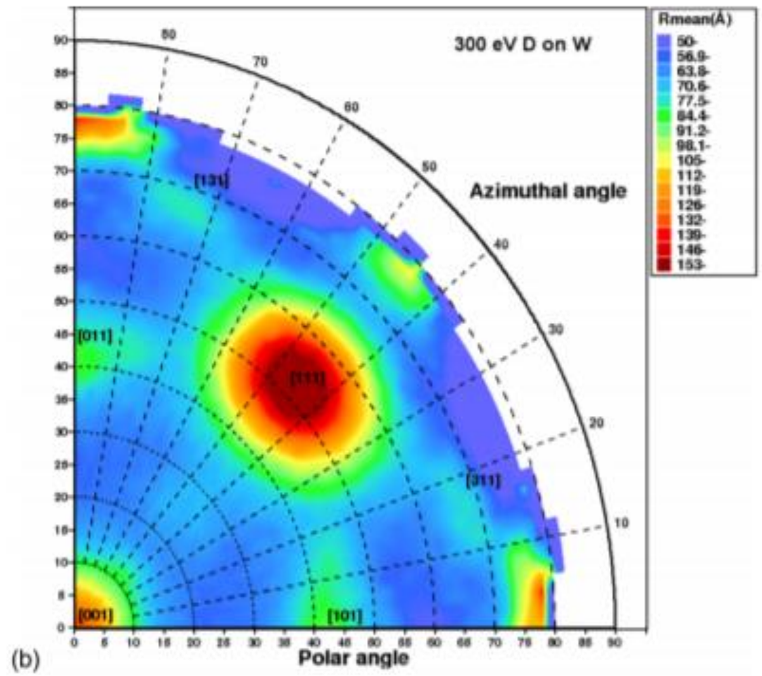
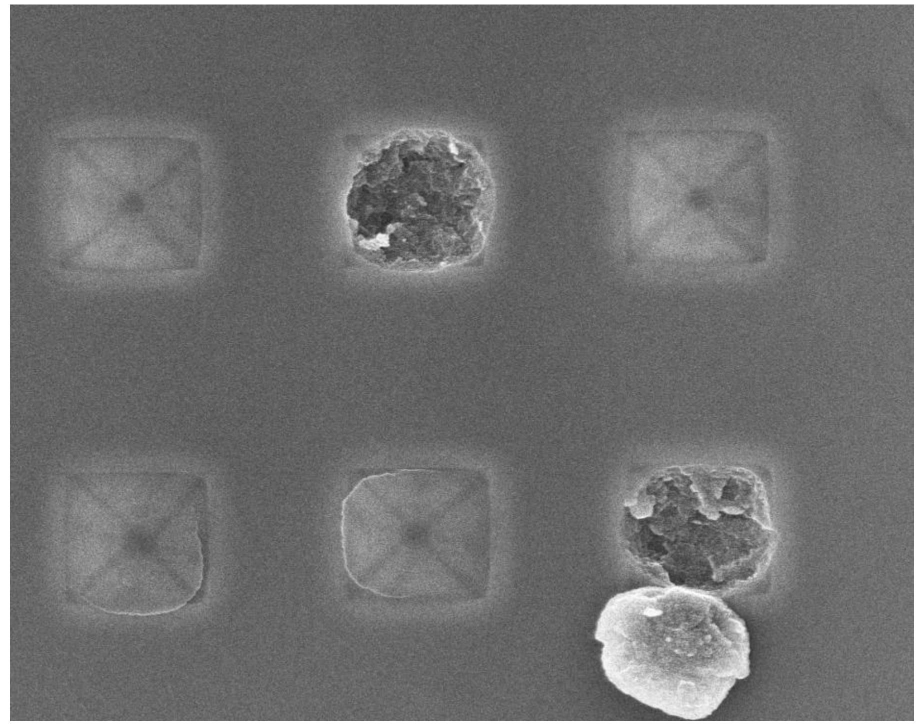
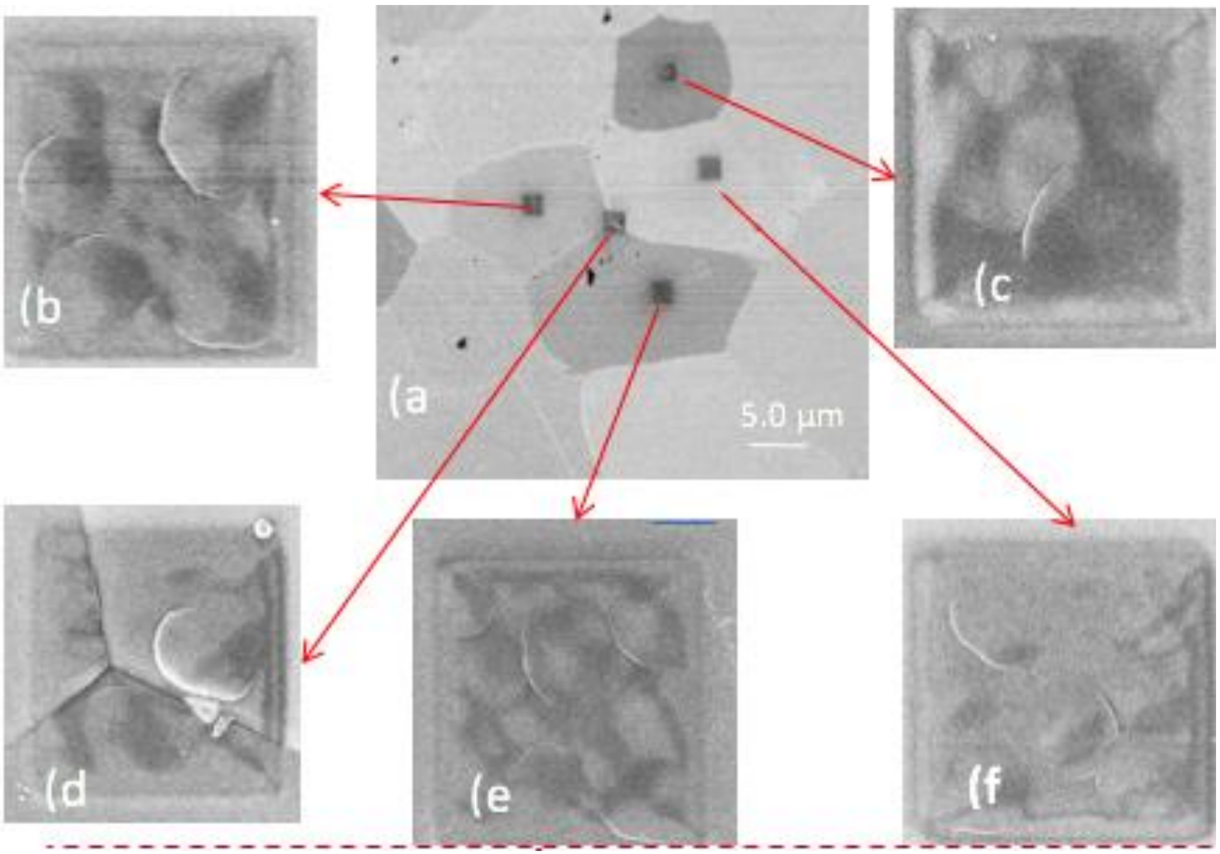

150 keV W on W
Azimuthal angle
Rmean(Å)
0-
75-
150-
225-
300-
375-
450-
525-
600-
675-
750-
825-
900-
975-
1050-
1130-
[131]
[011]
[111]
[311]

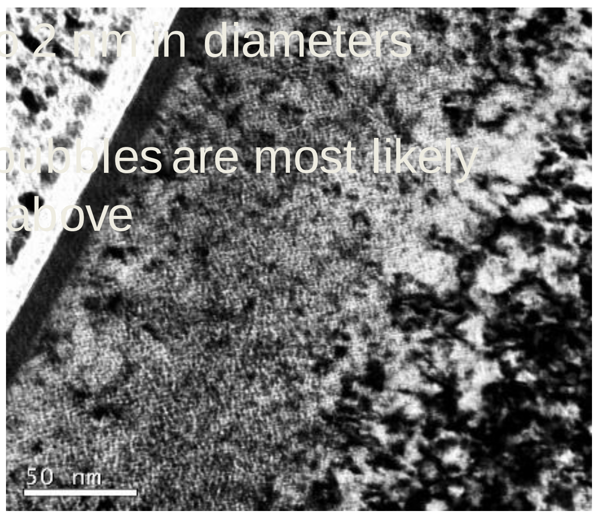

300 eV D on W
Azimuthal angle
Rmean(Å)
50-
56.9-
63.8-
70.6-
77.5-
84.4-
91.2-
98.1-
105-
112-
119-
126-
132-
139-
146-
153-
[131]
[011]
[111]
[311]
[001]
[101]
Polar angle
(b)

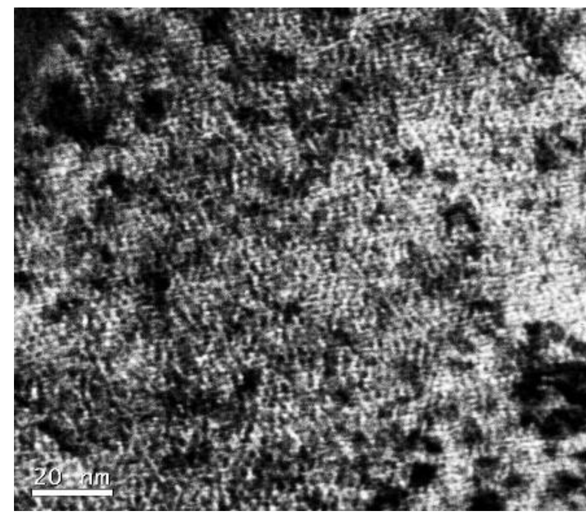



(a
5.0 µm
(b
(c
(d
(e
(f

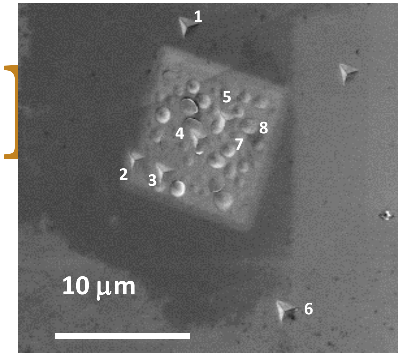
Bubbles formation

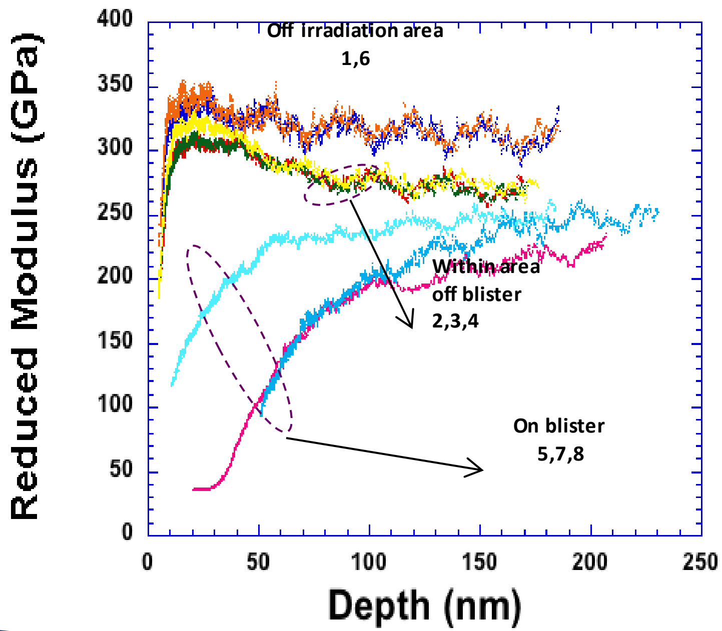
- Bubbles with 1 to 2 nm in diameters are formed.
- Pressure in the bubbles are most likely in equilibrium or above

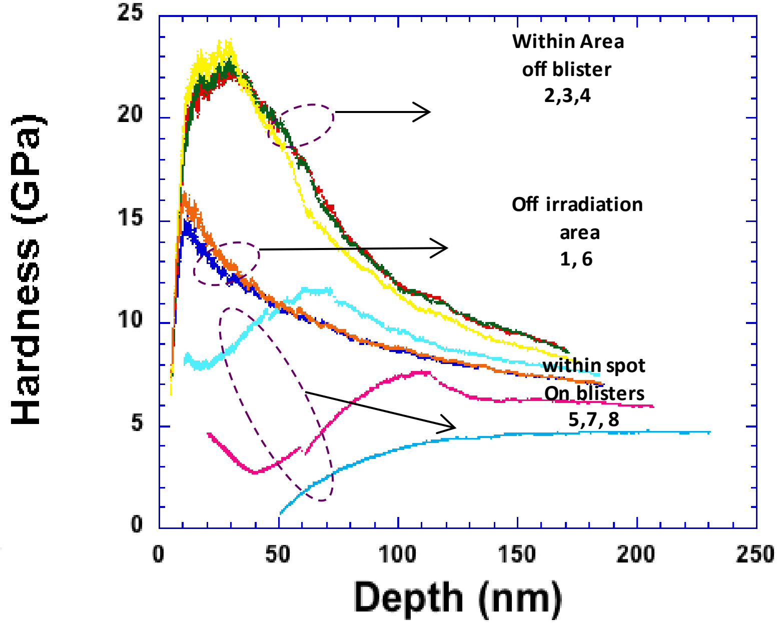




Mechanical ties



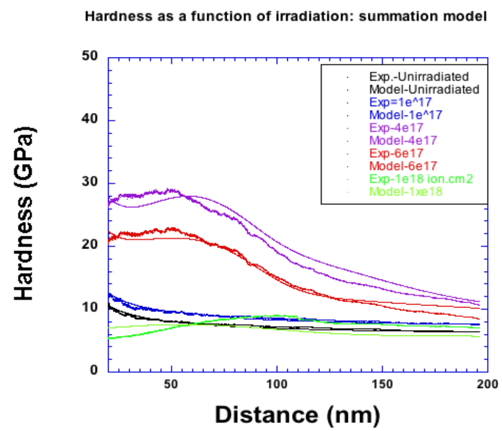


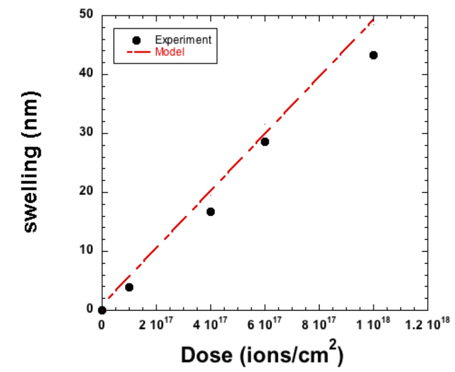


Analysis of mechanical properties

- At low doses H increases with dose first but reduced as dose approach high values studies.
- The results of H versus depth and swelling versus dose explained well by a phenomenological equation

$$N_{He} = \frac{N_0}{2S}\sum_{d=1}^{10}\left[erf\left(\frac{x + \frac{10^3 \times S}{N_0} - (36 + 23.3d)}{\sqrt{2} \times \Delta R_p} \right) - erf\left(\frac{x - (36 + 23.3d)}{\sqrt{2} \times \Delta R_p} \right) \right] \quad (6)$$

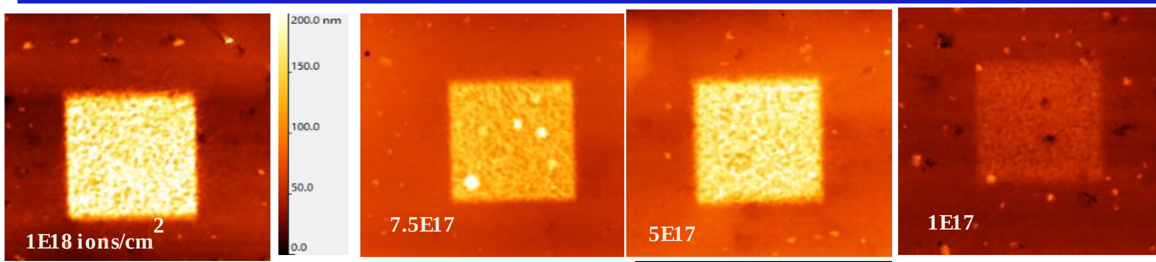




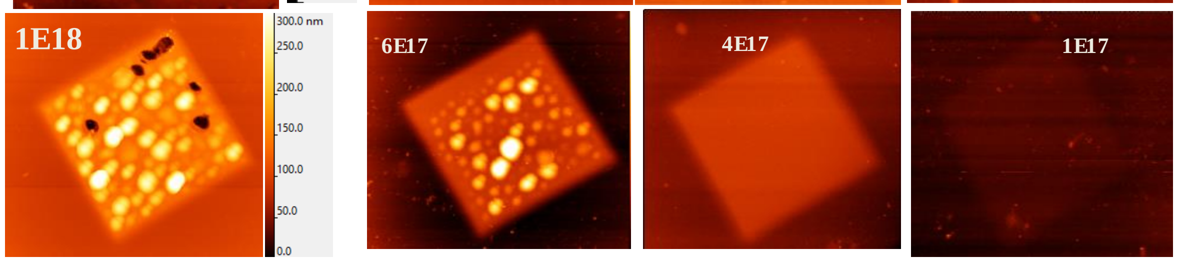
Conclusion

- Swelling and blistering of tungsten irradiated with 25 keV helium ion has been investigated in a site-specific manner
- Bubbles near the surface with 1-2 nm in diameters are identified
- Channeling phenomena was observed on low-index planes
- E and H versus depth, on and off the implanted area, have been measured by nano-indentation technique
- A simple phenomenological equation has been developed to explain the measured profiles of nano-indentation and swelling assuming the implanted ions are deposited with Gaussian depth distribution and that the helium atoms quickly diffuse forming bubbles keeping the initial depth distribution intact

Similar study on other materials

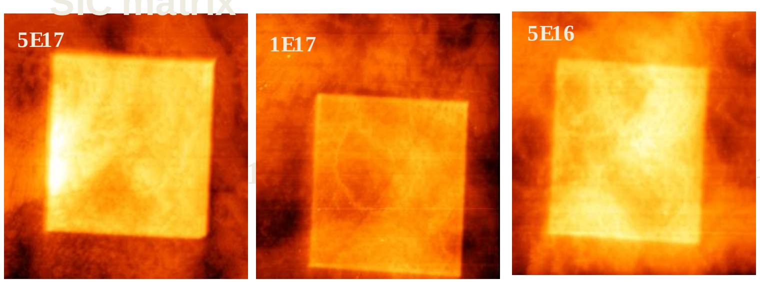


Titanium

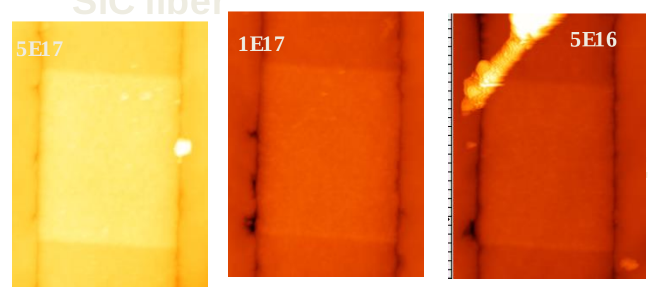


Tungsten

SiC matrix

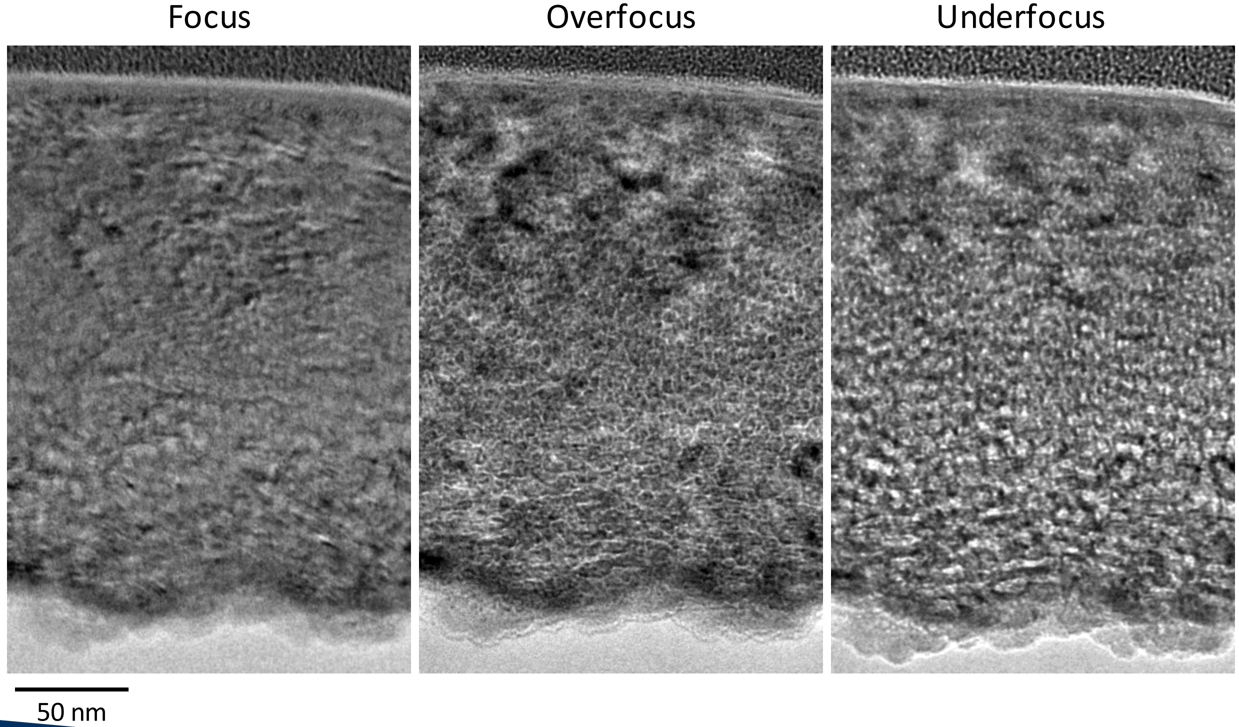


SiC fiber



Bubble ditribution (TEM): Imaging

7×10^{17} ions/cm^2 implant



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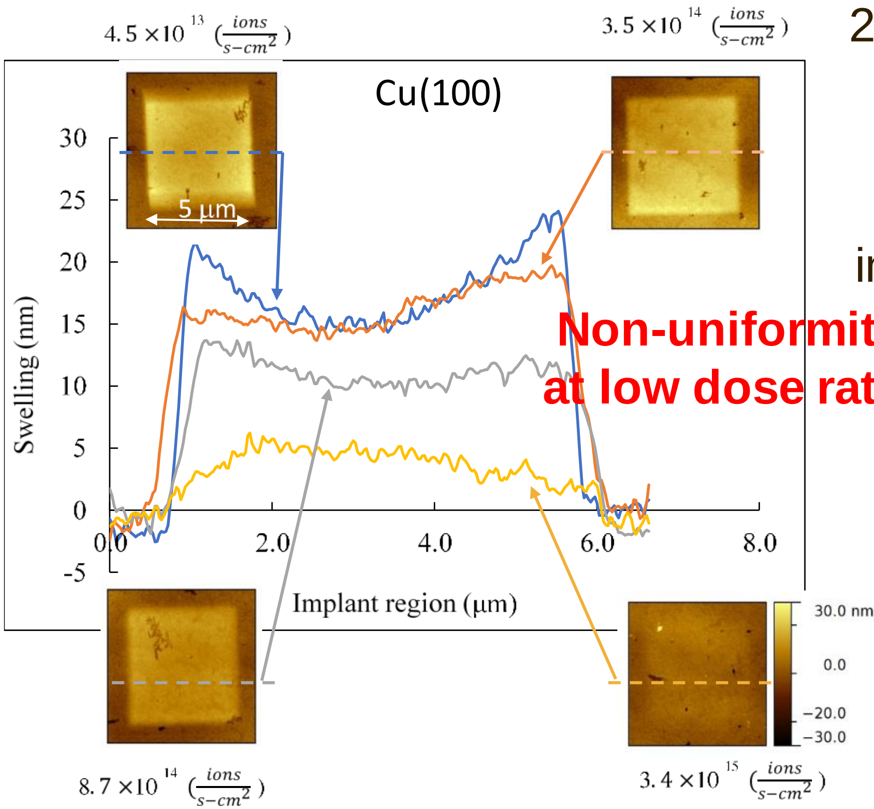
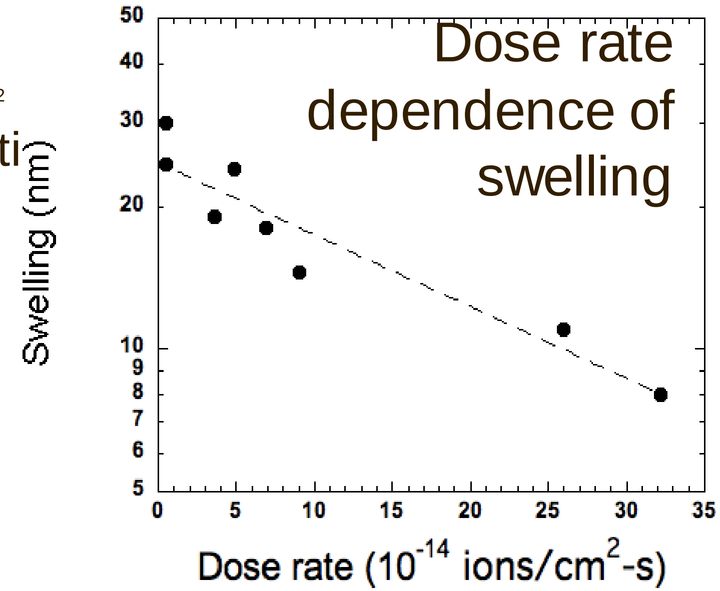
Effect of dose rate

2×10^{17} ions/cm^2



25 keV He ions 2×10^{17} ions/cm^2 implantation

Non-uniformity at low dose rates



TEM underway

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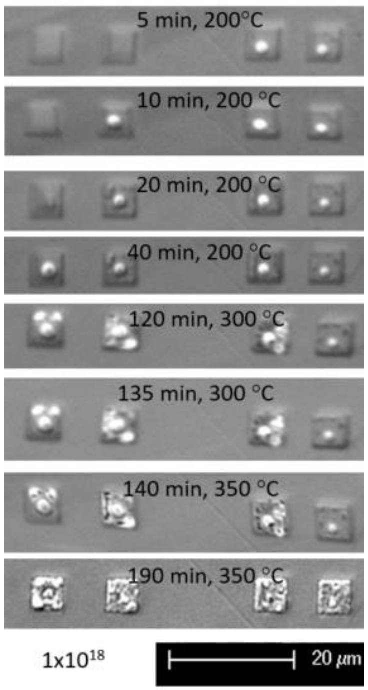
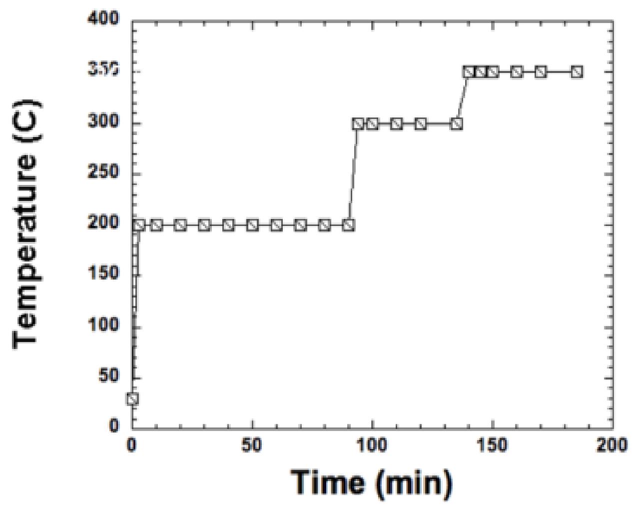
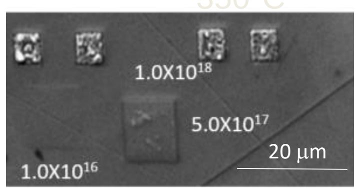
Results: Effects of post-irradiation annealing

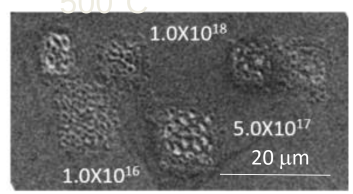




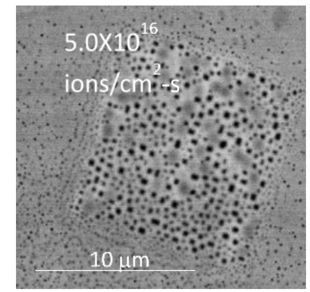
350°C

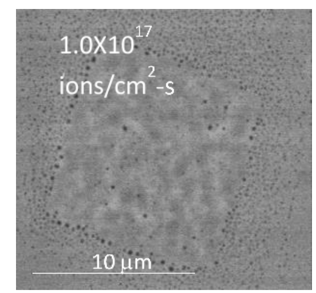


500°C



Blisters and cavities:
5 min. annealing at 500°C





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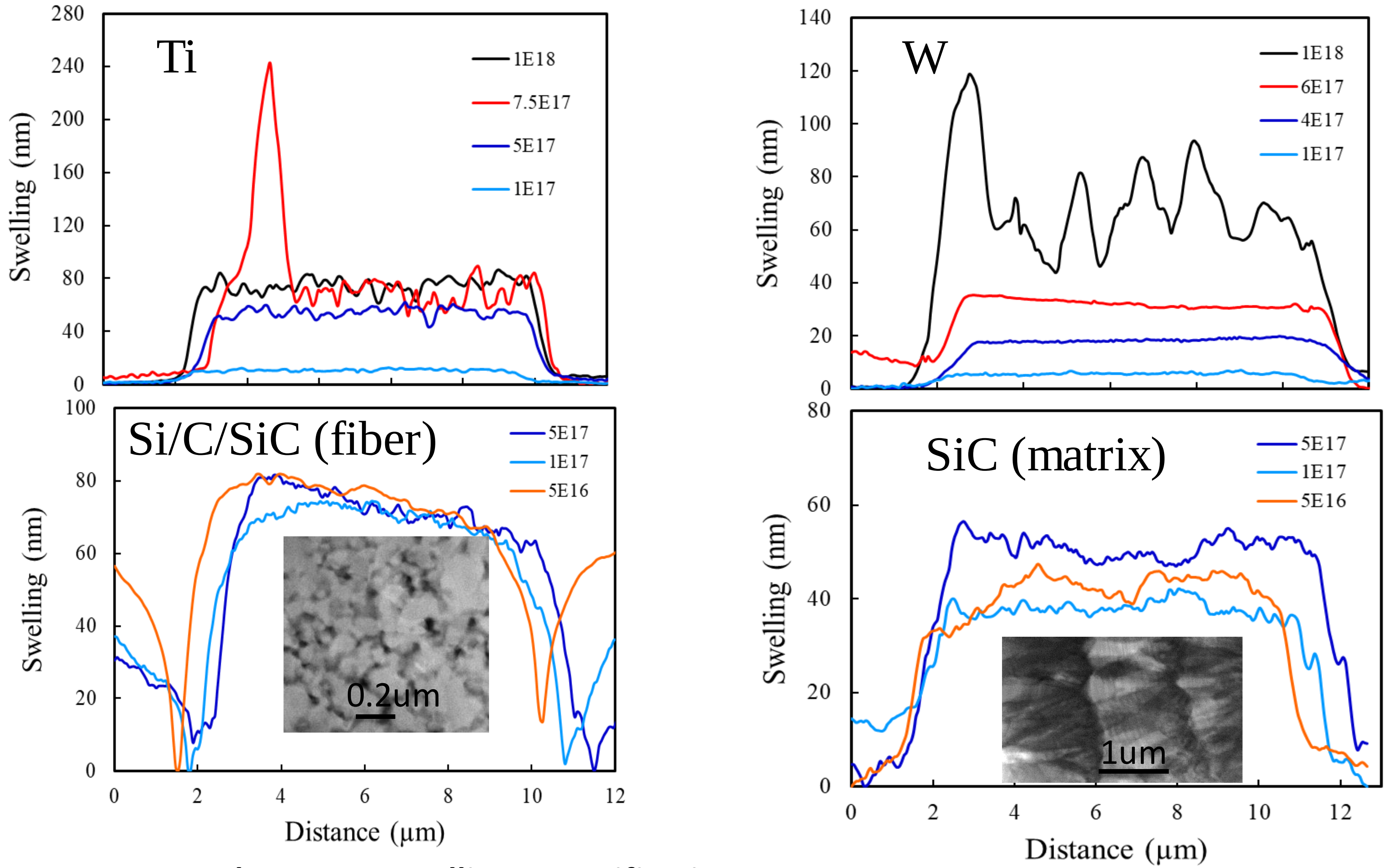
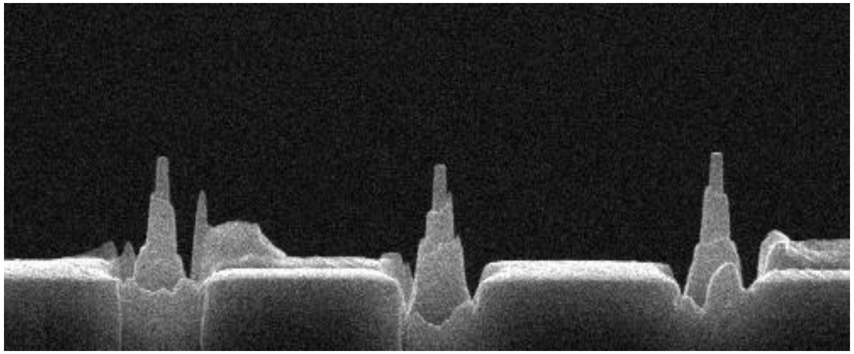
Similar study on other materials



POSTER: M. Ambat; F-10: Swelling Quantification of High Dose Helium Implantation in Different Materials Using a Helium Ion Beam Microscope

→ Expand this rapid screening approach to other materials systems

EFFECT OF HELIUM PILLAR IMPLANTATION



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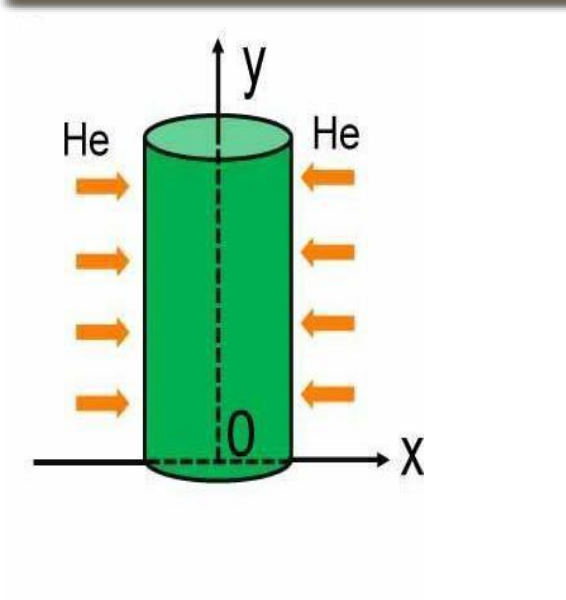
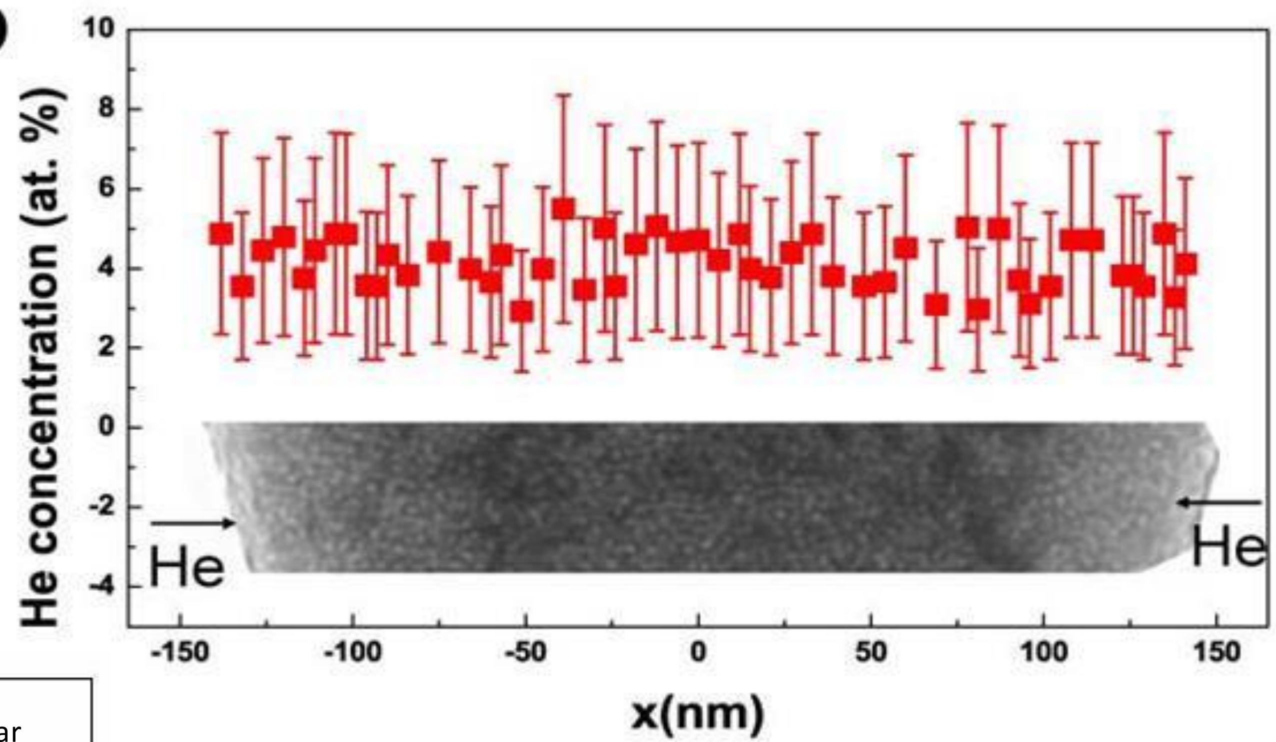
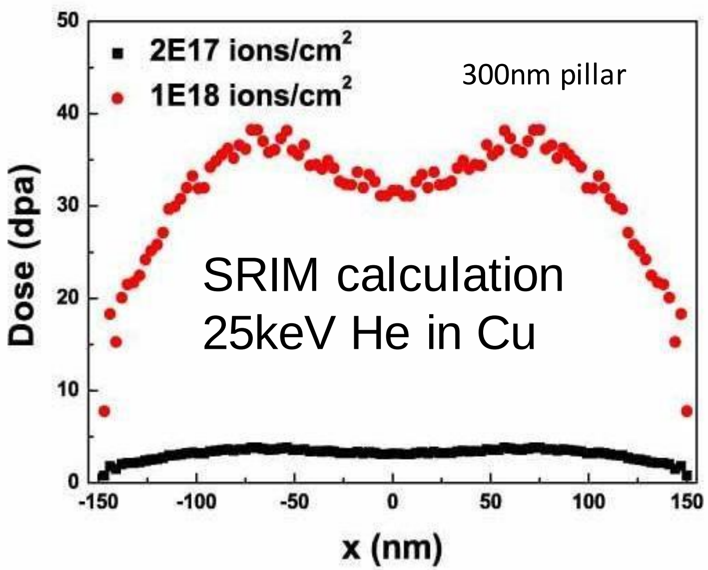
Micro area implantation in pillars



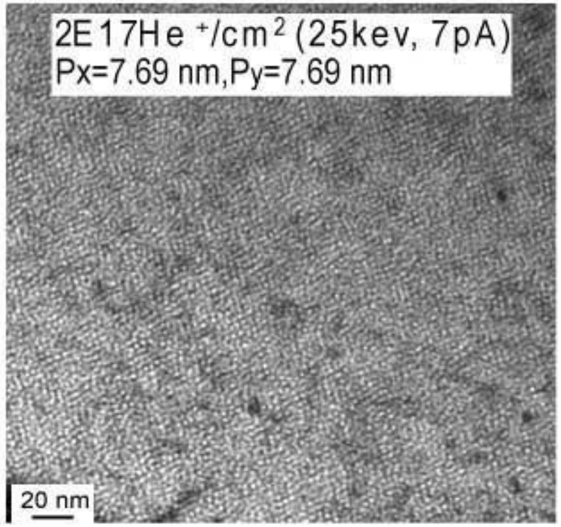


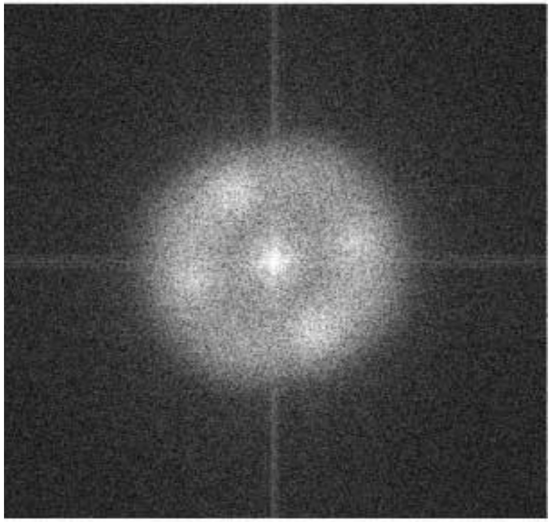


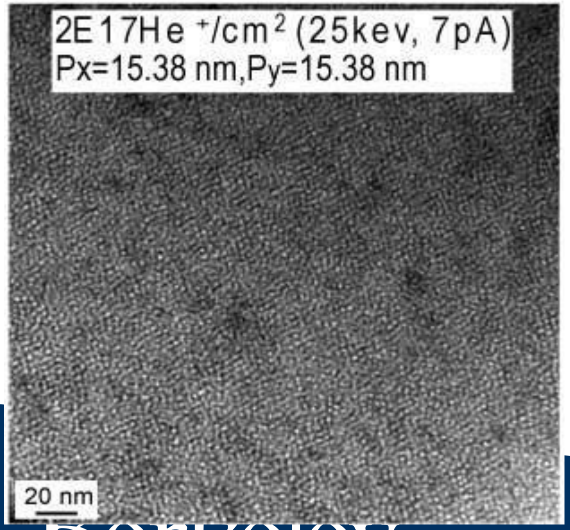
Helium implantation was conducted from two sides to achieve a deeper Helium beam penetration and a h***omogeneous He Bubble distribution!***

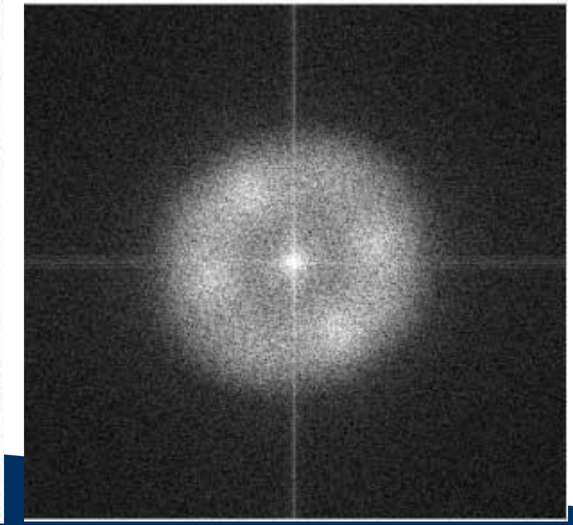
No large cavities were observed

No influence of scanning parameters on bubble structure was found



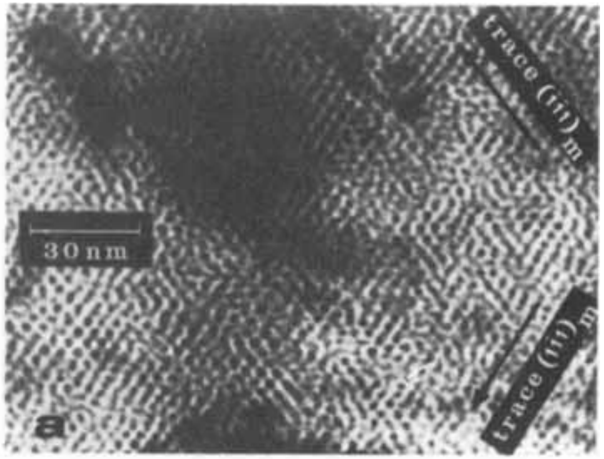






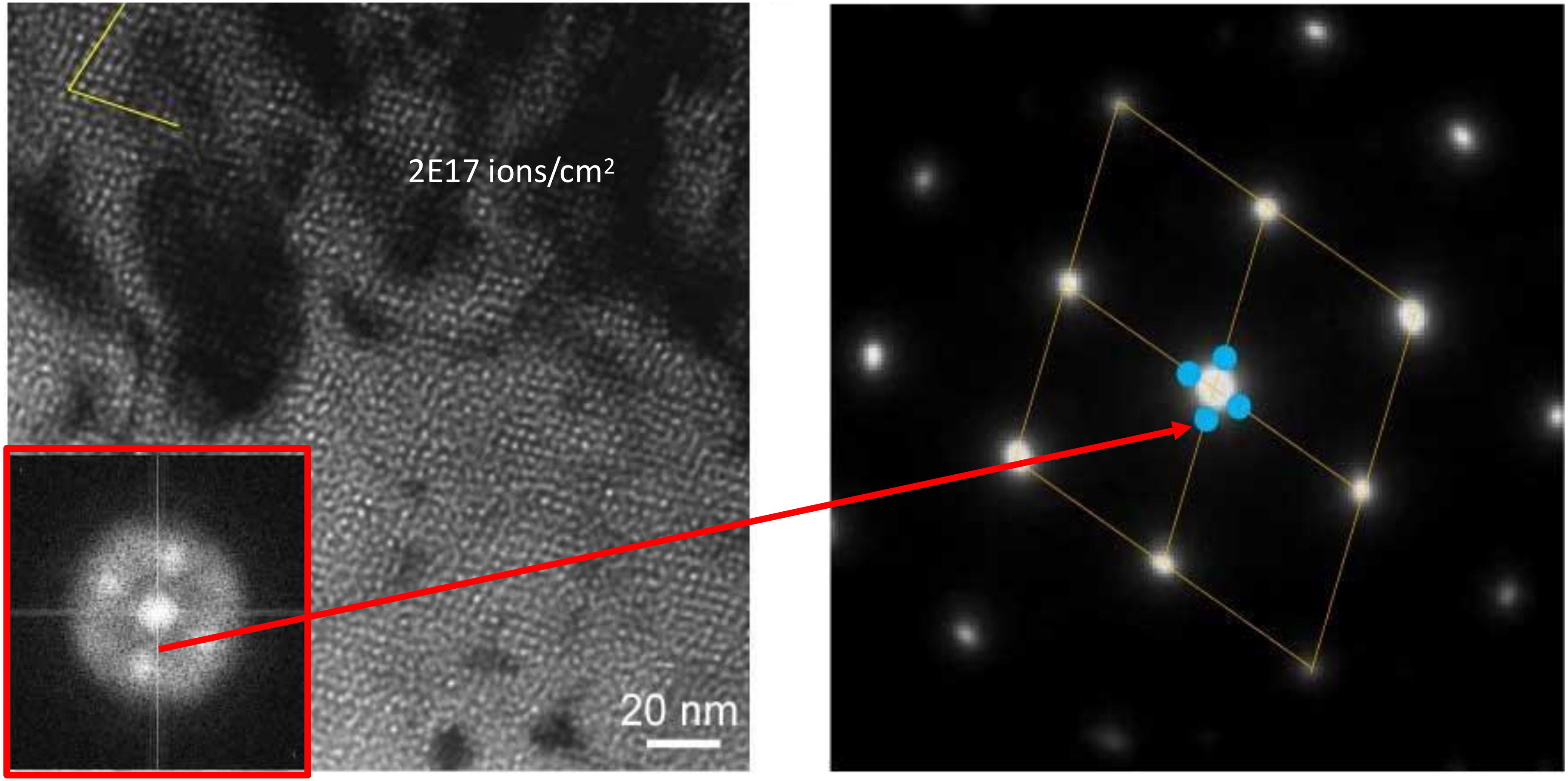
No large effect from the scanning parameters can be found on parameters we examined.

Good agreement with the literature:



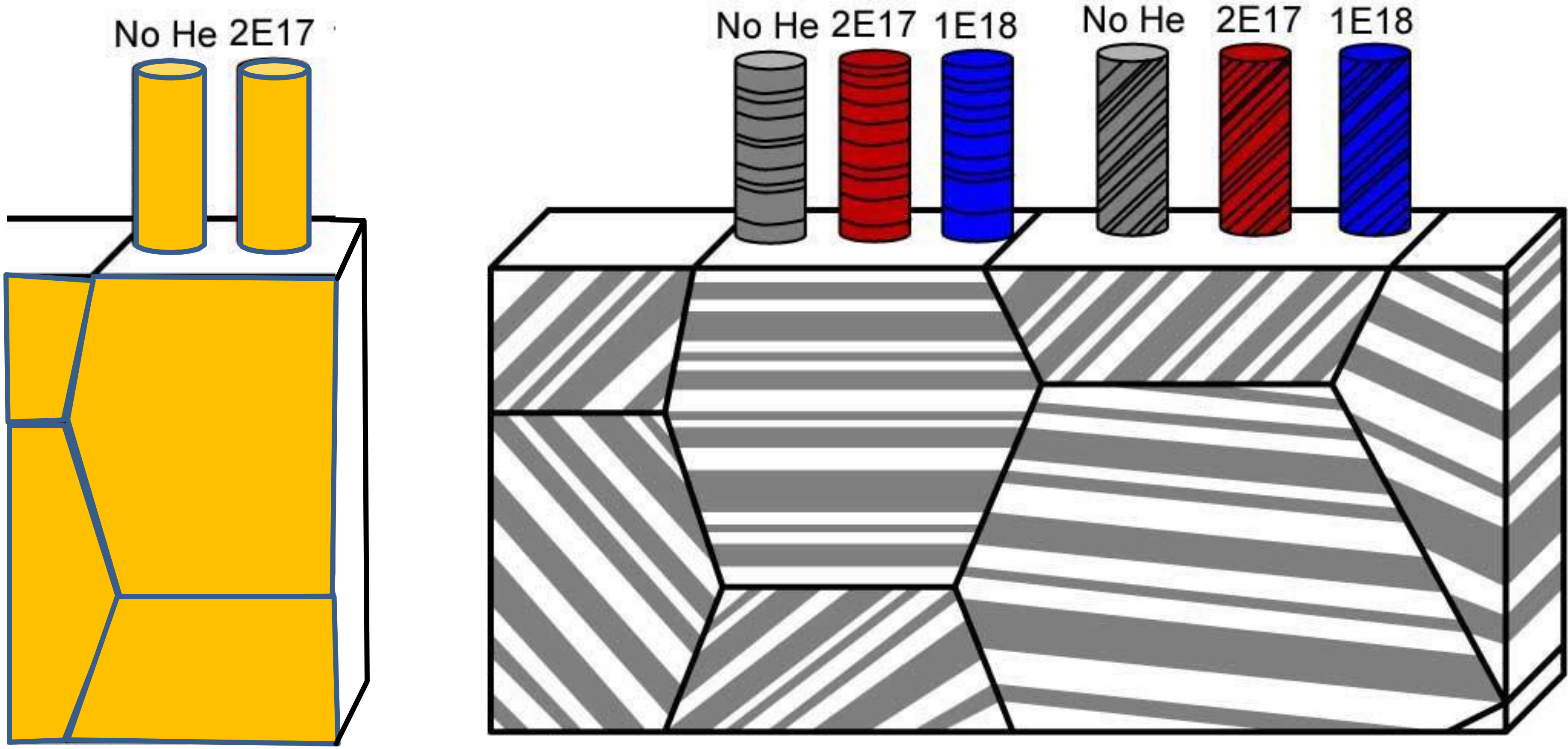
P.B. Johnson, et al Radiation Effects, 1980, 53, pp. 195-202

A simple way to display the orientation relationship



Z. Wang et al; Acta Mat 2016

Implanting nanopillars to different doses.



Demonstrated in single crystal Cu and nanotwinned structures

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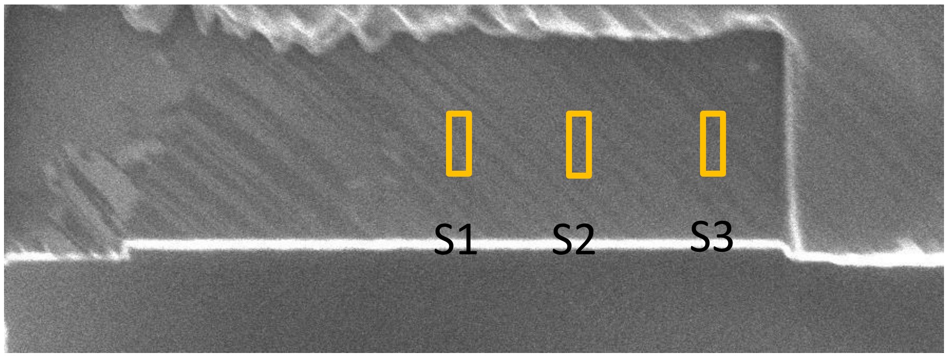
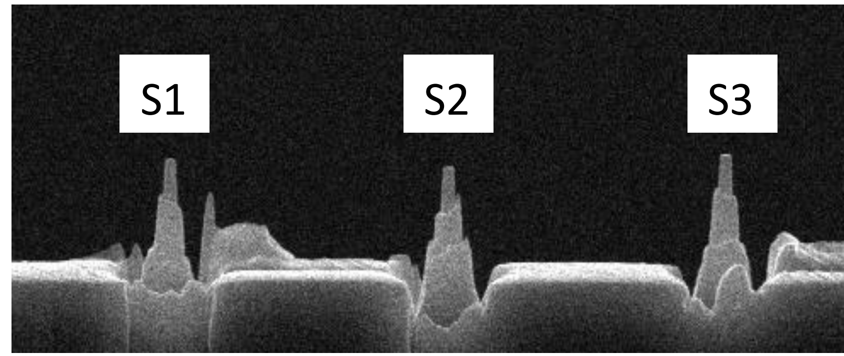
Implanting nanopillars to different doses.

Ga source: 30Kev, from 1nA to 1.6pA

Cut Cu nano pillars from one grain

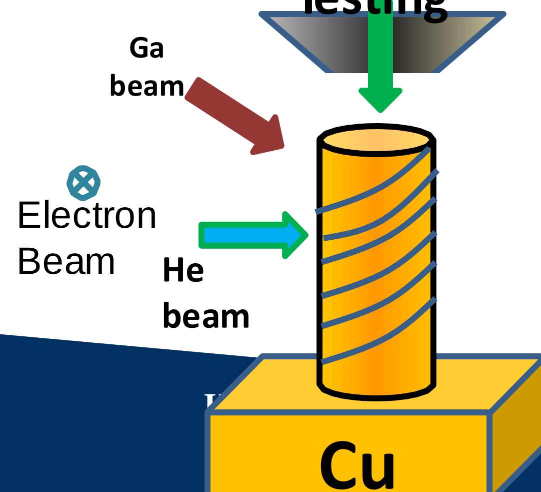




He source: 25Kev, 40pA Implanted to different doses

Compression Testing

Hysitron PicoIndenter (PI95) 3010JEOLNCEM



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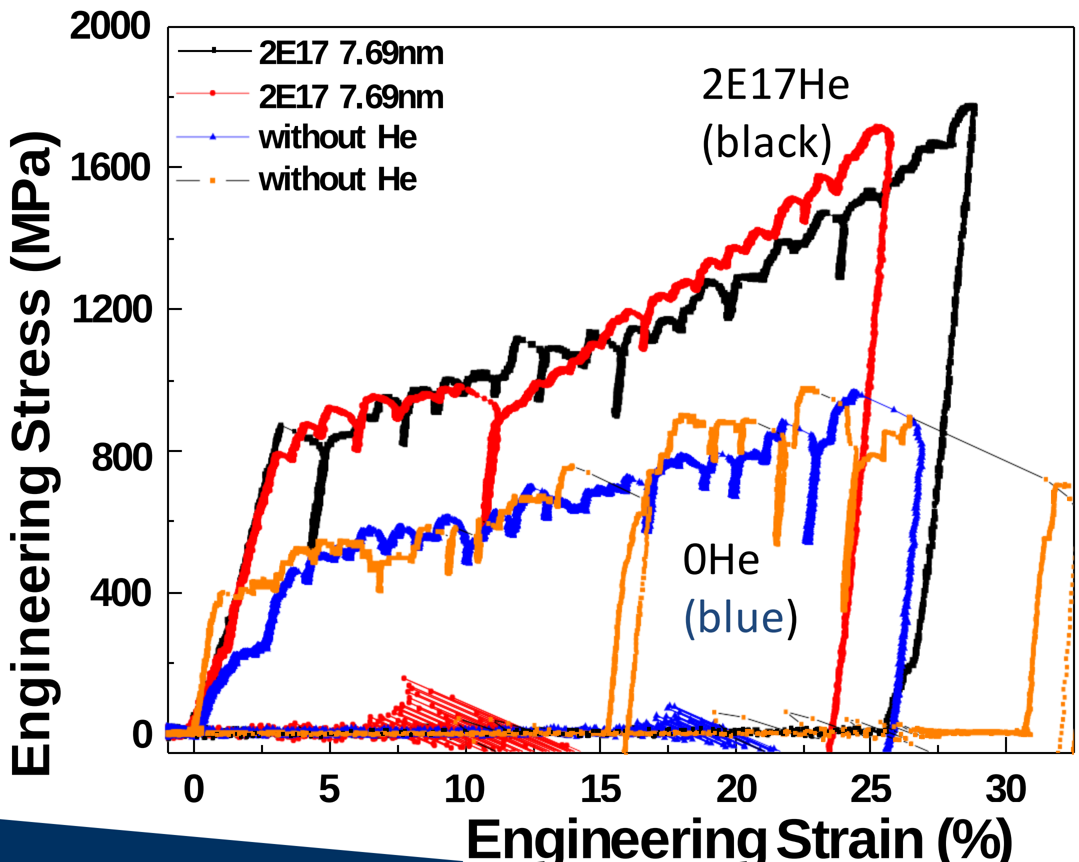
Post implantation mechanical testing

Loading Information: <u>Single crystal Copper</u>: D=300 nm

Loading along [100]; Beam direction:[110]



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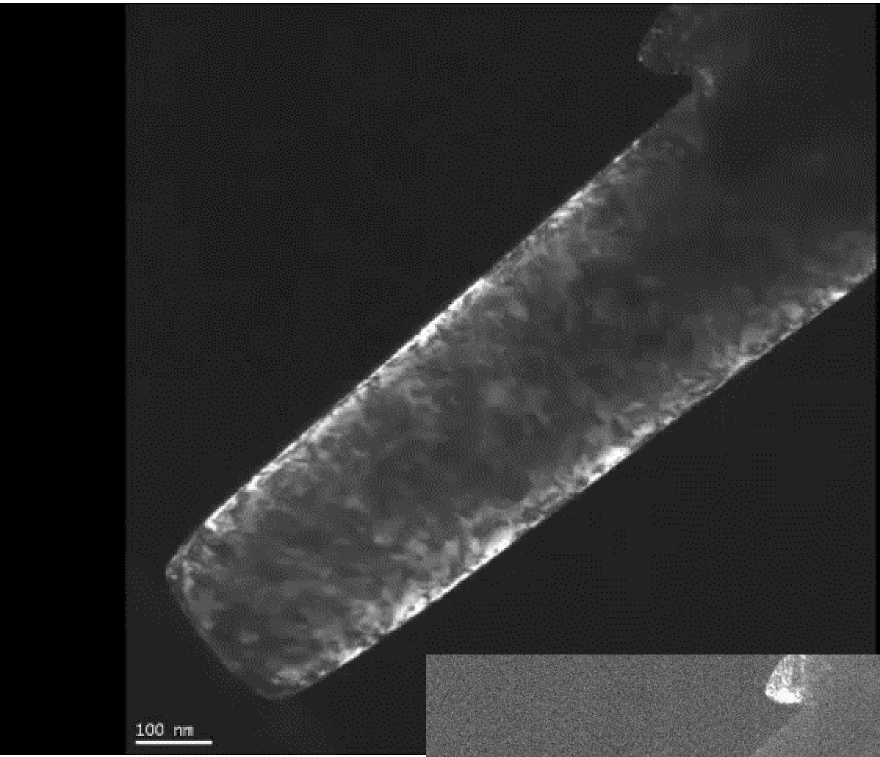
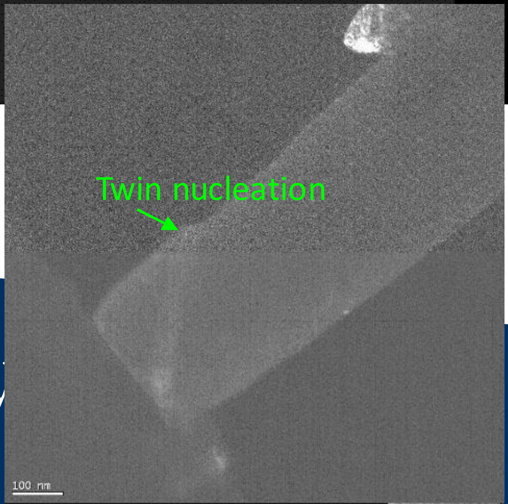
Z. Wang et al; Acta Mat 2016

Insitu compression testing reveals different deformation mechanisms on pillars ~200nm in size

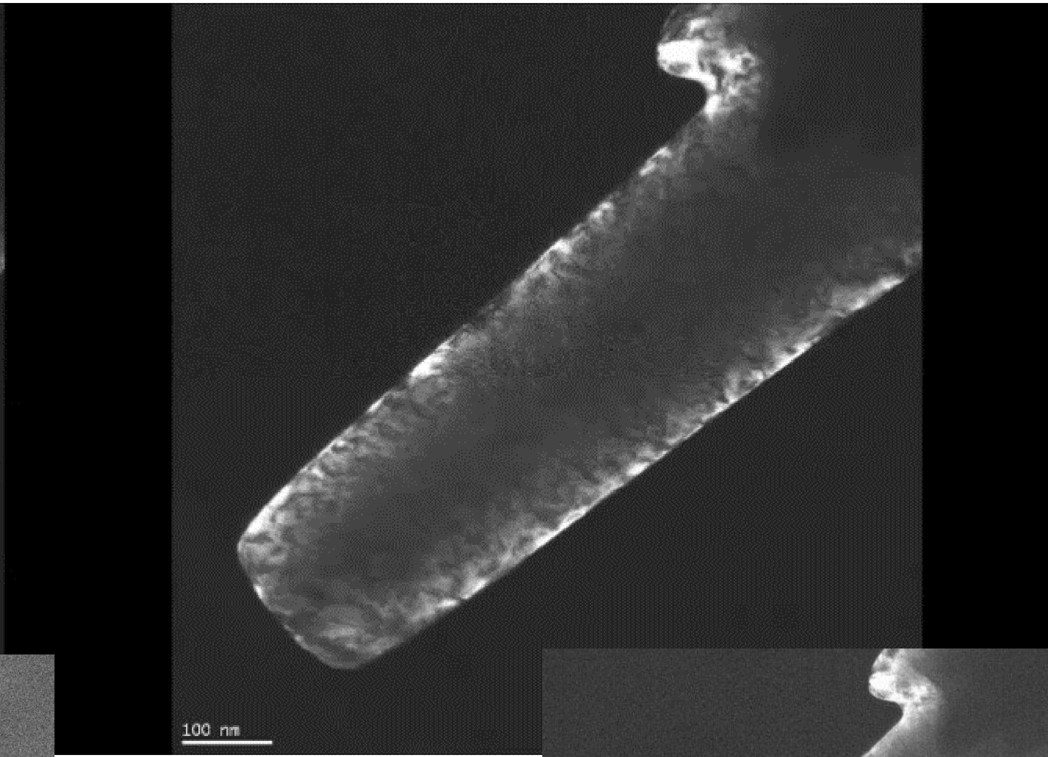
210nm diameter pillars single crystal twinning

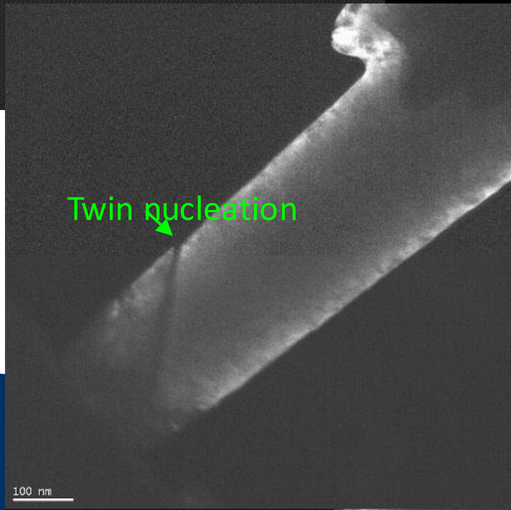
Not implanted





10^{17} He implanted





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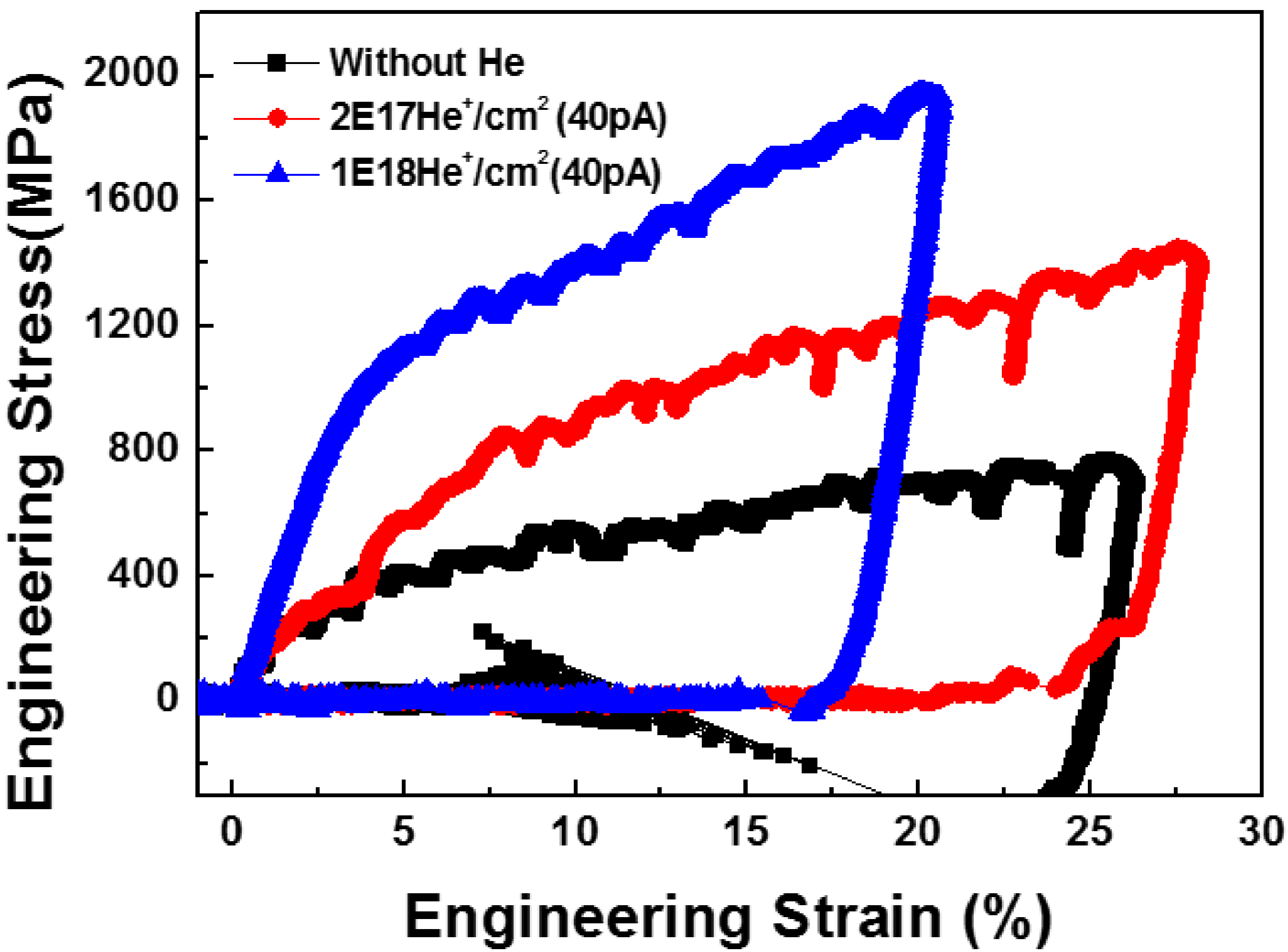
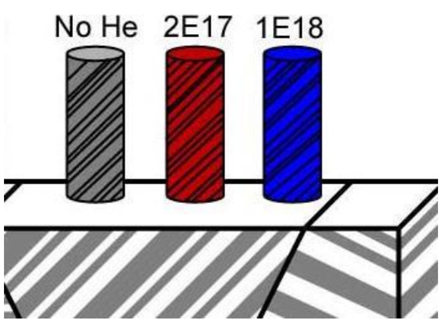
Z. Wang et al;
Acta Mat 2016

Stress strain curves on nanotwinned materials

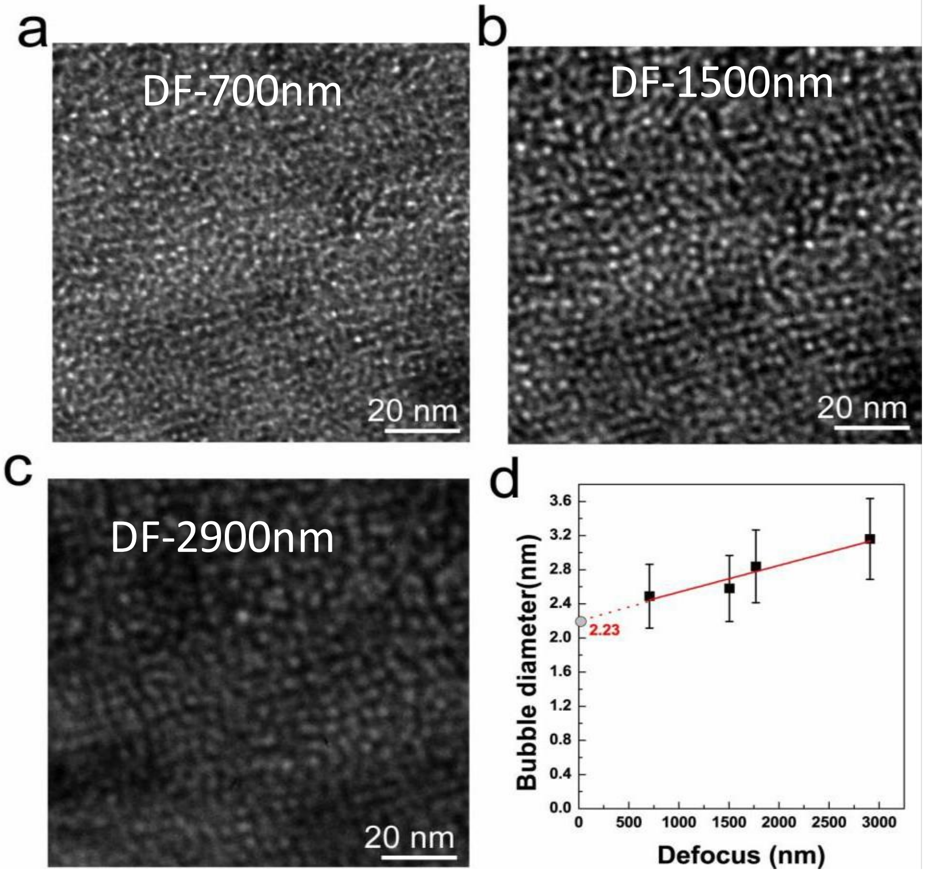




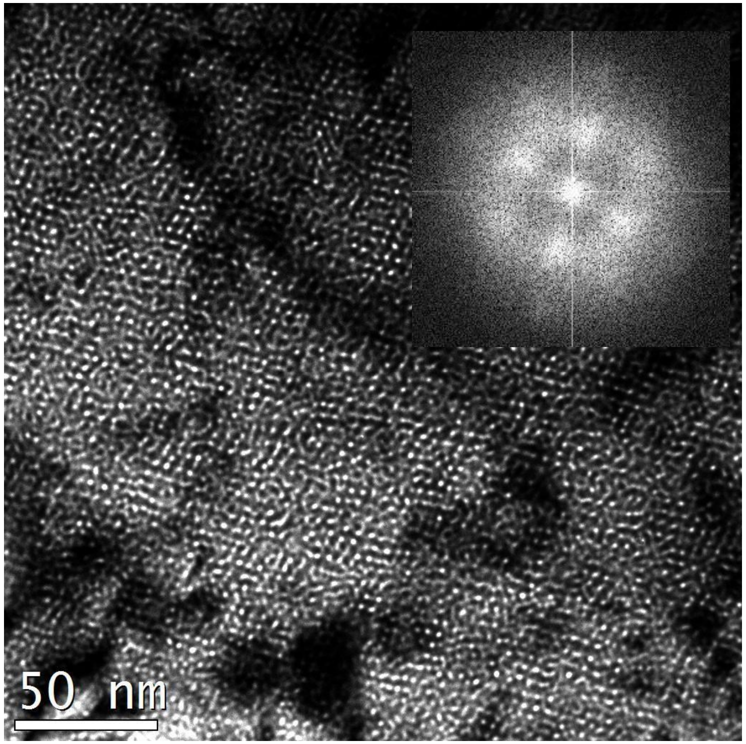
Z. Wang et al; Acta Mat 2016

Quantifying Bubble structure

Bubble diameter



Density



$1/d_{\{111\}He}$->a_{He}->Density

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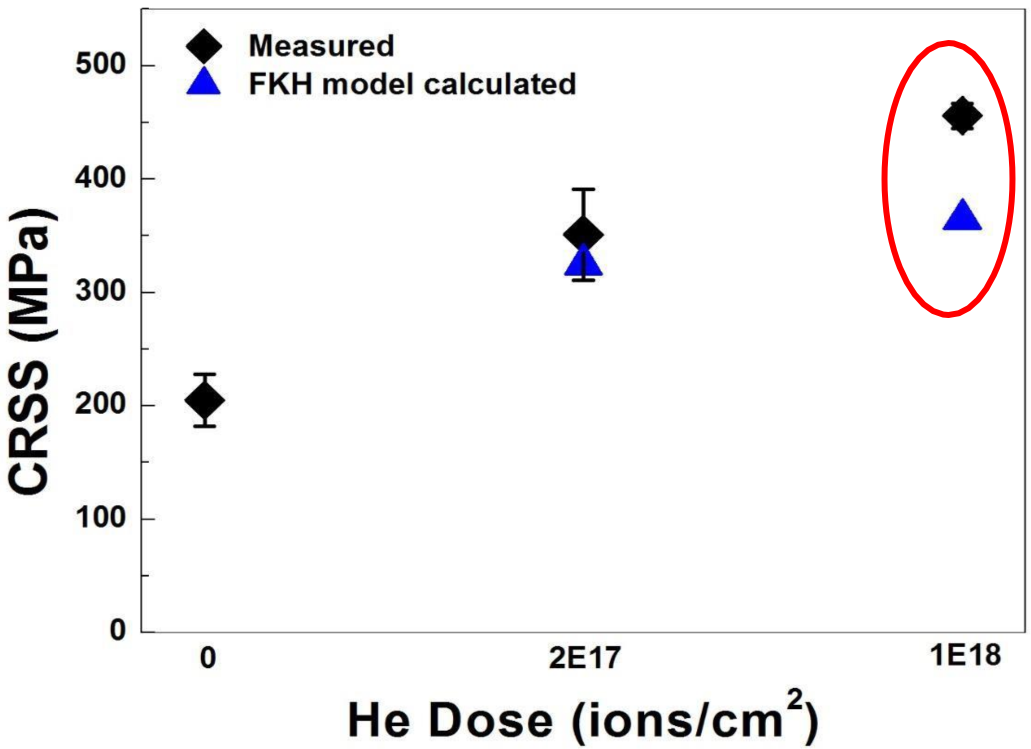
Comparison between model and experiment



Model and experiment not in good agreement

Obstacle-controlled strengthening:

Weak Obstacles: $\Delta\tau = \mu b d N^{2/3}/8$ （$\alpha<0.25$）

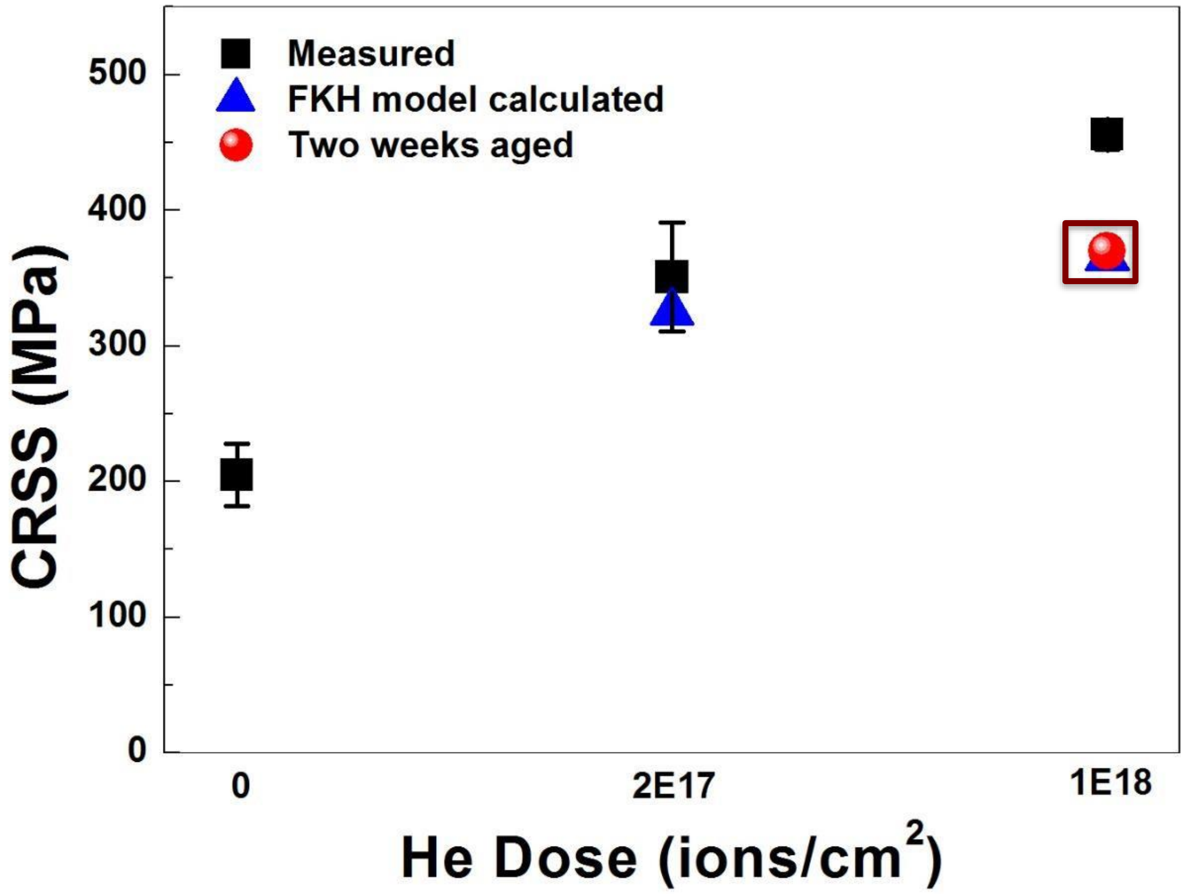
Friedel-Kroupa-Hirsch (FKH)

μ is the shear modulus, b is the Burgers vector of the twinning partial dislocations, and N and d are the defect density and diameter.

Calculated value is smaller than the measured value(1E18 ions/cm^2)

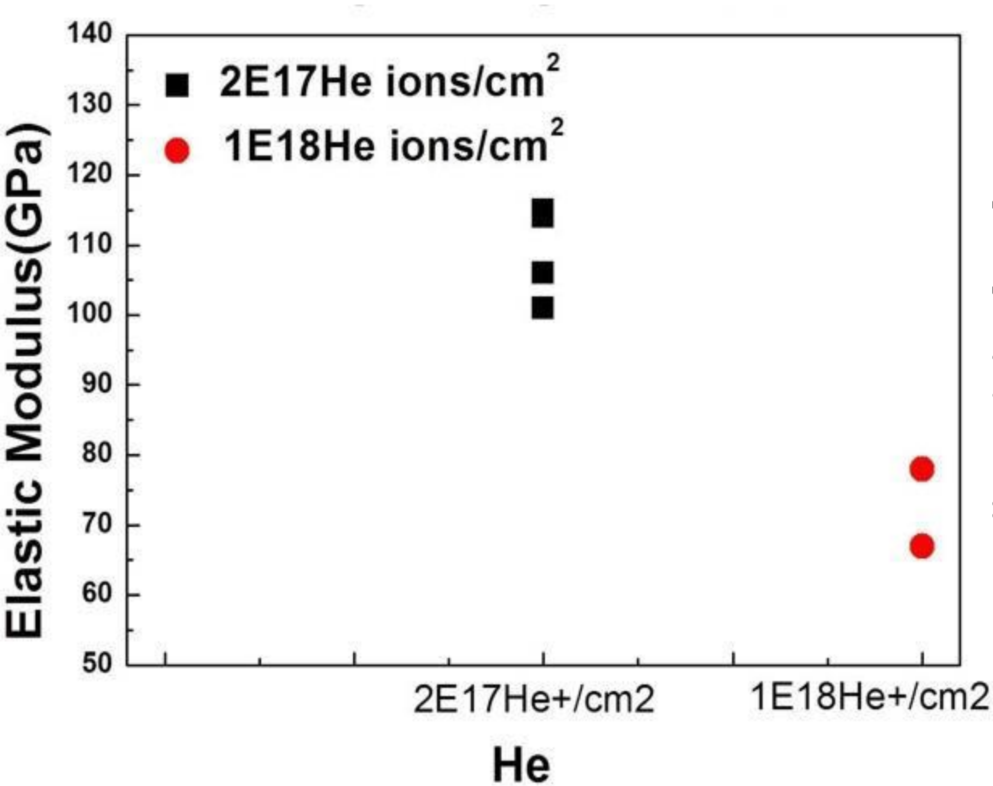
2 weeks aging of the sample draws a different picture

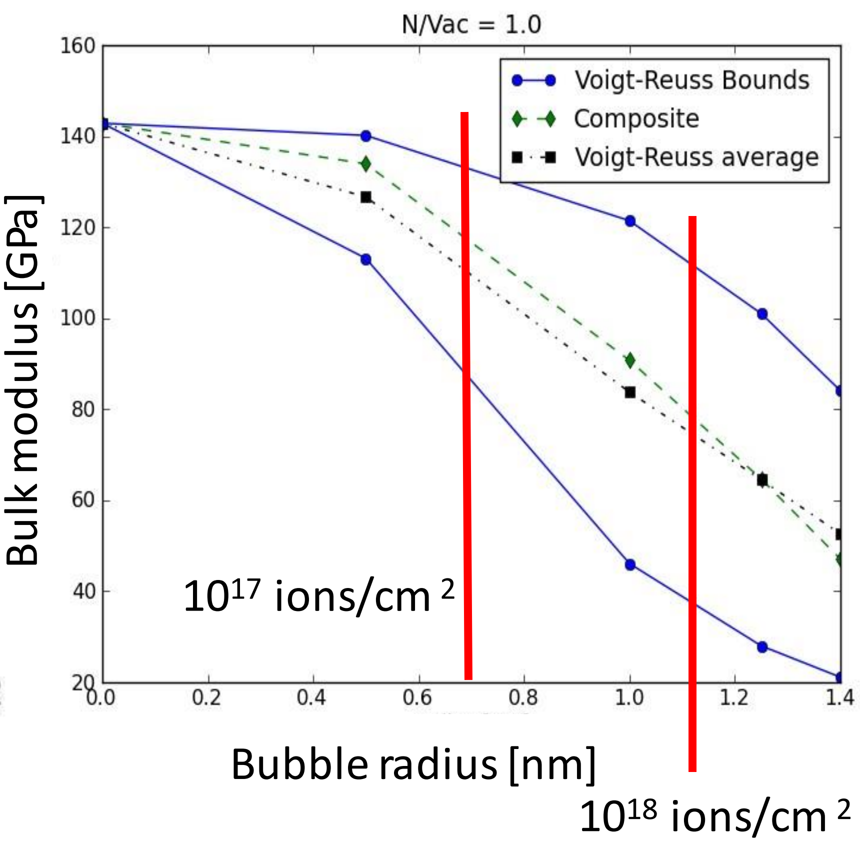
Performing the test on the pillar implanted at the same time <u>2 weeks later</u>
Pillar in the same grain.



Comparing elastic modulus modeling/experiment

I. Winter and D. Chrzan





Modulus measured from the unloading slope

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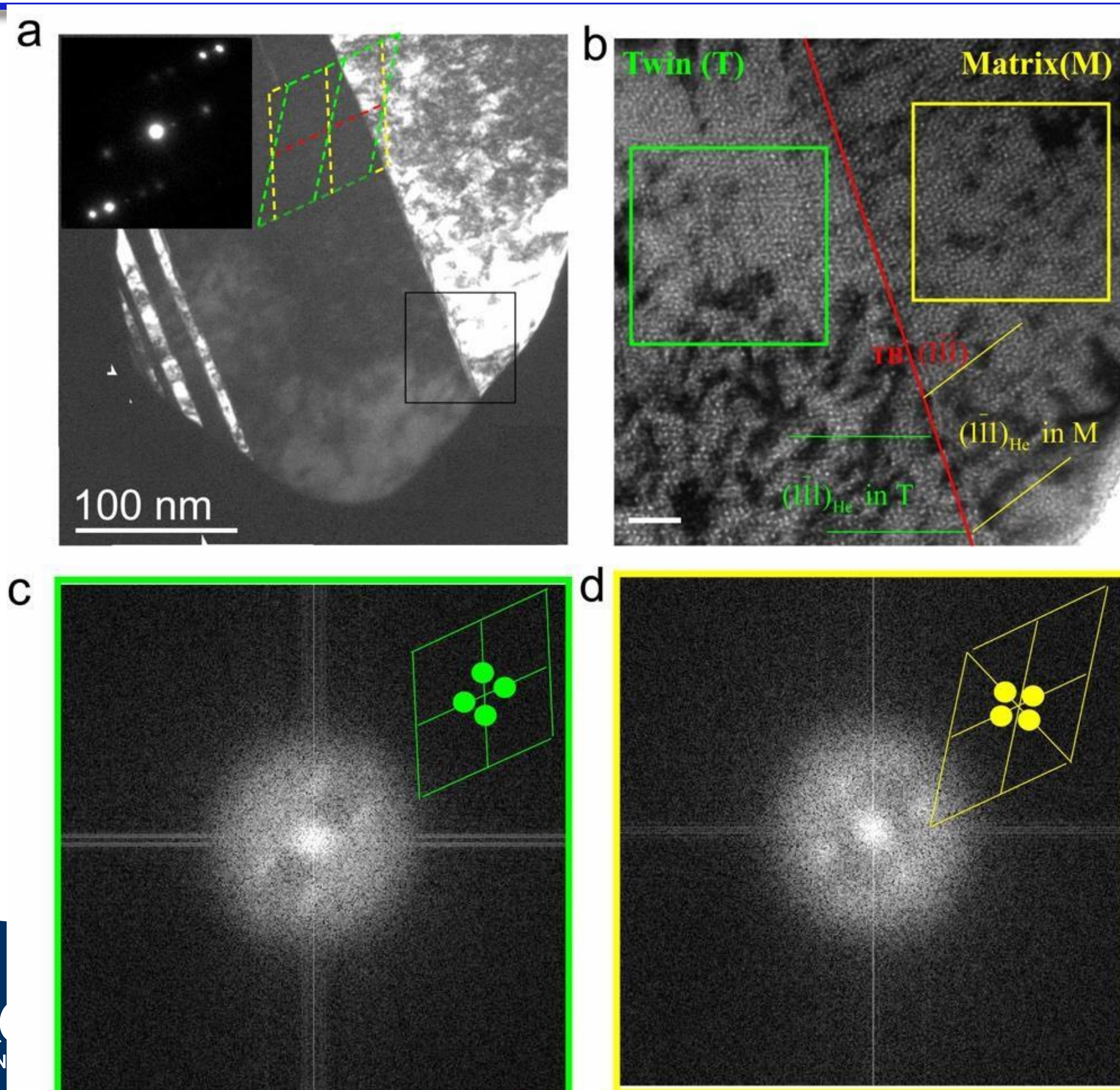
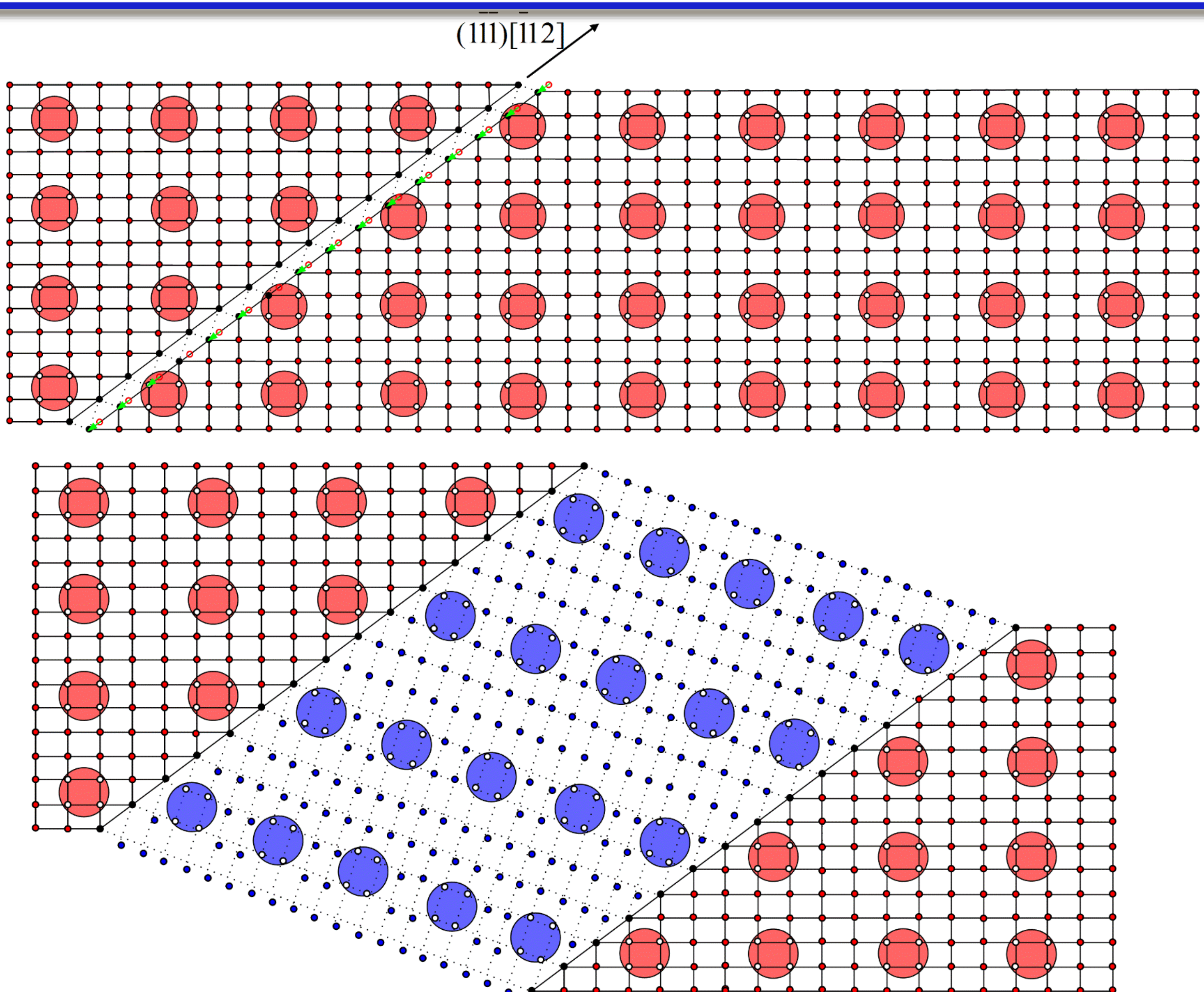
Deformation changes the He bubble superlattice



Schematic of the mechanism



Summary

- Introduce the benefits of nanoscale He implantation

- Demonstrate a rapid implantation approach in materials using the ORION NanoFab Helium ion beam microscope.

- Demonstrated He implantation in Cu nanopillars and TEM foils (NT-Cu and SC-Cu), the novel technique makes it feasible and efficient to evaluate He ion damage and its effect on small volume materials.

- The resistance of TB migration was significantly improved by He implantation.
- He implantation fosters the development of twins in small pillars.

- The He supper lattice after TB migration(NT-Cu) and deformation twinning(SC-Cu) was still kept due to the directional cutting of the He bubbles through twining partial dislocations. While, the He supper lattices preferred to be destroyed by the random ordinary dislocation motion.

Future work: Expand to other materials; incorporate heating; tensile testing, etc.

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Thank you for your attention!

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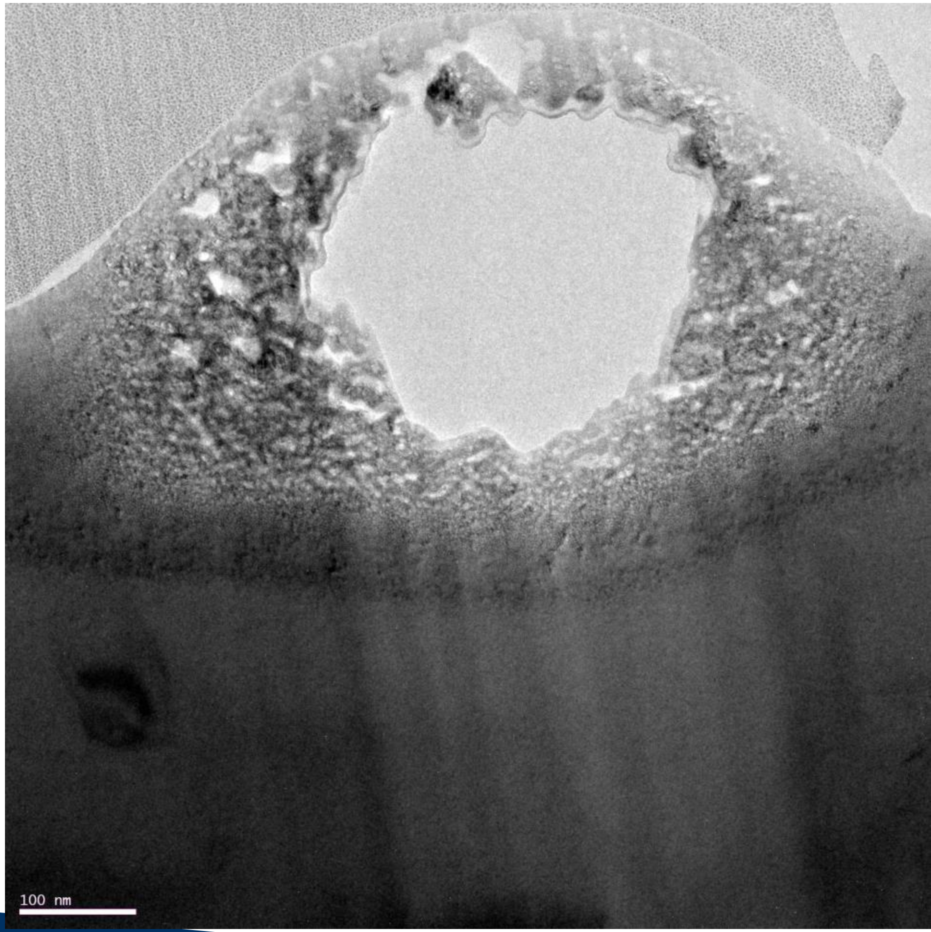
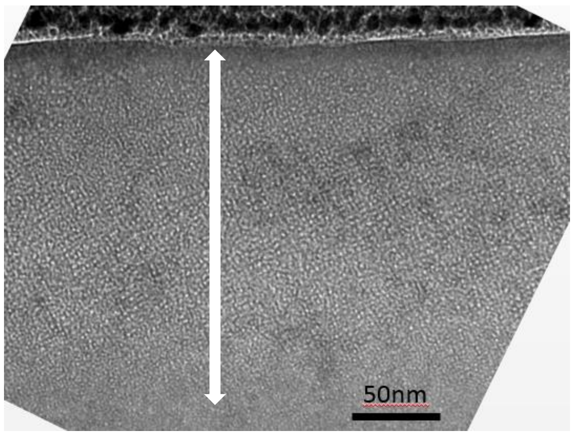
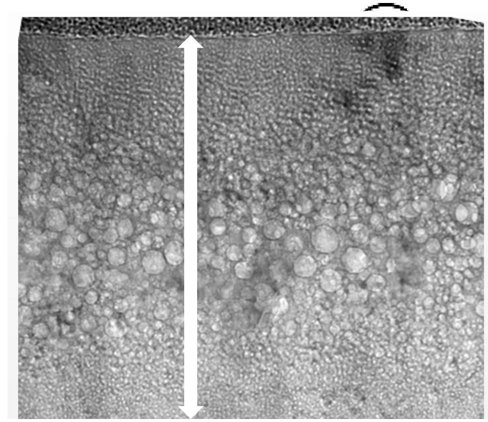
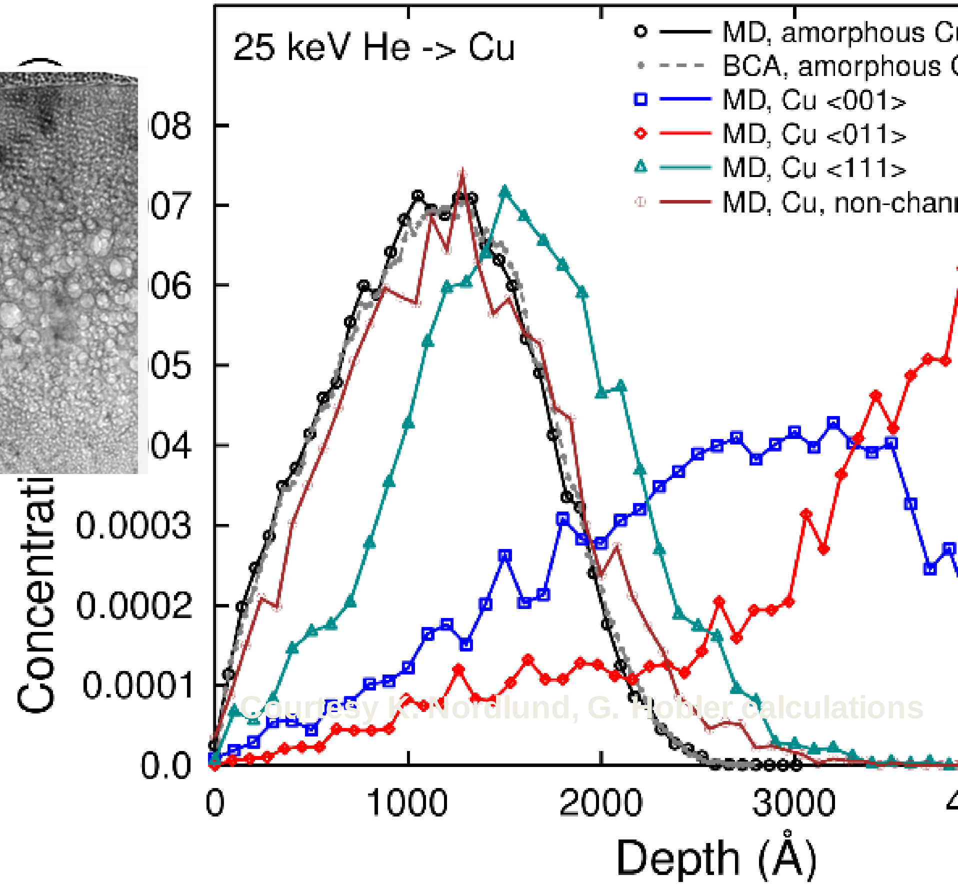
TEM tomography of the Blister



Penetration depth of Helium considering ion channeling







- Mean range for amorphous cells:

  112 ± 1 nm (MDRANGE calculation)

  114 ± 1 nm (BCA CASWIN code)

Measured depth:

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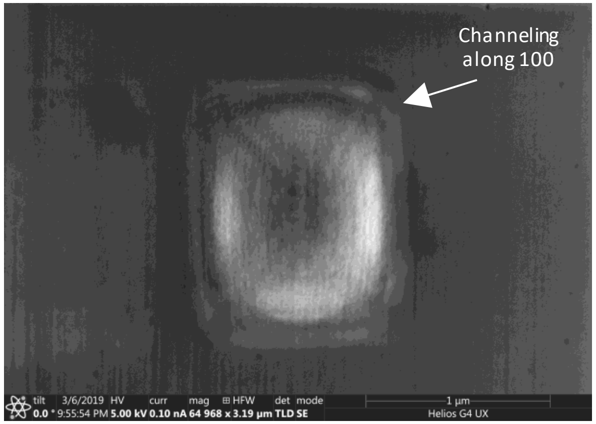
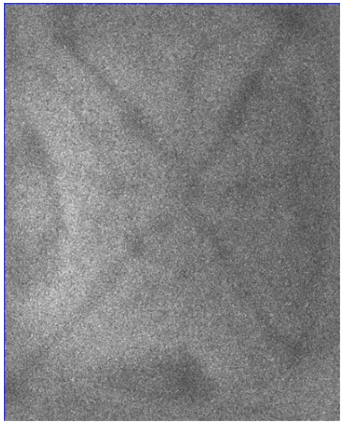
Channelling is observed for the does of $1{\times}10^{18}$ ions/cm^2

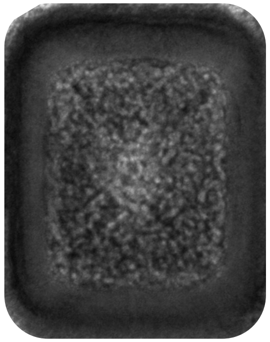
E-beam SE image



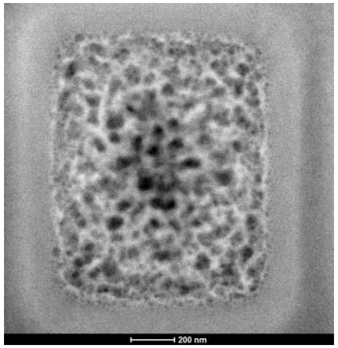
HIM SE image



TEM bright-field



STEM HAADF



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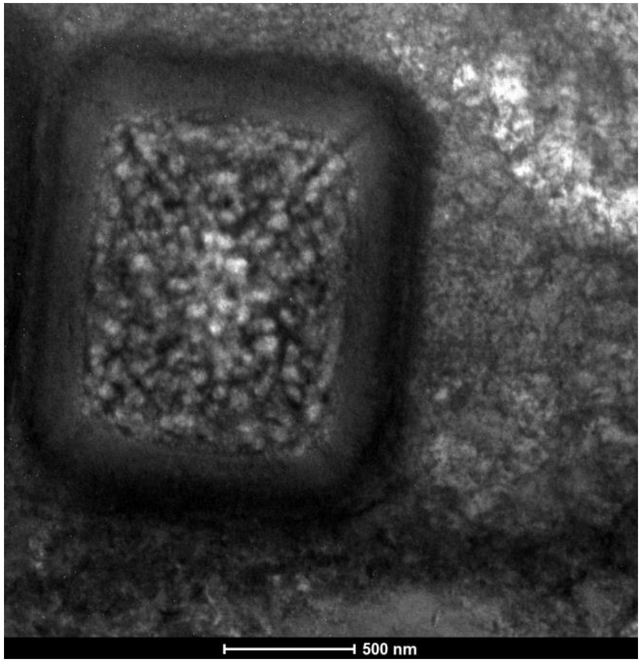

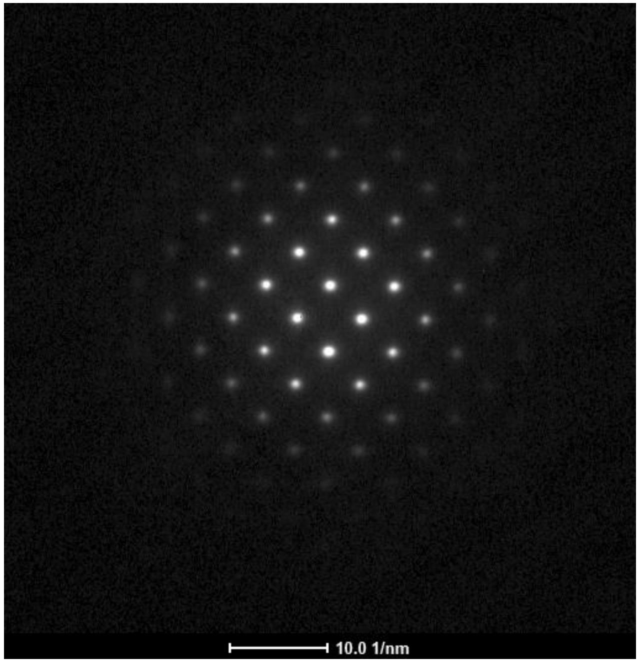

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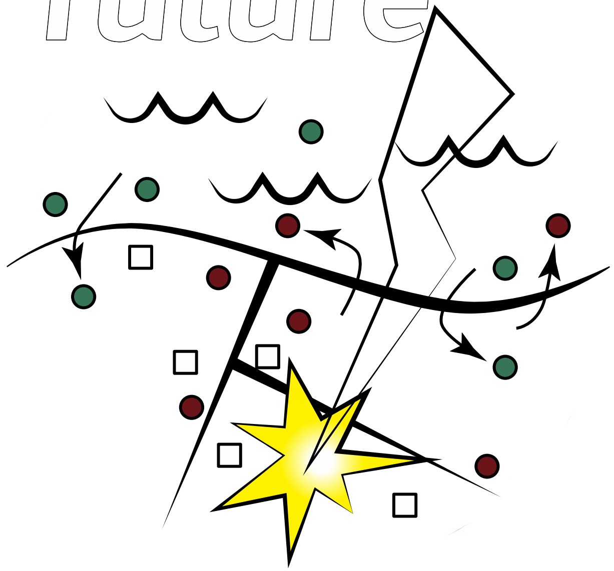
Quantification of Pressure in Helium Bubbles via 4DSTEM and Computer Simulations



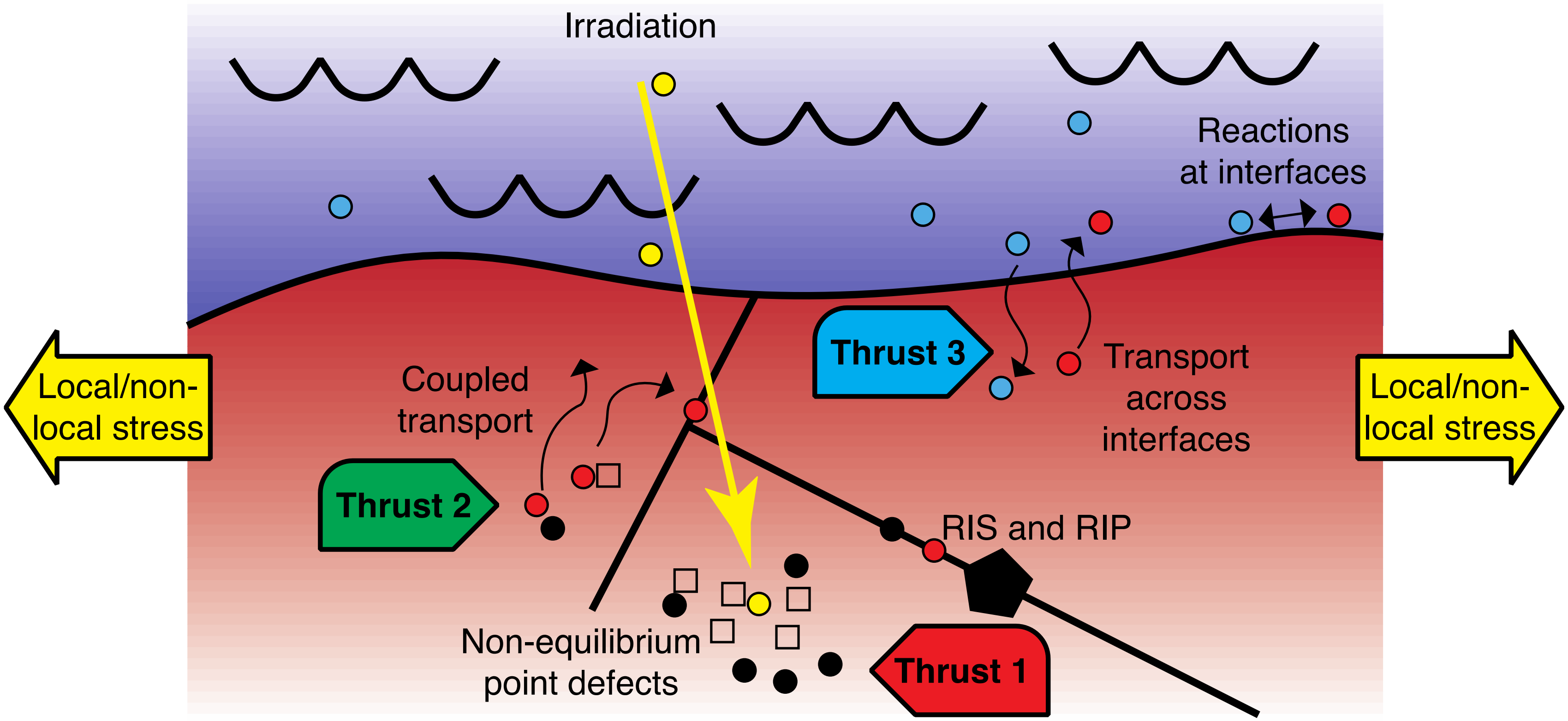
A. Kohnert, A. Minor, *P. Hosemann*, L. Capolungo

Fundamental Understanding of Transport Under Reactor Extremes (FUTURE)

An Energy Frontier Research Center funded by the U.S. Department of Energy, Office of Science

“Captain Future”
“Capitaine Flam”

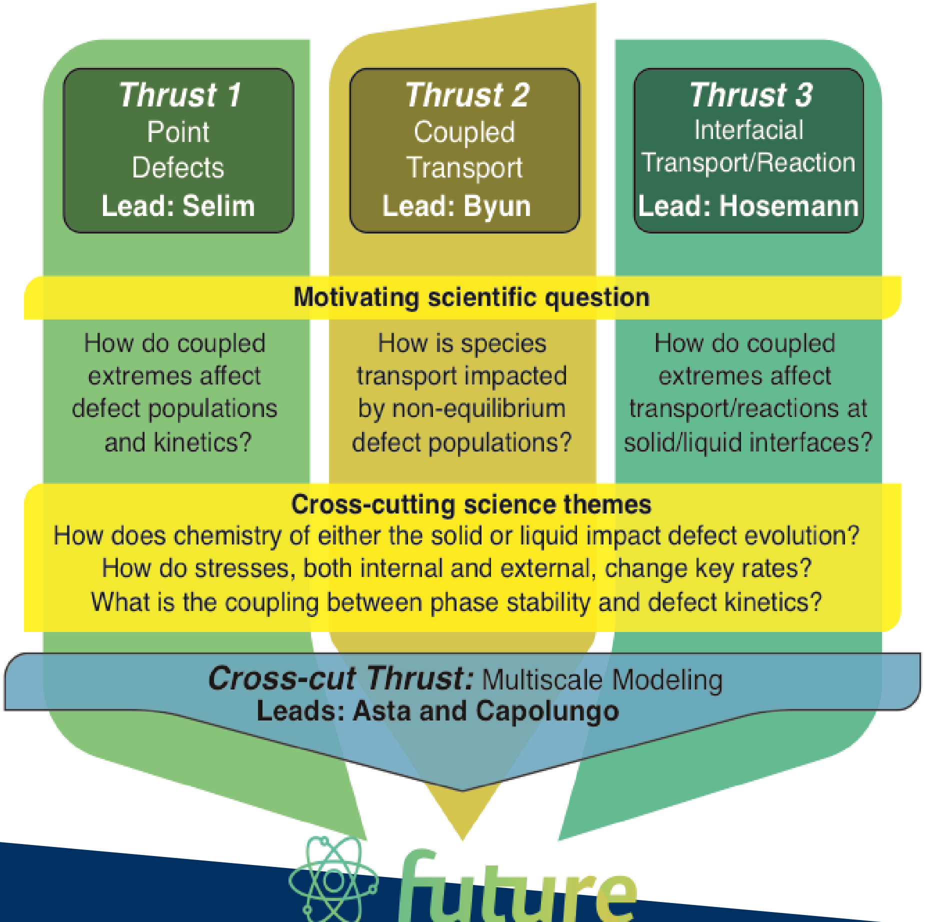
FUTURE EFRC key points

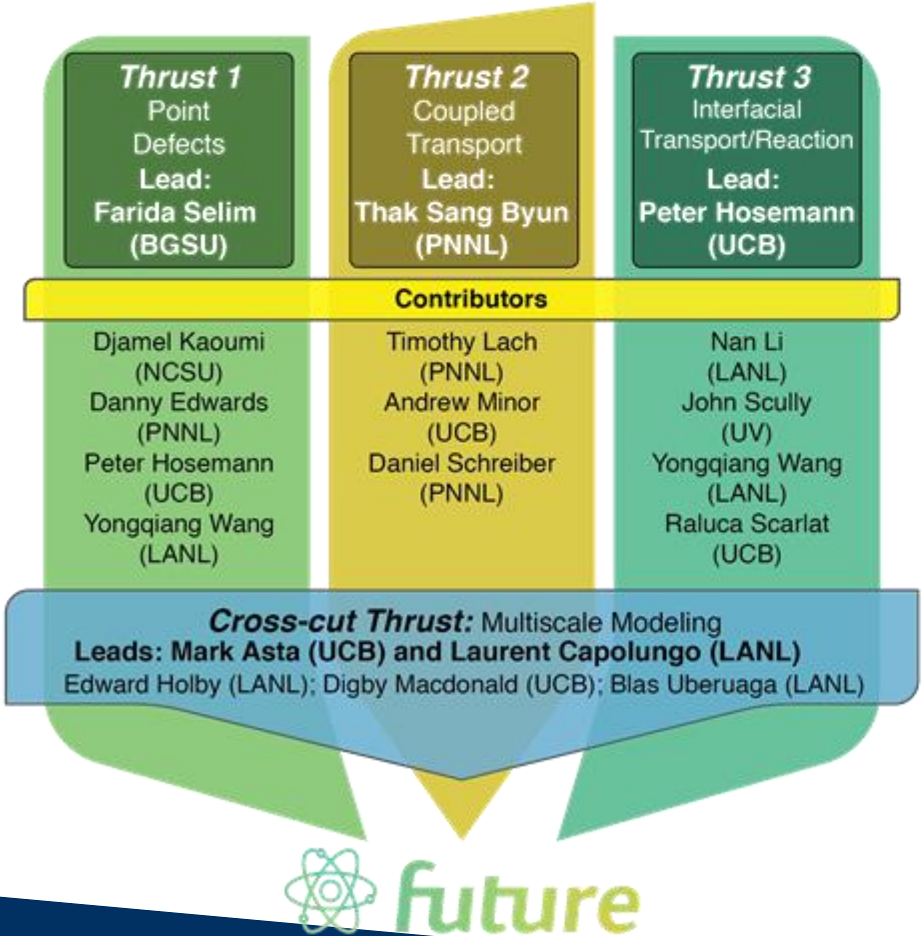


The post cascade surviving defects are responsible for transport phenomena and therefore alter the materials microstructure and composition.
We aim to understand the effects of the post cascade damage evaluation and their meaning for the environment.

FUTURE EFRC

PI: Blas Uberuaga





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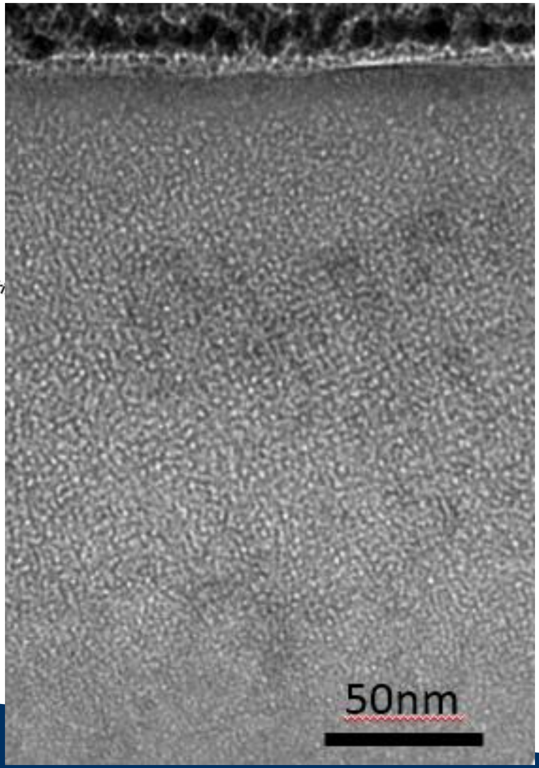
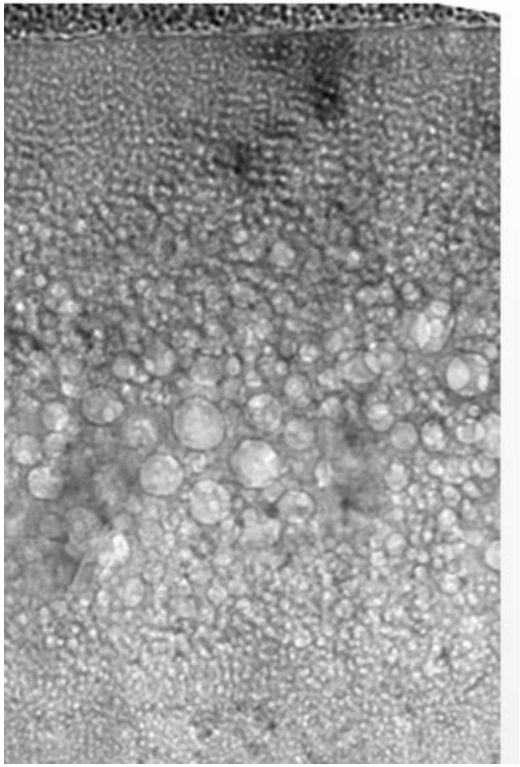
Generation of Helium filled cavities

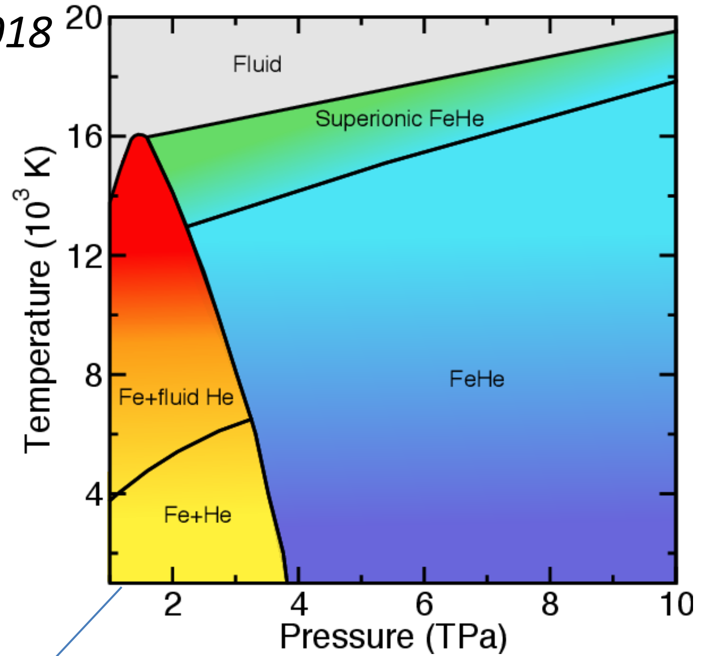
- Helium bubbles are studied since decades and occur due to (n,α) reactions and Helium implantations in fusion and fission.

What stresses surround Helium bubbles and can they be related to the gas content and pressure?



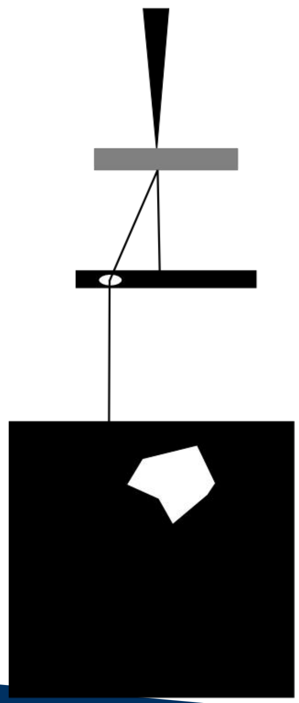


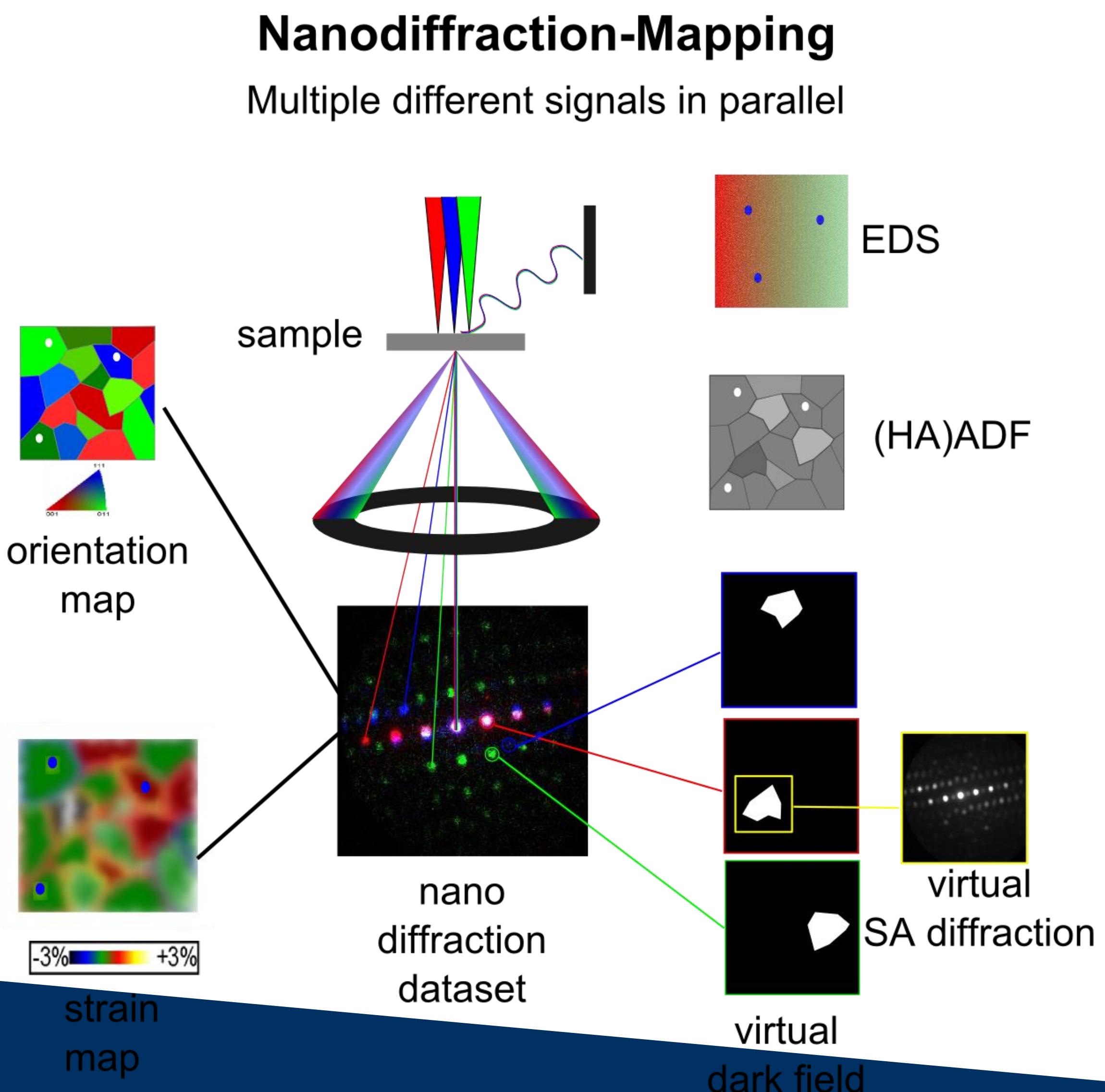
B. Monserrat et al, Phys Rev Lett 2018



ROI here Insolubility of He in Fe leads to helium bubble formation

4D STEM- A method to mapping method to provide more inside into the samples.





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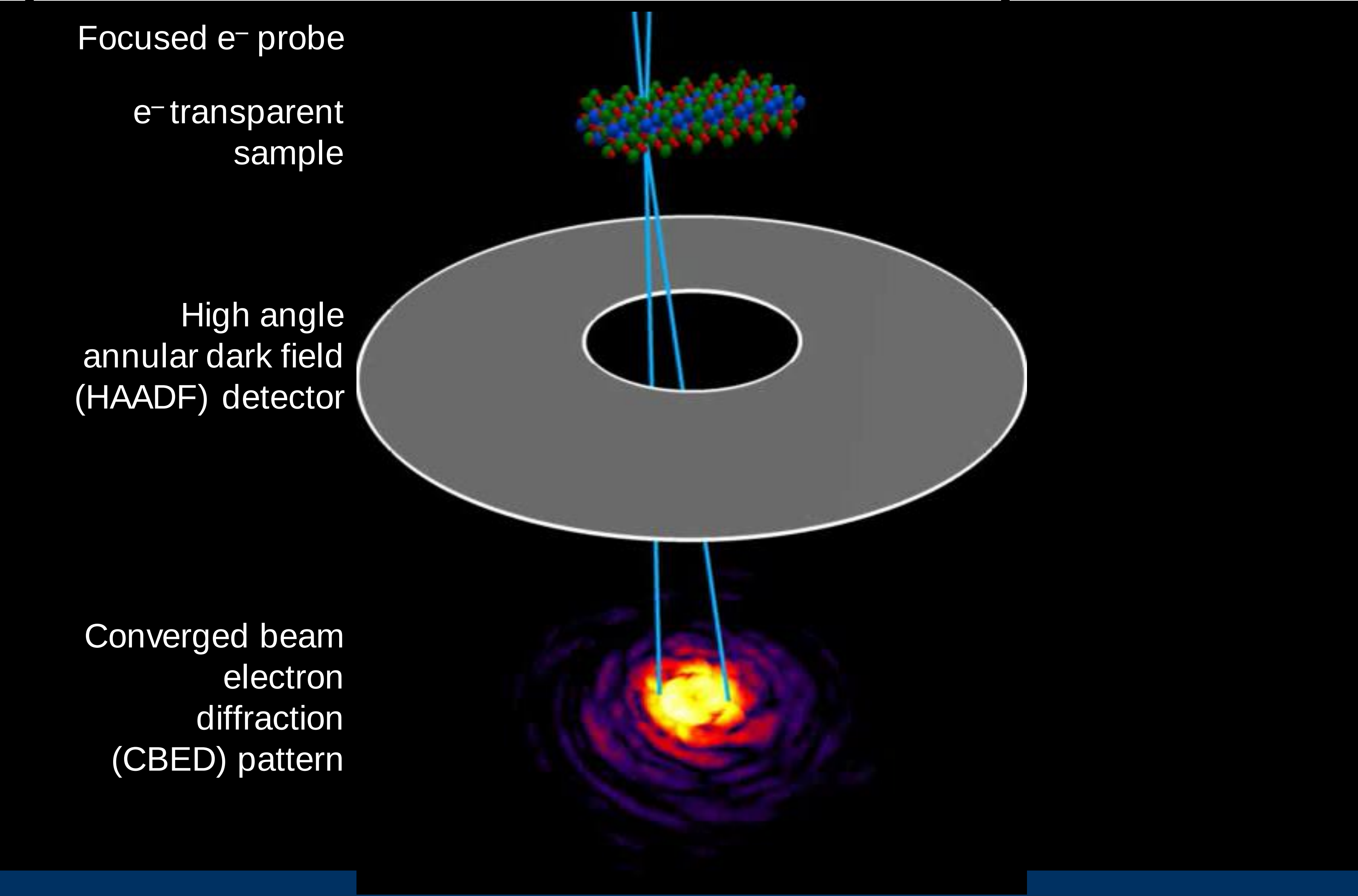
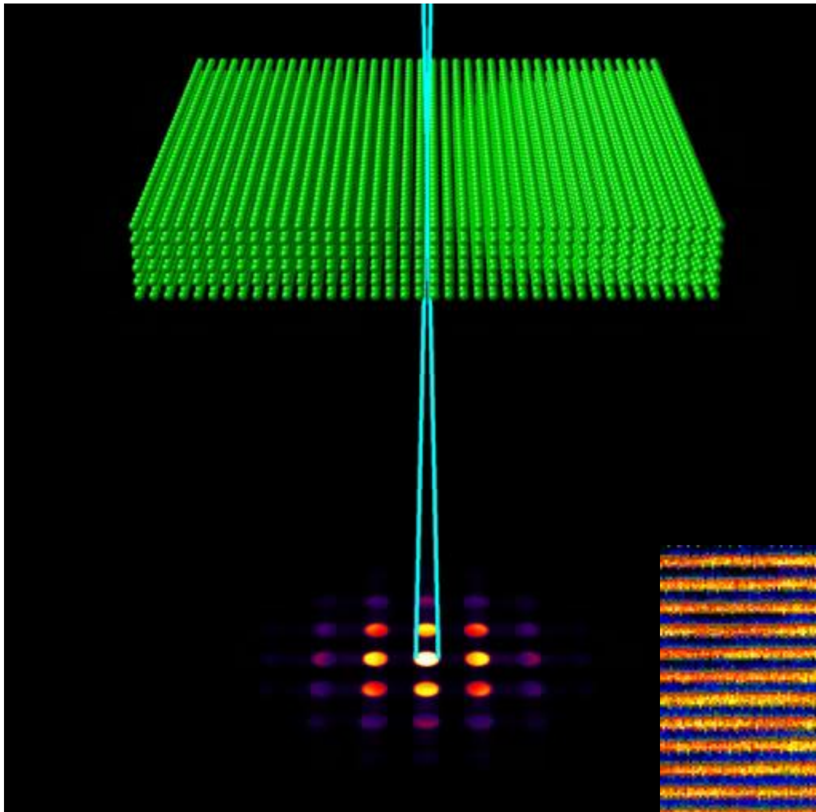
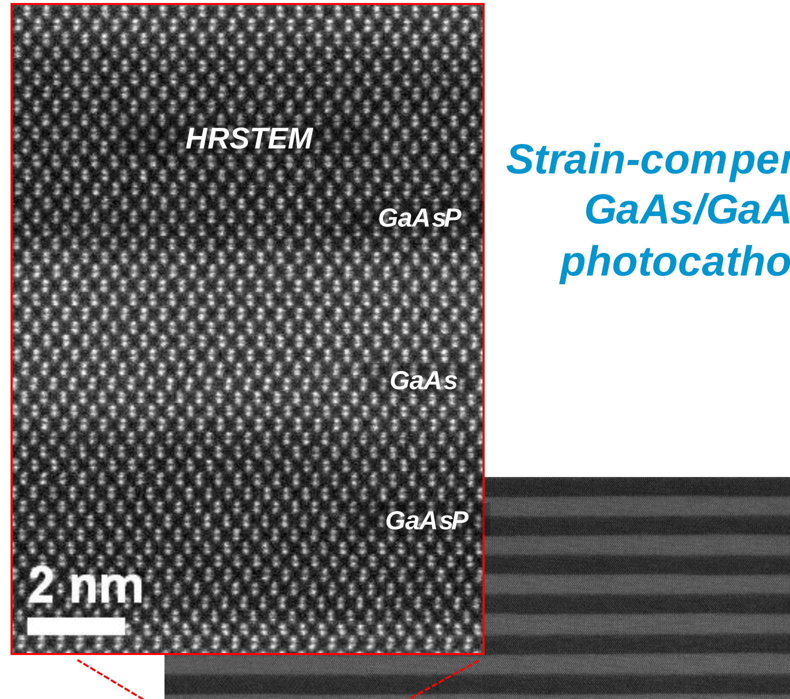
4D STEM- A method to mapping method to provide more inside into the samples.

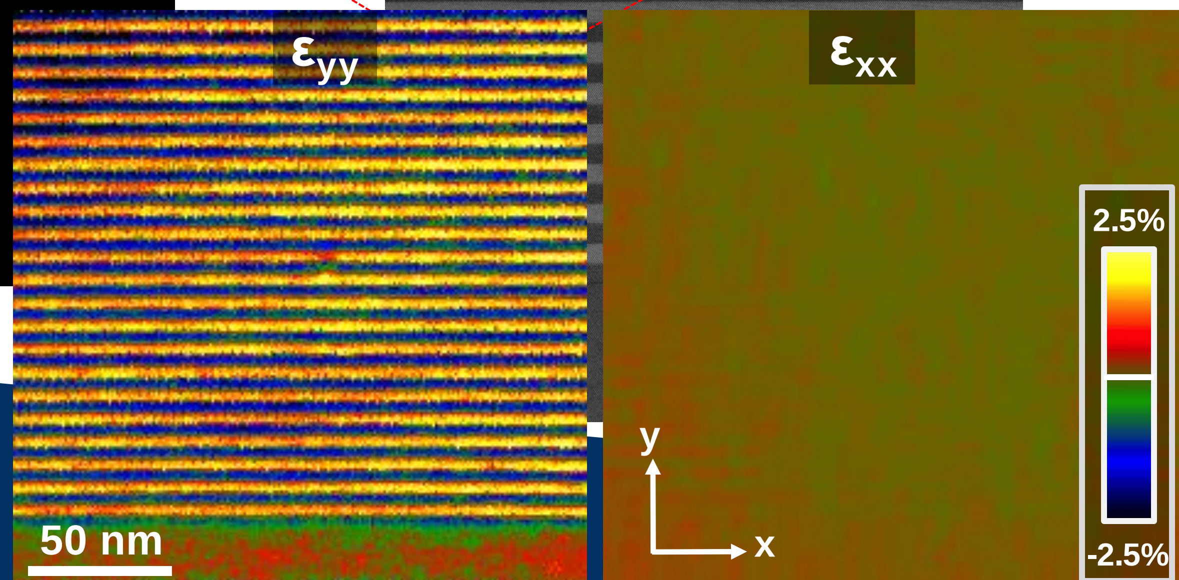


Nanobeam diffraction strain mapping



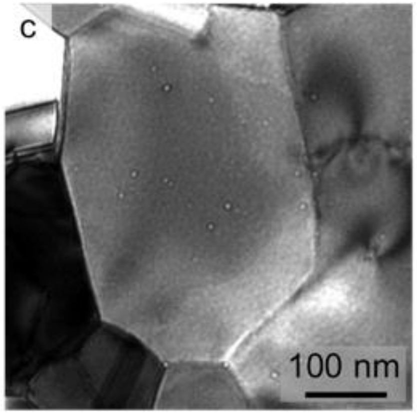


Strain-compensated GaAs/GaAsP photocathodes

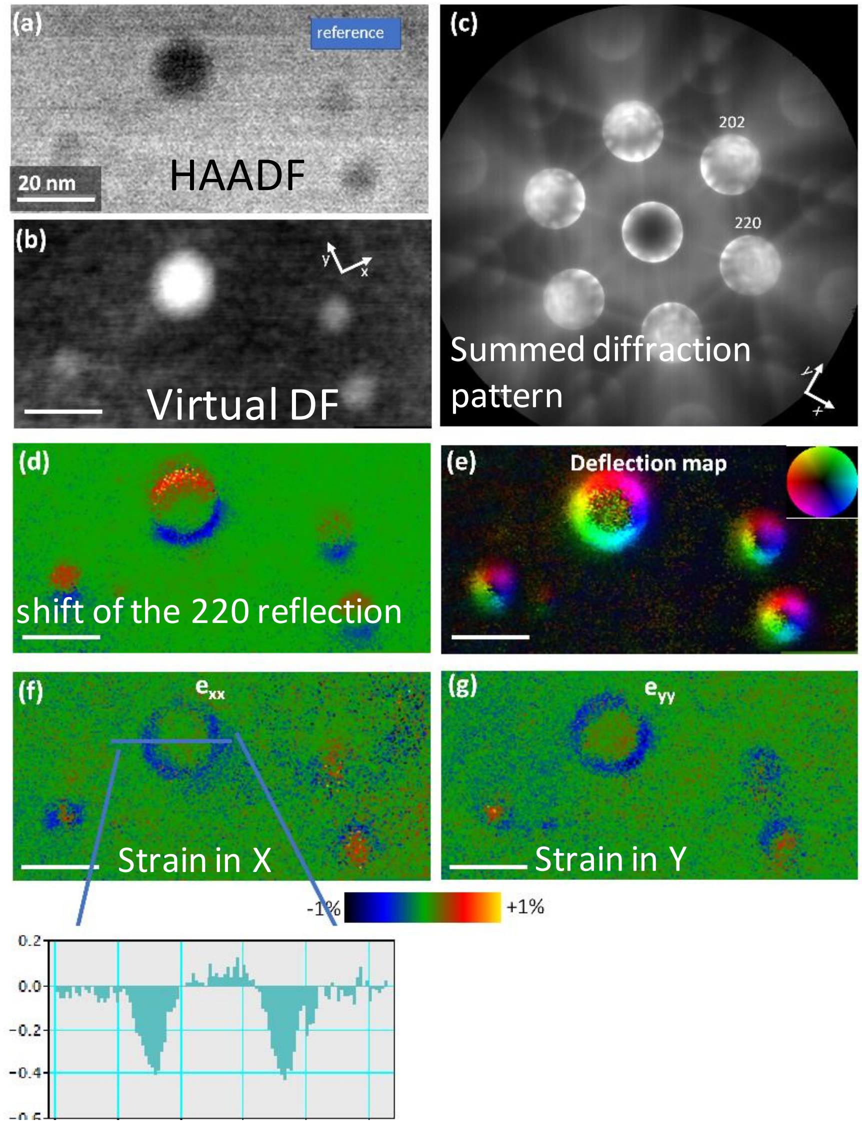


- B. Ozdol, C. Gammer, C. Ophus, P. Ercius, J. Ciston, and A. Minor, *APL* (2015)

Connecting with Experiments



4D STEM



He implanted Au at CINT Sandia (K. Hattar) and post implantation annealing at 360°C

- 4D STEM provides 2D map of strain around helium bubbles
- Modeling Question: Can we use this information to determine the **pressure** in the bubbles?
- → validate the models
- → dislocation bubble interaction

-Chisholm, Gammer, Kohnert, Ozdol, Hattar, Capolungo, Hosemann, Minor, (to be submitted)

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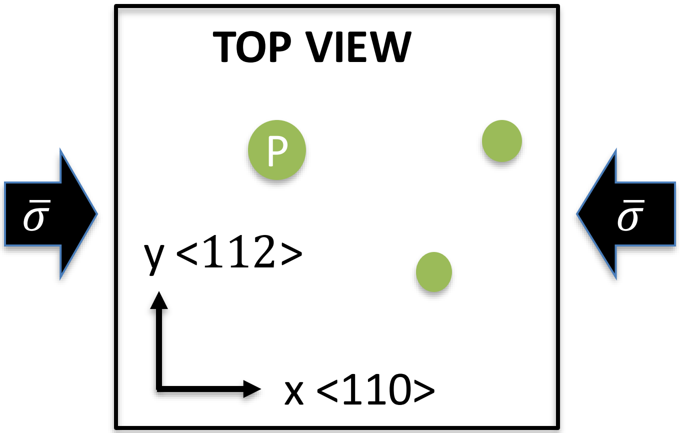
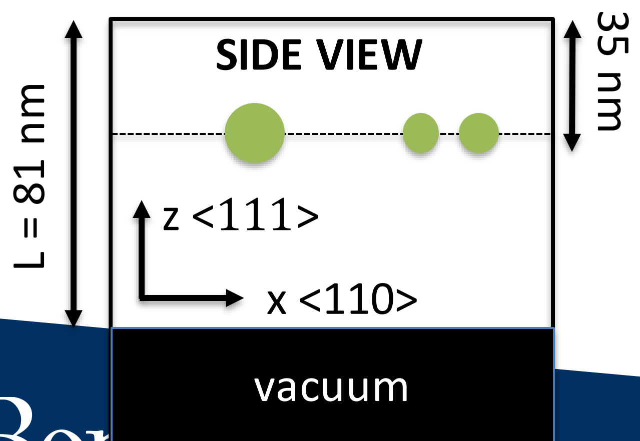
Simulation Setup

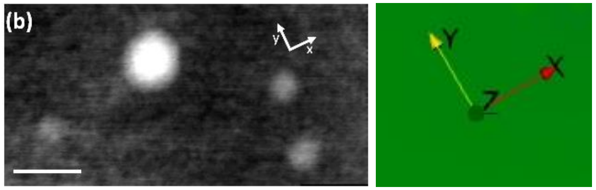
Periodic boundaries





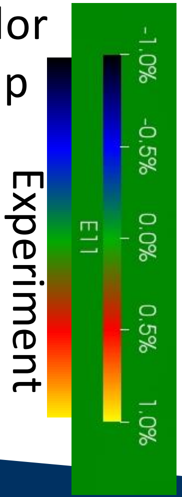
- Experimental conditions reproduced as closely as possible
- Three variables available in the model
  - Bubble pressure (P)
  - Average stress in the grain ($\bar{\sigma}$)
  - Foil thickness (L)
- Generates a **3D map** of stresses and strains

Orientation



Experiment Model

Color map



Virtual Experiment

1. Place bubbles with pre-defined pressure & average stress state
2. Generate 3D fields of stresses and strains
3. Apply several formulas to convert 3D fields to 2D image

In Plane

Report the strain values within the plane the bubbles sit on

$$\varepsilon(x, y) = \varepsilon(x, y, z_0)$$

Average

Report the through foil average of the strain state

$$\bar{\varepsilon}(x, y) = \frac{1}{L}\int_0^L \varepsilon(x, y, z)dz$$

Max strain

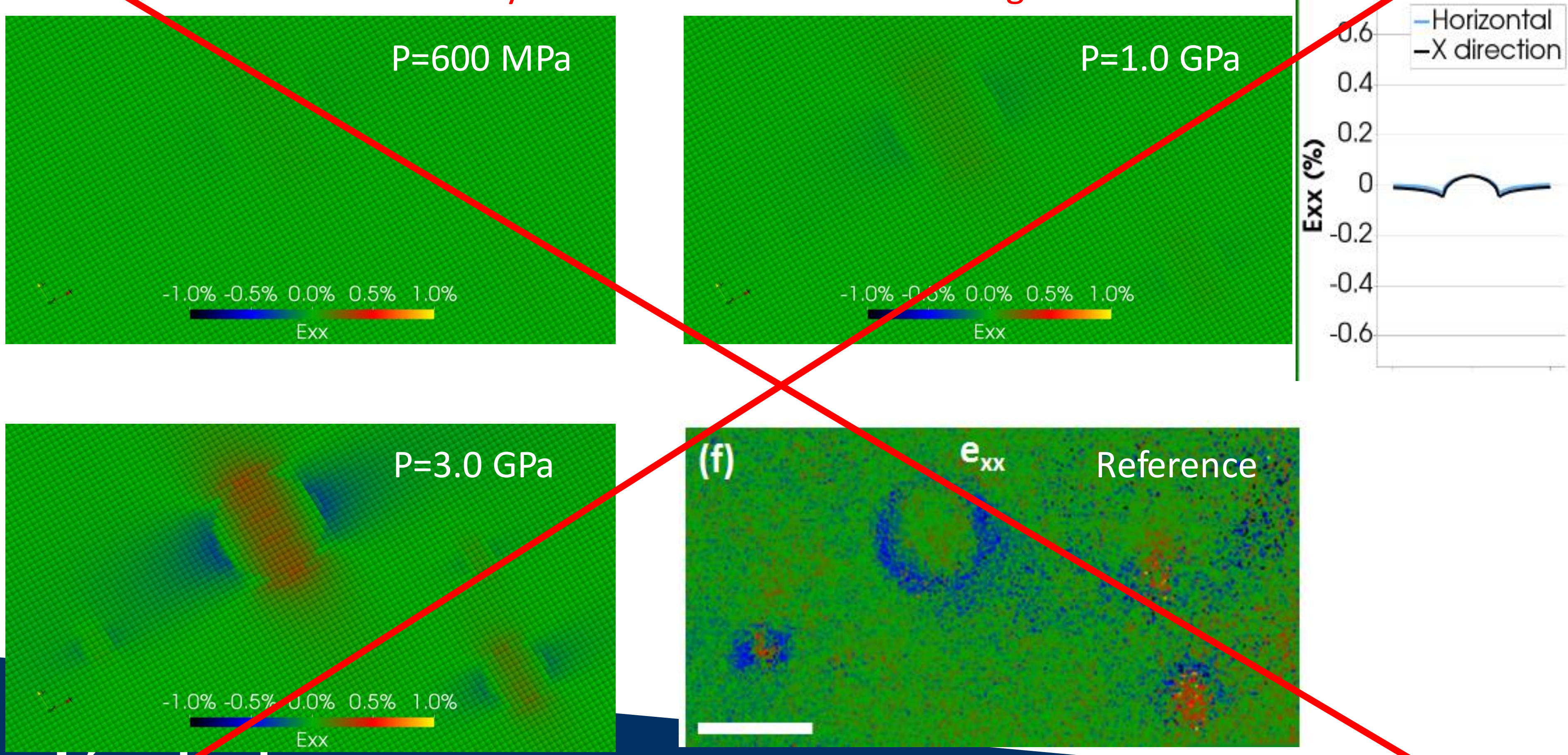
Report the largest magnitude of strain through the foil

$$\hat{\varepsilon}(x, y) = \max_z |\varepsilon(x, y, z)|$$

Through Foil Average Strain

- In order to obtain similar strain fields 3GPa pressure would be needed (non physical)

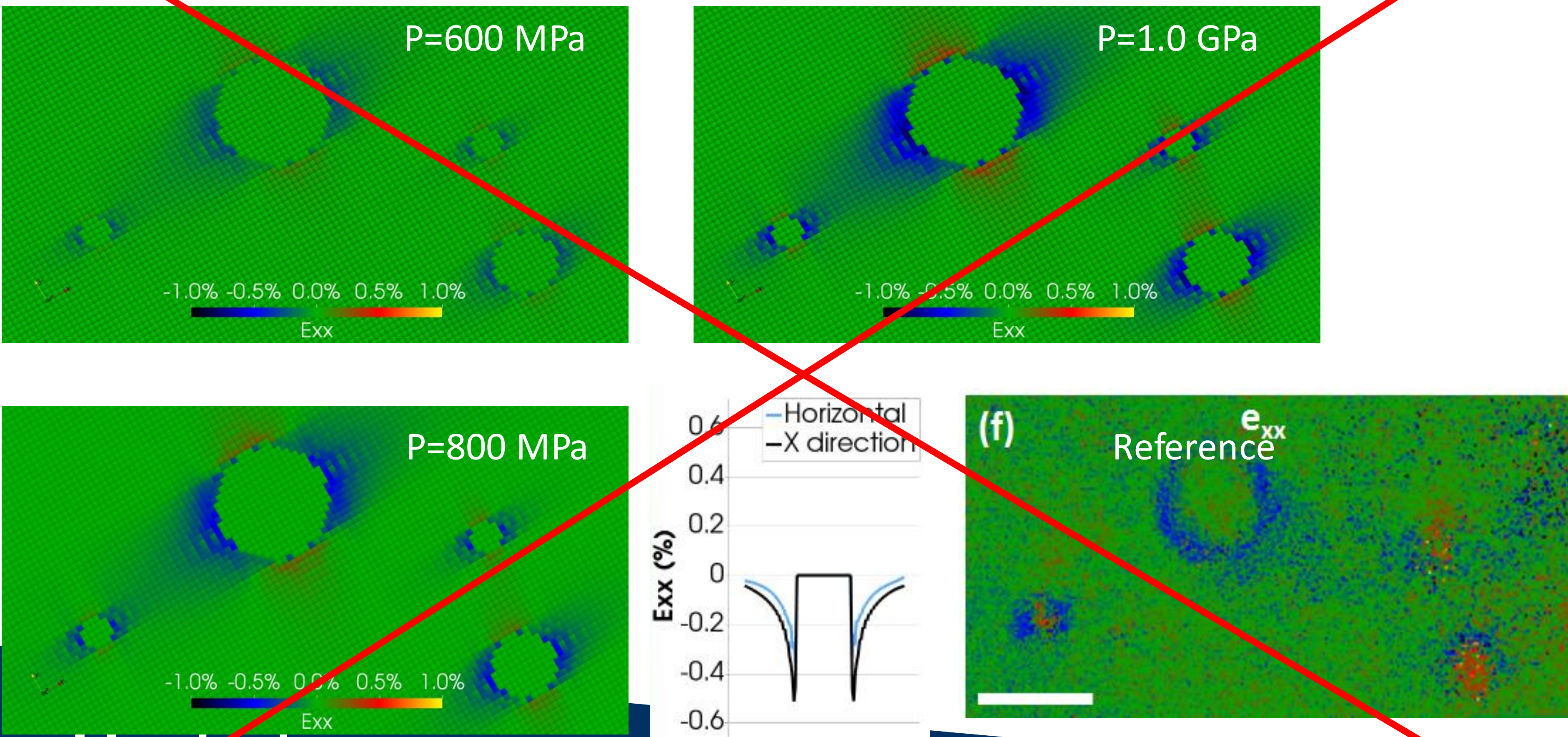
Likely we do not measure the average strain



In Plane Strain

- Analytical analysis indicates pressure of ~700 MPa in largest bubble to achieve experimental strain state (in-plane)

This would assume there is no contribution from the thickness



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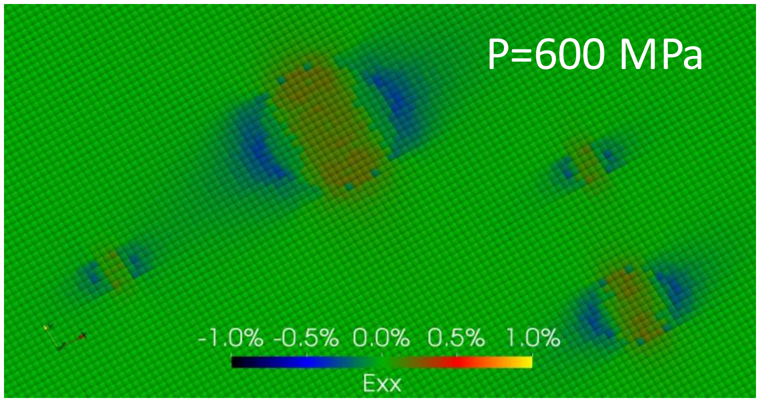
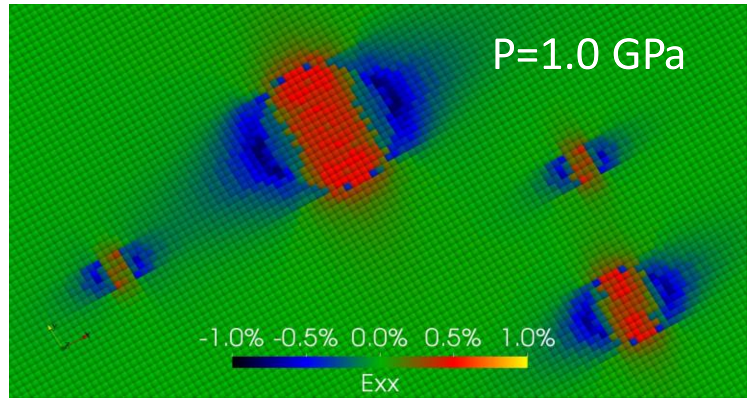
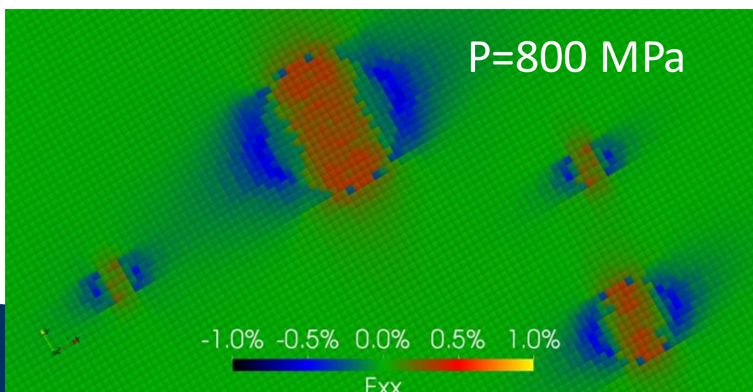
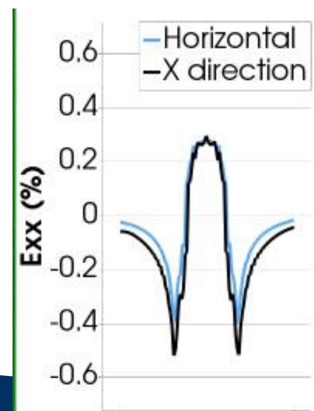
Maximum Strain

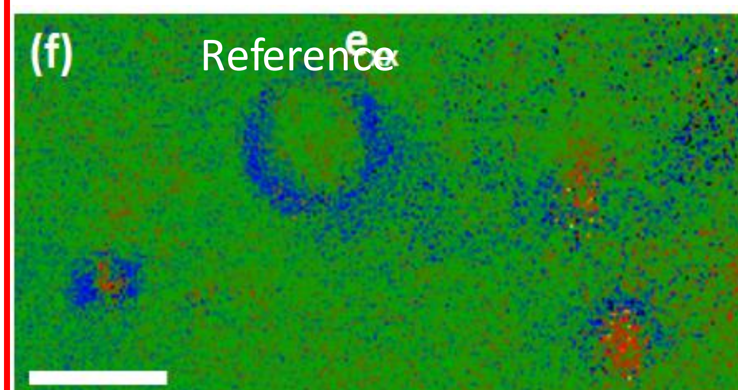
- Tensile values appear within bubble perimeter
  - Pressure magnitudes remain reasonable







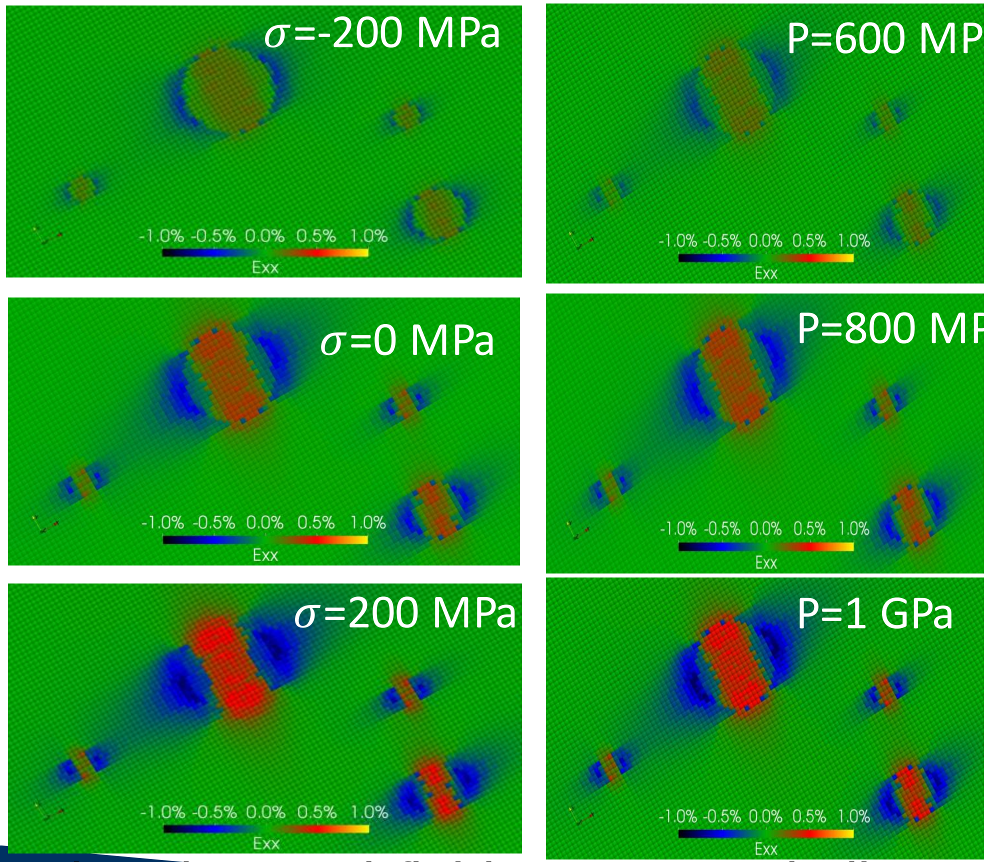




TEM foil under external stress?

P=800 MPa

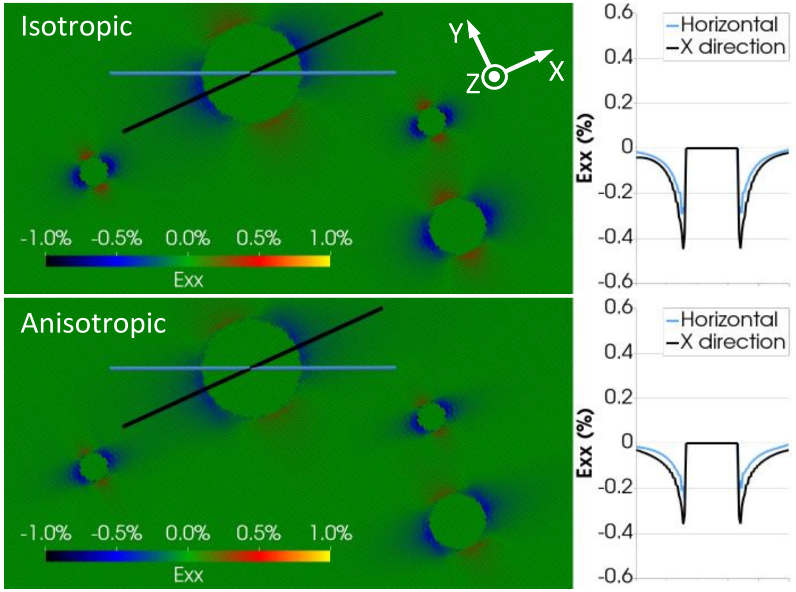
σ=0



- The observed fields are very similar to the state with no strain but with effective pressure $P' = P + \sigma$

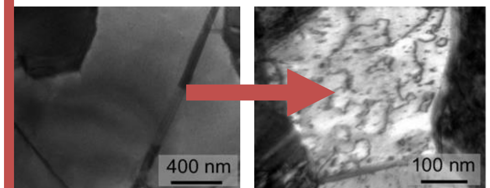
Isotropic vs. Anisotropic elastic properties

Elastic constant considerations



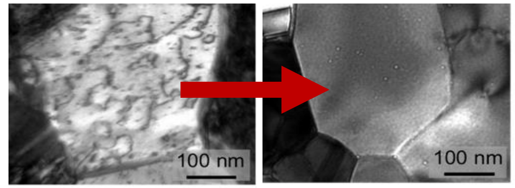
Compare the data with loop punching

1) Implant helium

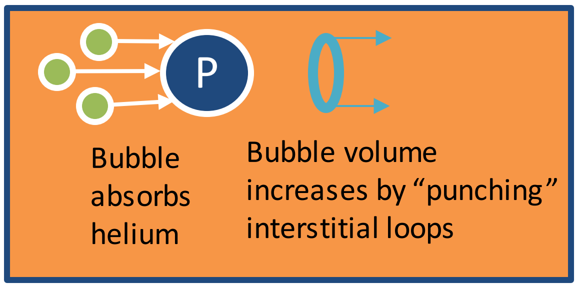


- Helium introduced
- Radiation damage forms
- Bubbles not visible

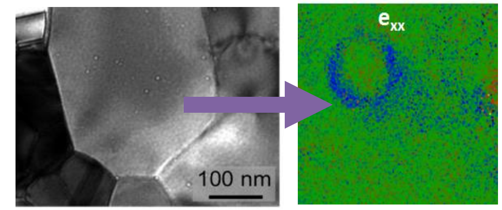
2) Anneal implanted sample



- Damage annealed out
- Bubbles grow
- Pressure limited by loop punching



3) Cool and measure strain



- Temperature reduced
- Gas pressure drops
- Maximum pressure in bubbles is less than loop punching pressure

$$P_2 \approx P_1 \frac{T_2}{T_1}$$

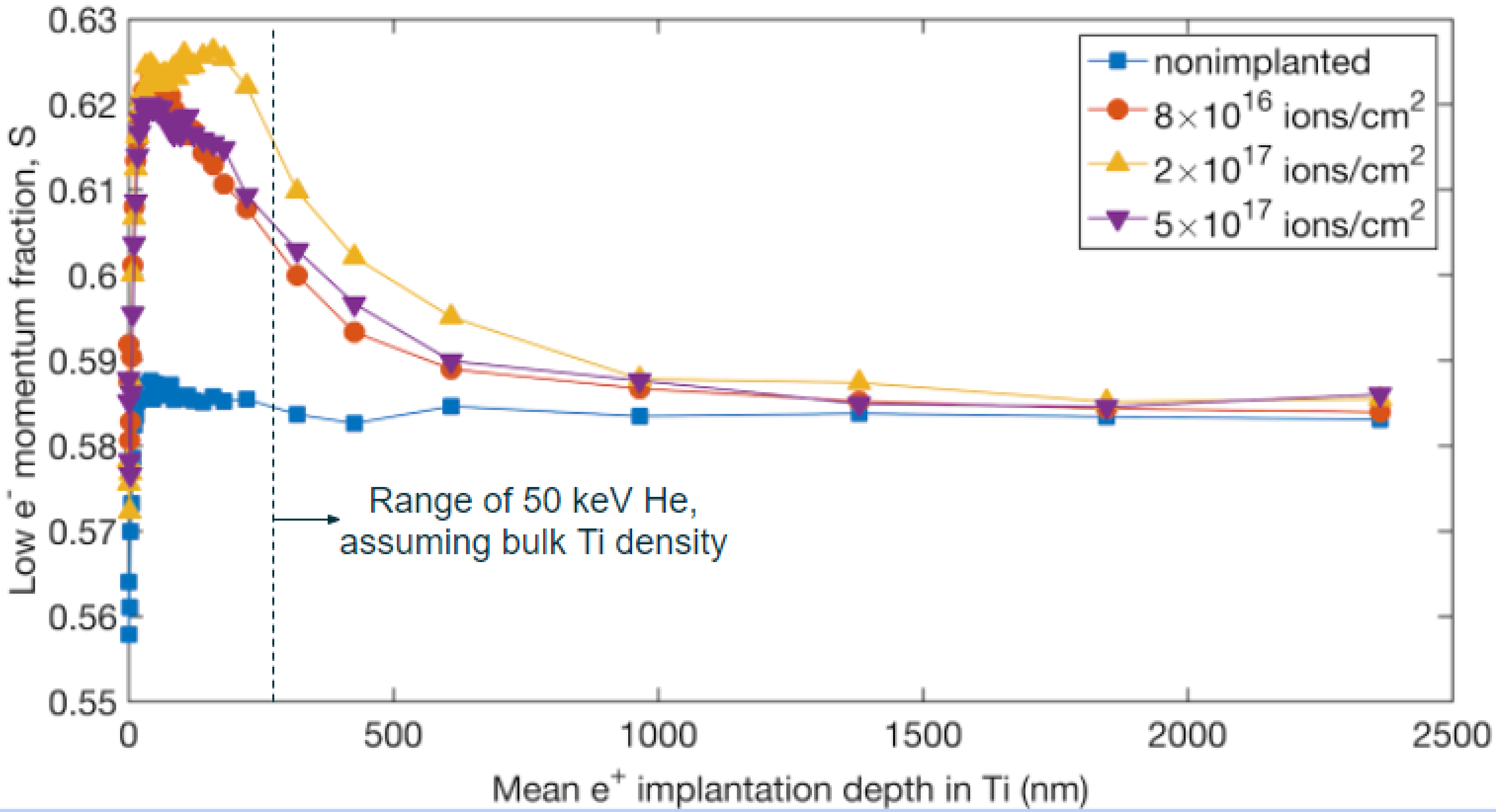
Physical limit on pressure

Loop Punching (at anneal 360°C)	1450 MPa
Max Pressure (at measurement)	**690 MPa**

Agree within measurement error

Pressure from measured strain

600 MPa	Isotropic Analysis
720 MPa	Anisotropic Analysis



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4D STEM is able to map the strain around microstructural features and calculate a pressure.

A model allows to further allowing to calculate the pressure in a Helium bubble and further the V/He ratio using EOS.

EOS	**He per V (@720 MPa)**	**He per V (@1100 MPa)**
Ideal Gas	2.05	3.14
Stoller ‘85	1.05	1.29
Stoller ‘14	1.01	1.21
Trinkaus ‘83	0.85	1.10

The virtual experiment agrees well with the actual experiment.

The EFRC allows for <u>modeling and experiments to be coupled</u> and establishes a method for further in the EFRC and other programs.

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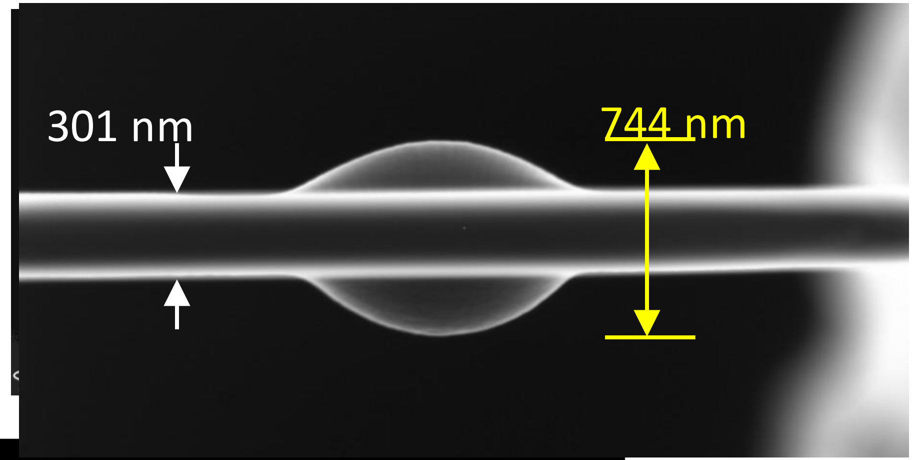
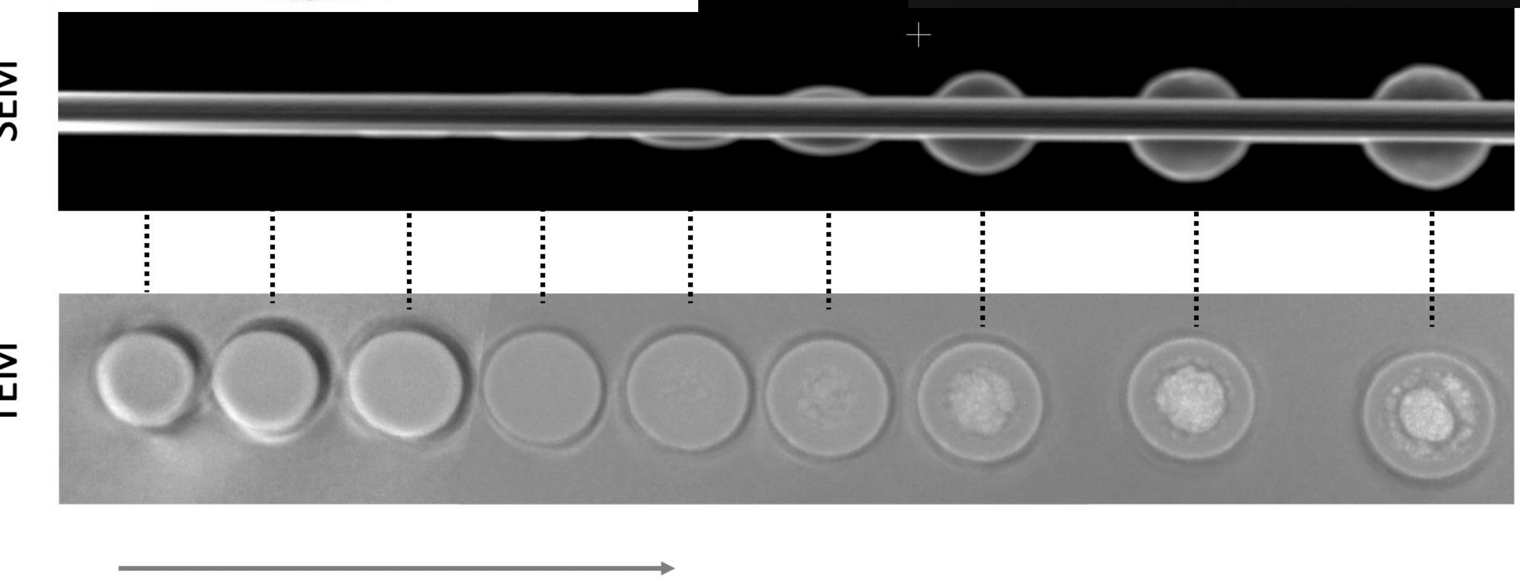
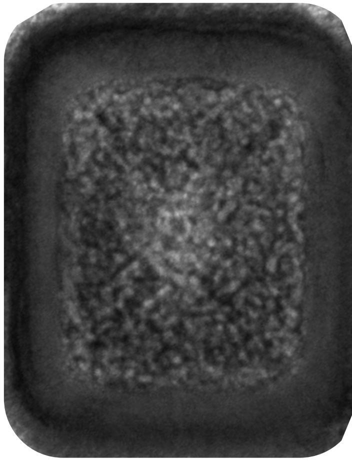
FUTURE inspired new work:

What is the stress at onset of blistering ?



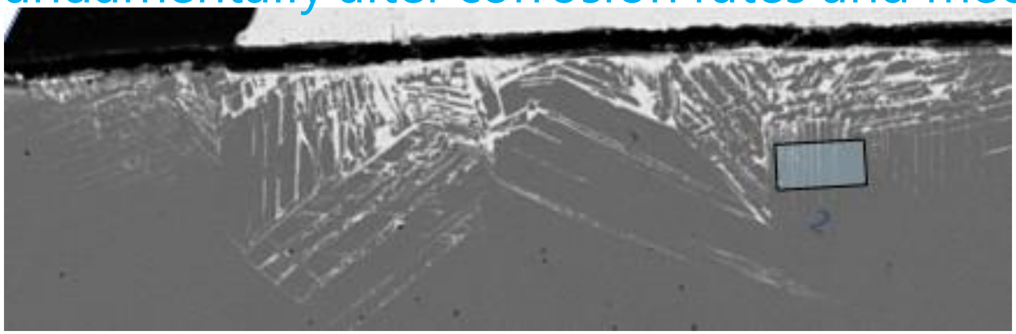


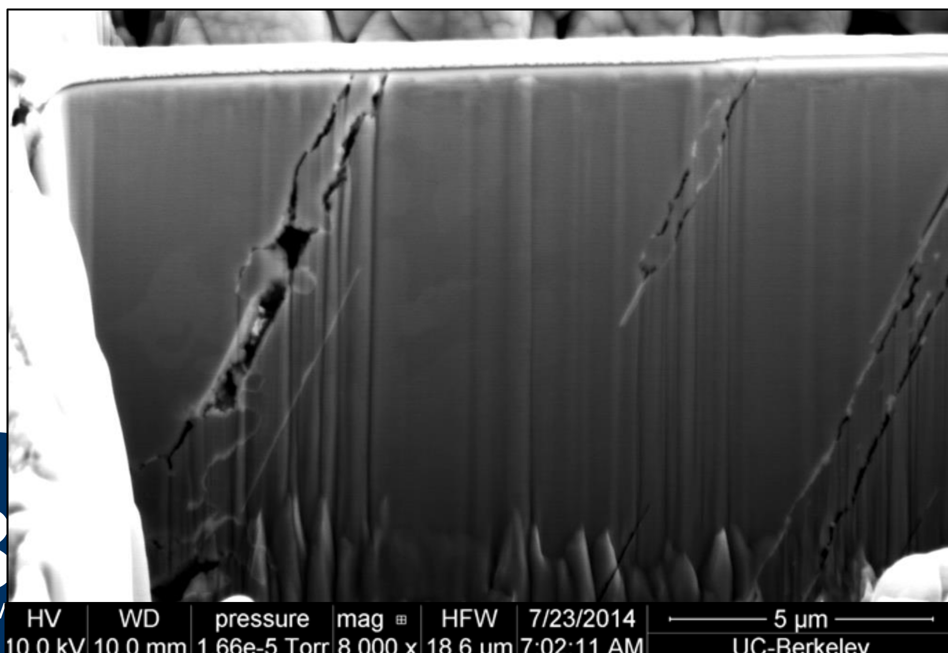


Why strain mapping in FUTURE EFRC Thrust III → environmental interaction??

- Hypothesis:

3) Radiation and corrosion related (electrochemically-induced) stresses may fundamentally alter corrosion rates and mechanisms.





316l, exposed to Lead Bismuth Eutectic (LBE) at 450C, 1000h <<10^{-8}wt% oxygen

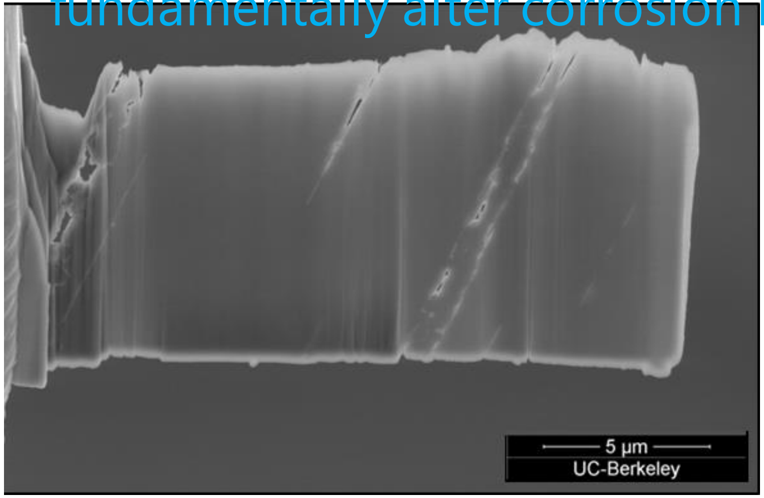
Deep penetration of LBE in the metal:

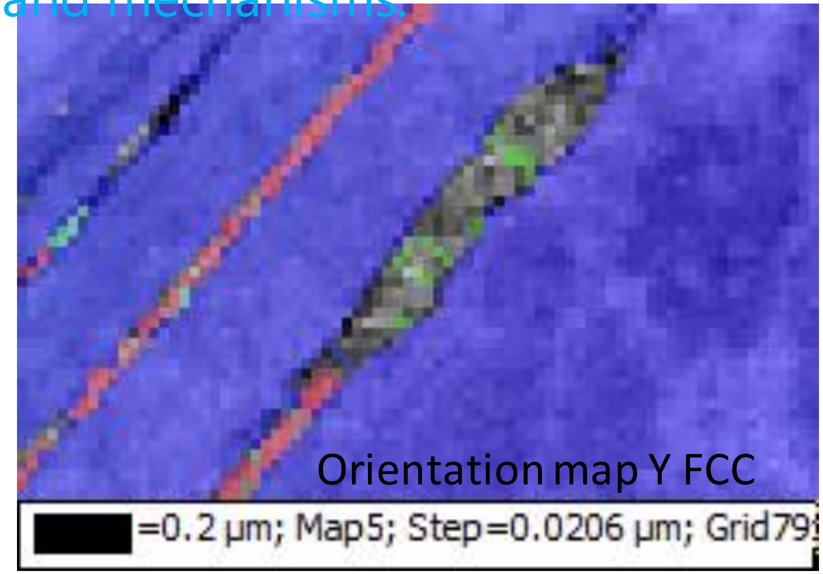
Ni has a high solubility in LBE

Why strain mapping in FUTURE EFRC?

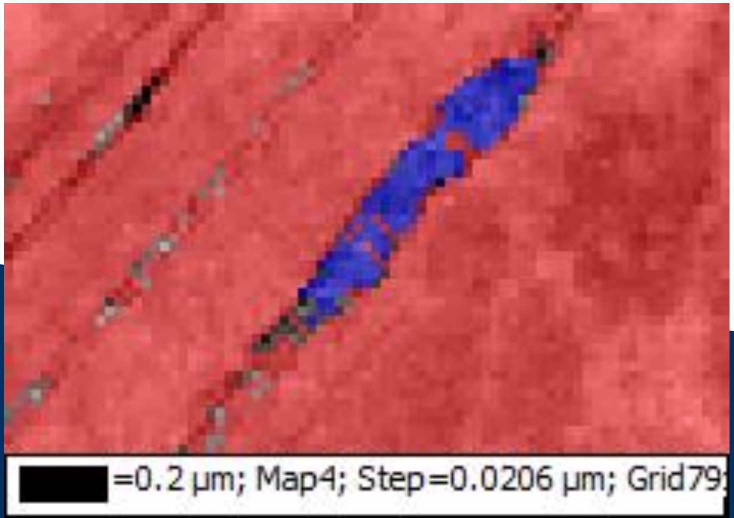
- Hypothesis:

3) Radiation and corrosion related (electrochemically-induced) stresses may fundamentally alter corrosion rates and mechanisms.





Phase map (red fcc, blue bcc)



At leaching paths bcc phase is found.

P. Hosemann, D. Frazer, E. Stergar, K. Lambrinou; Scripta Materialia. 118 (2016) 37-40

Hypothesis: does the leaching induced phase transformation lead to lattice stress and cracks?
4D STEM will help answer this question

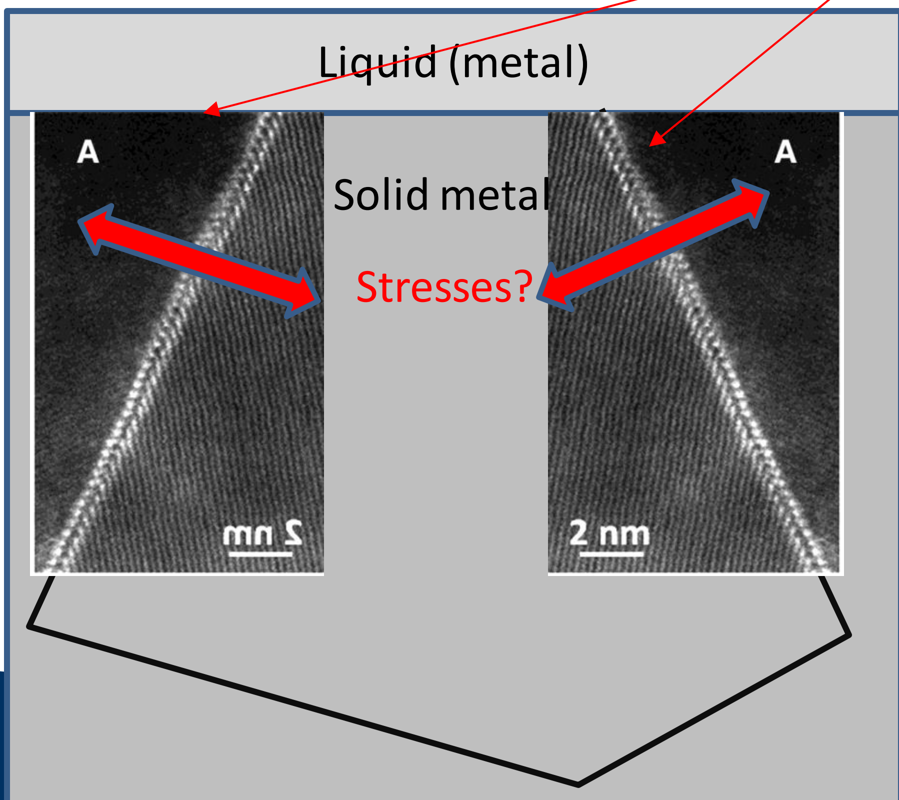
Why strain mapping in FUTURE EFRC?

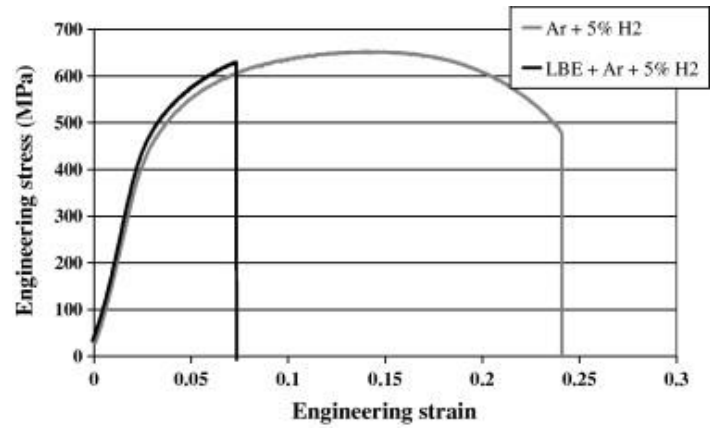
- Hypothesis:

3) Radiation and corrosion related (electrochemically-induced) stresses may fundamentally alter corrosion rates and mechanisms.

Bi atoms decorating Nickel GB

Jian Luo, Science 23 2011





Stress strain response showing LME in steels

J. Van den Bosch, P. Hoemann et all, J Nucl. Mat.

Elements originating from the liquids (Bi shown here) can decorate GB. What is the role of stresses in Liquid metal Embrittlement?

4D STEM will help us answer this question.

Comparison AFM-TEM data

Consider implanted He and equilibrium pressure

From TEM: 1E18 sample of **97nm** and 5E17 sample of **39nm**

From AFM :1E18 sample of **91nm** and 5E17 sample of **33nm**.

Consider implanted He and equilibrium pressure

$$P = \frac{2\gamma}{R} = \qquad [P + \frac{an^2}{V^2}](V - nb) = nRT$$

Only using TEM data (5E17 and 1E18)

$5*10^{17}$ ions/cm^2 should contain $2.37*10^{18}$ Helium atoms/cm^2 to account for the bubble size

1E18 ions/cm^2 should contain $7.28*10^{18}$ Helium atoms/cm^2 to account for the bubble size.

The bubbles are a factor 4 (5E17) and factor 7 (1E18) too large to account for equilibrium.

→ **Under pressurized bubbles**

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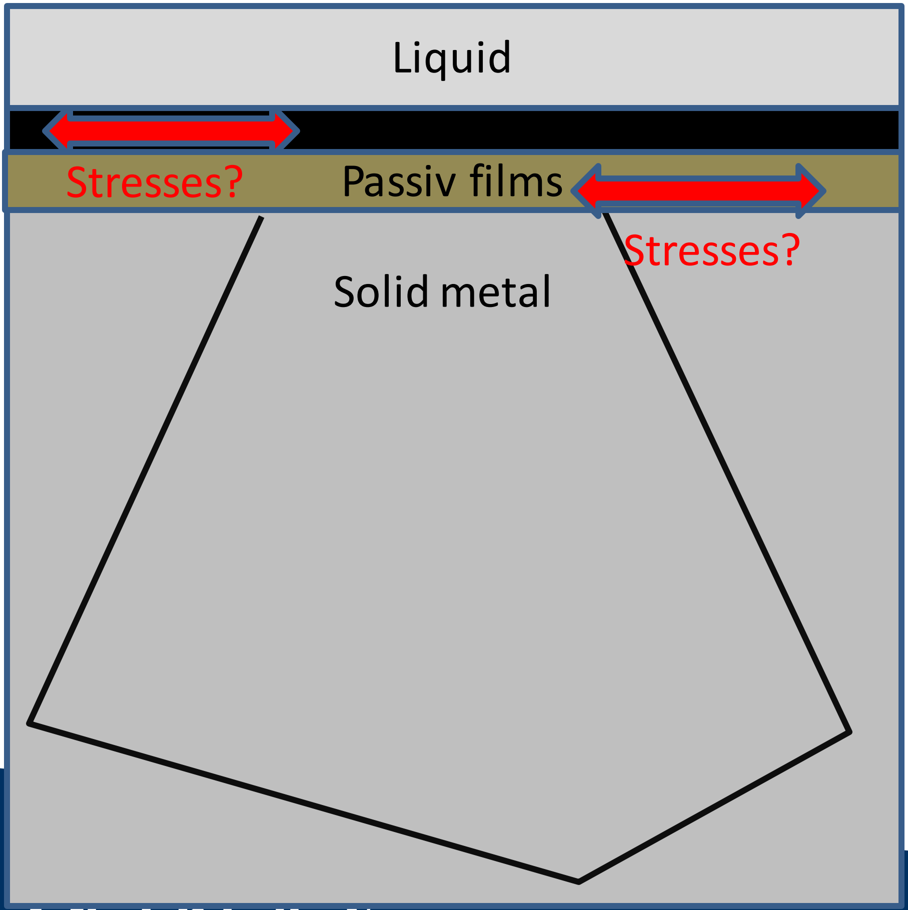
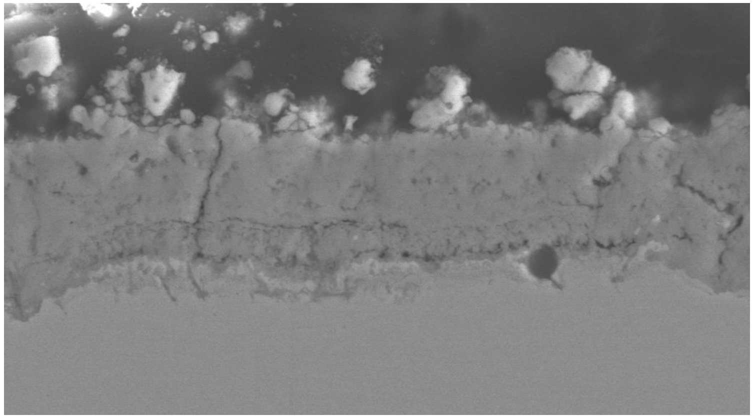
Why strain mapping in FUTURE EFRC?

- Hypothesis:
    3) Radiation and corrosion related (electrochemically-induced) stresses may fundamentally alter corrosion rates and mechanisms.





Quantification of the stresses formed in passive films in extreme environments (molten metal, aquas, etc)

→ Spalling of passive films.
→ Does radiation relax these stresses ?
→ Can these stresses contribute to transport problems in the metal/environment interaction?

Summary

Demonstrated how 4D-STEM strain mapping and modeling combined

a) Help us understand the data (maximum strain vs. average strain)
b) Quantify the pressure in a Helium bubble
c) Quantify the stress around the bubble which helps understand dislocation bubble interactions
d) Will help us understand blistering effects at very high Helium doses better.

How 4D-STEM will be utilized in environment thrust in the FUTURE EFRC

a) Quantify grain boundary stress with environmental segregates in the material (Pb, Bi, Te, etc..) and contribute to the understanding of LME phenomena

b) Quantify the stress in passive films formed under different conditions and relate to film spalling with and without irradiation.

c) Stress is a contributing factor to transport mechanism. Being able to quantify it on a local scale will allow to enhance the transport problem understanding.

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Thank you for your attention