Using *in-situ* TEM Triple Ion Beam Irradiations to Study the Effects of Deuterium, Helium, and Radiation Damage on TPBAR Components

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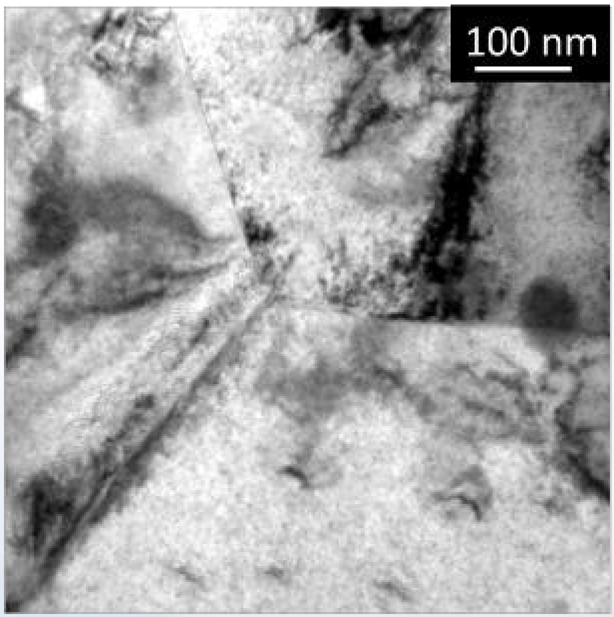
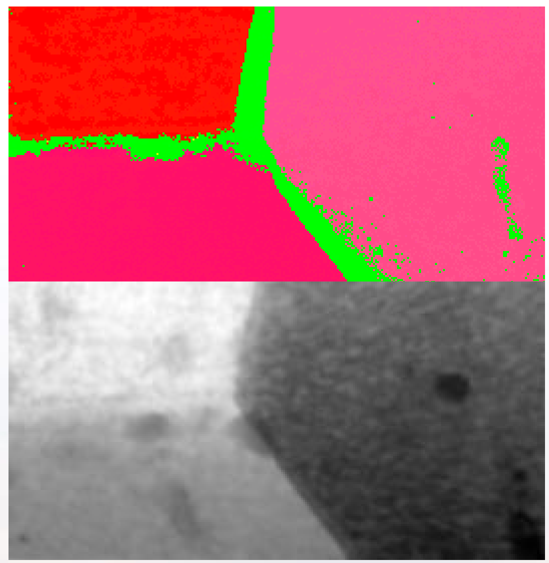
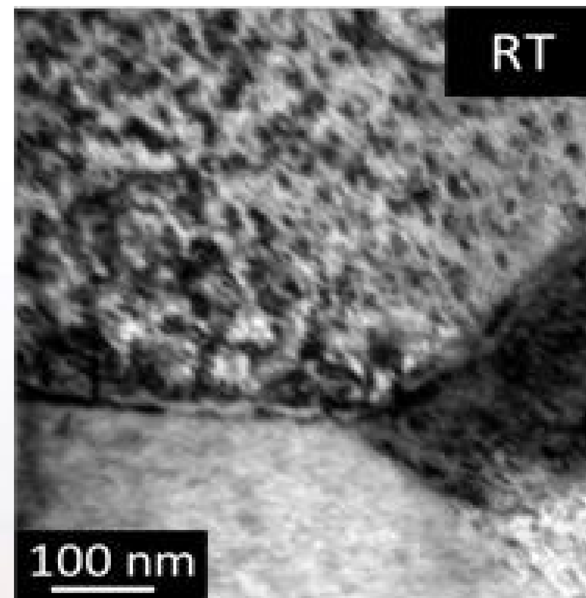
August 10th, 2017







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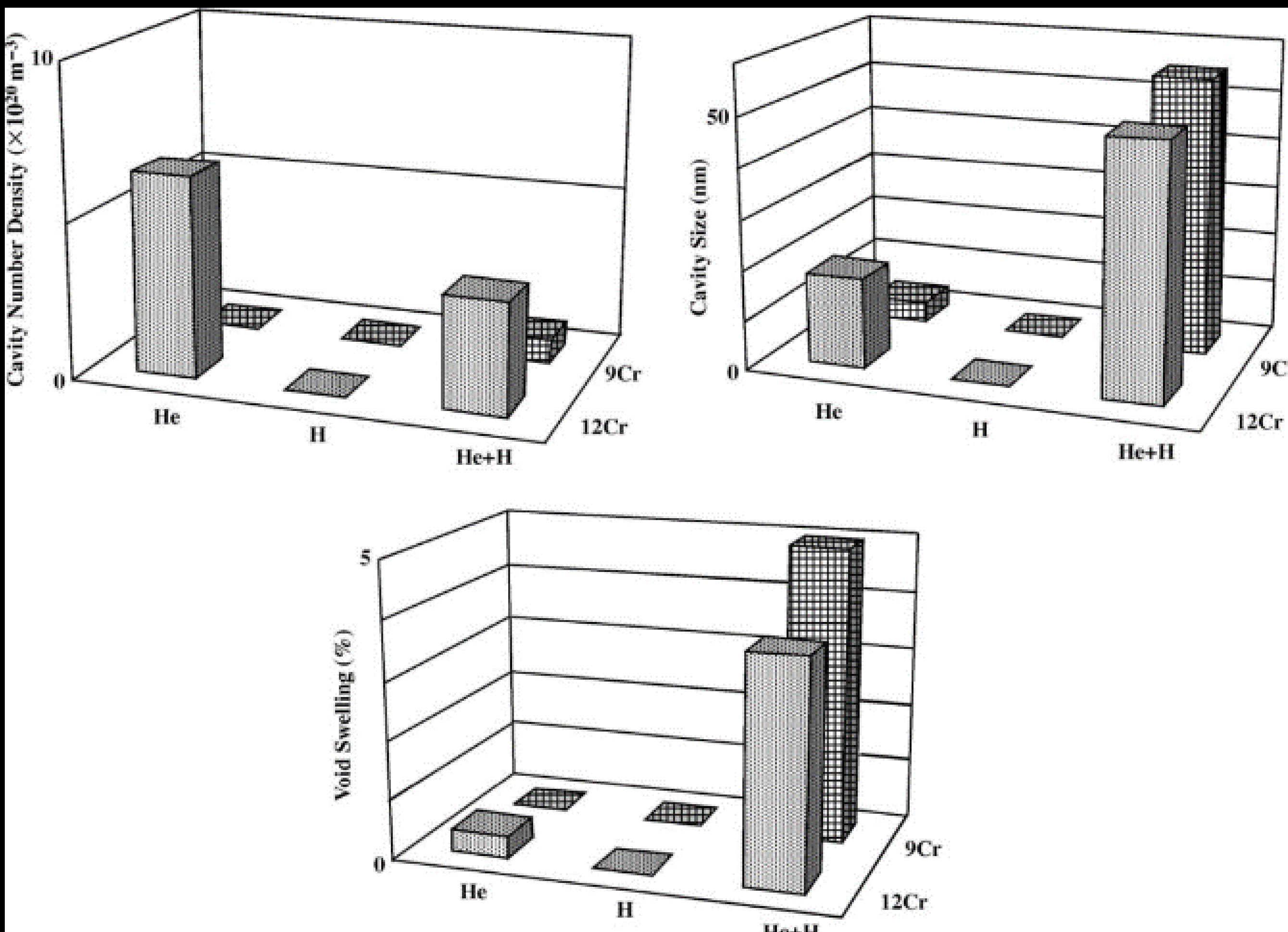
H, He, and Displacement Damage Synergy



T. Tanaka et al. "Synergistic effect of helium and hydrogen for defect evolution under milt-ion irradiation of Fe-Cr ferritic alloys"
J. of Nuclear Materials 329-333 (2004) 294-298

Coupling Effect

- H and He are produced as decay products
- The relationship between the point defects present, the interstitial hydrogen, and the He bubbles in the system that results in the increased void swelling has only been theorized.
- The mechanisms which governs the increased void swelling under the presence of He and H have never been experimental determined

Difficulty of performing triple-beam irradiation has resulted in a limited number of facilities world wide

In situ Ion Irradiation TEM Facility

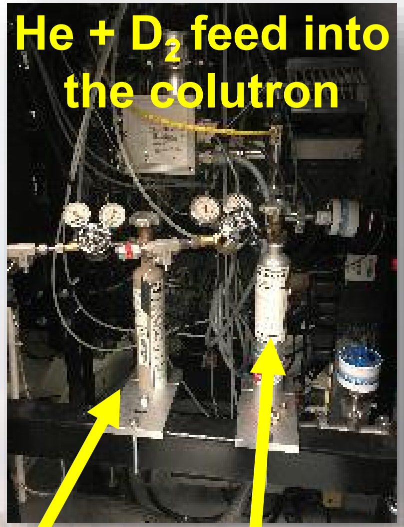
Capabilities

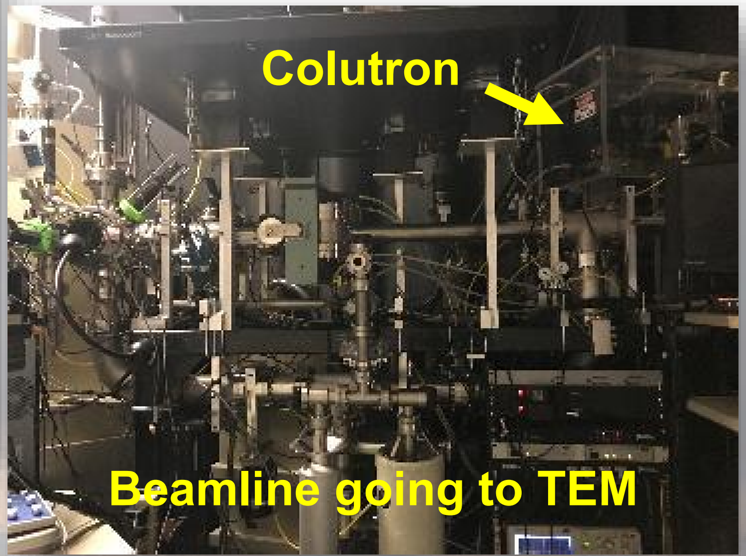
- 200 kV LaB_6 TEM
- Ion beams considered:
  - Range of Sputtered Ions
  - 10 keV D_2^{2+}
  - 10 keV He^+
- All beams hit same location

- Nanosecond time resolution (DTEM)
- Precession scanning (EBSD in TEM)
- *In situ* PL, CL, and IBIL

- *In situ* vapor phase stage
- *In situ* liquid mixing stage
- *In situ* heating
- Tomography stage (2x)
- *In situ* cooling stage
- *In situ* straining stage

We have produced 430 eV He with the Colutron –below knock-on energy!





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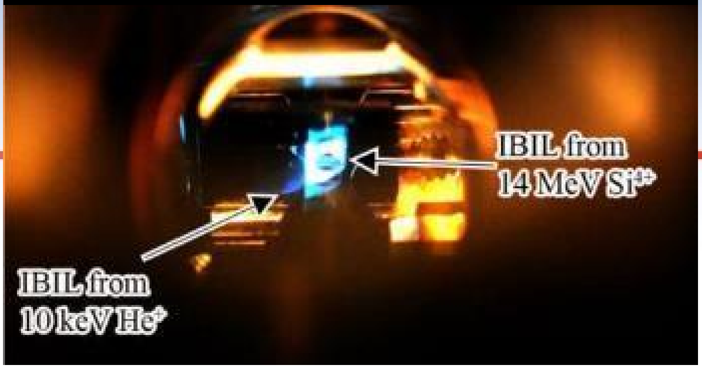
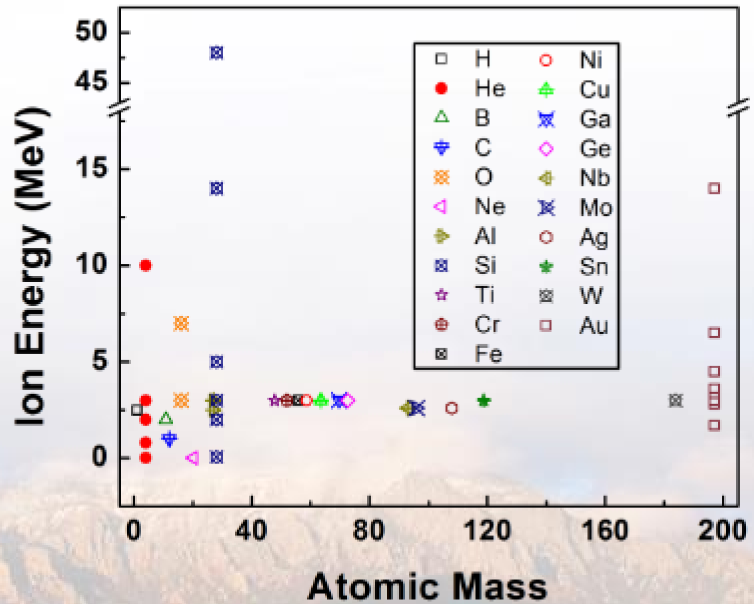
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

10 kV Colutron - 200 kV TEM - 6 MV Tandem

IBIL from a quartz stage inside the TEM

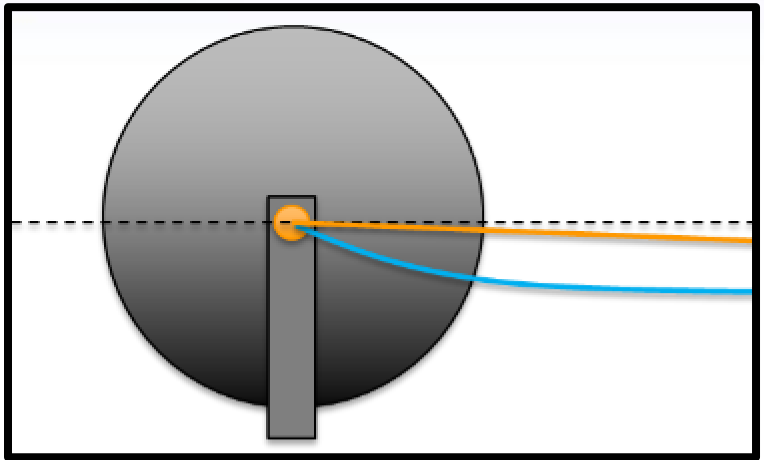


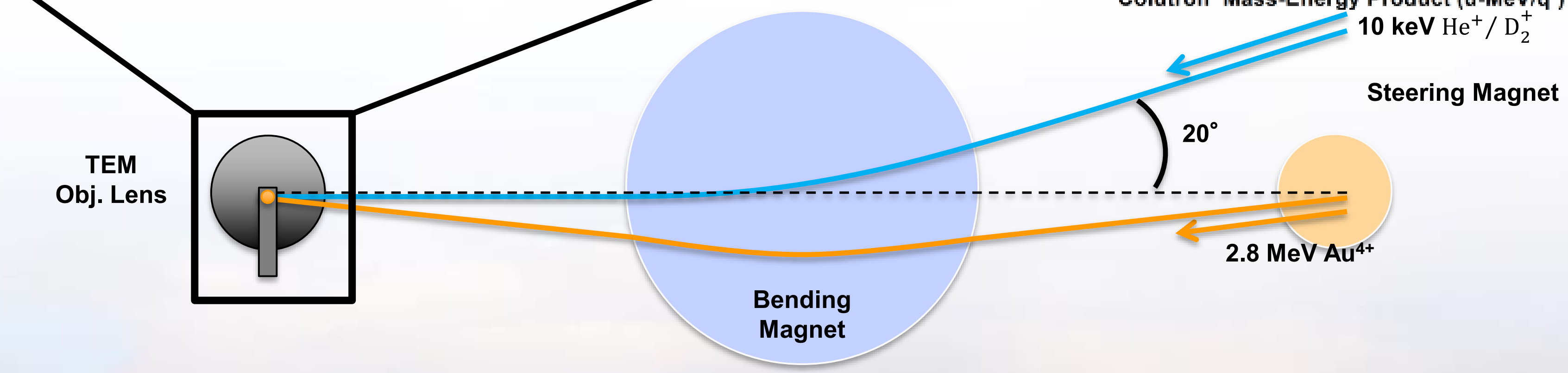
Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

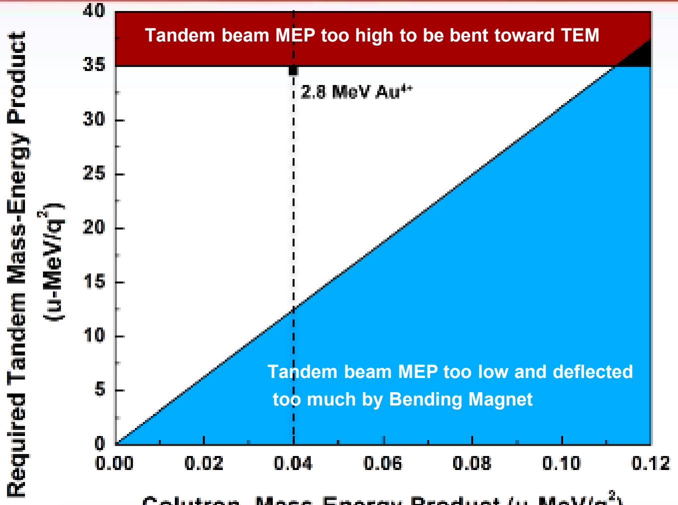


Modeling Beam Mixing and Deflection

Collaborators: M. Steckbeck, D.C. Bufford, & B.L. Doyle



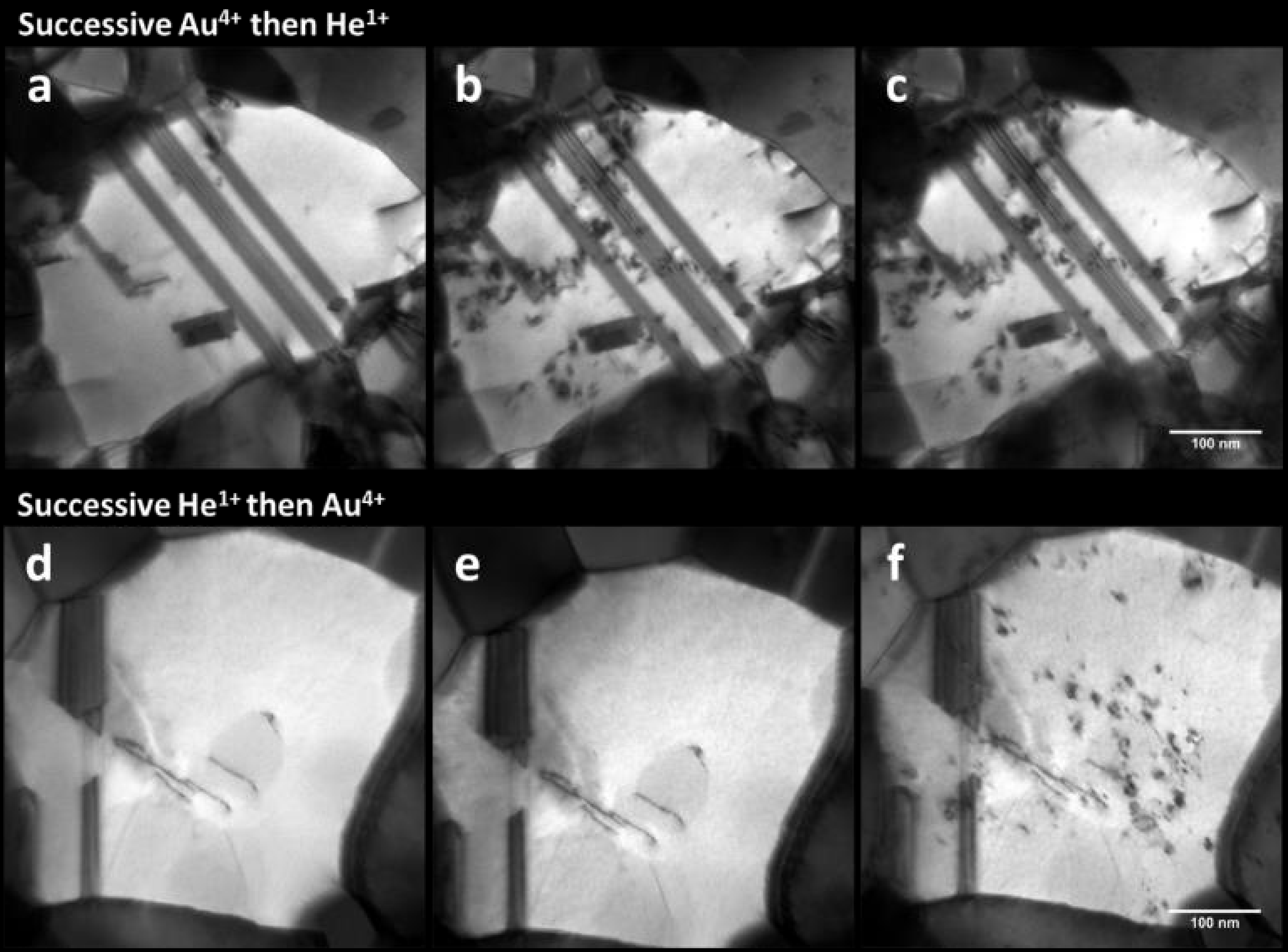




- Must compensate for deflection of Tandem beam by bending magnet
  Colutron beams deflected by the TEM objective lens
- Insignificant deflection of Tandem beams
- With 10 keV He/D_2 we can use Tandem beams ≳13 MeV/q^2
- Au, He, and D_2 ions all reach the sample concurrently

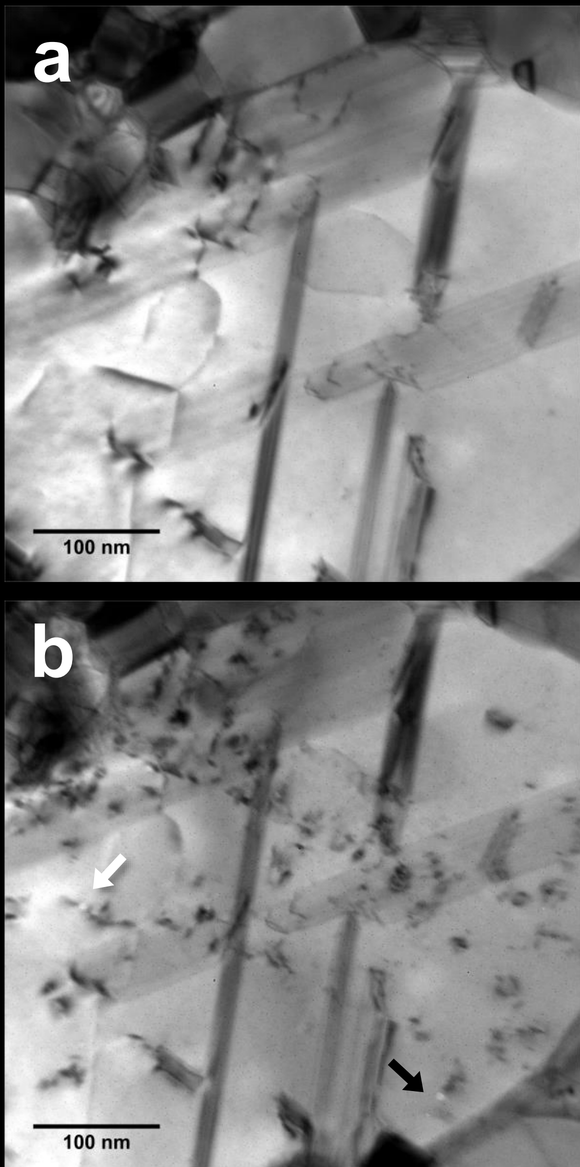
In situ Successive Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor

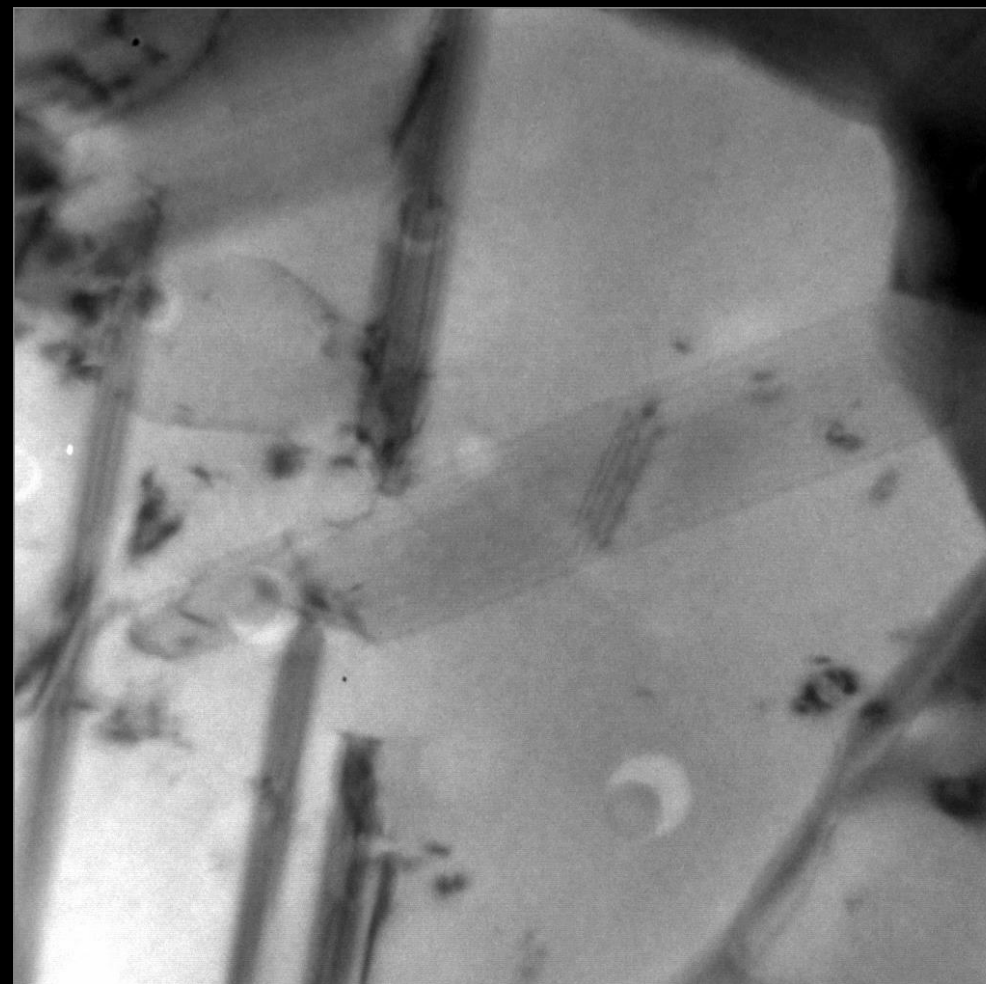


In situ Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor



He^{1+} implantation and Au^{4+} irradiation of a gold thin film



Single Ion Strikes During Concurrent Irradiation: Nucleation of Helium Cavities

Collaborators: C. Chisholm, P. Hosemann, & A. Minor

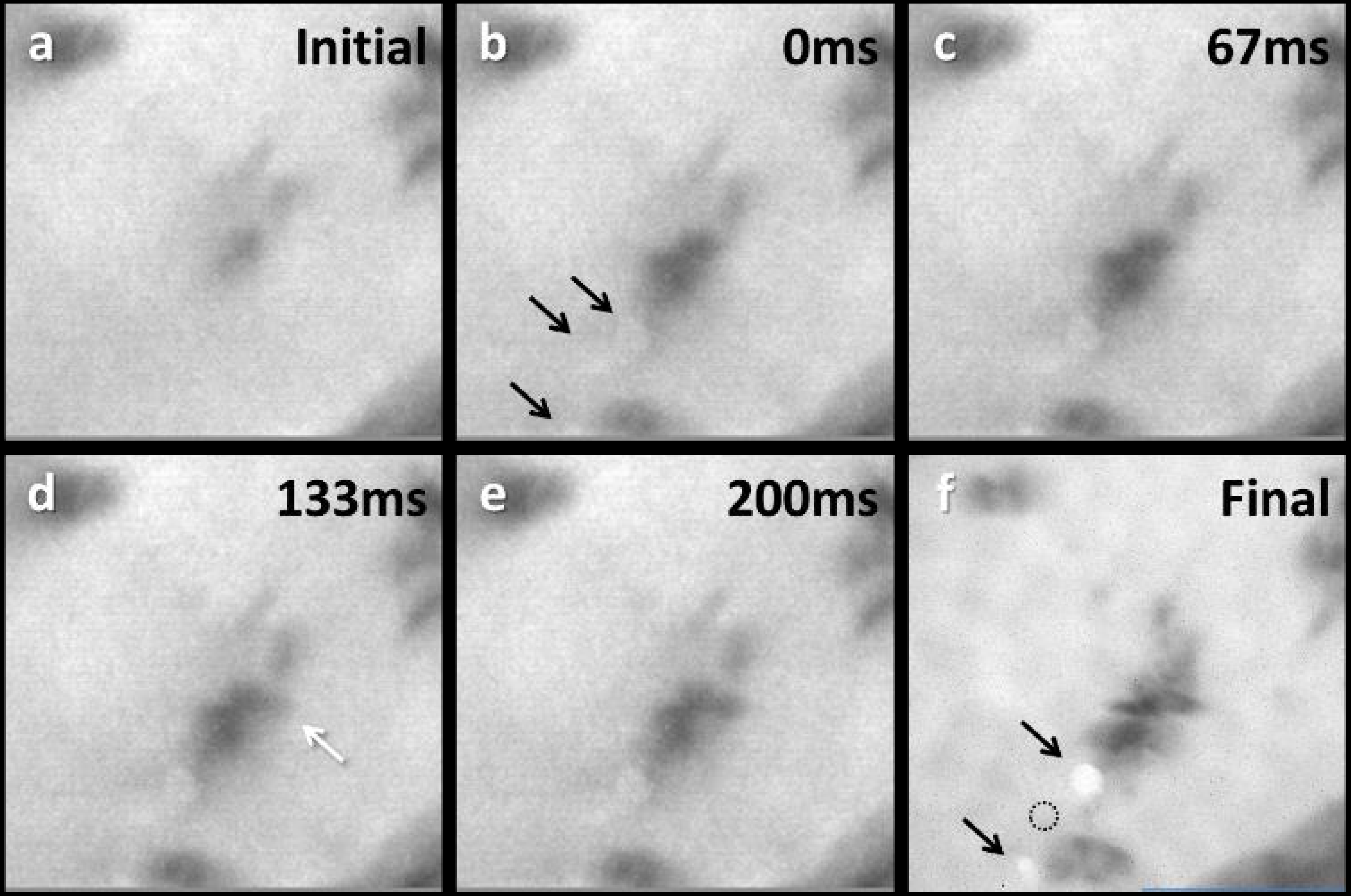
a) Initial microstructure

b) Cascade: Creation of dislocation loops, vacancy clusters, and three cavities

d) Cascade damage still evolving

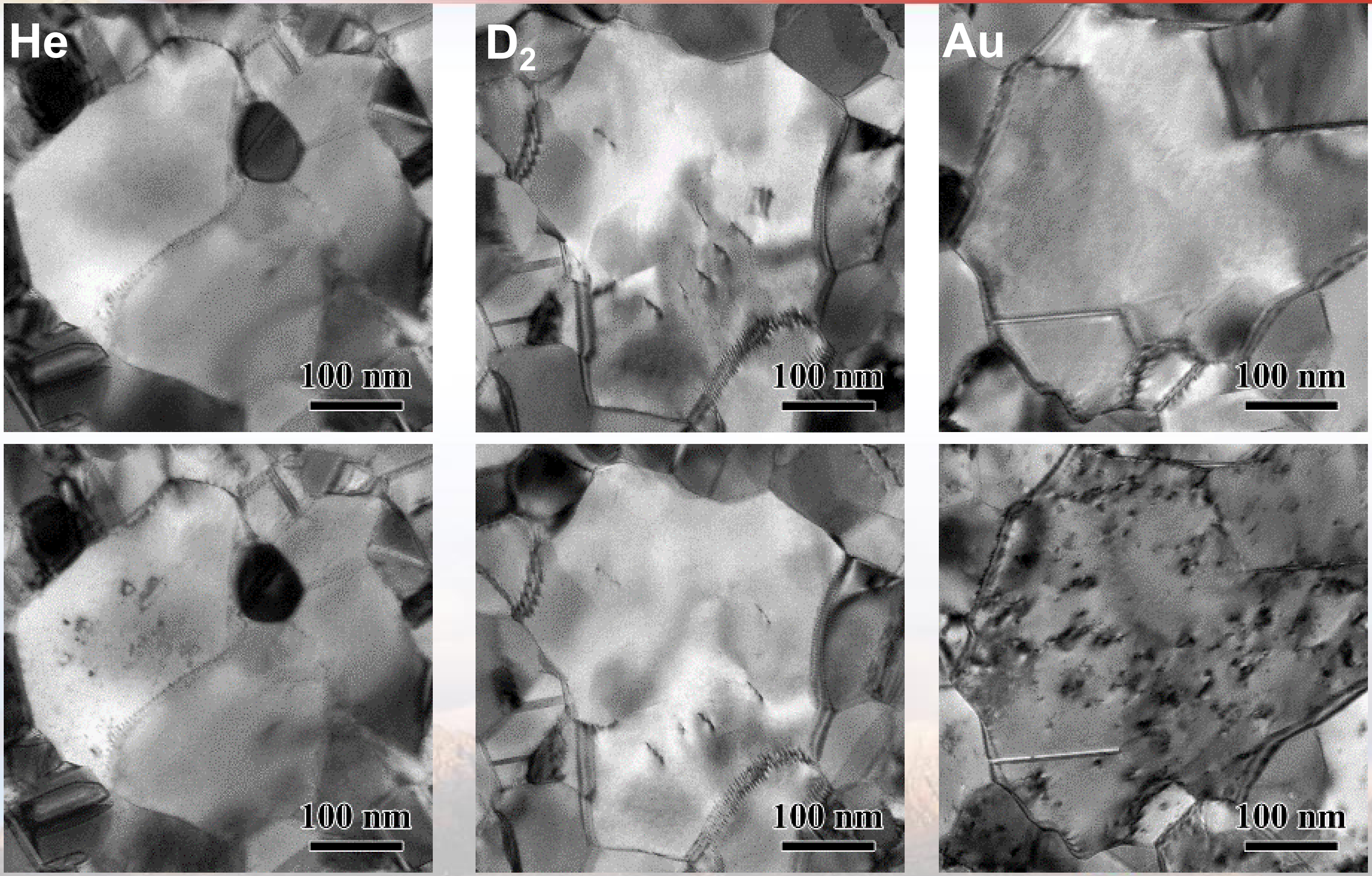
e) Apparent stability

f) Final microstructure: Only two remaining cavities



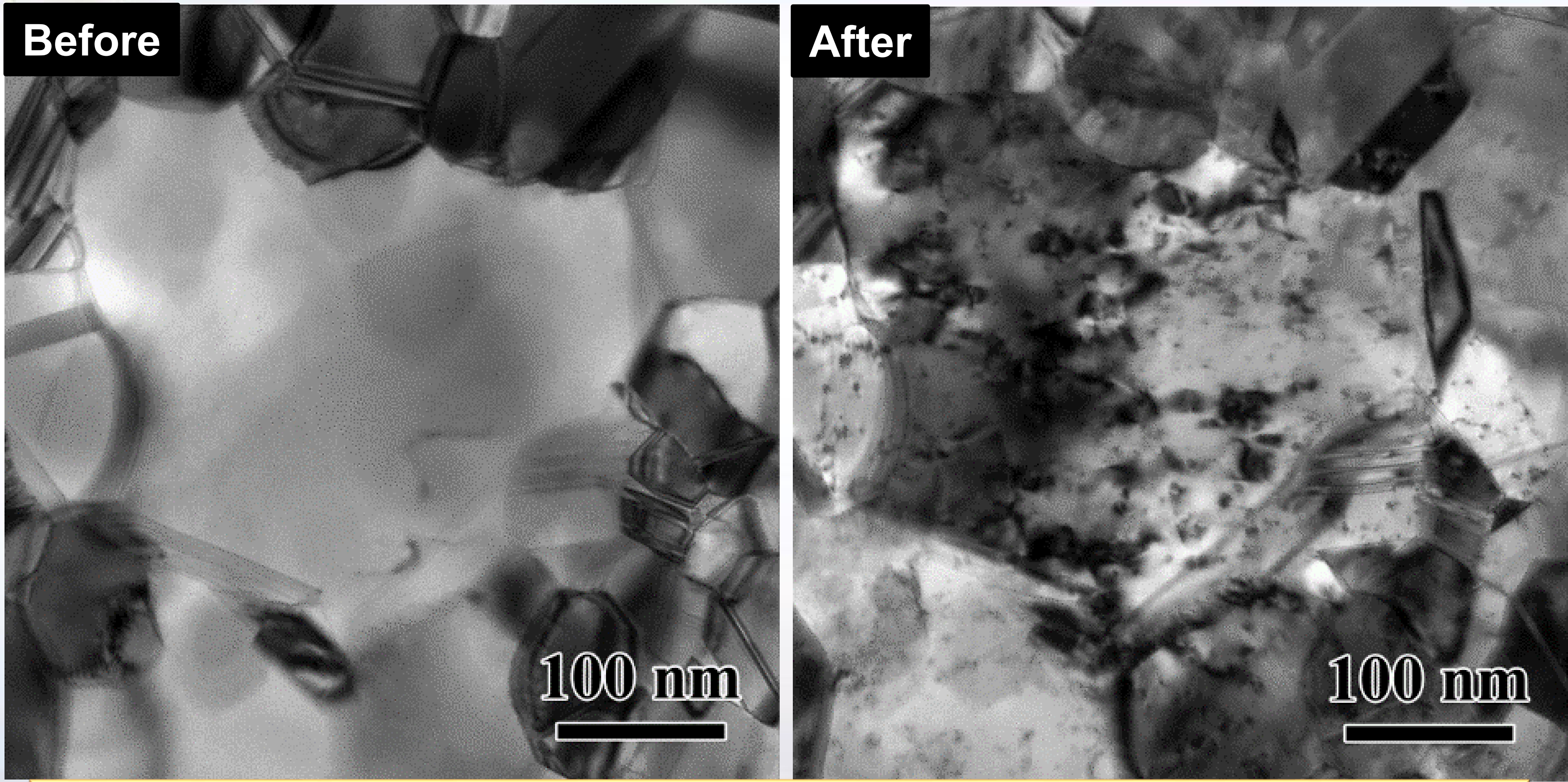
Aligned Individual Colutron and Tandem Beams

Collaborators: D.C. Bufford



Concurrent 10 keV He, 10 keV D_2, and 3 MeV Au

Collaborators: D.C. Bufford

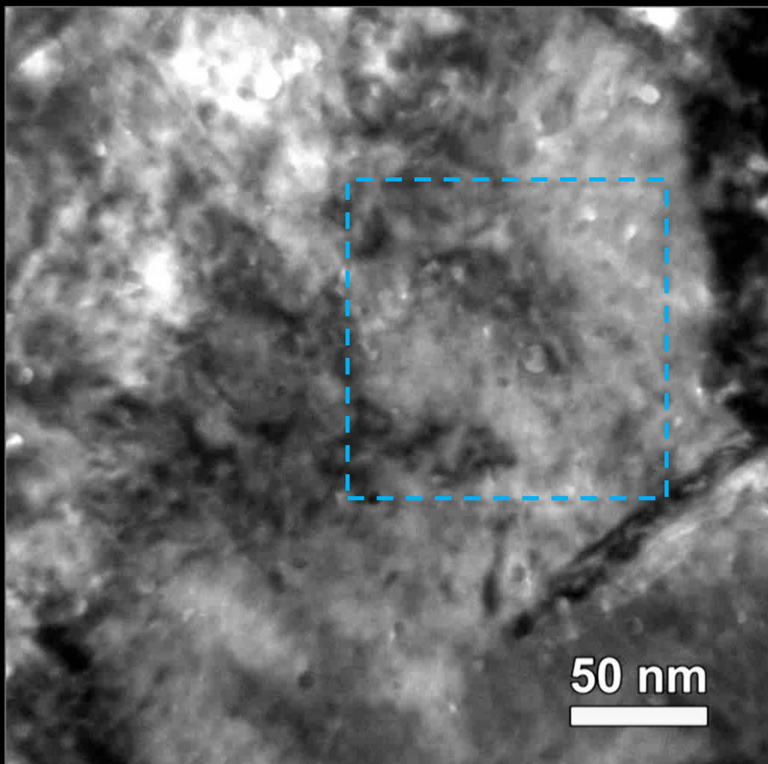


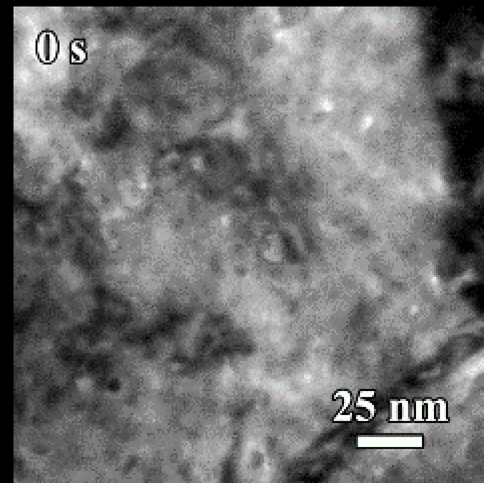
In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I[3]TEM! Intensive work is still needed to understand the defect structure evolution that has been observed.

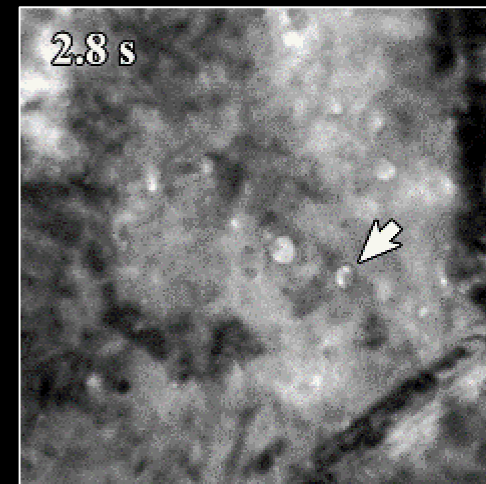
Simultaneous *In situ* TEM Triple Beam: 2.8 MeV Au^{4+} + 10 keV He^{+}/D_2^{+}

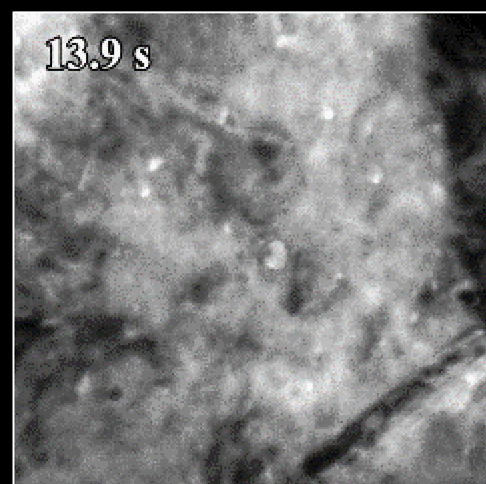
Collaborator: D.C. Bufford

Video playback speed x1.5.





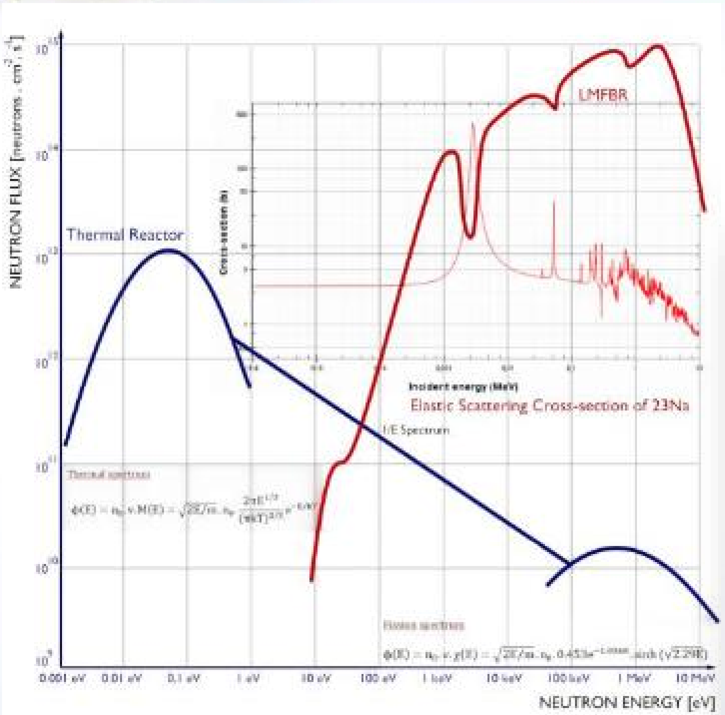


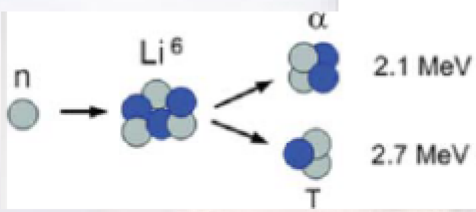


In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I^3TEM! Intensive work is still needed to understand the defect structure evolution that has been observed.

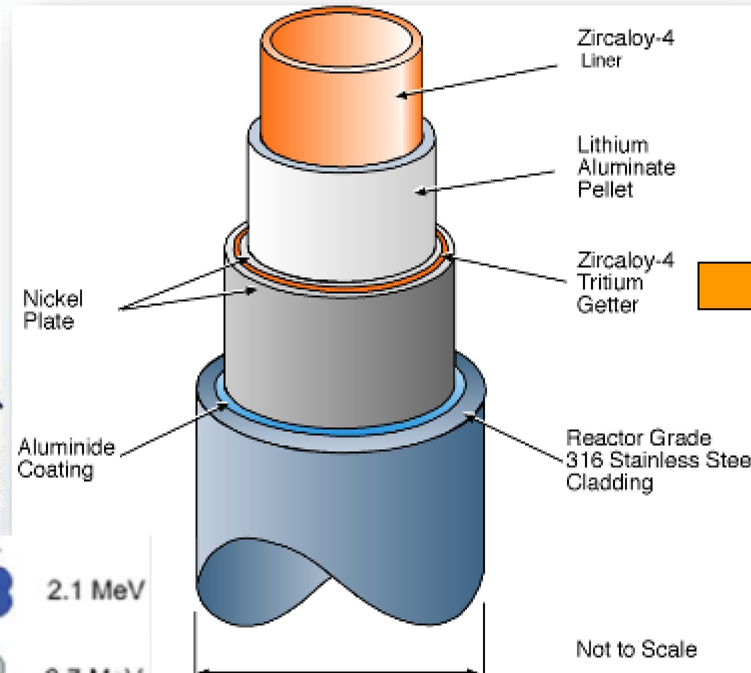
- **Approximate fluence:**
  - Au 1.2 $\times 10^{13}$ ions/cm^2
  - He 1.3 $\times 10^{15}$ ions/cm^2
  - D 2.2 $\times 10^{15}$ ions/cm^2
- **Cavity nucleation and disappearance**

Applying the Triple Beam Irradiation to Deconvolute Reactor Enviroments





Tritium Producing Burnable Absorber Rod (TPBAR)



- Displacement Damage
- Helium Implantation
- Tritium Implantation
- Elevated Temperatures

Simulating neutron irradiation in a reactor is complicated, and TPBAR adds the additional complication of ^{3}H production

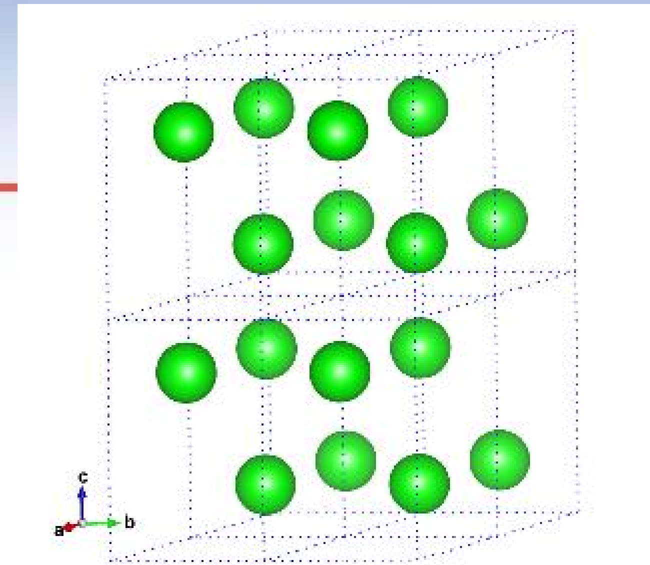
Zircaloy Background

<u>What is Zircaloy?</u>

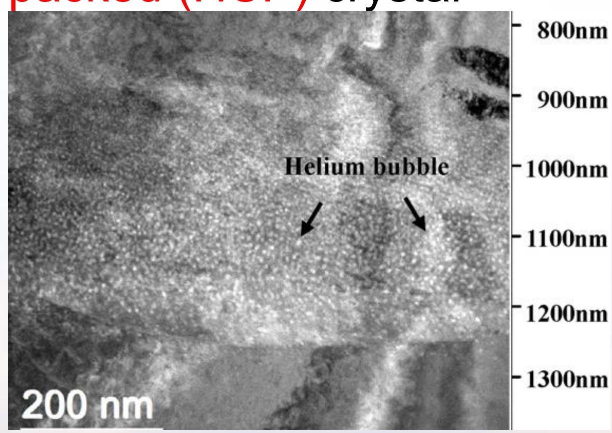
- Zircaloy-2: predominantly used as fuel cladding for BWRs
  - α-Zr, 1.5% Sn, 0.15% Fe, 0.1% Cr, 0.05% Ni
- Zircaloy-4: Removed the Ni and increased Fe content for less H uptake in certain reactor conditions
  - α-Zr, 1.5% Sn, 0.2% Fe, 0.1% Cr
- Zr-Nb alloys (e.g. Zirlo) are also common
- α-Zr has a hexagonal close-packed (HCP) crystal structure up to 810ºC

<u>Gas and defect behavior in Zr/Zr alloys</u>

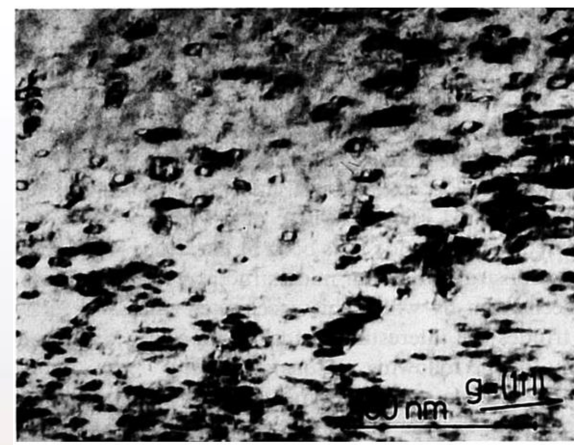
- ^{3}H, H, and He diffusion and release
- Bubble formation
- Irradiation induced metallic precipitate formation



Crystal Structure of α-Zr (HCP)

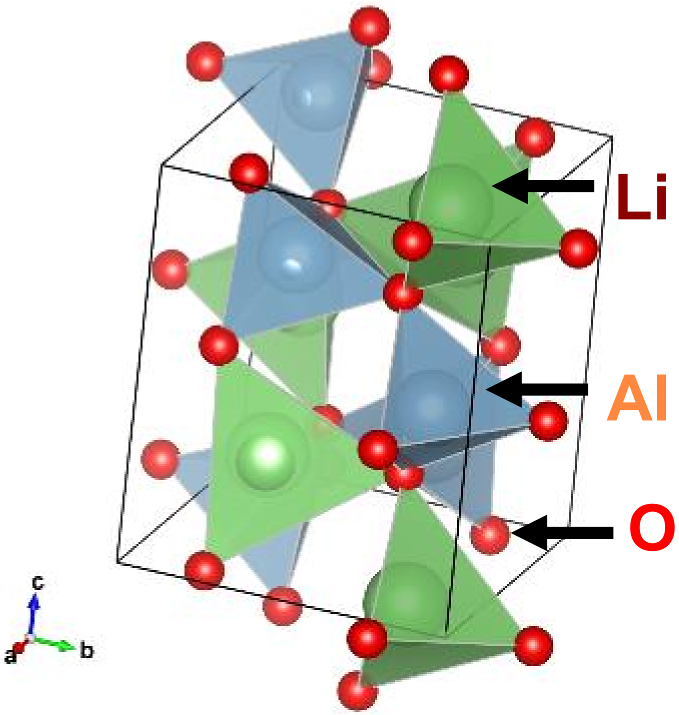


He bubbles in Zr-Nb alloy
Shen et al Mat Char 107 (2015) 309-316



TEM of Zr tritide after 325d
Schober et al JNM 141-143 (1986) 453-457

$LiAlO_2$ Background



γ–$LiAlO_2$ is tetragonal (space group: P 41 21 2)

$LiAlO_2$ transforms to /precipitates out $LiAl_5O_8$ (cubic spinel) under electron irradiation and some ion irradiation conditions.

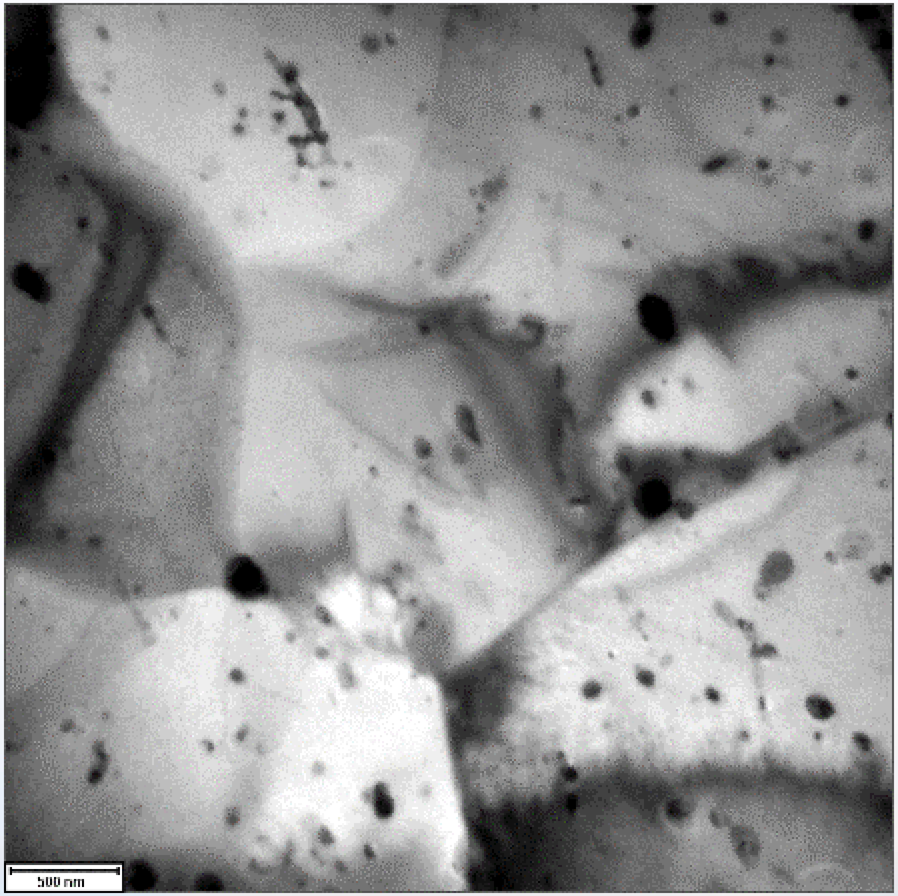
<u>How is $LiAlO_2$ used in the TPBAR?</u>

- $^6Li(n,\alpha)^3H$, emitting **^3H** (~2.75 MeV) and **^4He** (~2.05 MeV)
- ^3H β-decays to **^3He**
- Experiences displacive damage and gas accumulation at high temperature in reactor
- In addition to TPBAR, $LiAlO_2$ has been considered as a candidate for ^3H production in fusion reactors

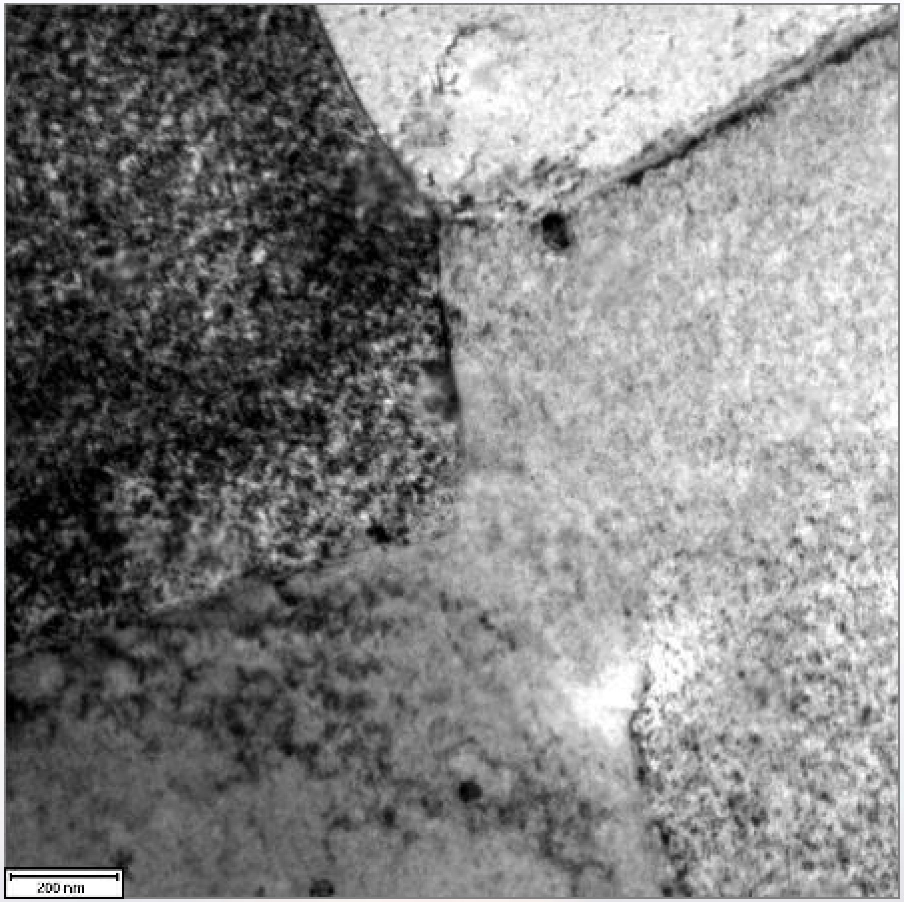
<u>Previous Work</u>

- Structural defects
  - Luo *et al* JNM 372 (2008) 53-58
- Volume swelling
  - Noda JNM 179-181 (1991) 37-41
- ^3H detrapping
  - Oyaidzu *et al* JNM 375 (2008) 1-7
- Gas diffusion and release
  - Raffray *et al* JNM 210 (1994) 143-160

3 MeV Self Ion Irradiation at 310 C

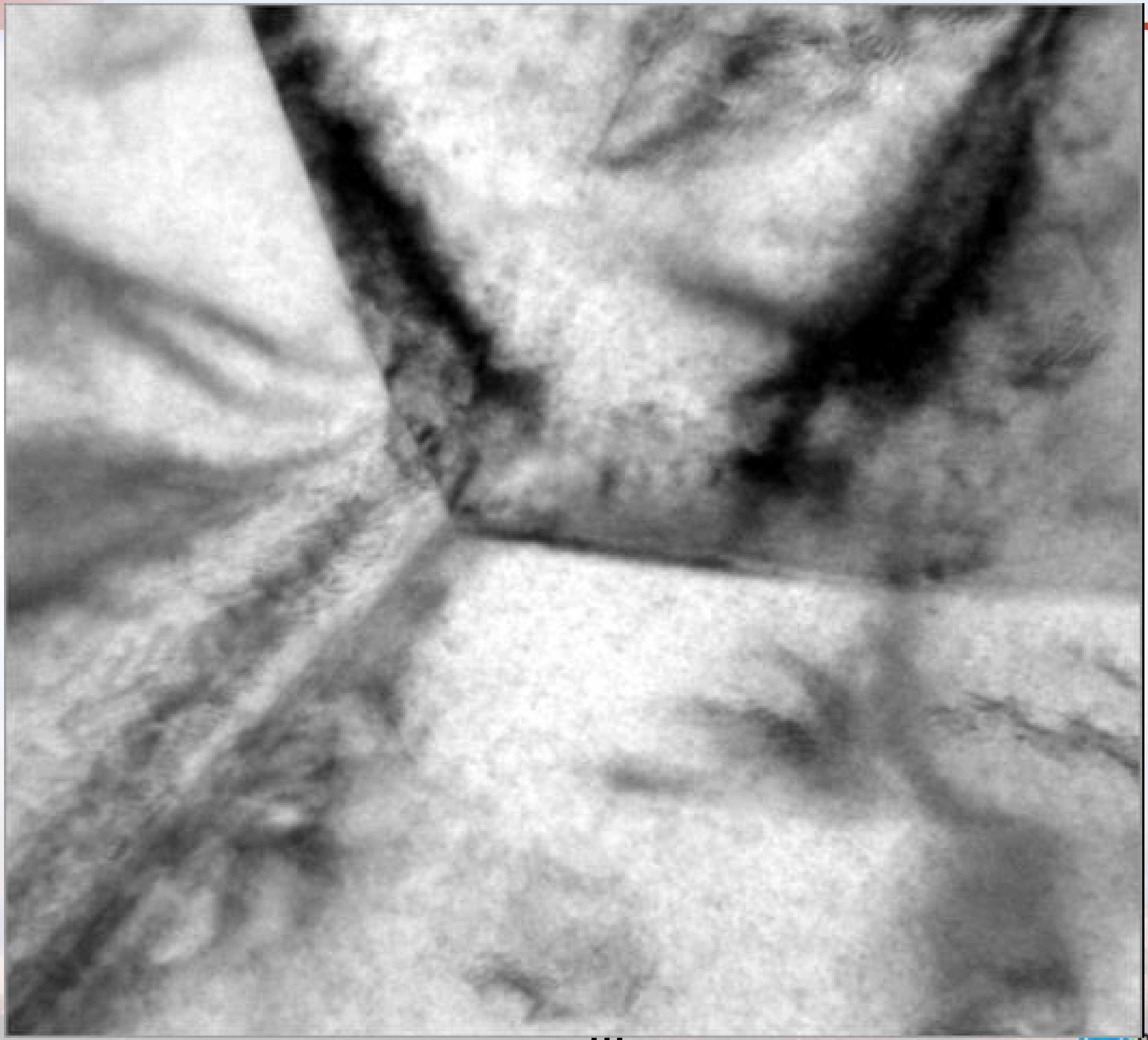


Before Irradiation

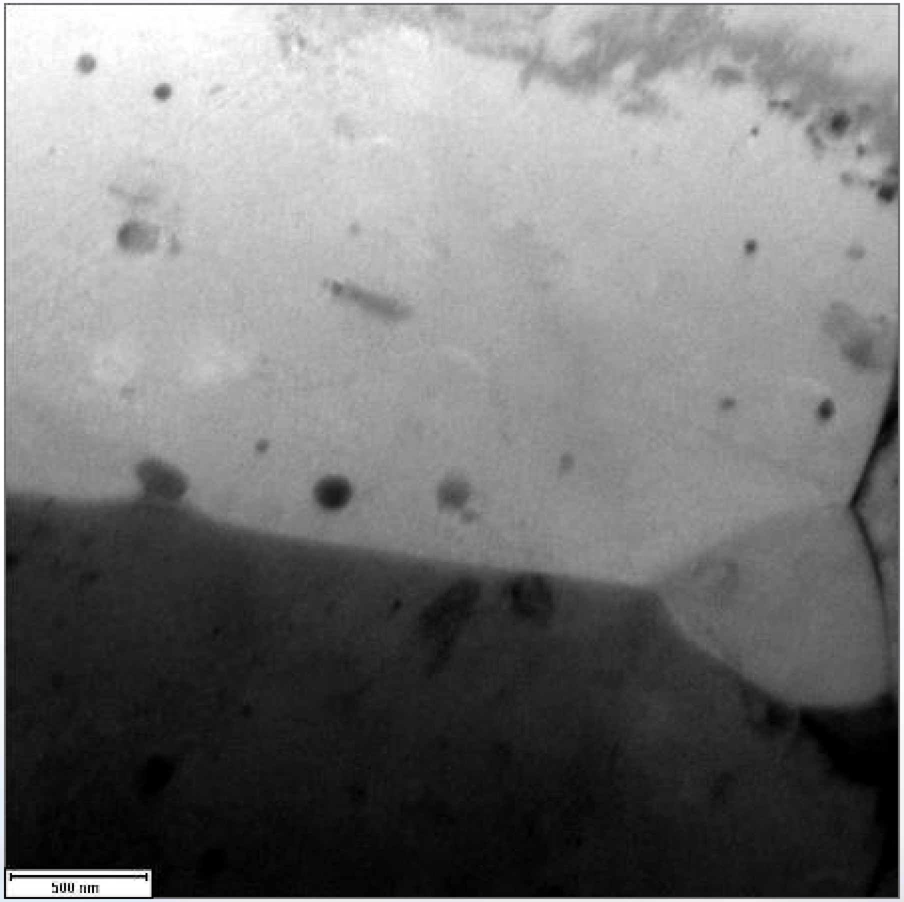


After Irradiation ≈ 7 DPA

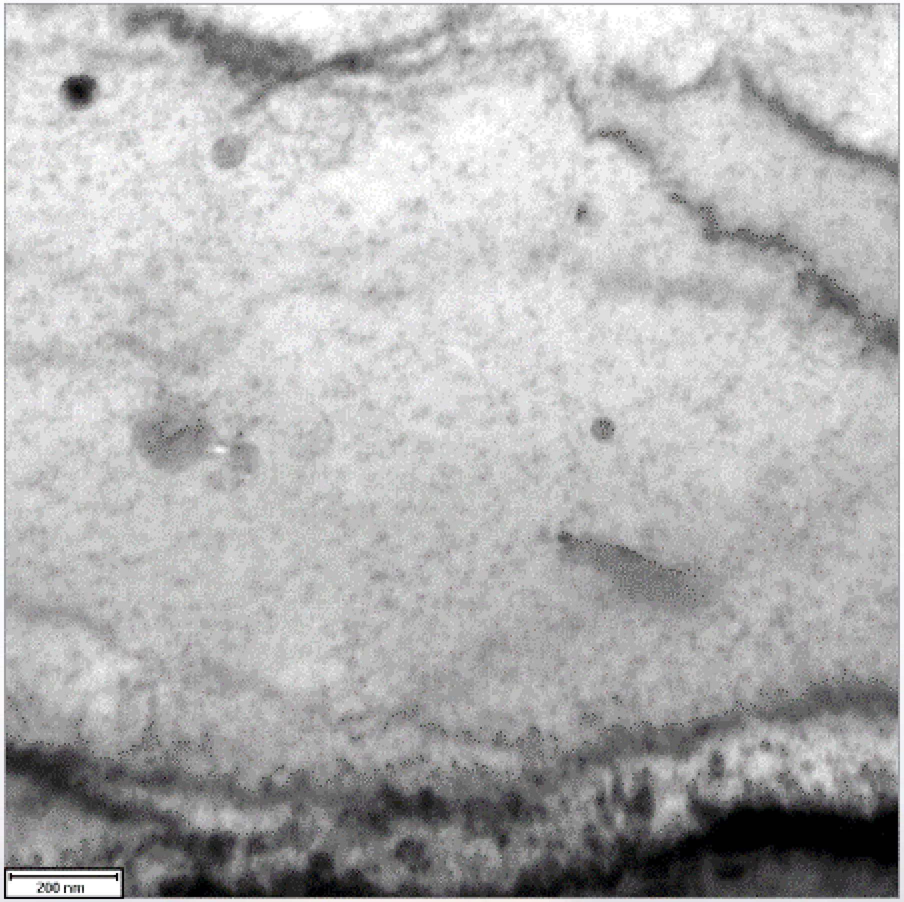
3 MeV Self Ion Irradiation at 310 C



10 keV He^{+} Implantation at 310 C



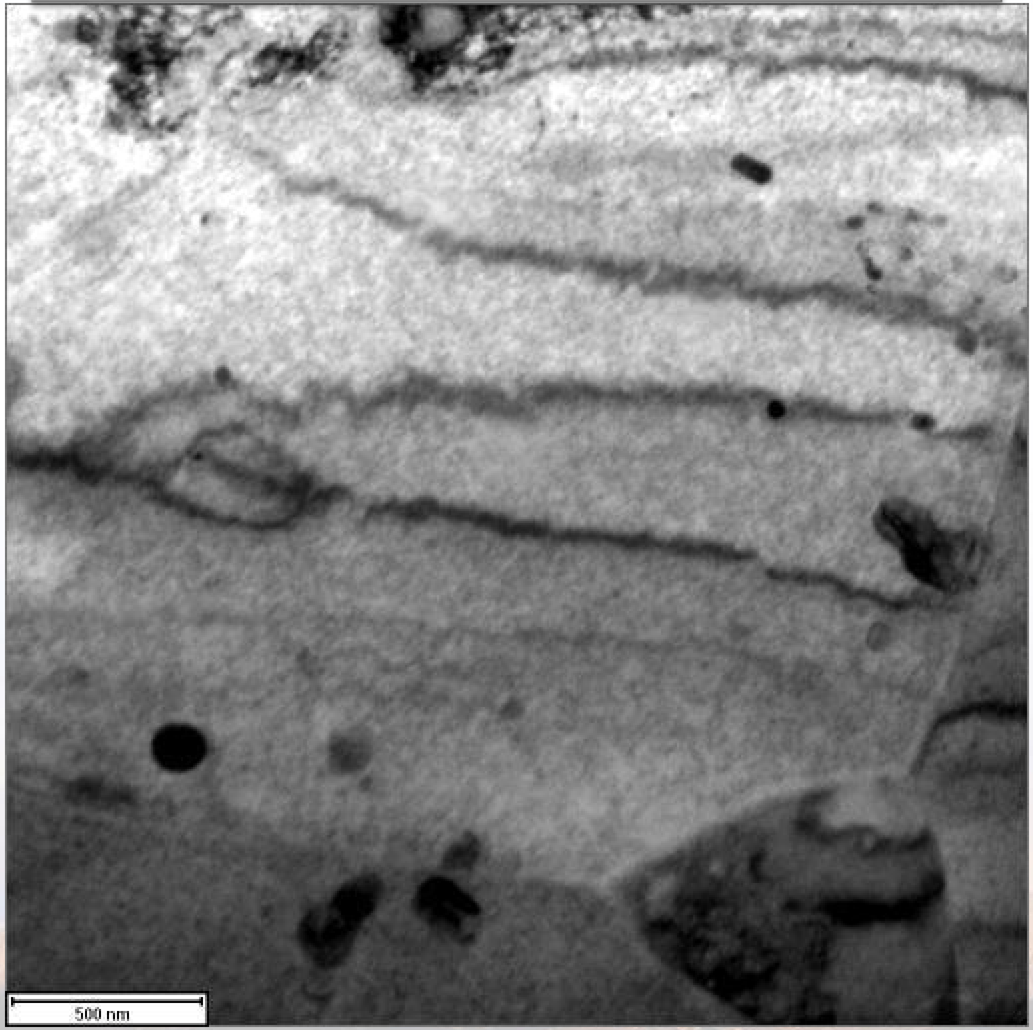
Before Implantation



After Implantation Damage, No Cavities

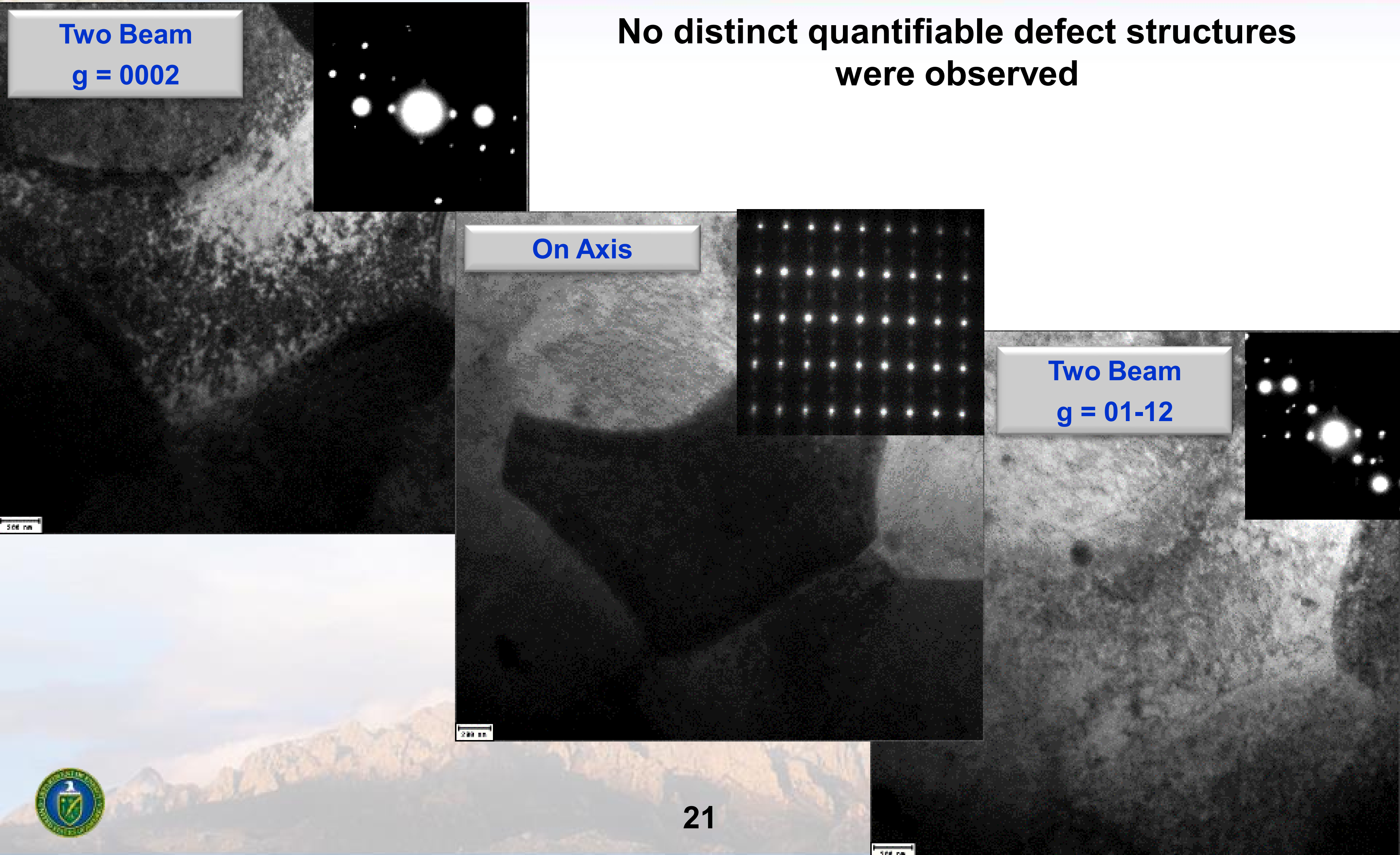
3 MeV Self Ion Irradiation after He^+ Implantation

High Density of Defects but No Cavities

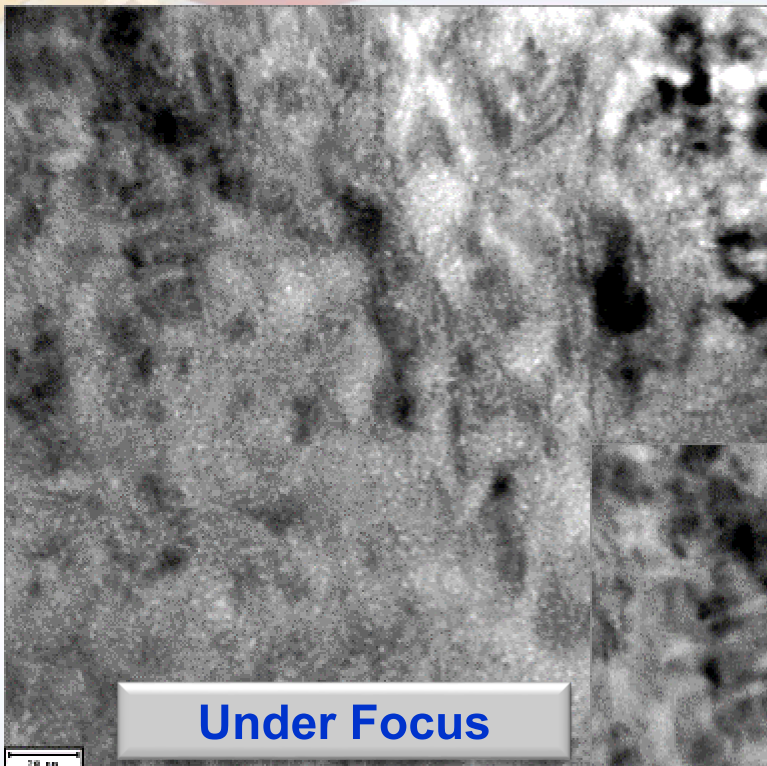


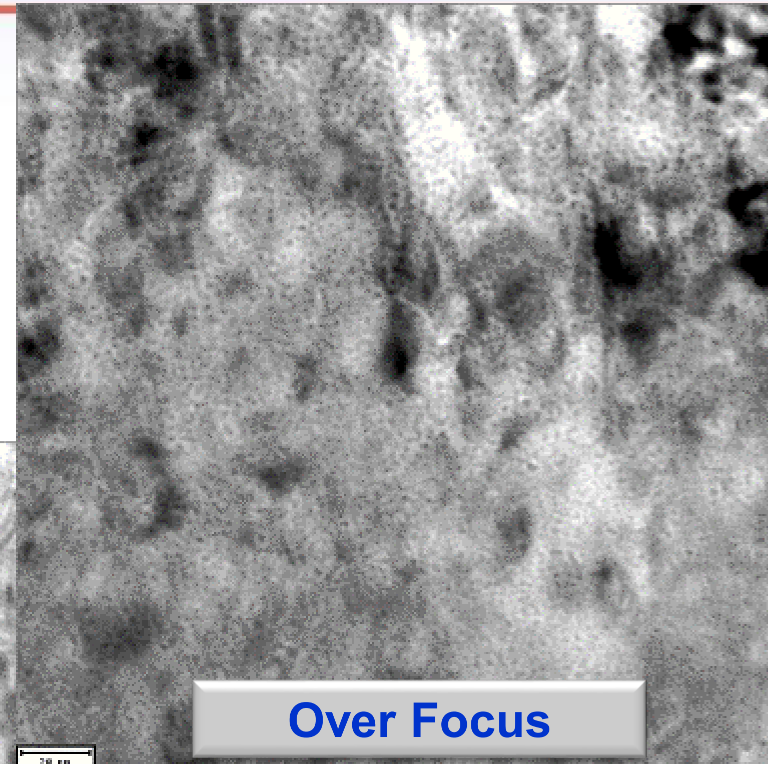
3 MeV Self Ion Irradiation after He^+ Implantation

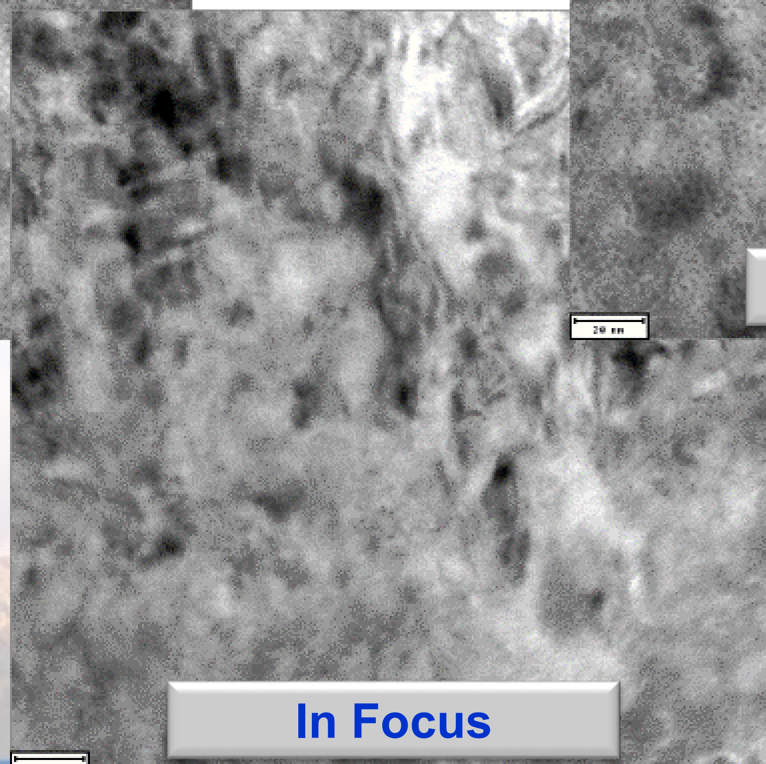
No distinct quantifiable defect structures were observed



Through Focus Imaging of Cavities: 30 Days Later

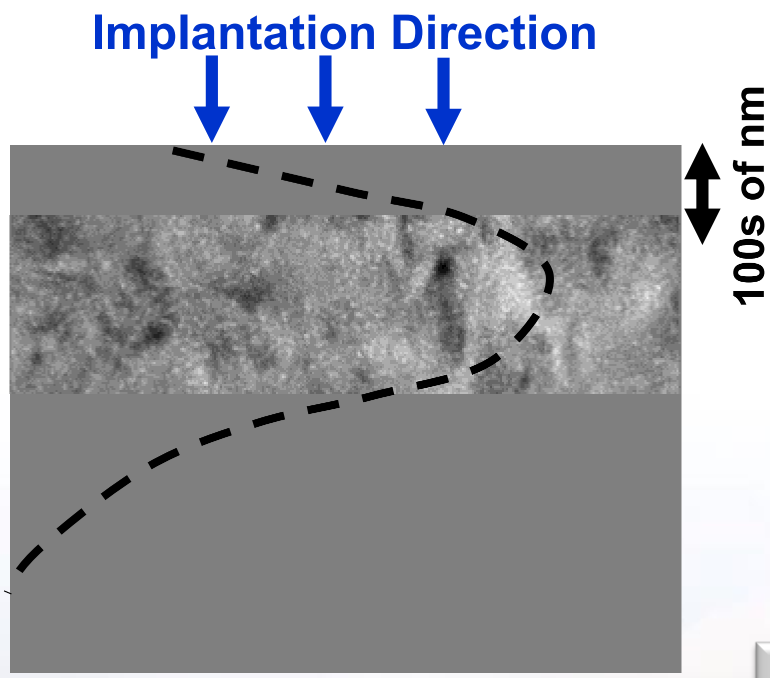




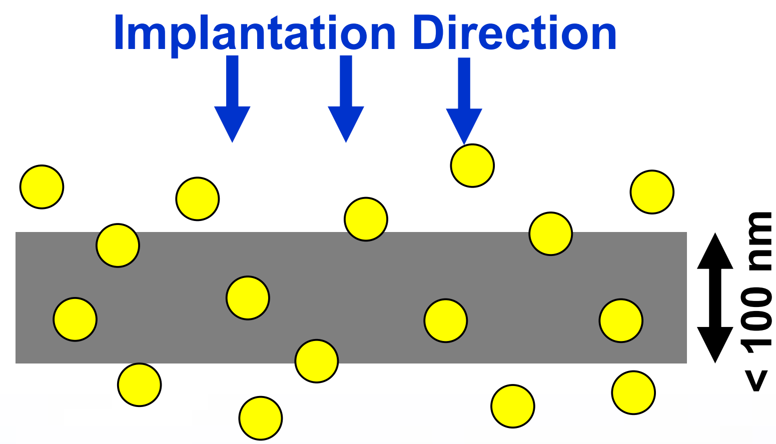


Why is finding bubbles after 30 days interesting?

- In-situ ion irradiation produces a different set of issues due to surface effects



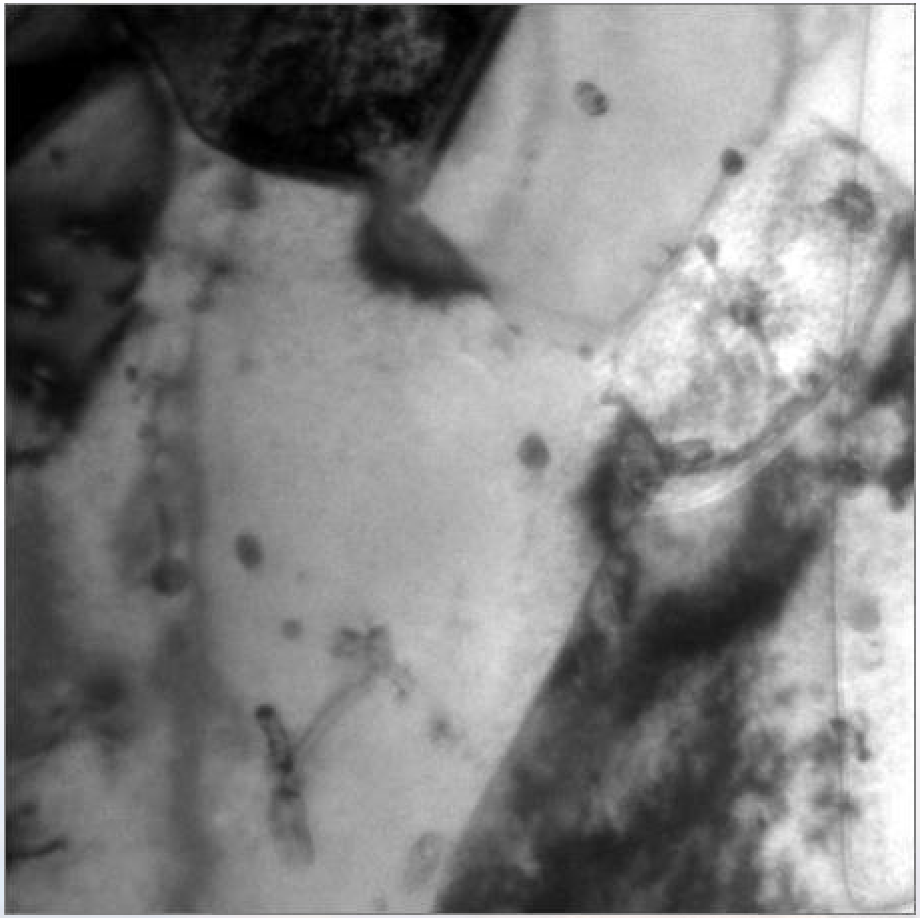
Bulk Irradiation: He/D diffuses at the same rate, but becomes trapped by defects before reaching surface.



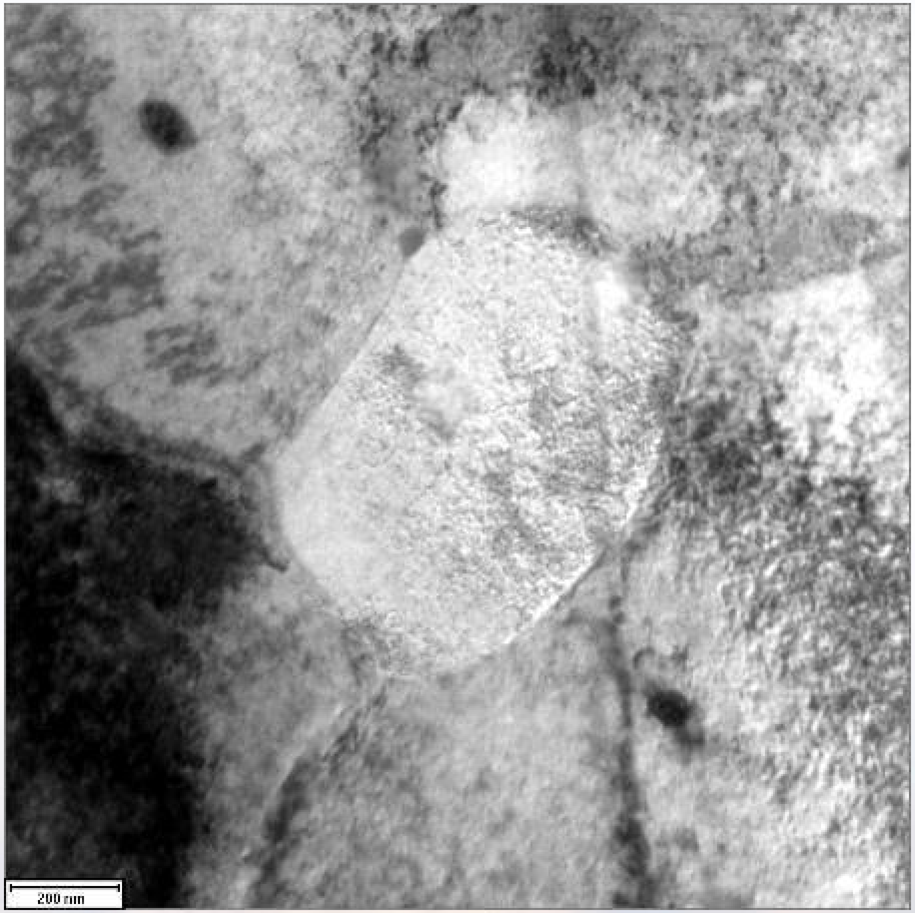
Thin-Film Irradiation: Much of the He/D diffuses to the surface before being trapped by a defect

Bubbles form in bulk at a much lower fluence that in thin-films. If there was not enough He/D to form bubbles in-situ, why did they form after 30 d? Some other mechanism is occurring.

Concurrent He^{+} Implantation and Self Ion Irradiation



Before Implantation/Irradiation

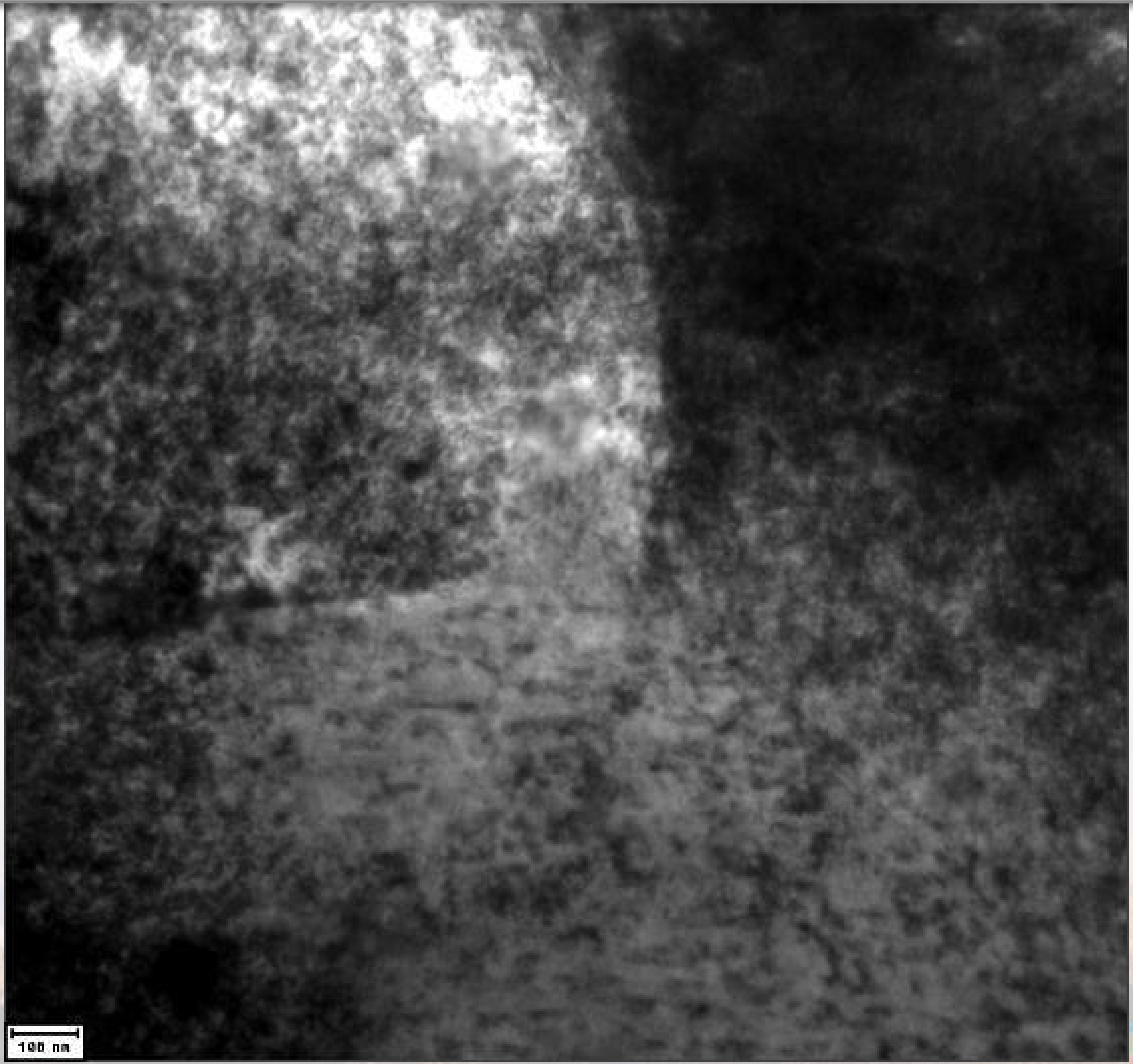


After Implantation/Irradiation

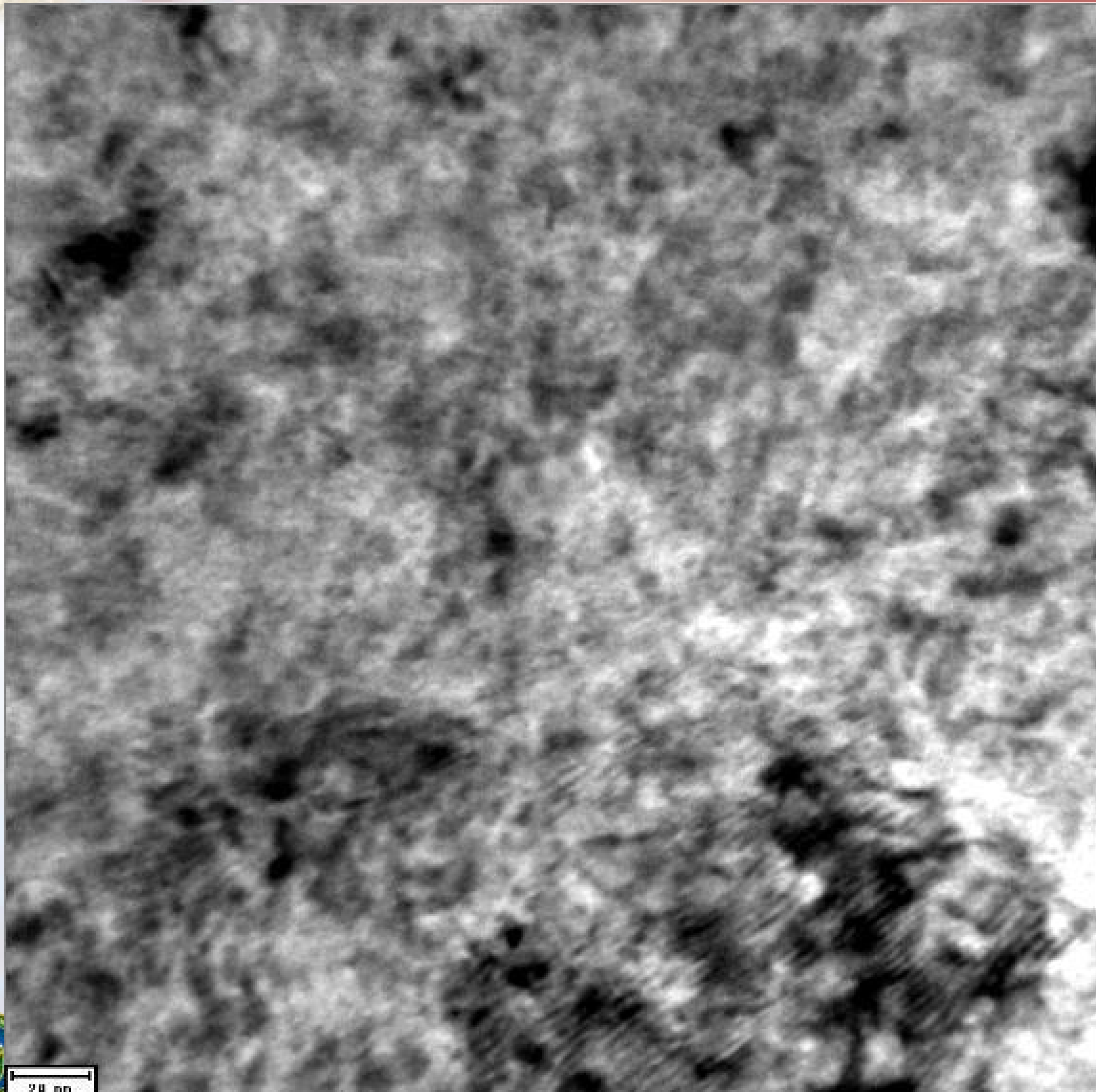
Damage, No Cavities

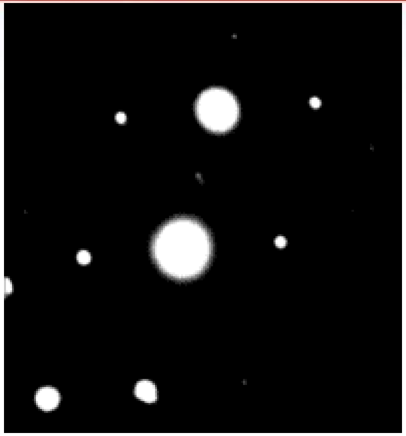
Concurrent D & He Implantation & Zr Irradiation

After Implantation/Irradiation Damage, No Cavities



Concurrent D & He Implantation & Zr Irradiation

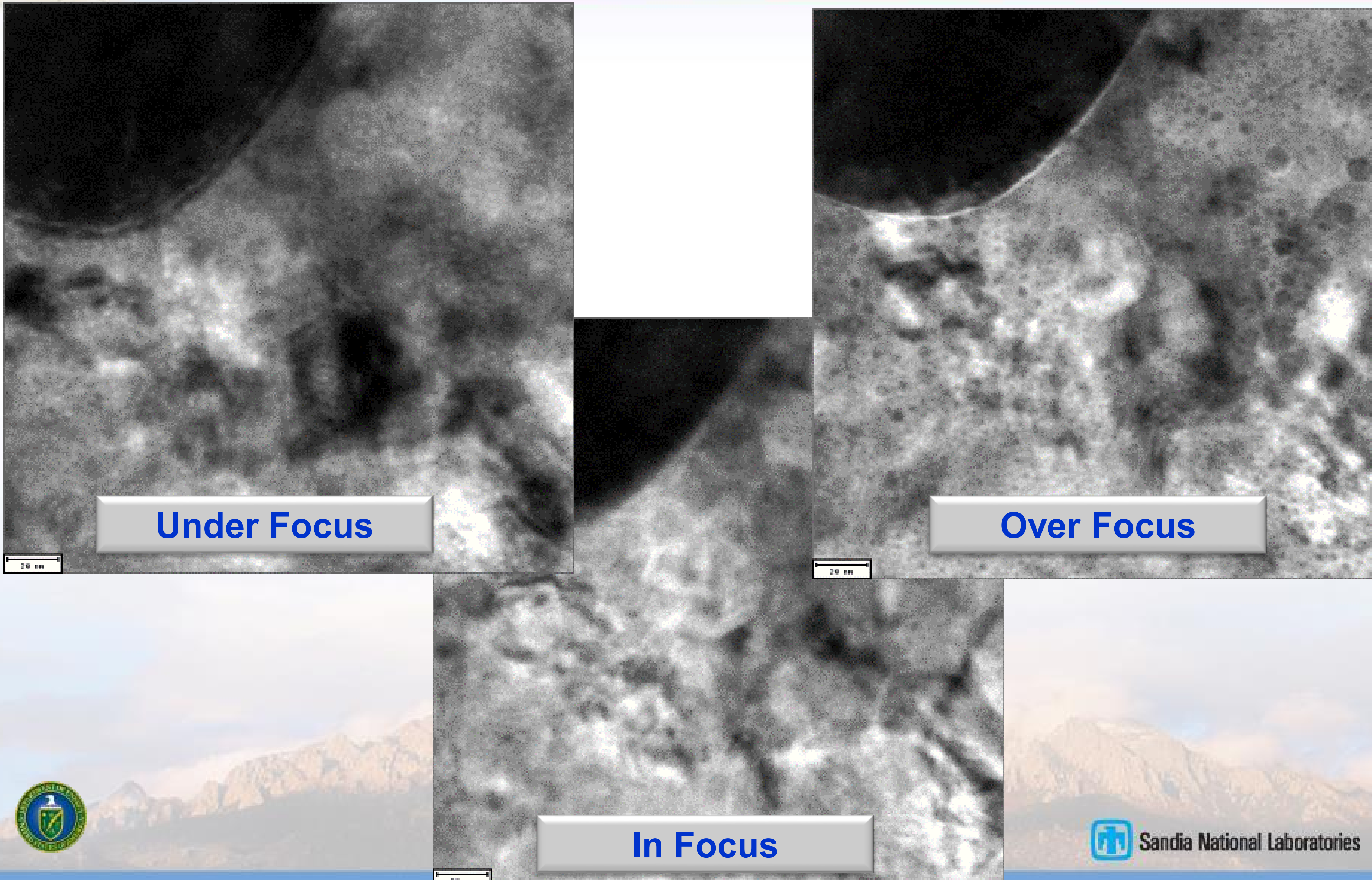




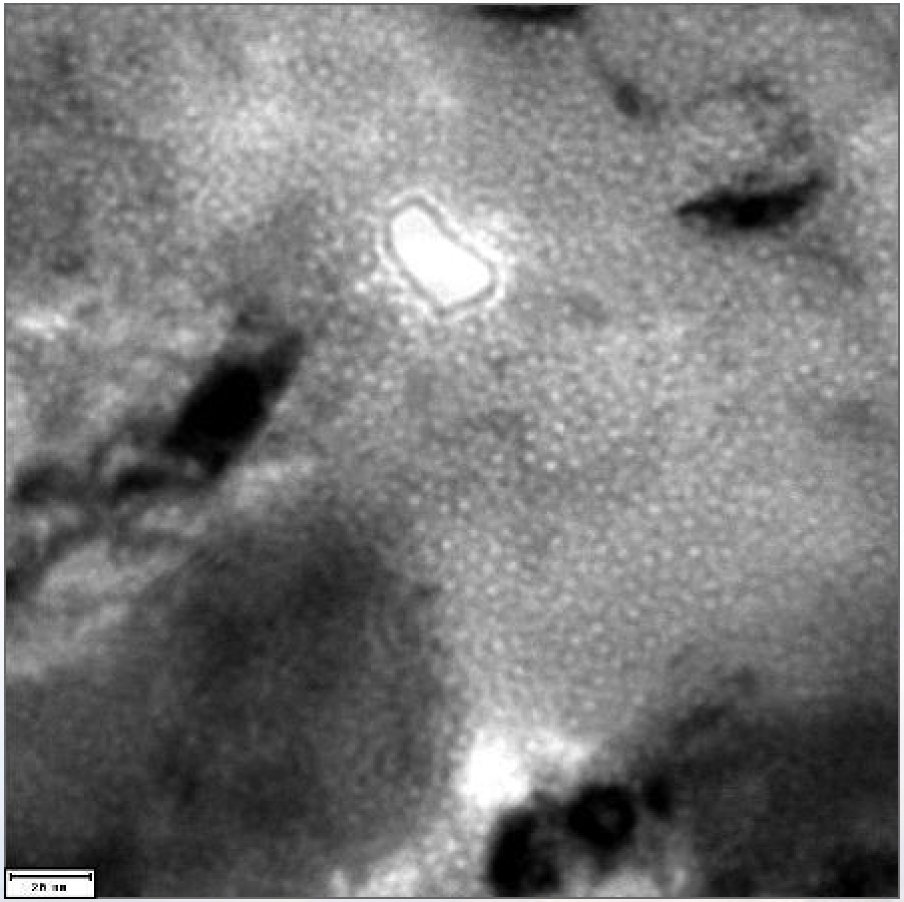
Two Beam

$g = 1\bar{1}01$

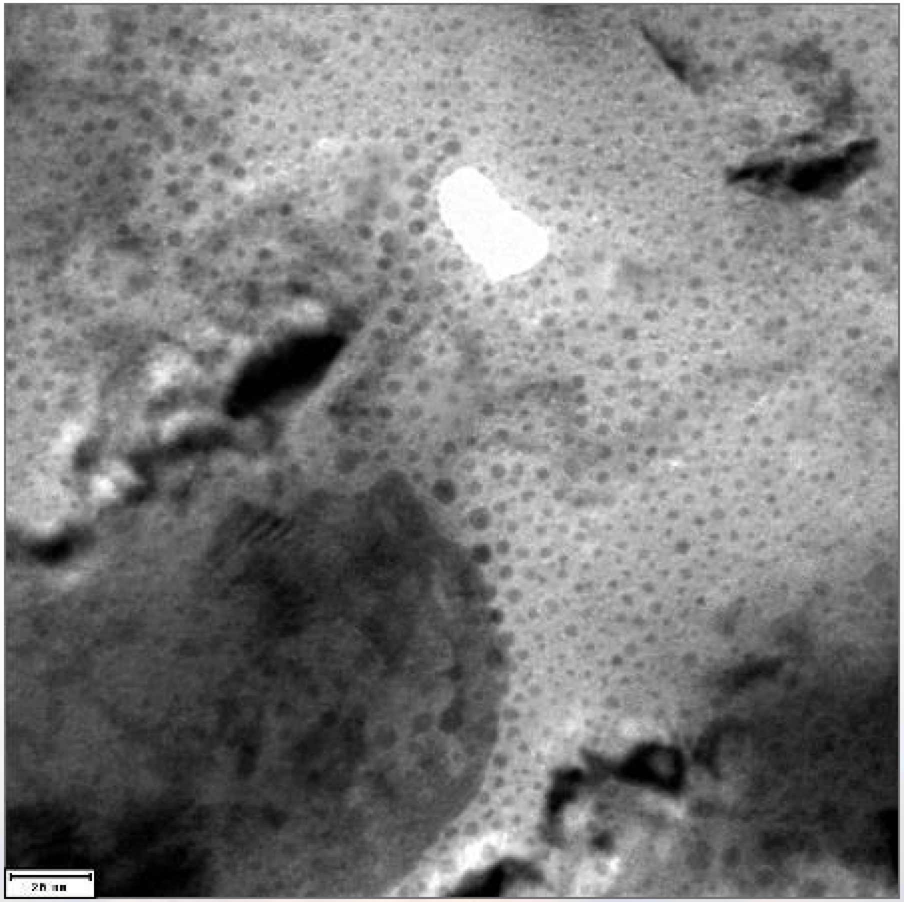
Through Focus Images: 30 Days Later



Through Focus Images: 30 Days Later



Under Focus



Over Focus

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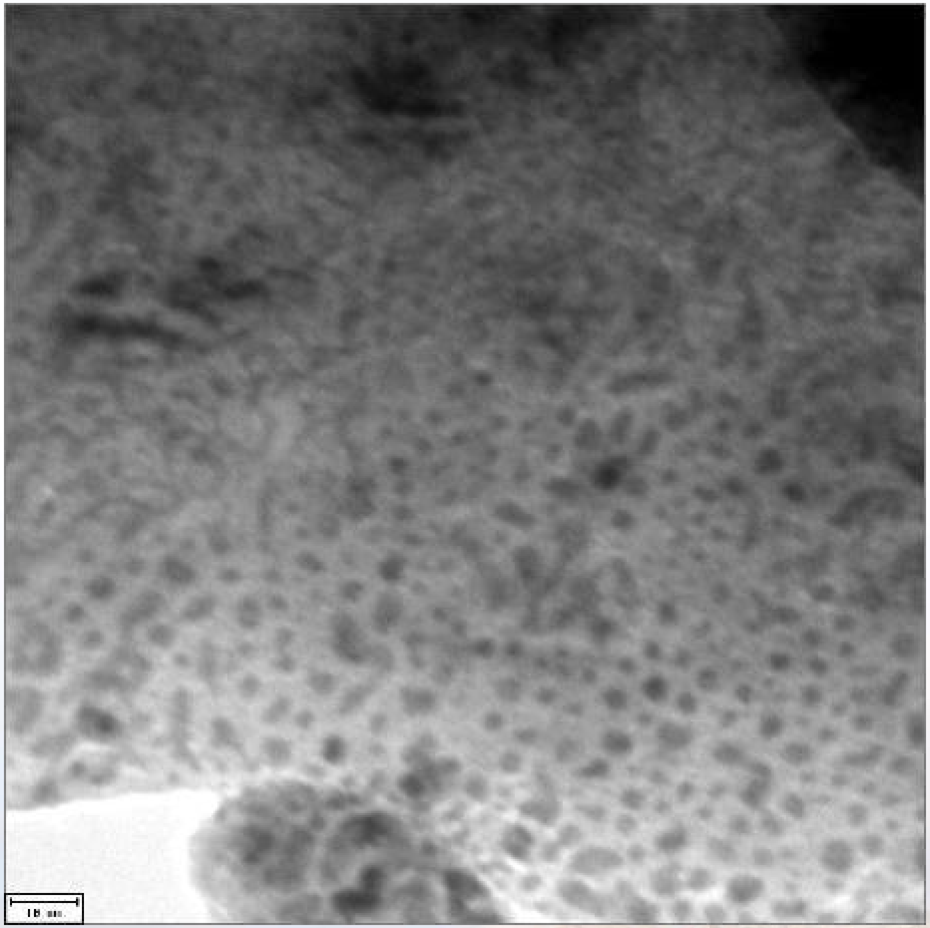
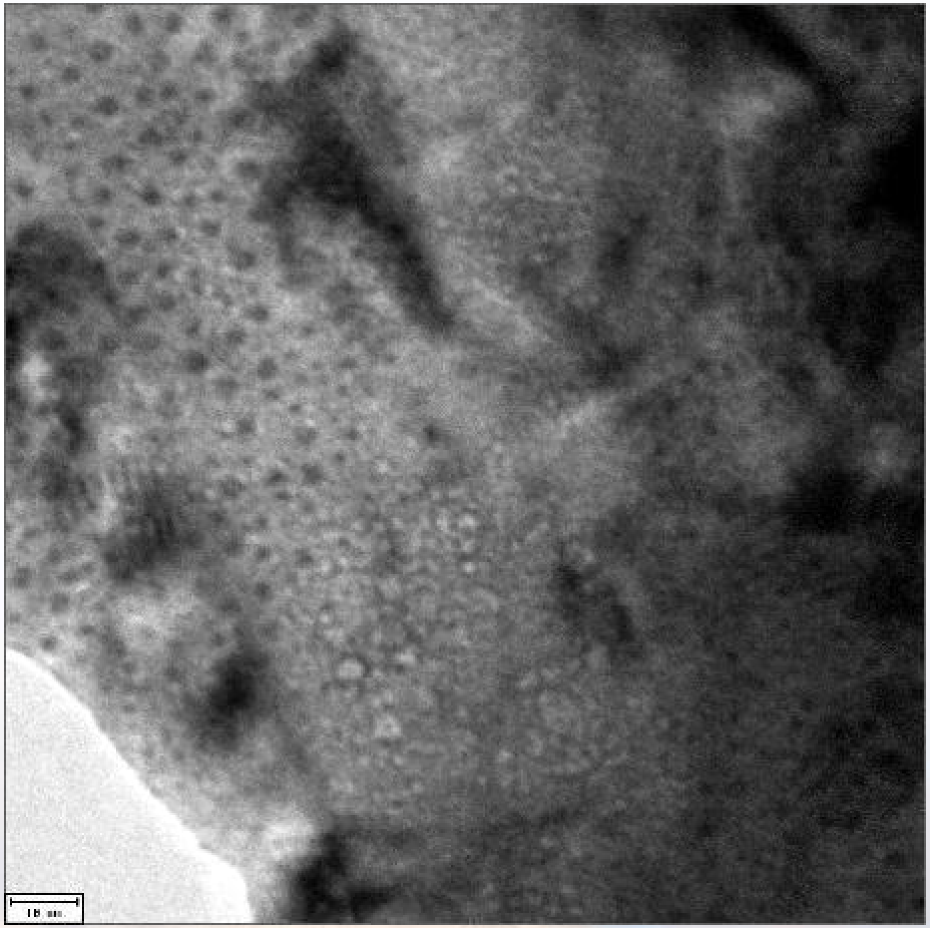
Surface Effects?



Under Focus



Under Focus

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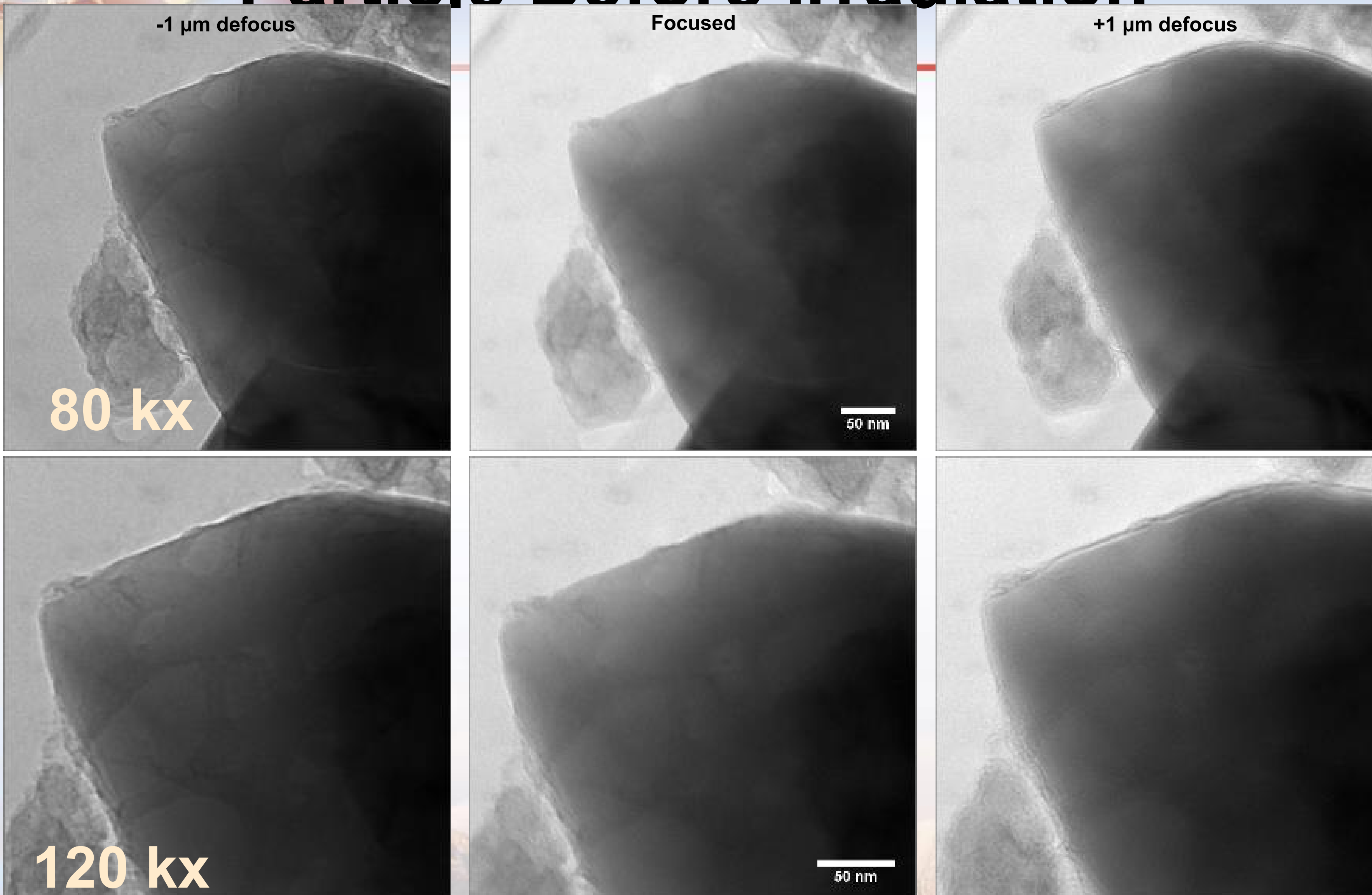
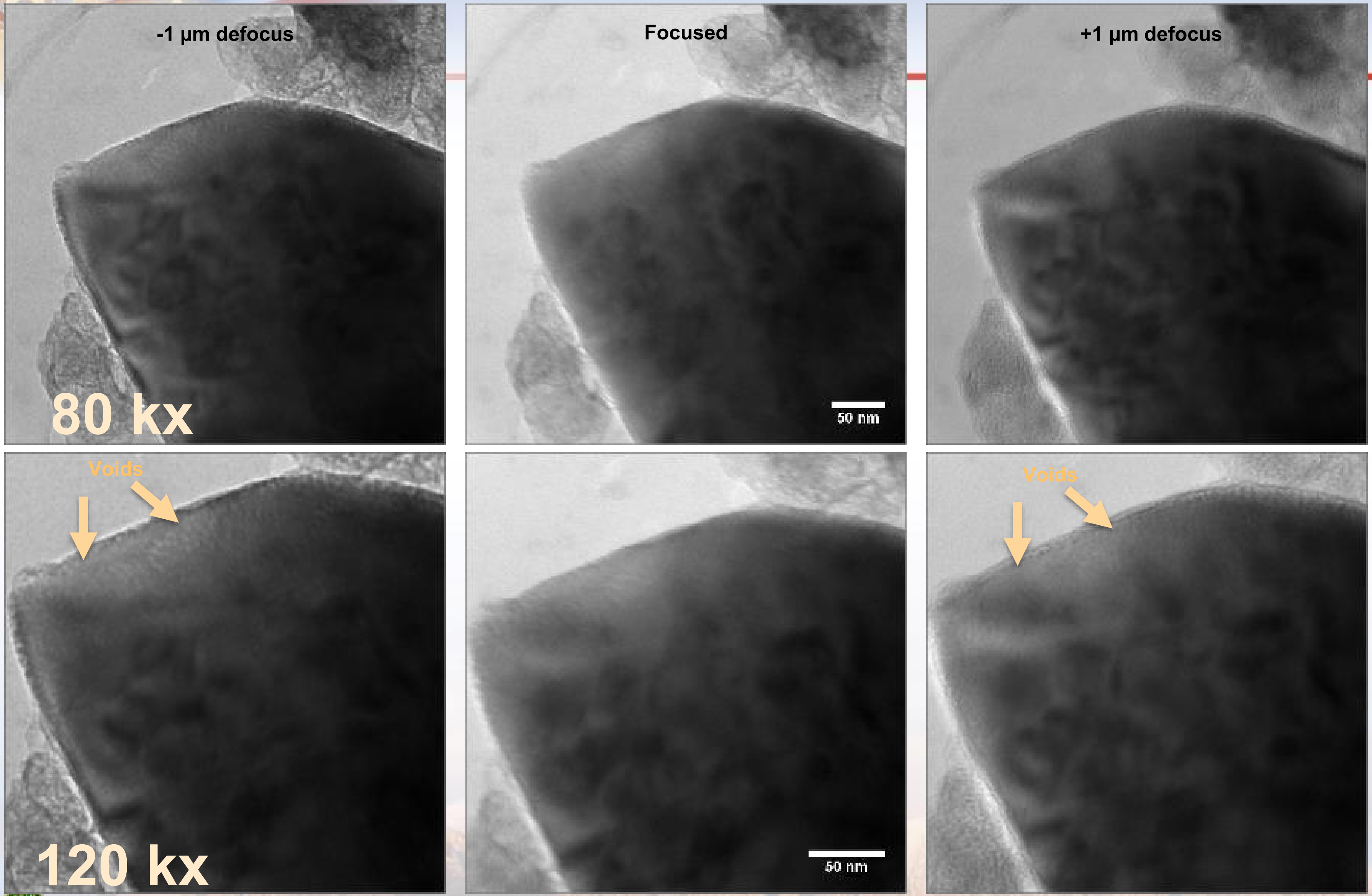
Particle Before Irradiation

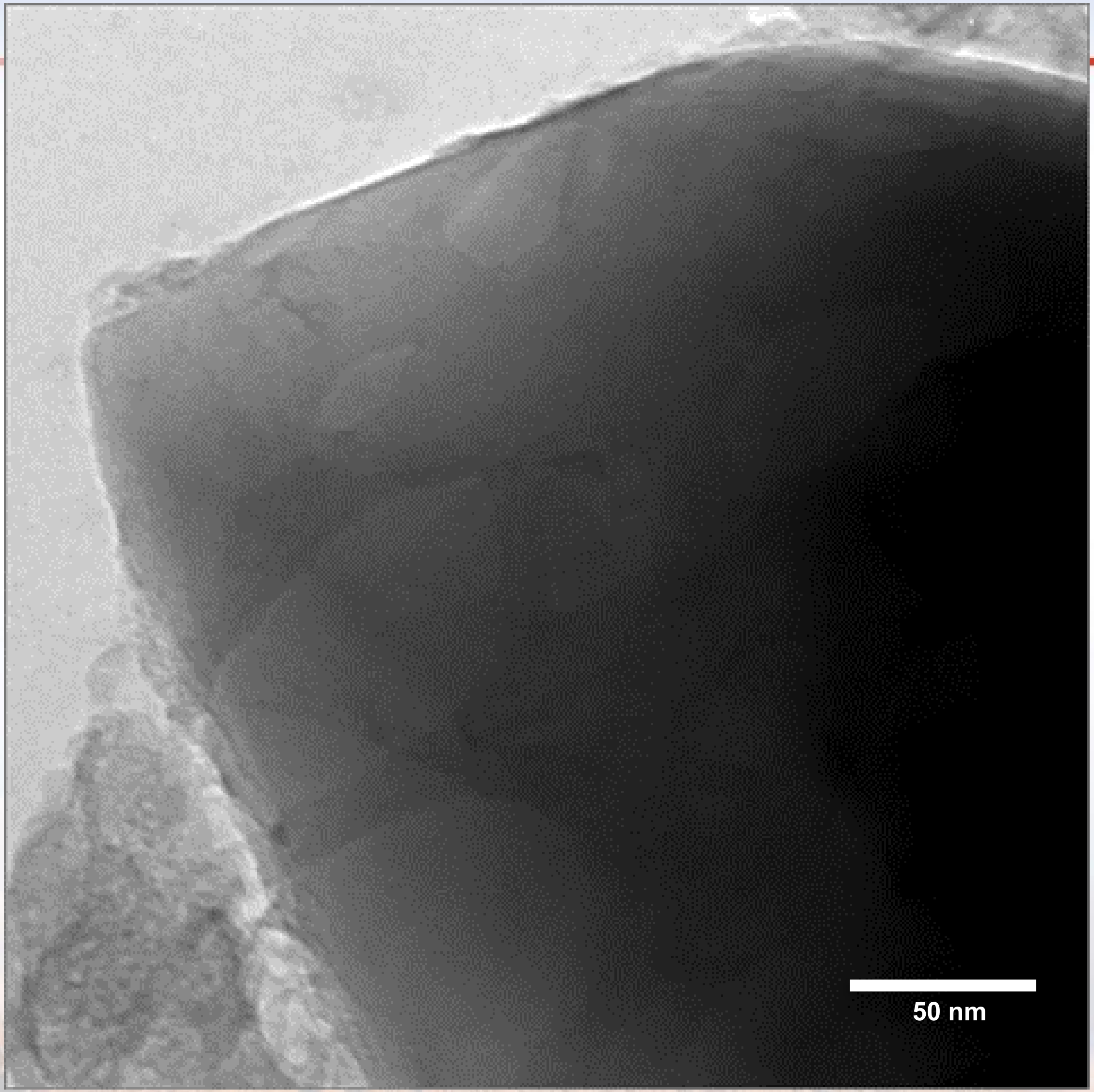


Particle After Irradiation



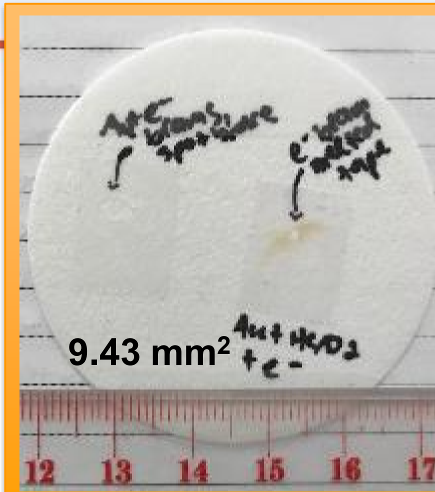
In-situ Void Formation Video

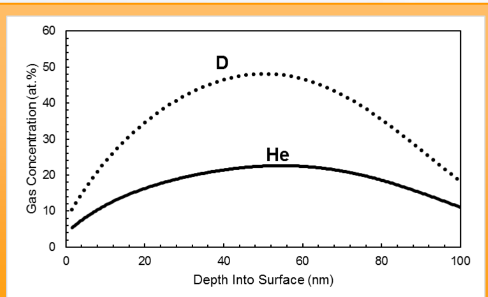
- **Each frame is 5 min of irradiation.**
- **Images taken at -518 nm defocus.**
- **Initial void formation appears after ~60 min.**
  - This would be ~1.13×10^{17} He/cm^2 (~11 at.% He) and ~2.25×10^{17} D/cm^2 (~25 at.% D)
  - It is difficult to determine when voids actually form based on the images—experiment needs to be repeated a few times.
  - Could be due to electron beam.

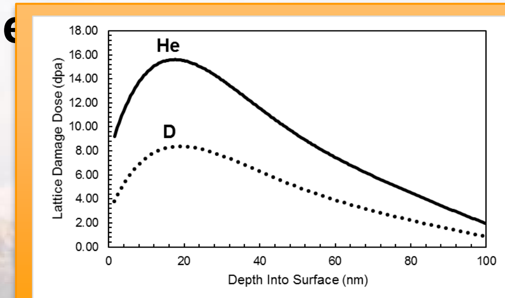


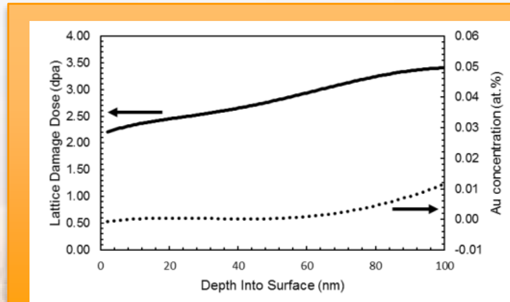
Experimental Parameters

- **Samples: drop-cast on 2.3 mm Mo grids w/C film**
- **Irradiation Parameters: 10 keV He/D_2 & 1.7 MeV Au^{3+}**
  - Beam current: 3 μA He/D_2, ~1.08 nA Au particle current
  - Flux: 7.18×10^{10} Au/cm^2/s, 9.38×10^{13} (He+D_2)/cm^2/s
  - Total irradiation time: 2 hours
  - Total Fluence: 5.17×10^{14} Au/cm^2, 6.75×10^{17} (He+D_2)/cm^2
    - D fluence = 2/3* 6.75×10^{17} = 4.5×10^{17} D/cm^2
    - He fluence = 1/3* 6.75×10^{17} = 2.25×10^{17} He/cm^2
  - Total dose: 28 dpa (He+D+Au), 25 at.%He, 50 at.%D
- **Temperature (HB HT stage): 310°C**
- **Electron Beam Exposure: Series acquisition every 1 min (100 ms exposure/image) with a few minutes total re-aligning sample. Do not think beam blank between images was working properly-probably** ... **s the**



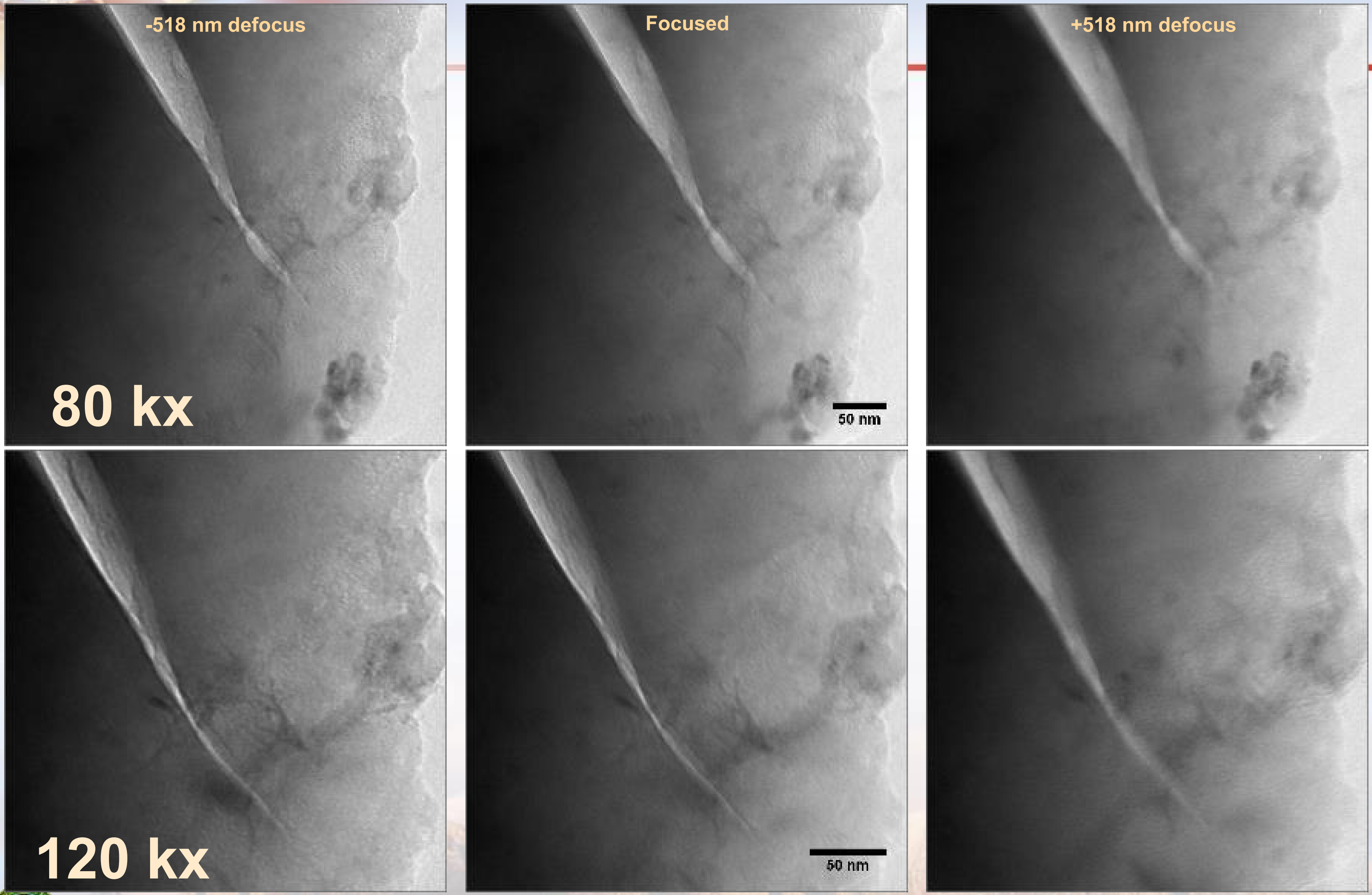




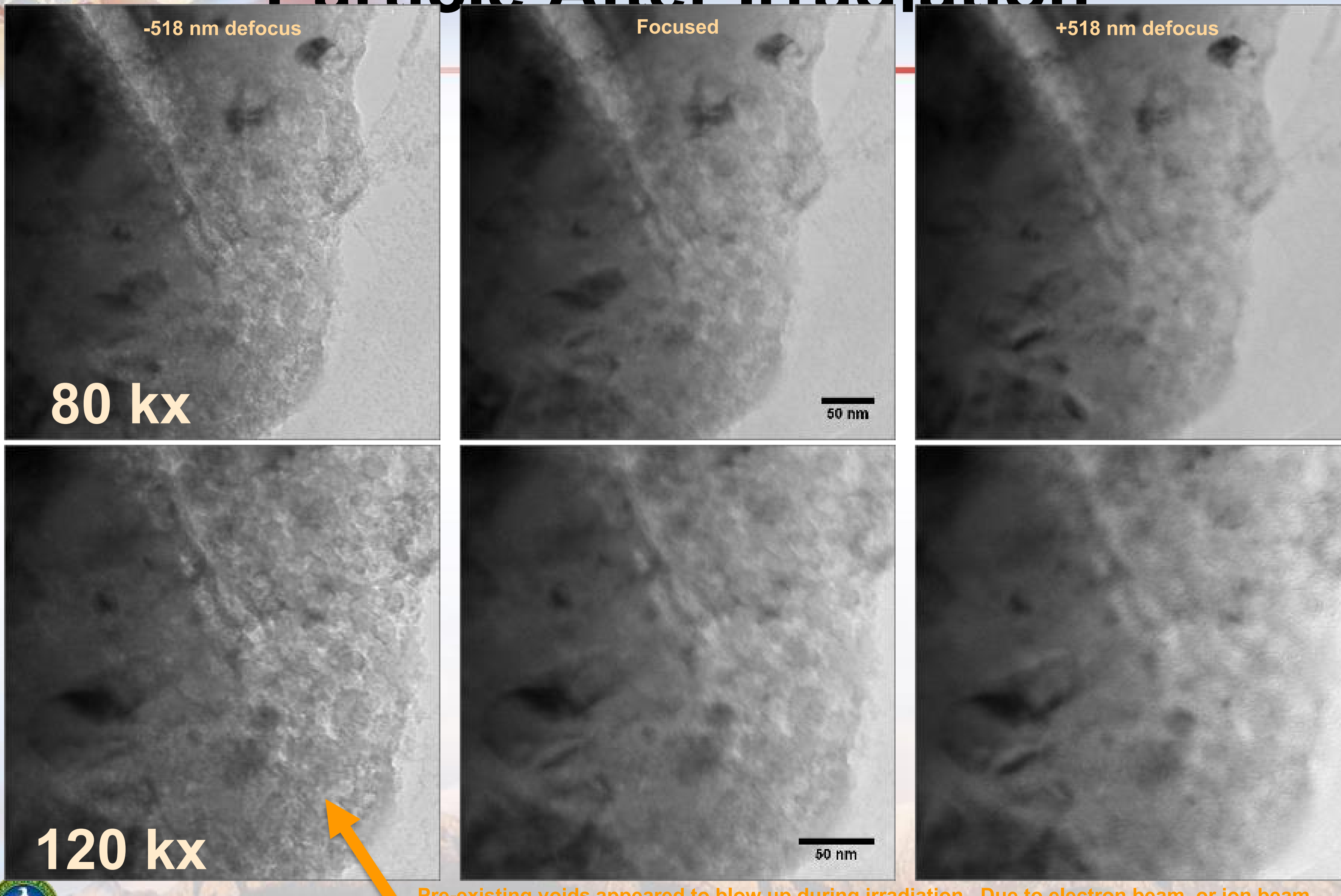


Could not find a particle w/o voids in this sample

Particle Before Irradiation



Particle After Irradiation



Pre-existing voids appeared to blow up during irradiation. Due to electron beam, or ion beam, or both??

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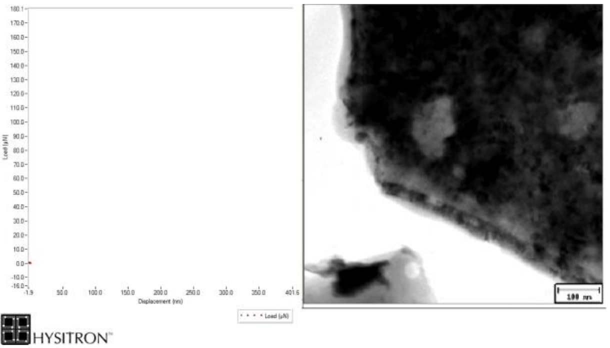
Radiation & Potential Synergistic In- Situ Capabilities

Mechanical Effects



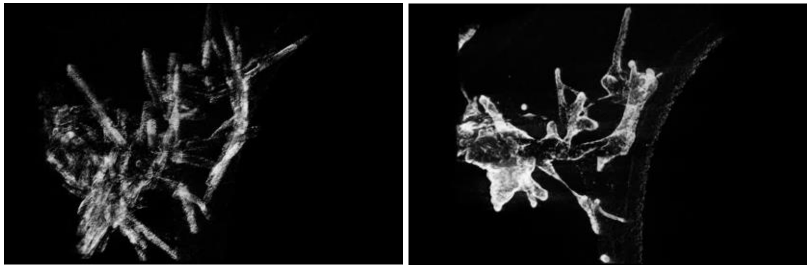
Hysitron PI95 TEM Picoindenter
Gatan 654 Straining Holder
Allows for direct correlation of dose and defect density with resulting changes in strength, ductility, and defect mobility

Environmental Effects

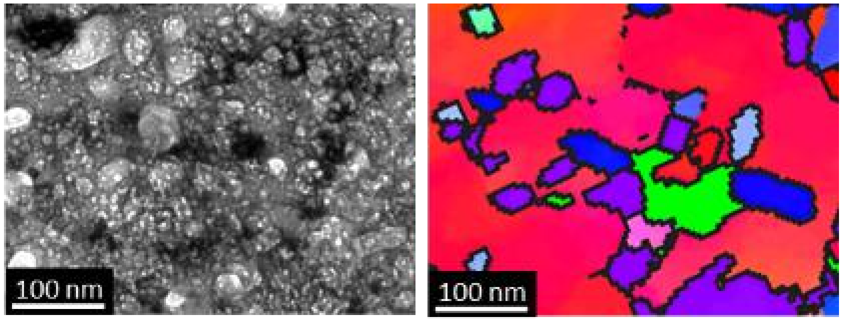
Protochips Liquid and Gas Flow
Study the material in different environments (flowing, mixing, temperature)

Structural Effects

Hummingbird Tomography Stage
Gatan 925 Double Tilt Rotate
Morphology changes as a result of radiation damage

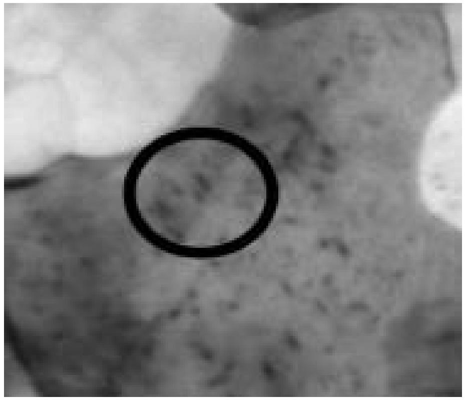


Nanomegas ASTAR
Grain structure changes as a result of radiation and implantation



Thermal Effects

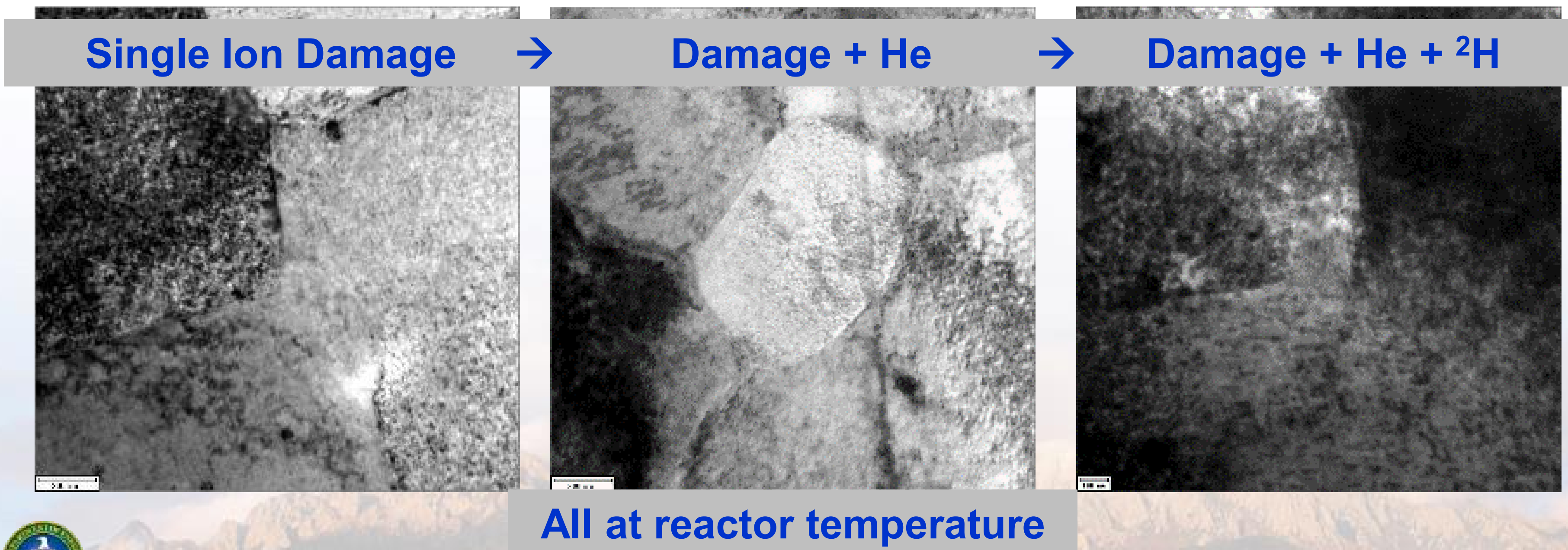
Hummingbird Heating Stage
Coupling effects of temperature and irradiation on microstructural evolution up to 800° C



The application of advanced microscopy techniques to characterize synergistic effects in a variety of extreme environments

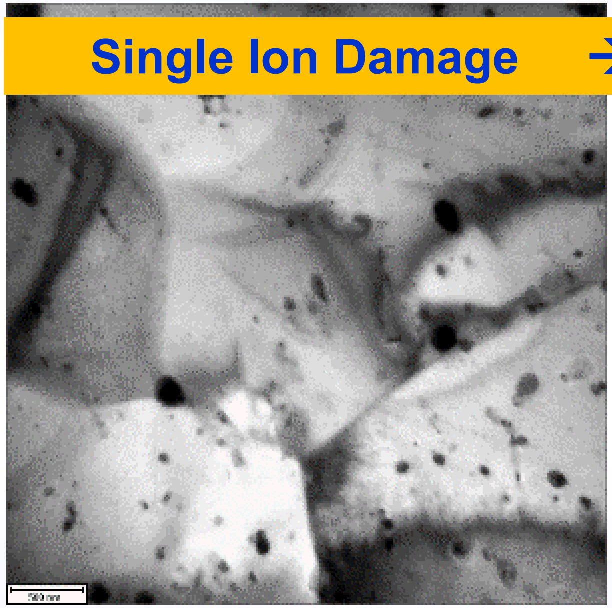
Summary

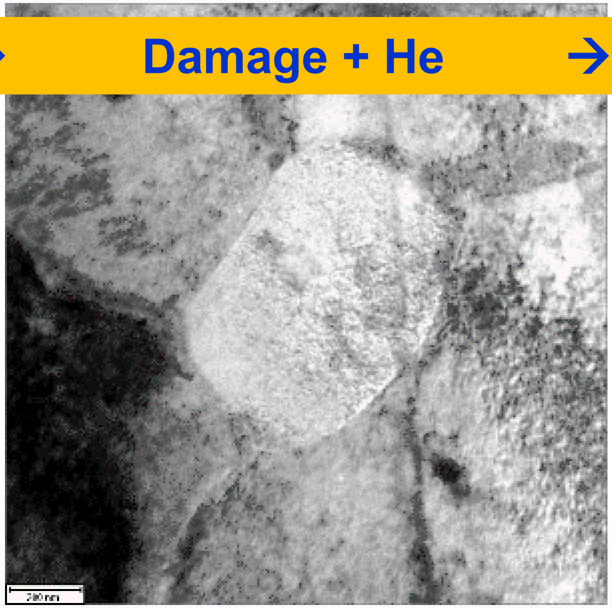
- Synergistic effects between damage and gas accumulation are being simulated in TPBAR materials, in-situ, at the SNL I^{3}TEM facility, using heavy ion irradiation and D_2 + He implantation
- Aimed at understanding fundamental defect interactions that affect ^{3}H retention
- In-situ triple beam irradiations can be coupled with **HT TEM stage** for more accurate simulation of reactor conditions
- In Zr alloys, various irradiation/implantation conditions resulted in no bubble formation in-situ, but bubbles were observed in irradiated thin foils 30 days later

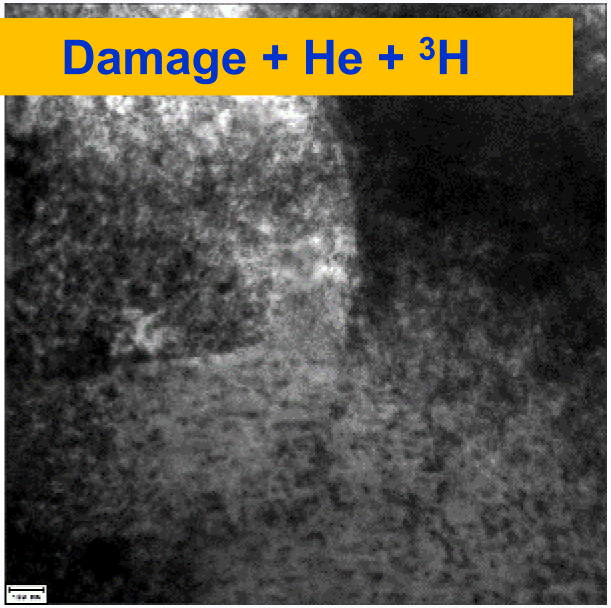


Summary

Single Ion Damage → Damage + He → Damage + He + ^3H



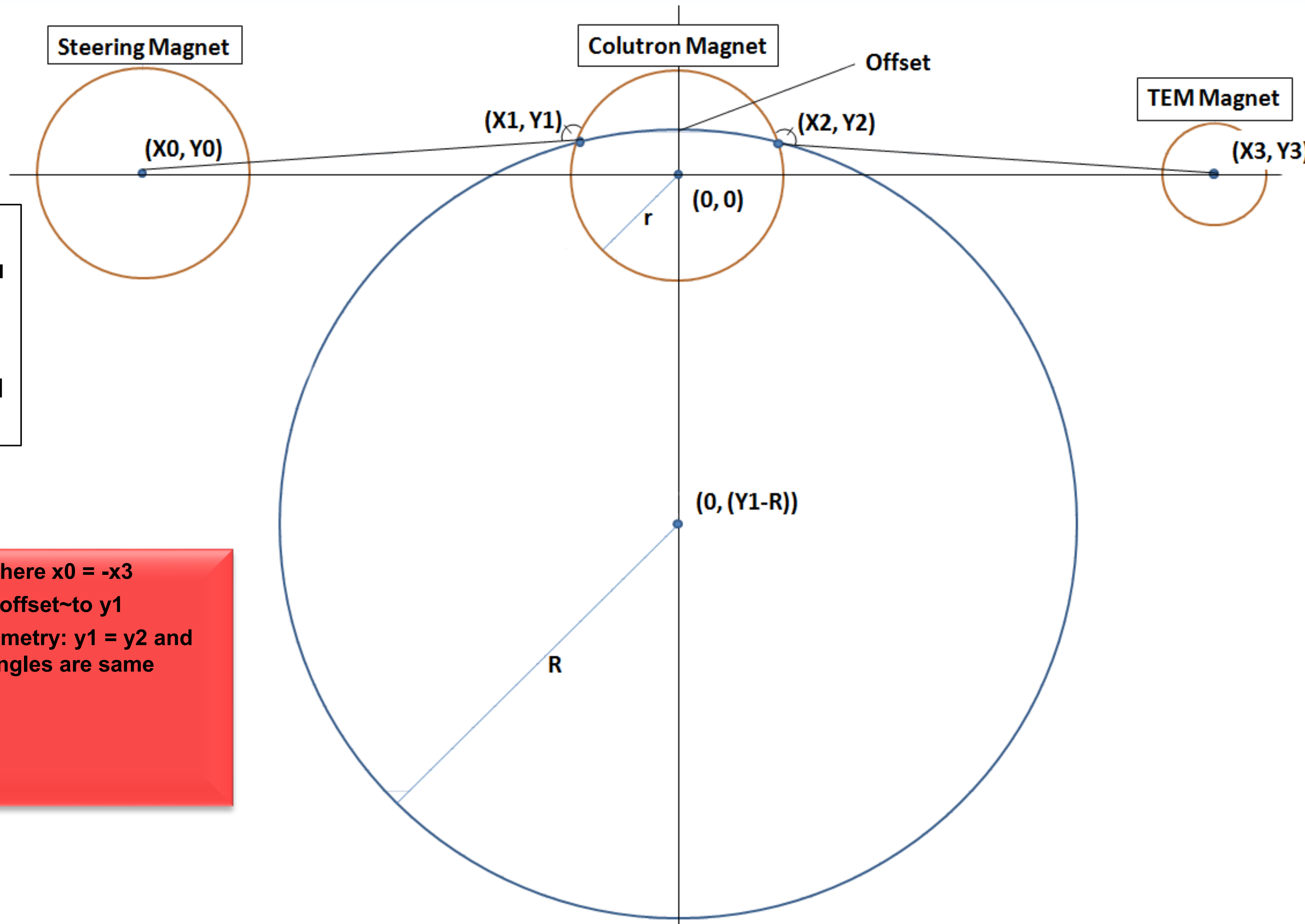




All at reactor operating temperature

This work demonstrates that the I^3TEM is capable of simulating the synergistic effects of damage, gas accumulation, and high temperature occurring in reactor-like conditions, *in situ*

Analytical Set-up



Where:

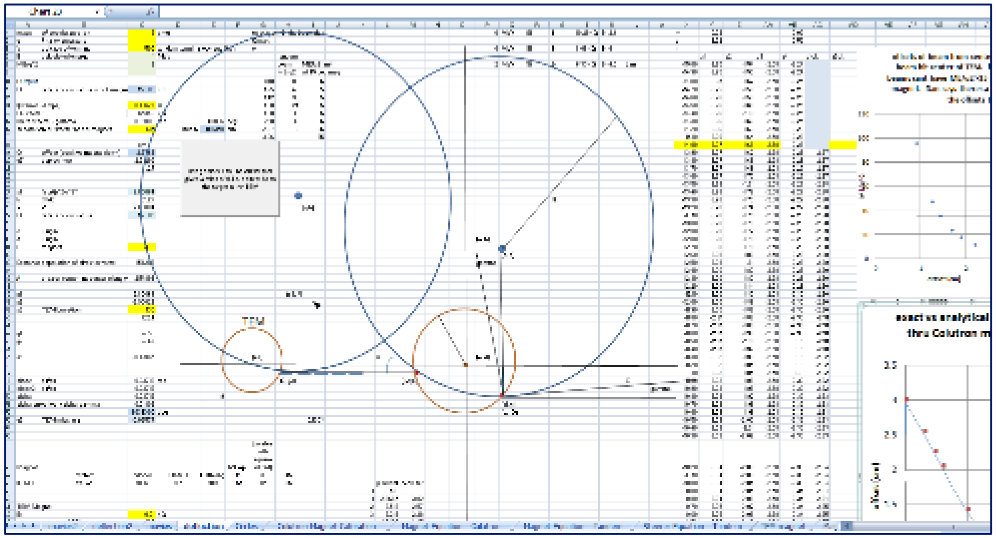
- M is ion mass amu
- E is energy MeV
- q is ion charge
- B is magnetic field kG

$$R = \frac{\sqrt{2mE}}{qB}$$

- Symmetry where x0 = -x3
- R >> r, and offset~to y1
- Time reversal symmetry: y1 = y2 and entry and exit angles are same

Calculating Offset

Exact:

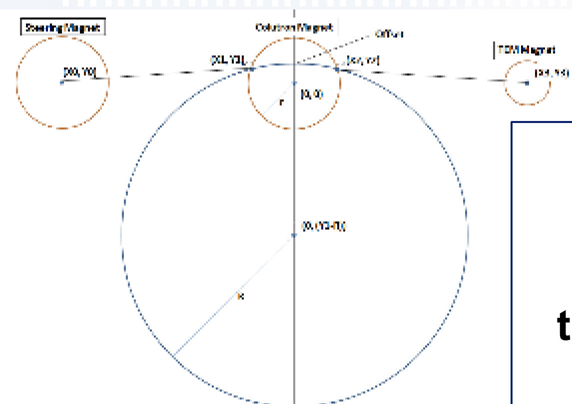


Analytical:

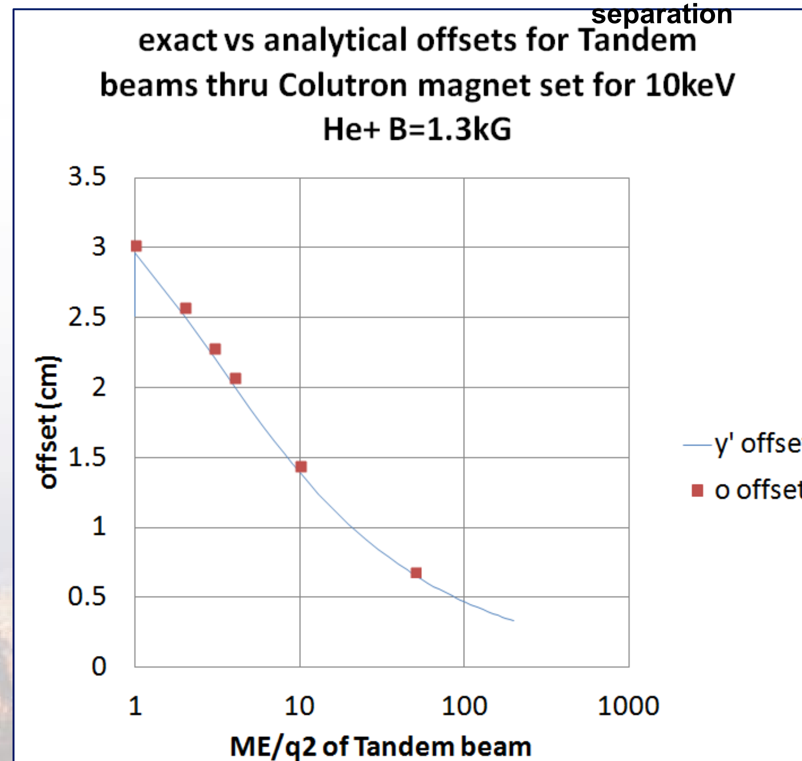
$$y_1 = \frac{r}{\sqrt{\left(\frac{R}{x_0}\right)^2 + 1}}$$

Where:

- y1 is ~ offset
- r is the radius of Col. Mag.
- R is radius of curvature of ion
- X0 is TEM Col. mag separation



- Ion path must be continuous
- Set slope of 1) ion trajectory line and of 2) radius of curvature at (x1,y1) equal



Beam Limits

Use offset equation to find tandem beam rigidity limit as function of magnetic field, B:

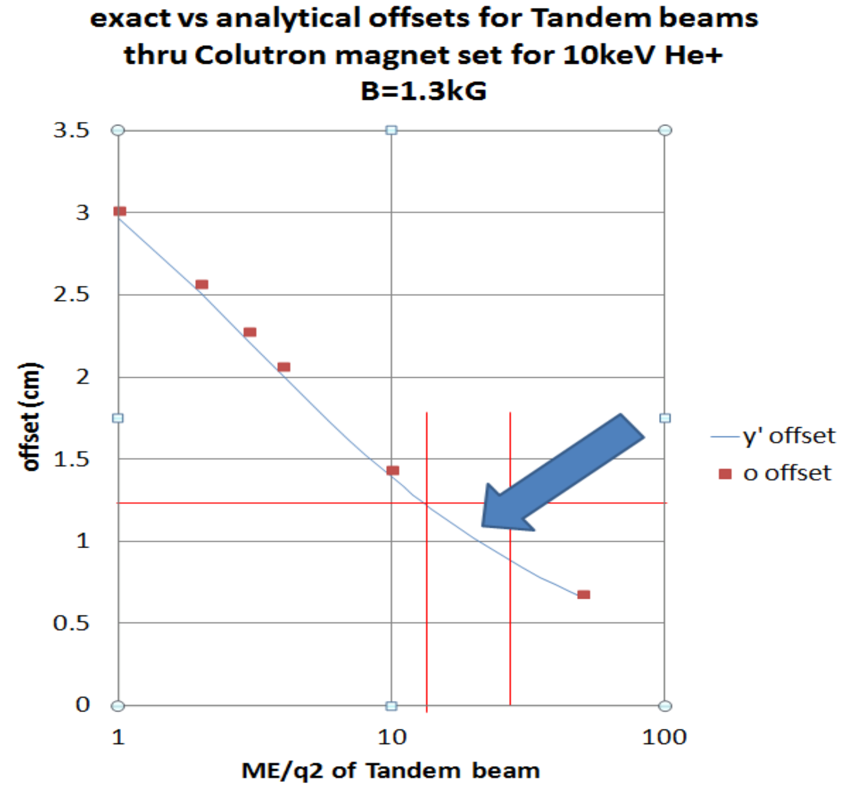
$$R = \frac{\sqrt{2mE}}{qB}$$

$$y_1 = \frac{r}{\sqrt{\left(\frac{\sqrt{2mE}}{x_0 qB}\right)^2 + 1}}$$

$$\left(\frac{mE}{q^2}\right)_t = \frac{B_c^{\,2} x_0^{\,2}}{2}\left[\left(\frac{r}{y_1}\right)^2 - 1\right]$$

Substituting in values for Colutron magnet set for 10keV He+ B=1.3kG we find:

$$\left(\frac{mE}{q^2}\right)_t > 13$$



As a Function of Colutron Energy

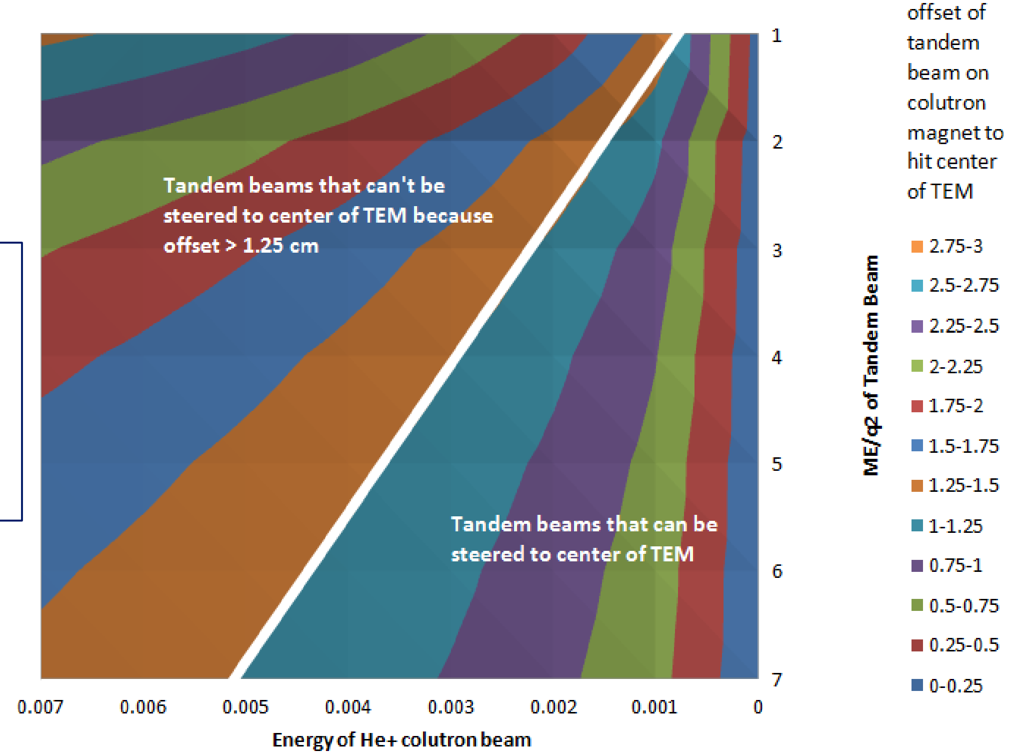
Solve for B as a function of Colutron beam rigidity:

$$B_c = \frac{\tan\frac{\theta}{2}\sqrt{2m_c E_c}}{rq}$$

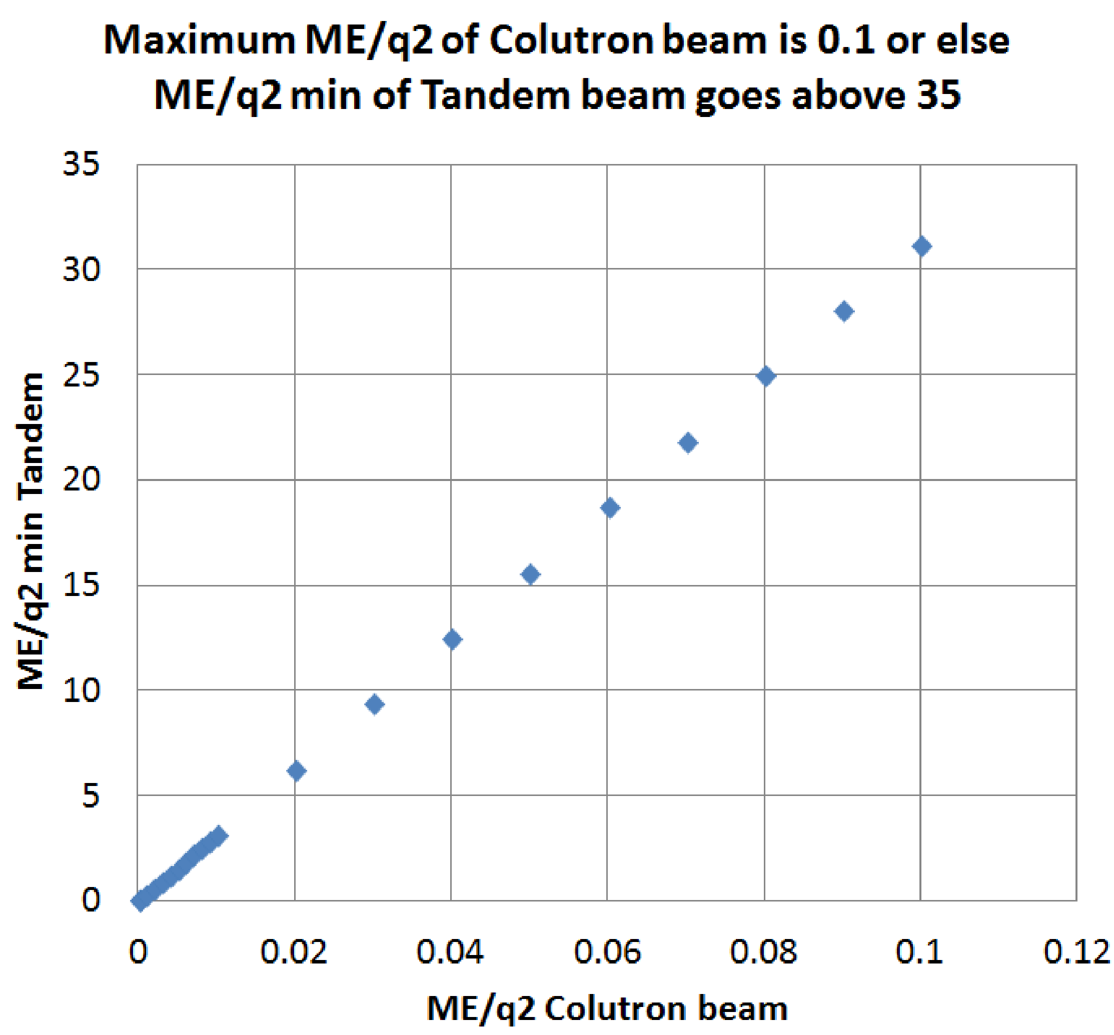
$$\left(\frac{mE}{q^2}\right)_t = \frac{B_c^{\,2} x_0^{\,2}}{2}\left[\left(\frac{r}{y_1}\right)^2 - 1\right]$$

Equation is first-order in respect to Colutron beam energy

$$\left(\frac{mE}{q^2}\right)_t = \frac{\left(\tan^2\frac{\theta}{2}\right)2m_c E_c (x_0)^2}{2r^2 q_c^{\,2}}\left[\left(\frac{r}{y_1}\right)^2 - 1\right]$$



Colutron Parameters



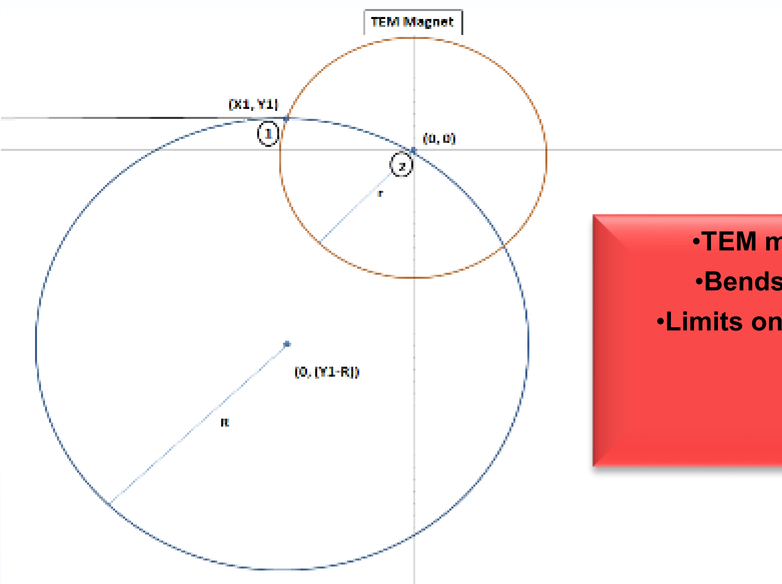
Colutron beam mass-energy product cannot exceed ~ .1

- Colutron magnetic field too high even for ^{10}B at 10 keV
- Requires higher Tandem beam rigidity
- Tandem rigidity outside of 35 limit on Colutron magnet
- … s

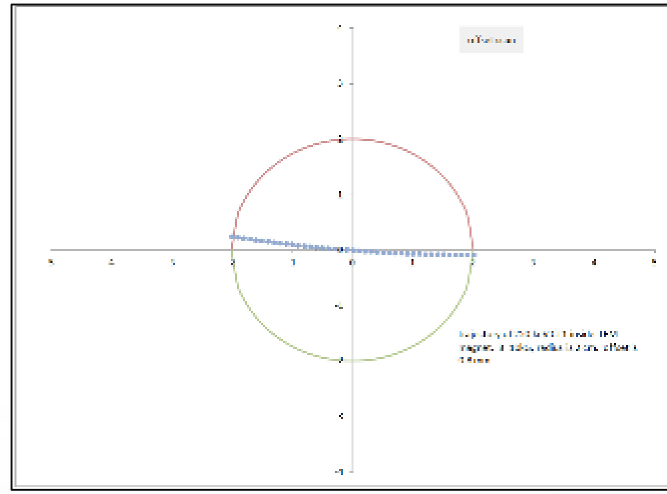
Use lower energy Colutron beams

$$y_1 = \frac{r^2}{2R}$$

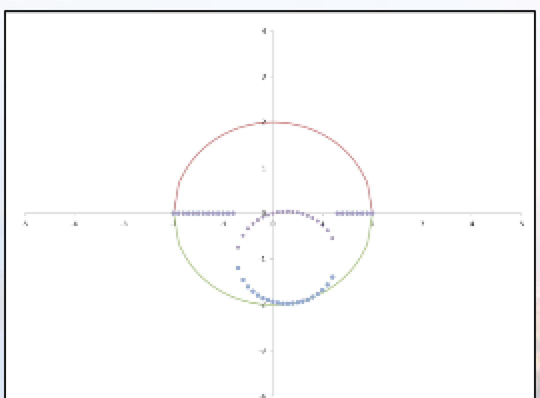
TEM Magnet and Colutron Beam



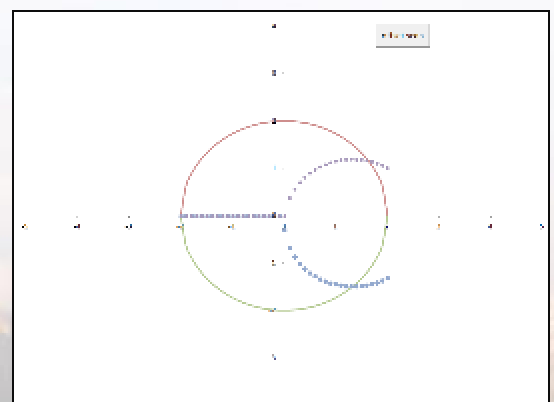
- TEM magnetic field
- Bends light beams
- Limits on Colutron beam



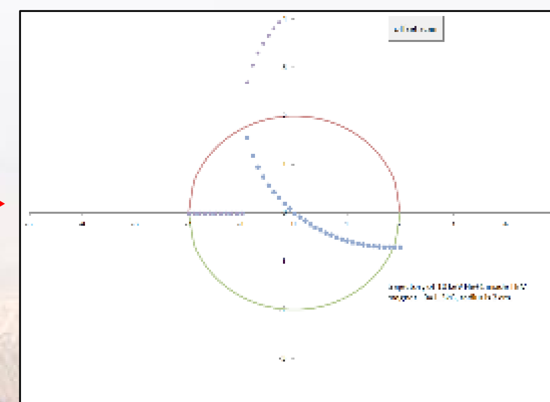
Tandem beam: mm deflection



$$\frac{r}{R} = 2$$



$$\frac{r}{R} = \sqrt{2}$$



10KeV He+ B=10kG

Benefits & Limitations of *in situ* TEM

Benefits

1. **Real-time nanoscale resolution observations of microstructural dynamics**

Limitations

1. **Predominantly limited to microstructural characterization**
   - Some work in thermal, optical, and mechanical properties
2. **Limited to electron transparent films**
   - Can often prefer surface mechanisms to bulk mechanisms
   - Local stresses state in the sample is difficult to predict
3. **Electron beam effects**
   - Radiolysis and Knock-on Damage
4. **Vacuum conditions**
   - 10^{-7} Torr limits gas and liquid experiments feasibility
5. **Local probing**
   - Portions of the world study is small

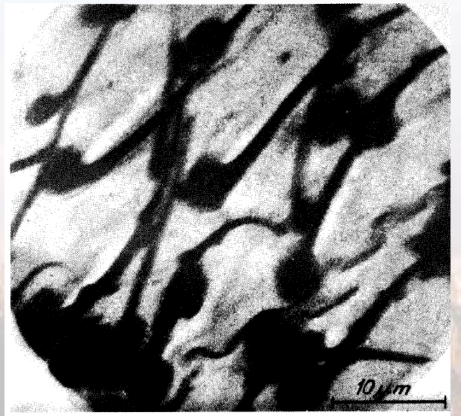


Fig. 6: Wing surface of the house fly.
(First internal photography, U = 60 kV, M_e = 2200)
(Driest, E., and Müller, H.O.: Z. Wiss. Mikroskopie 52, 53-57 (1935)

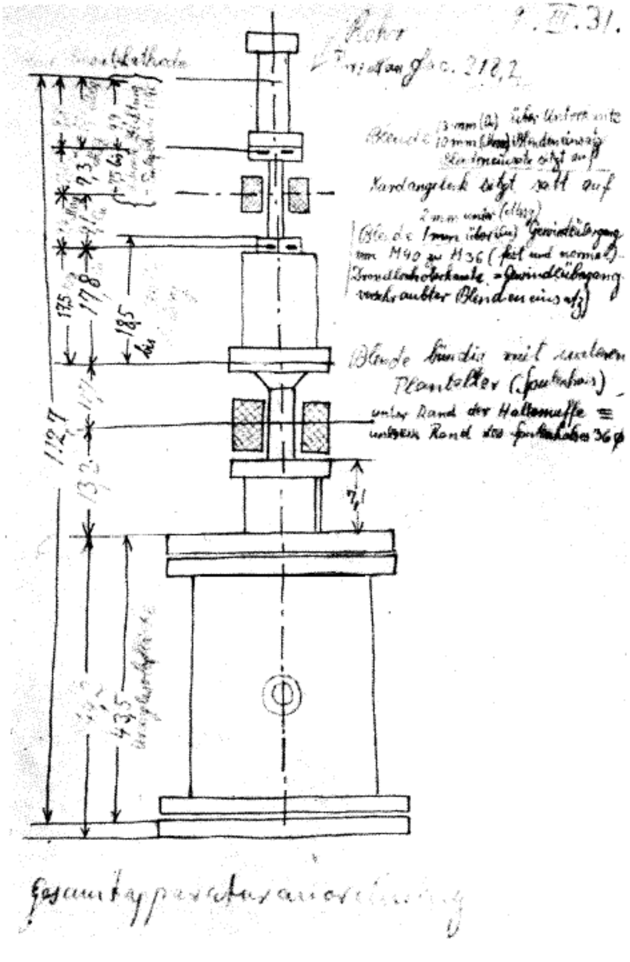
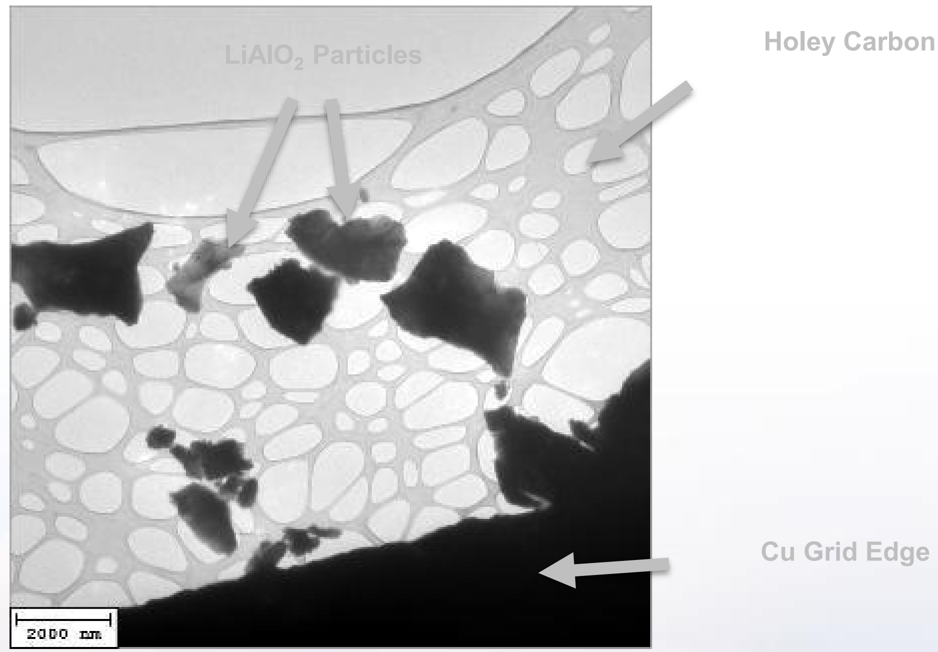


Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].

Section 1:
TEM sample preparation and characterization

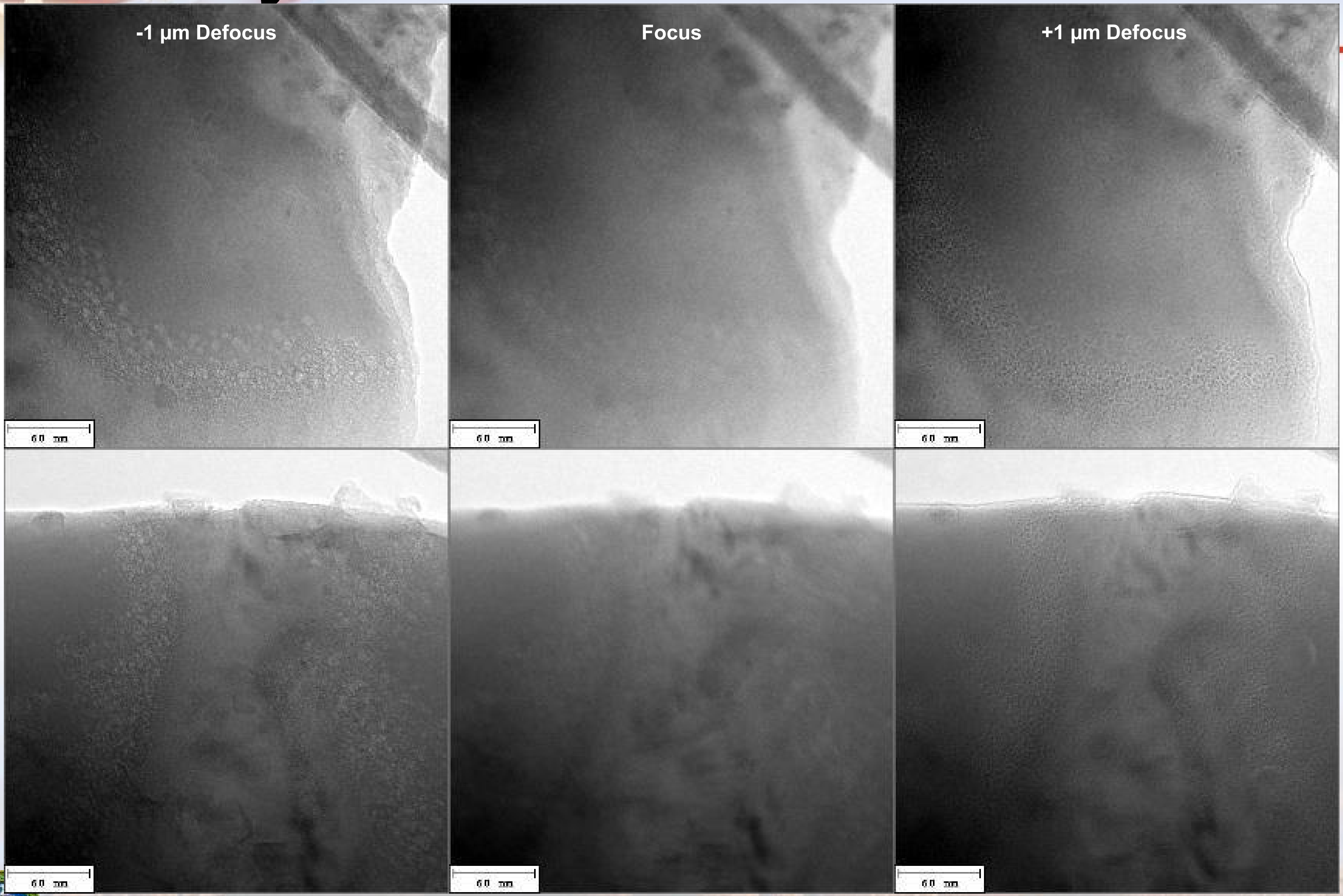
Sample Preparation

- Samples were prepared by drop-casting $LiAlO_2$ powders, obtained from PNNL, onto TEM grids (either 3 mm Cu grids with holey carbon film, or 2.3 mm Mo grids with a thin C film)



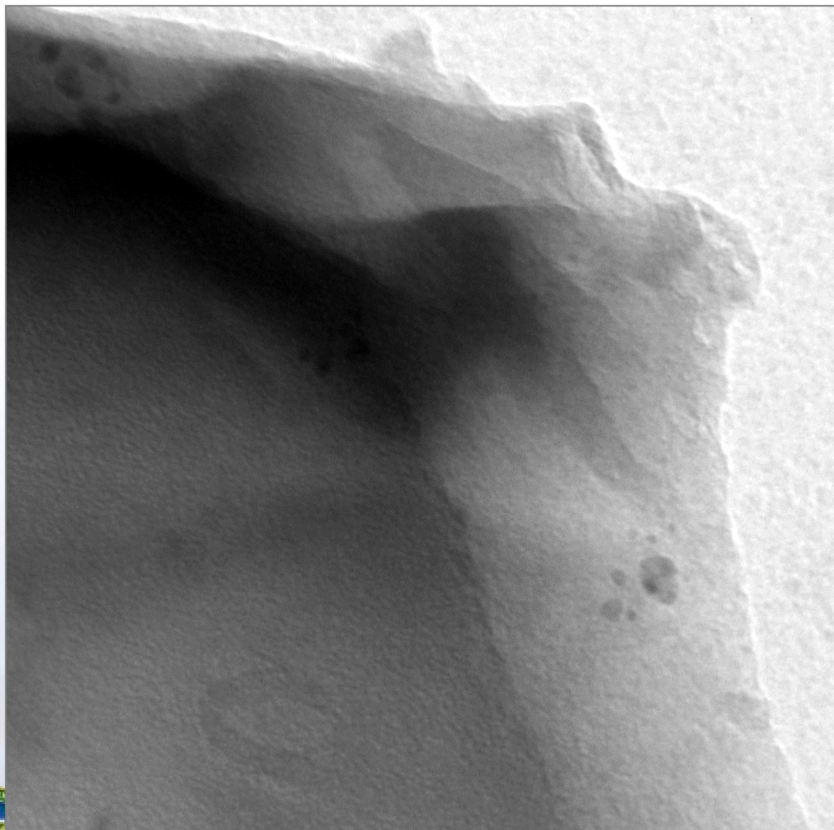
- No obvious difference between the two batches of powders
- No obvious difference after grinding powders with mortar and pestl

Many Particles Contained Voids



Material Response to Electron Beam

- Voids seem to be nucleating and growing under the electron beam both at room temperature and 310°C
- Timescale of void nucleation and growth varies greatly depending on the particle



Total time: 3 min 28 s

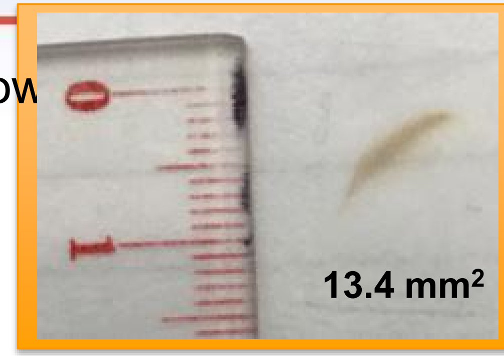
Total time: 4 min 22 s

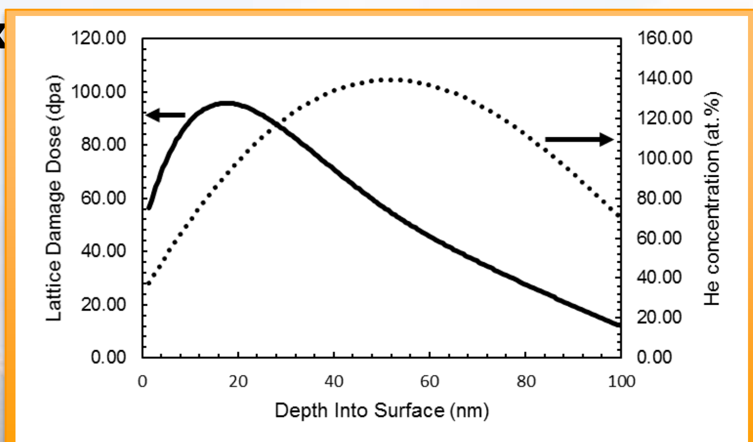
Section 2:
In-situ He implantation @ 310°C

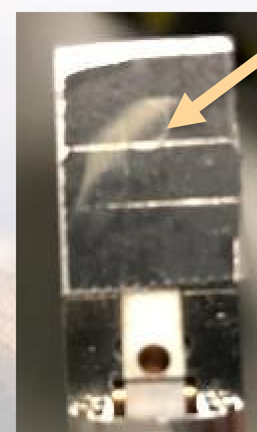
Experimental Parameters

- **Samples: drop-cast on 2.3 mm Mo grids w/C film**
- **Irradiation Parameters: 10 keV He**
  - Beam current: ~4 μA He—probably lower (see pic below)
  - Flux: 9.38×10^{13} He/cm^2/s
  - Total irradiation time: 2 hours
  - Total Fluence: 1.38×10^{18} He/cm^2
  - Total dose: 100 dpa, 140 at.%He
    - Note: He concentration is much too high for the size of bubbles observed; most of the He probably diffused out of particle upon impact.
- **Temperature (HB HT stage): 310°C**
- **Electron Beam Exposure: Series acquisition every 1 min (250 ms exposure/image) with a few minutes total re-aligning sample. Do not think beam blank between images was working properly-probably ex … e.**

He beam burn spot







He beam is only partially covering 3mm circle—may have been lower current at imaging location than measured in FC

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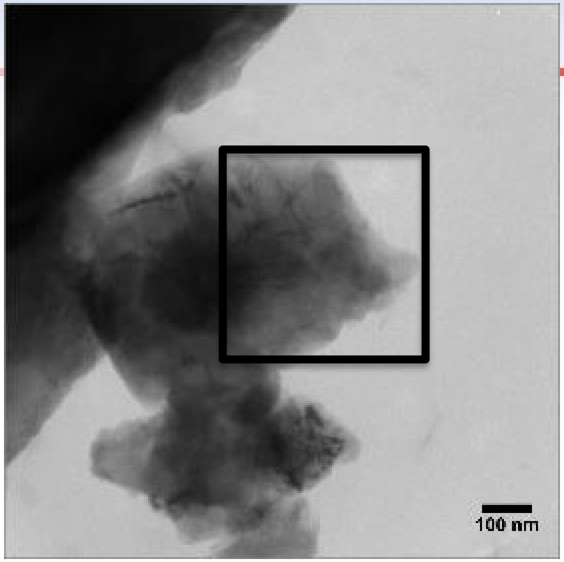
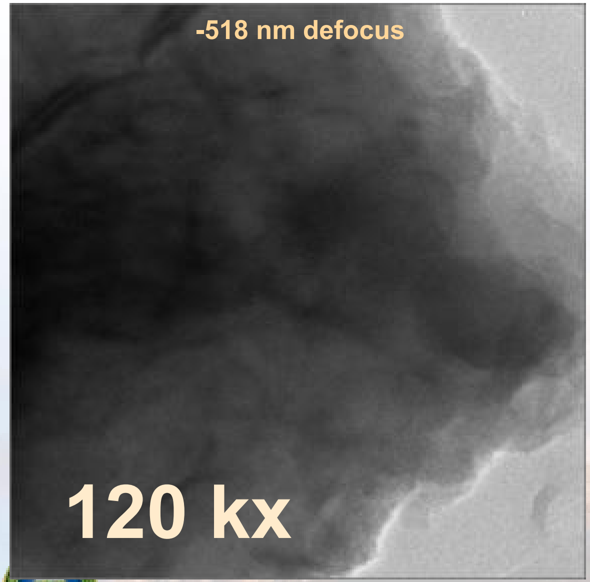
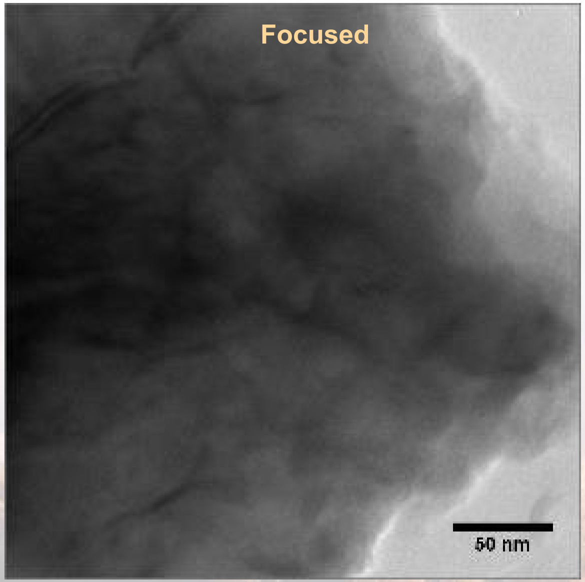
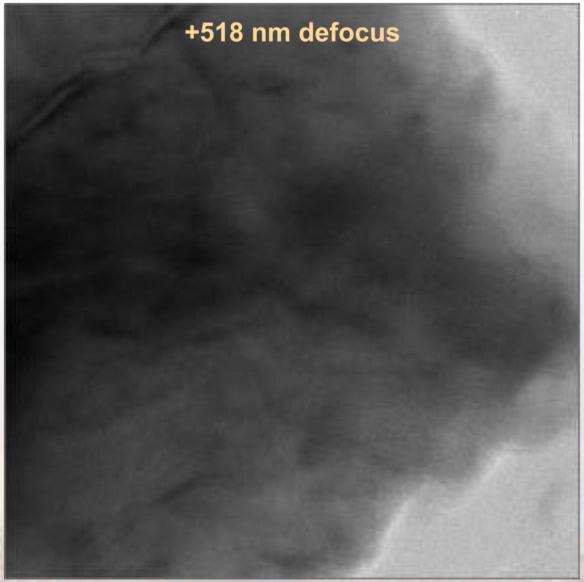
Particle Before Irradiation



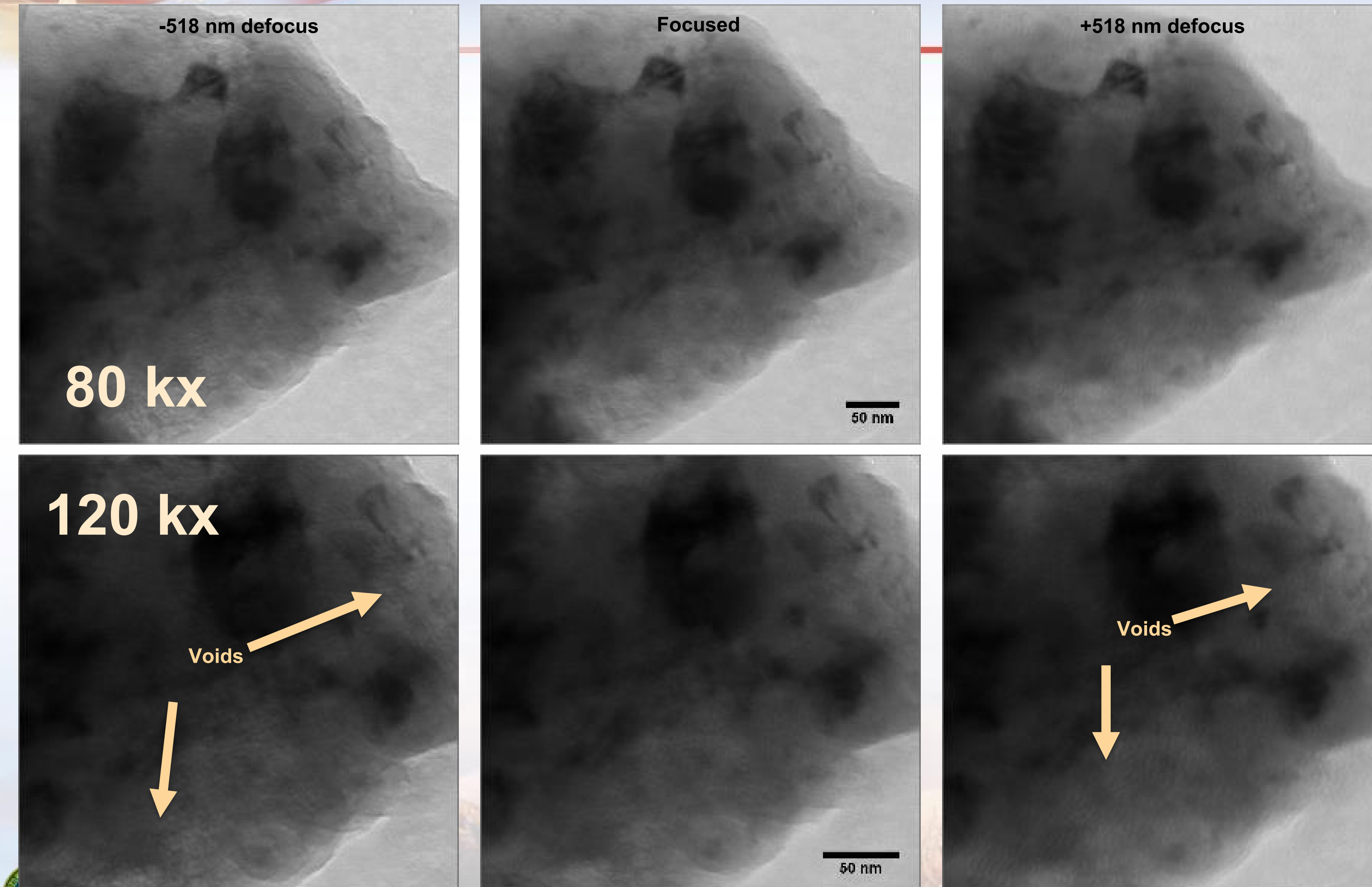
Note: Objective stig is not great in a lot of these images (as seen below). There is nothing amorphous in this sample, and I was trying to align quickly to minimize e-beam exposure.





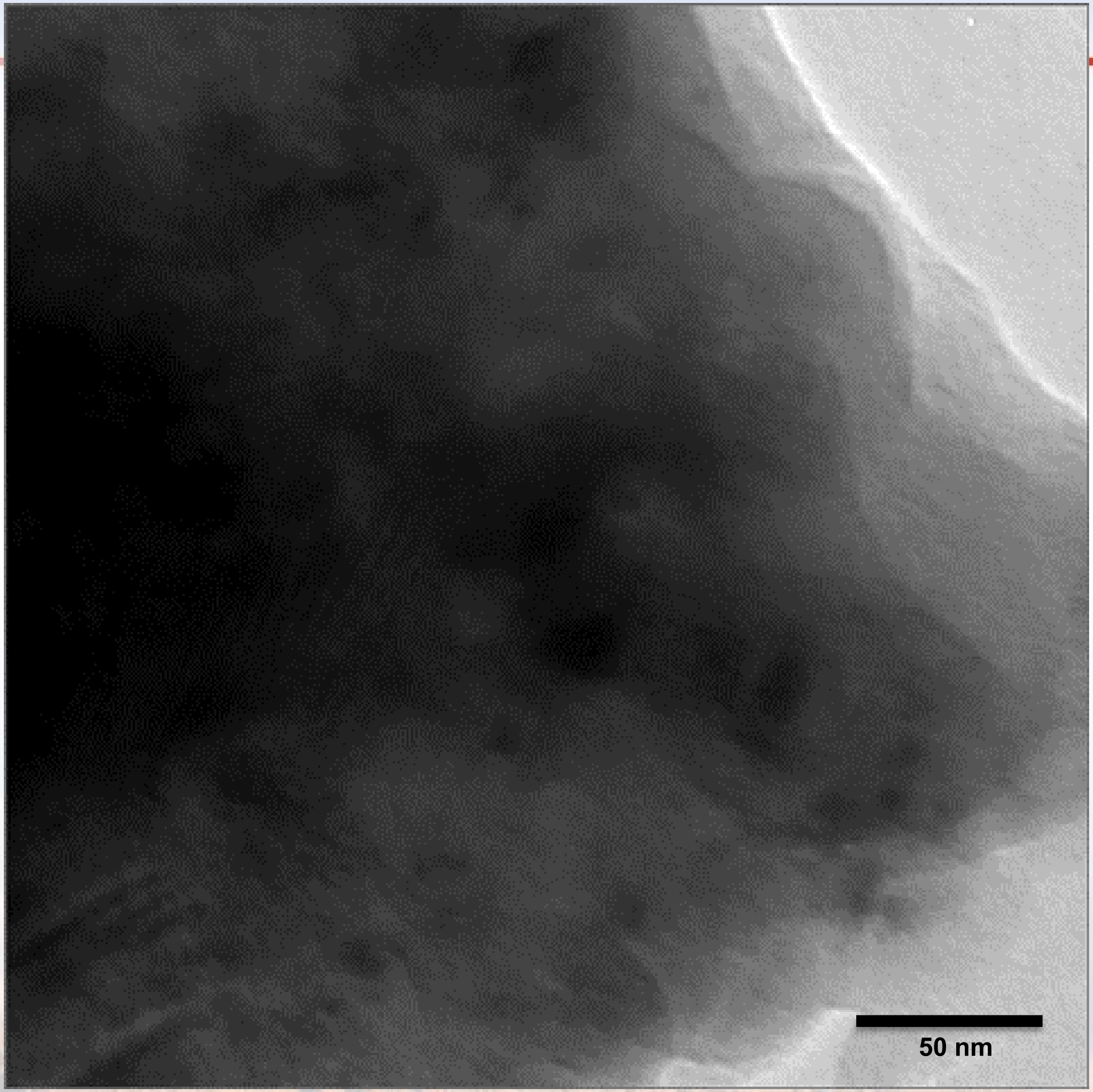


Particle After Irradiation



In-situ Void Formation Video

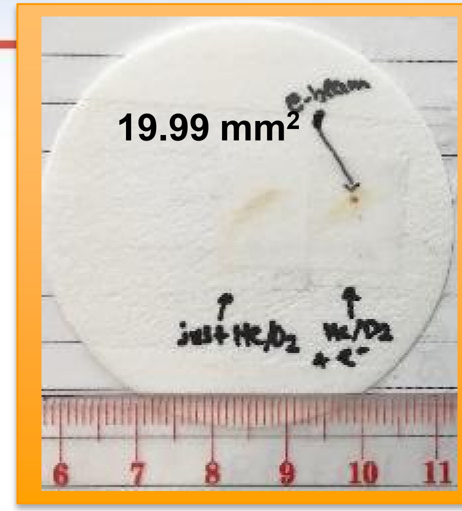
- **Video starts after 9 min (first several images had too much drift, I was experimenting with series acquisition). Each frame is 1 min of irradiation.**
- **Images taken at ~-1 μm defocus**
- **Void formation appears after ~13 min**
  - This would be ~7.32×10^{16} He/cm^2 (~8 at.% He)
  - It is difficult to determine when voids actually form based on the images—experiment needs to be repeated a few times.
  - Could be due to electron beam because He beam alignment wasn't great, and e-beam was on entire time



Section 3:
In-situ He+D_2 implantation @ 310°C

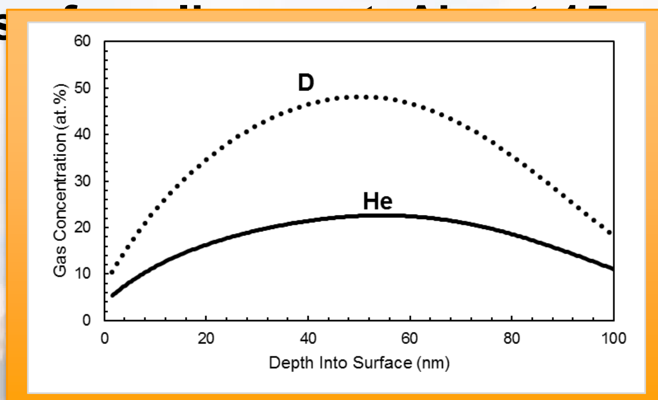
Experimental Parameters

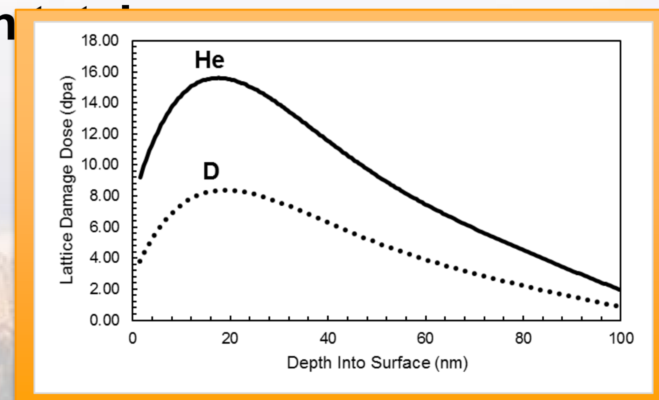
- **Samples: drop-cast on 2.3 mm Mo grids w/C film**
- **Irradiation Parameters: 10 keV He/D_2**
  - Beam current: 3 μA He/D_2
  - Flux: 9.38×10^{13} (He+D_2)/cm^2/s
  - Total irradiation time: 2 hours
  - Total Fluence: 6.75×10^{17} (He+D_2)/cm^2
    - D fluence = 2/3* 6.75×10^{17} = 4.5×10^{17} D/cm^2
    - He fluence = 1/3* 6.75×10^{17} = 2.25×10^{17} He/cm^2
  - Total dose: 25 dpa (He+D), 25 at.%He, 50 at.%D



- **Temperature (HB HT stage): 310°C**
- **Electron Beam Exposure: Because e^- beam seemed to be affecting sample during series acquisition in single and triple beam experiments, I opted for manual imaging every 5 min; exposure was ~ 30 s every 5 min, plus a few minutes … in …**

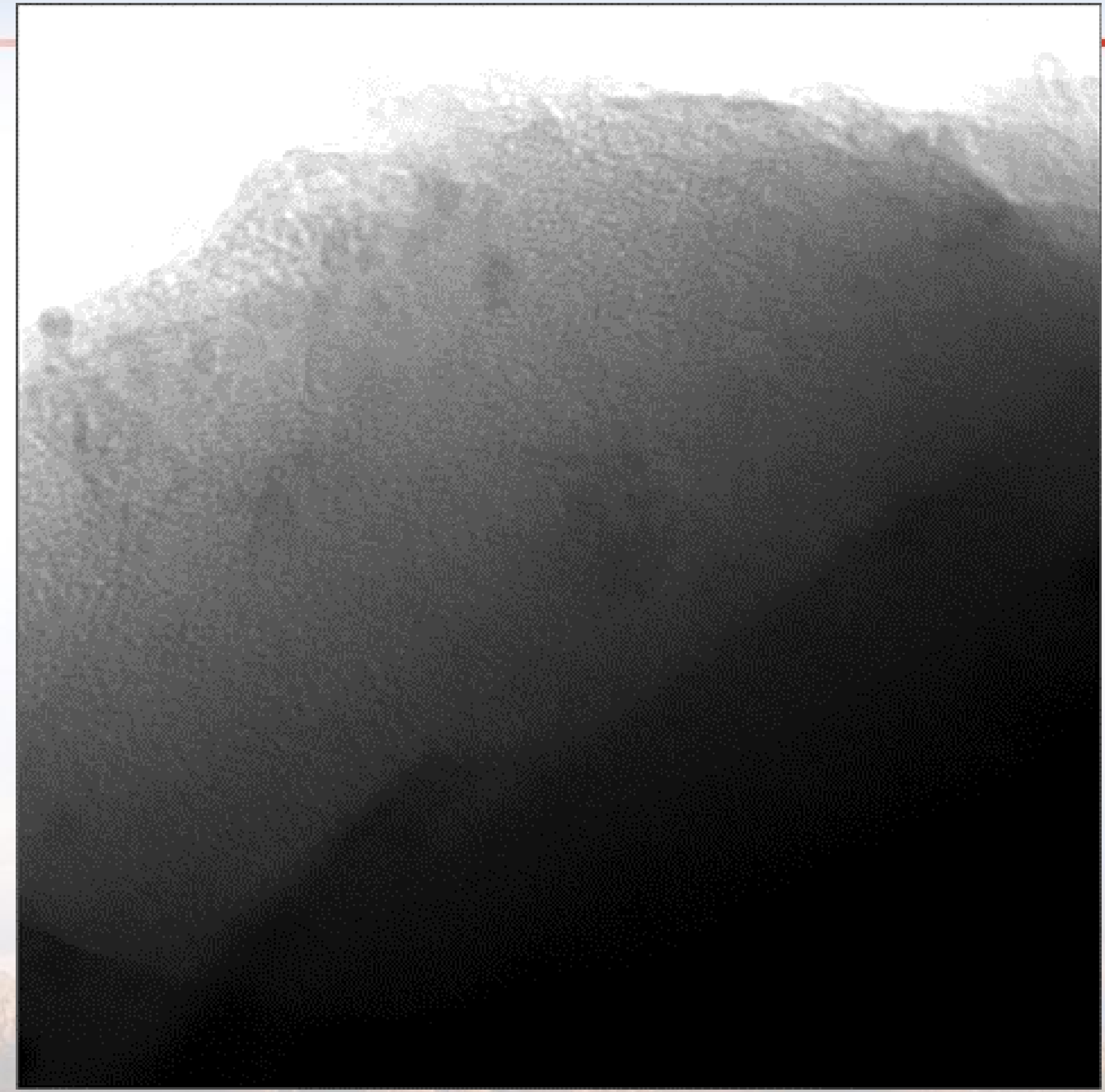
D_2 splits apart into two 5 keV D atoms upon hitting





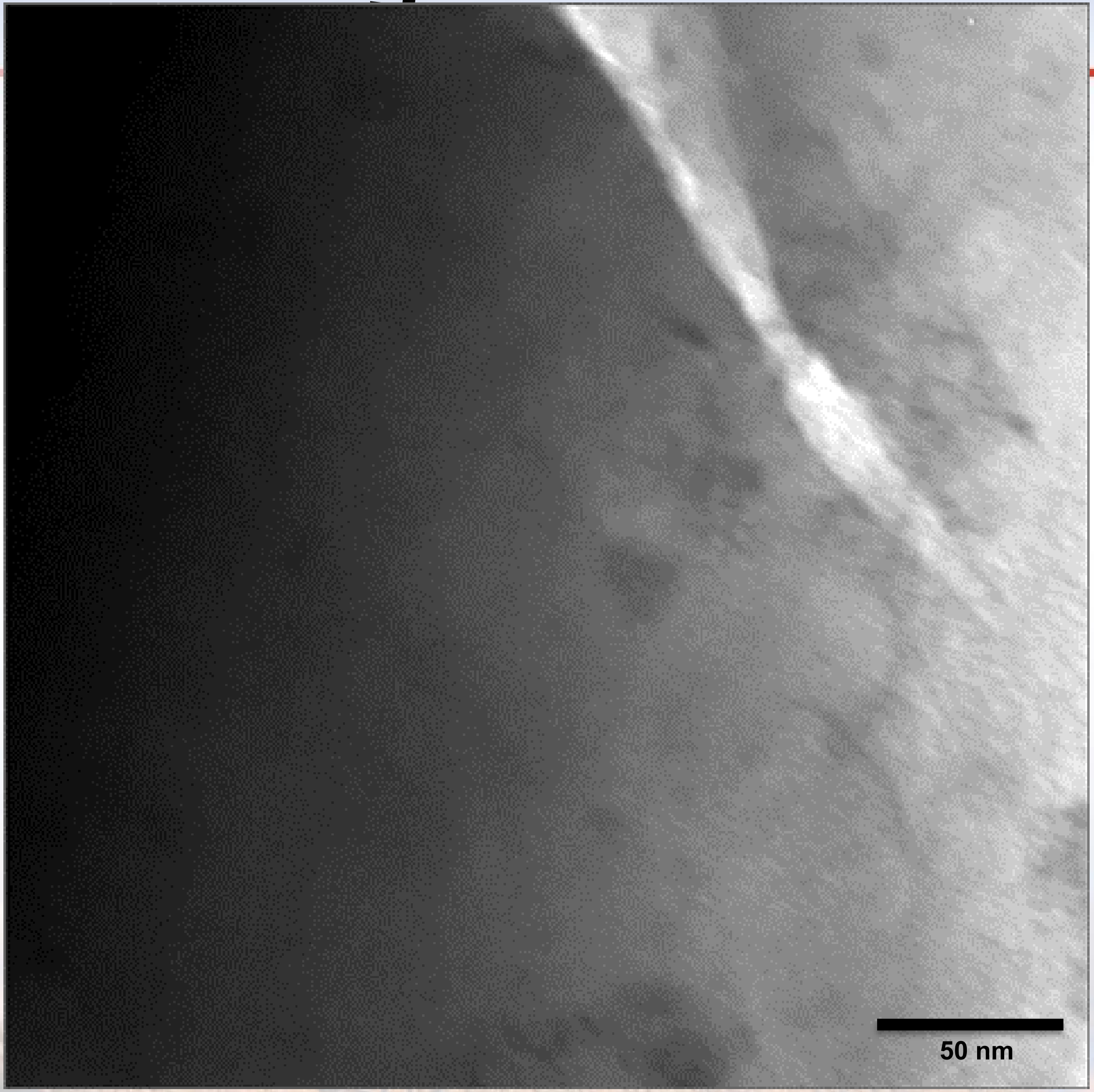
Electron Beam Effects

I recorded video on another nearby particle in the same sample under just the electron beam. Voids did seem to grow during the video, which is 2 min 37 s long.



In-situ Video Showing Void Growth

- Each frame is 1 min of irradiation
- Initially focused on crack because this region had no pre-existing voids.
- Voids seemed to rapidly expand under ion/electron beam irradiation.
- Could not see new void growth, because existing void expansion destroyed sample.



Section 5:
Summary and Planned Experiments

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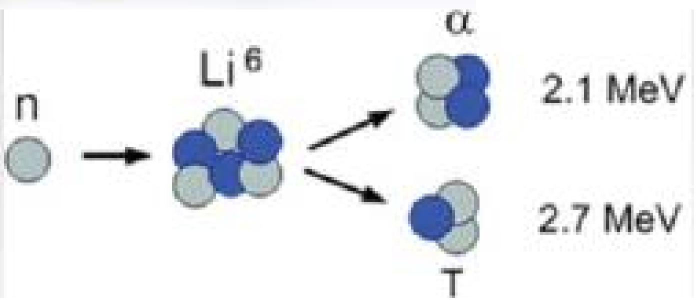
Summary

- **Irradiation Results:**
  - He @ 310°C: voids formed after ~13 min (7.32×10^{16} He/cm^2)
  - He+D_2 @ 310°C: voids formed after ~60 min (1.13×10^{17} He/cm^2, 2.25×10^{17} D/cm^2)
  - He+D_2+Au @ 310°C: Could not find a particle w/o pre-existing voids in this sample. Could not see new void formation because existing voids expanded so much during irradiation.
- **Very difficult to isolate irradiation effects from electron beam effects. The void nucleation and growth we are seeing could be entirely or partially due to the electron beam.**

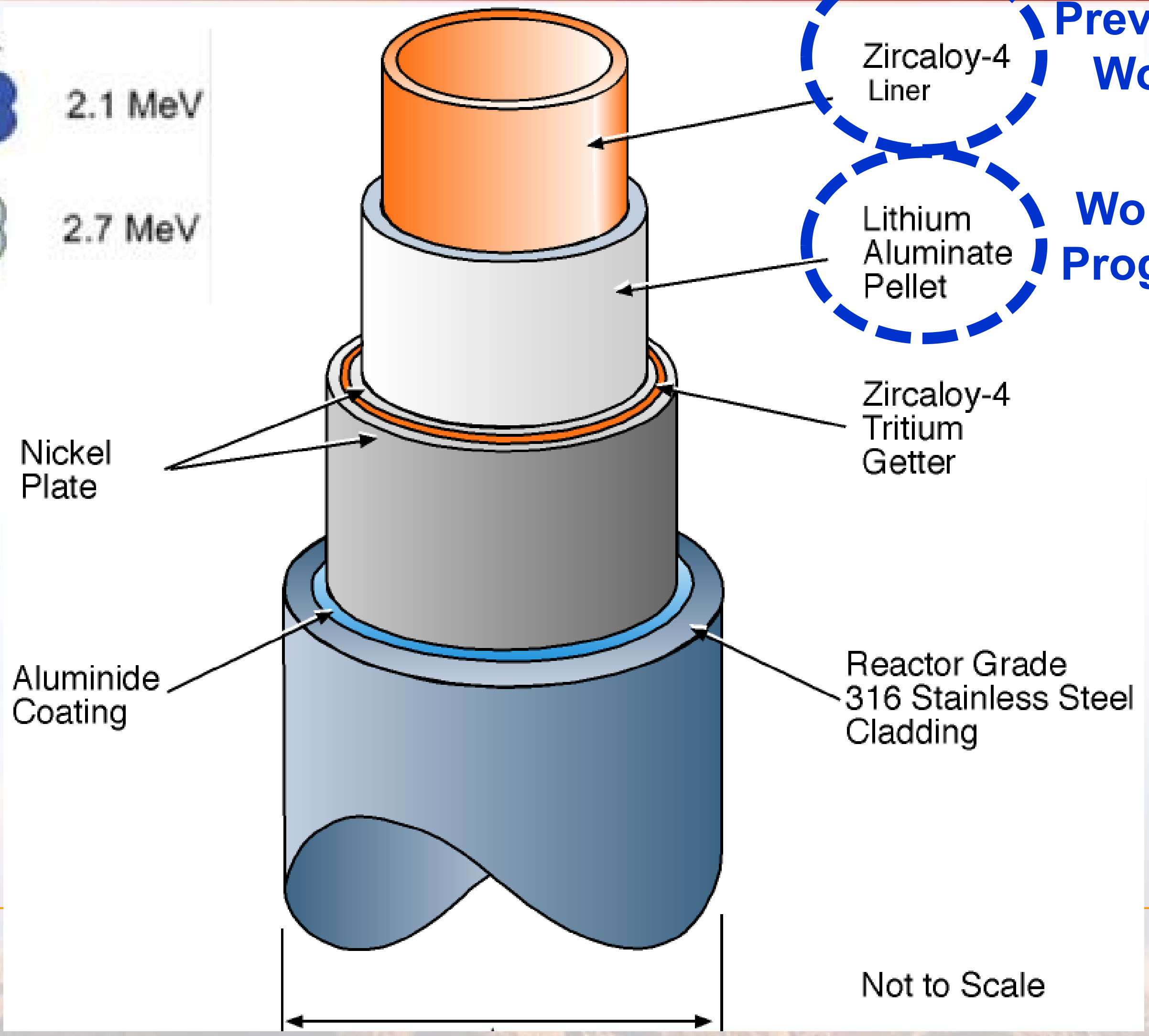
Planned Experiments

- **Isolate electron beam effects:**
  - Repeat He+D_2 @ 310°C experiment with the electron beam off for
    - The entire implantation (imaging only before and after)
    - Most of the implantation, imaging every 30 min
  - See if the void nucleation and growth behavior is the same with less or no electron beam exposure.
- **This work would benefit from bulk irradiation and in-situ irradiation with FIB samples**

Tritium Producing Burnable Absorber Rod (TPBAR)

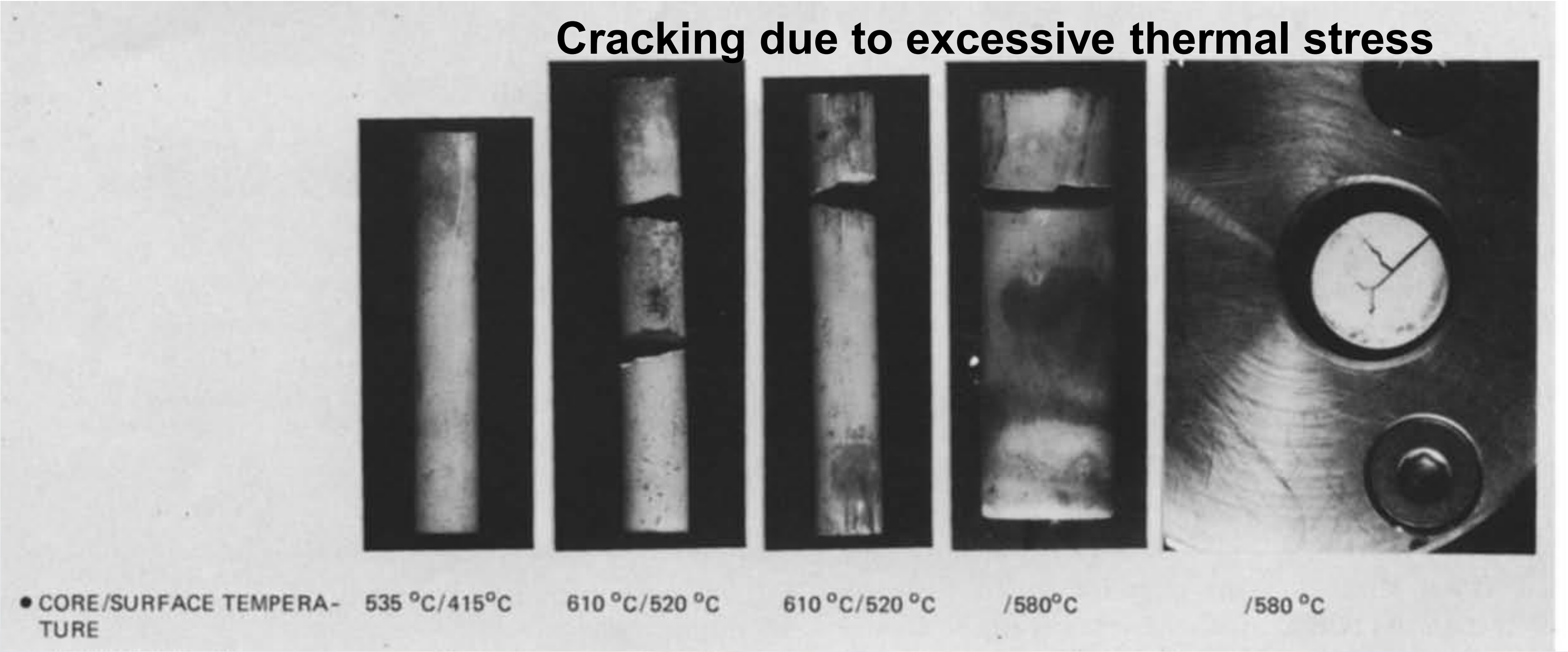


- Displacement Damage
- Helium Implantation
- Tritium Implantation
- Elevated Temperatures



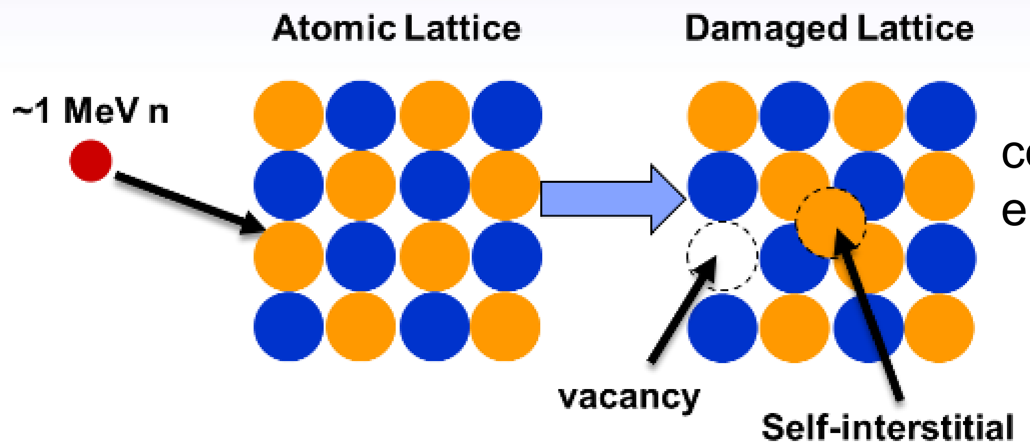
Traditional Experiments use Fast Reactor Irradiations

- In-reactor irradiations of bulk $LiAlO_2$ at high flux test reactors
- Typically quantify **macroscopic** (e.g. porosity, volume, cracking) and **mechanical** property changes

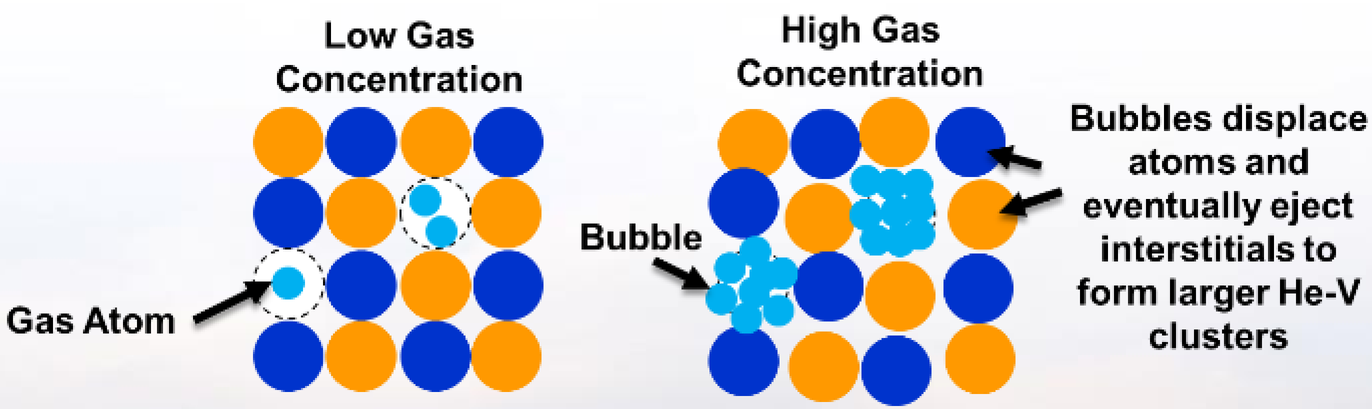


Botter *et al* JNM 160 (1988) 48-57

Fundamental Interaction of Neutron Irradiation Damage and Gas Accumulation May Play a Role



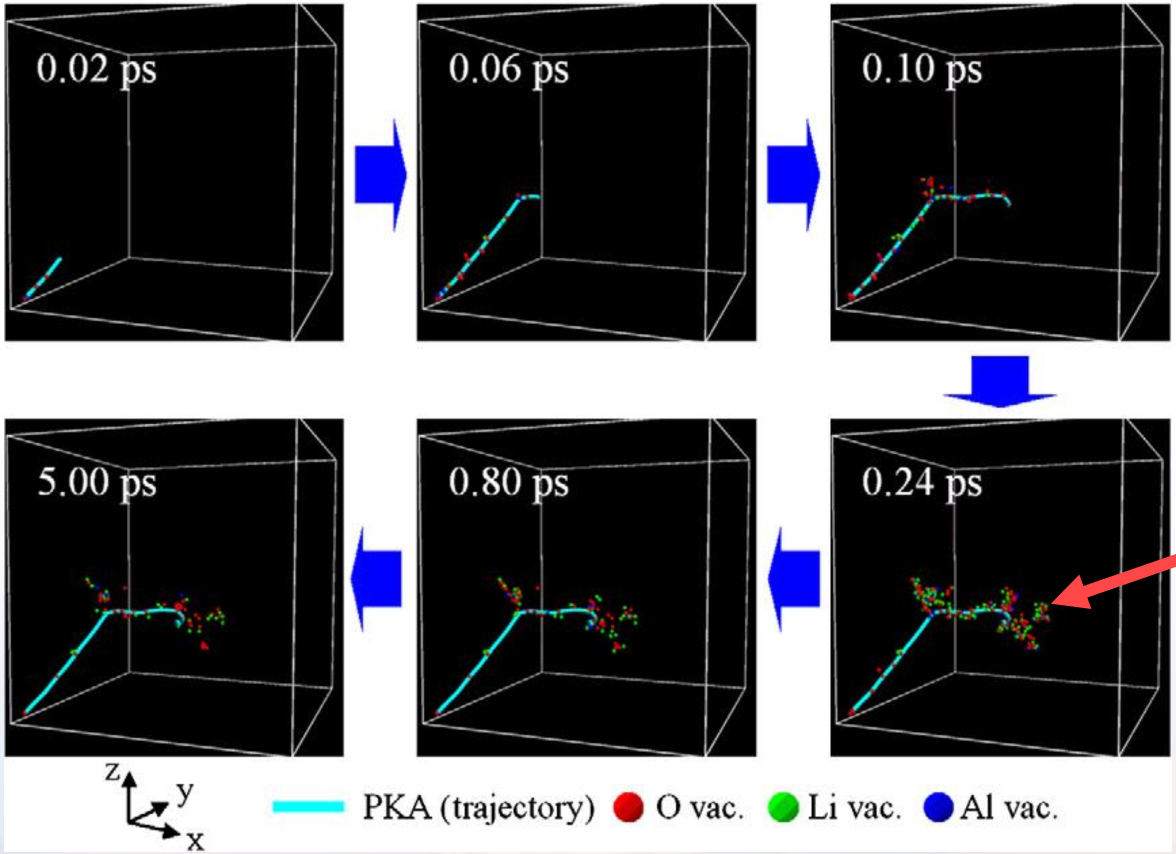
Over time more complicated defects, e.g. dislocations and voids, will form



The constantly changing damage state changes the way gas atoms accumulate in the material

High Energy Ions Can Produce Multiple Defects in Displacement Cascade

MD simulation of displacement cascade in $LiAlO_2$ (PKA = 5 keV)



Tsuchihira *et al* JNM 414 (2011) 44-52

Many defects form due to impact of ONE ion (most eventually recombine)

Accelerated Aging by Ion Beam Irradiation

Benefits

- Predict material behavior in radiation environment from a fundamental point of view
- Isolate specific variables (e.g. ion, damage, gas, temperature).
- Damage that would normally occur over several months or years in a reactor can be simulated in a matter of minutes or hours with an ion accelerator, **without activation**

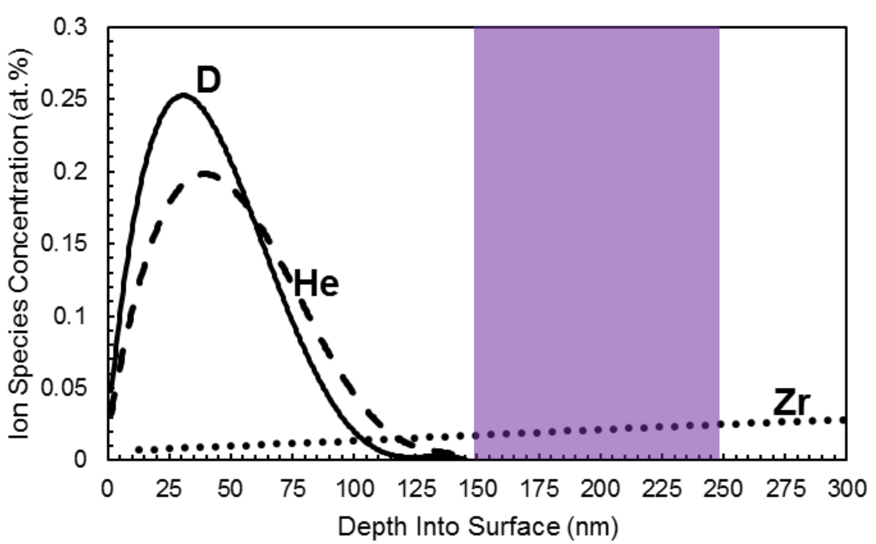
Ion irradiation is used to understand fundamental mechanisms occurring due to radiation damage at the atomic scale.

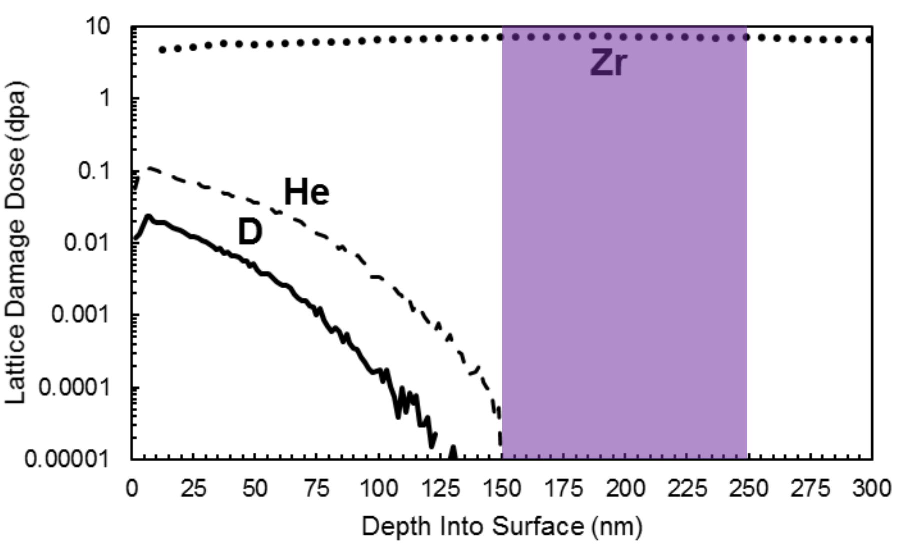
Limitations

- Higher ion flux than reality
- Difficult to predict dose rate effects
- Injected ions can influence the damage properties or chemistry of material
- Only irradiates surface layers

Relative Damage and Gas Distributions in Zr

- Ion concentration and damage are scaled based on the irradiation time
- Most Zr travels through entire TEM foil
- Zr produces two orders of magnitude higher damage than He
- These experiments were aimed at observing kinetic effects in-situ, so experiments were run overnight and the exact gas concentrations/damage doses are not all known

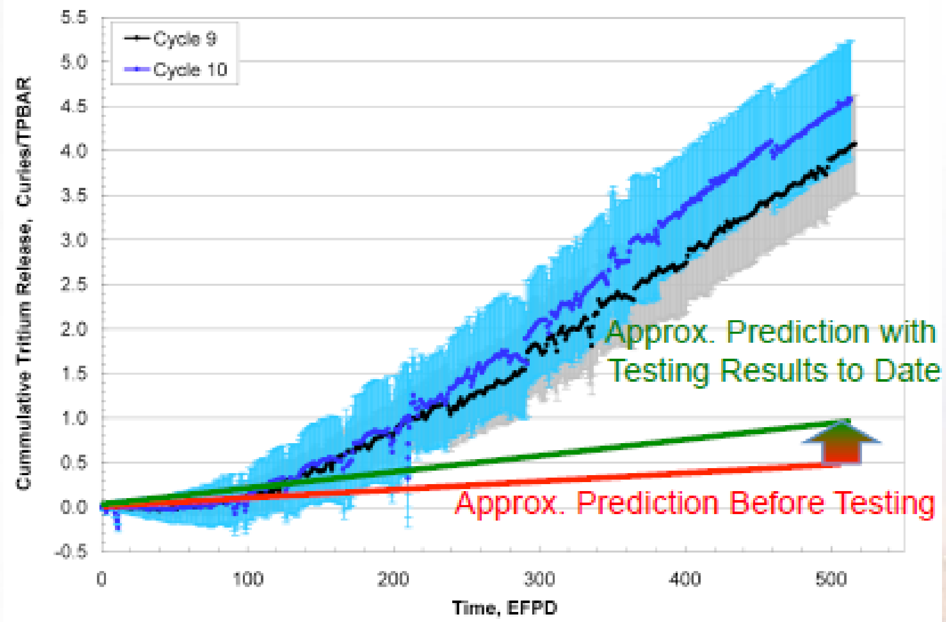




SRIM calculations of 10 keV He, 5 keV D, 3 MeV Zr implantation depth and damage

Understanding Tritium Permeation in TPBAR

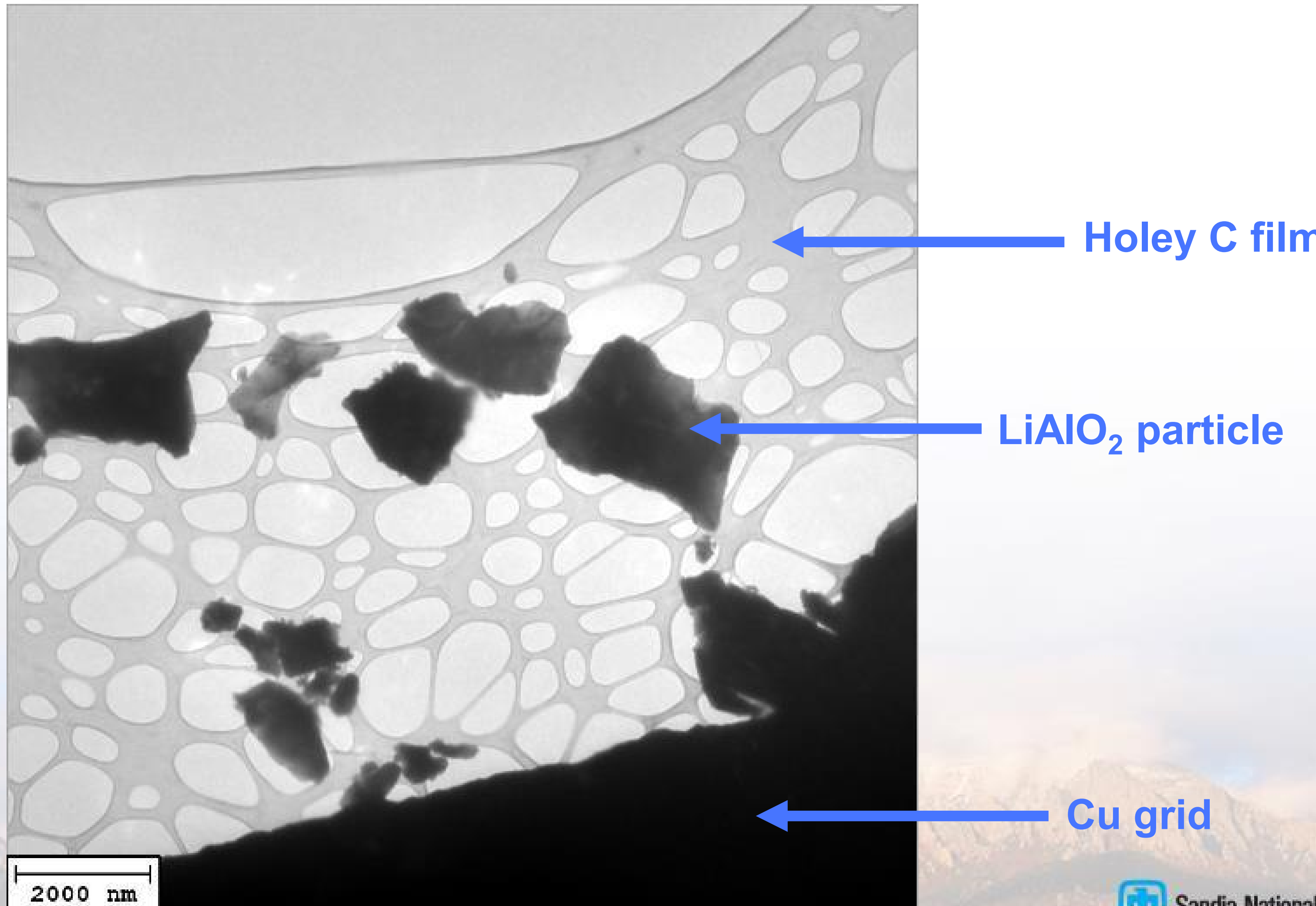
- TPBAR ^{3}H permeation is higher than predictive performance models
  - In 2004, during Cycle 6, the predicted levels were ~0.5 Ci/TPBAR/cycle and actual levels were ~4 Ci/TPBAR/cycle (0.04% of total ^{3}H produced)
- Mechanisms responsible for differences between predictions and observations are not well understood
- Currently building an understanding of fundamental ^{3}H-defect interactions



Burkes, Senor, Longoni and Johns, TFG Meeting 2016, Rochester, NY

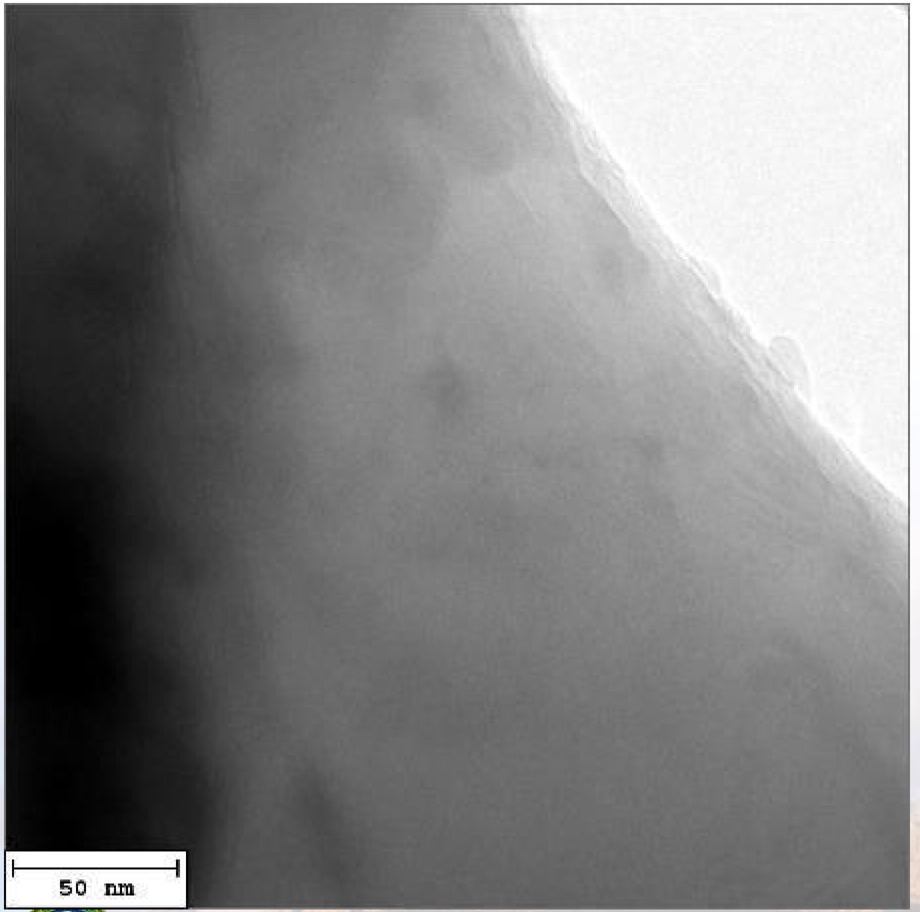
In-Progress TPBAR Work: $LiAlO_2$ Pellet

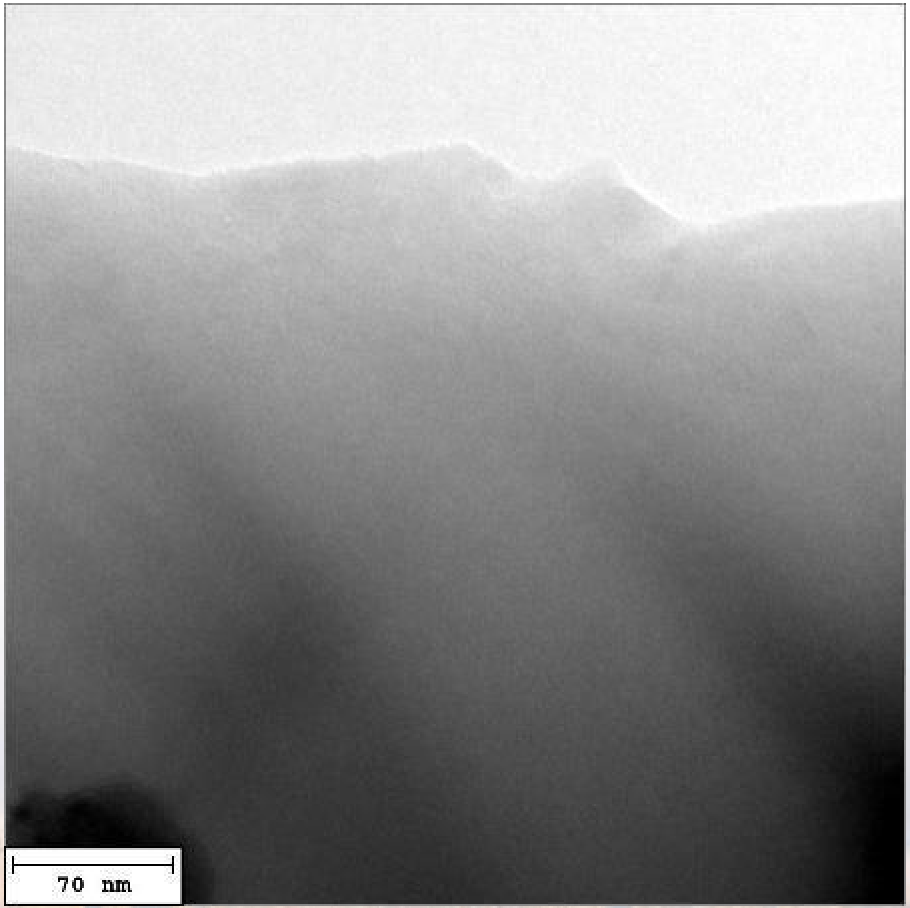
$LiAlO_2$ Powder Deposited on a TEM Grid



Current In-Progress TPBAR Work: $LiAlO_2$ Pellet

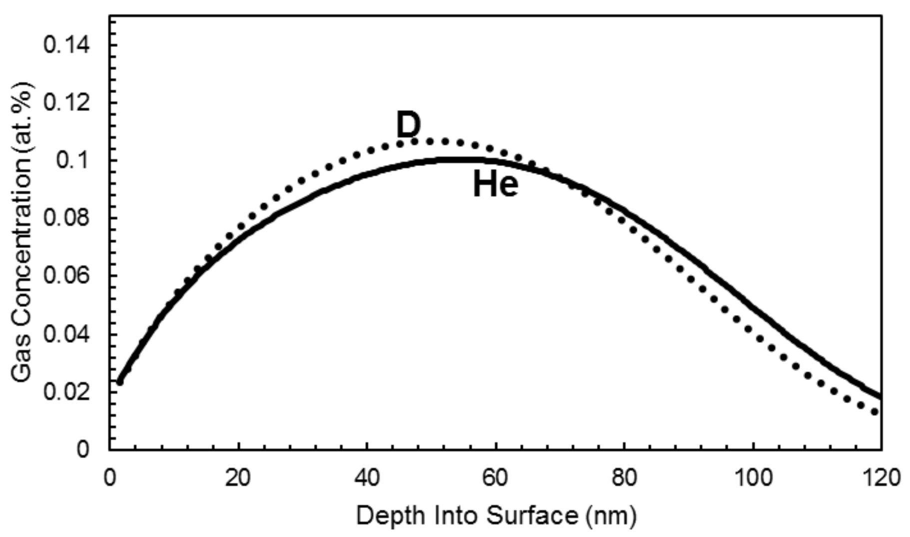
Some particles contain regions thin enough for TEM imaging of bubbles

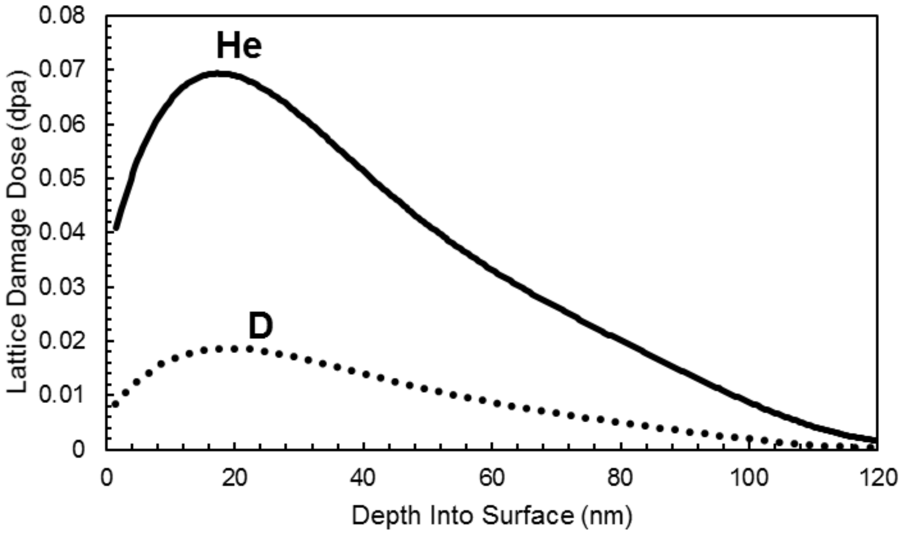




Understanding Synergy Between Damage and Gas Bubble Formation

- Helium is known to form bubbles in materials, especially when defect traps are present
- May be a synergy between He and ^{3}H behavior, so we are planning dual beam implantations using ^{2}H to simulate ^{3}H
  - He bubbles may form and affect ^{2}H diffusion or trapping
- Bubble nucleation will be observed *in-situ* with the TEM





SRIM calculations of 10 keV He and 5 keV D implantation depth and damage