

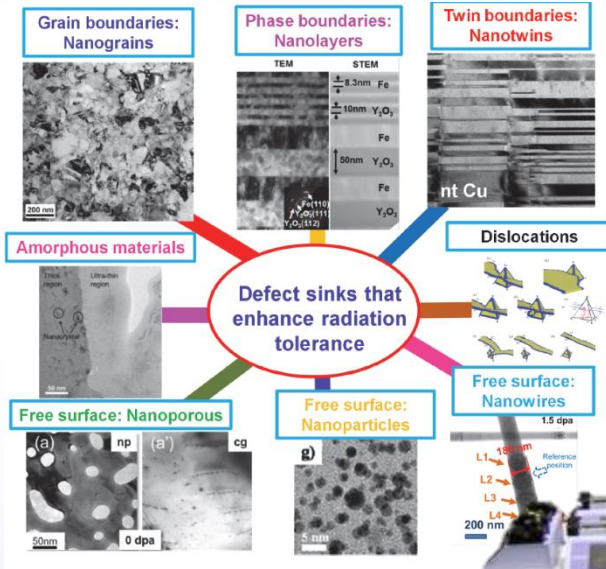
# Nanomaterials Evolution in Radiation Environment

SAND2017-5542PE

K. Hattar

Ion Beam Lab at Sandia National Laboratories

May 17, 2017

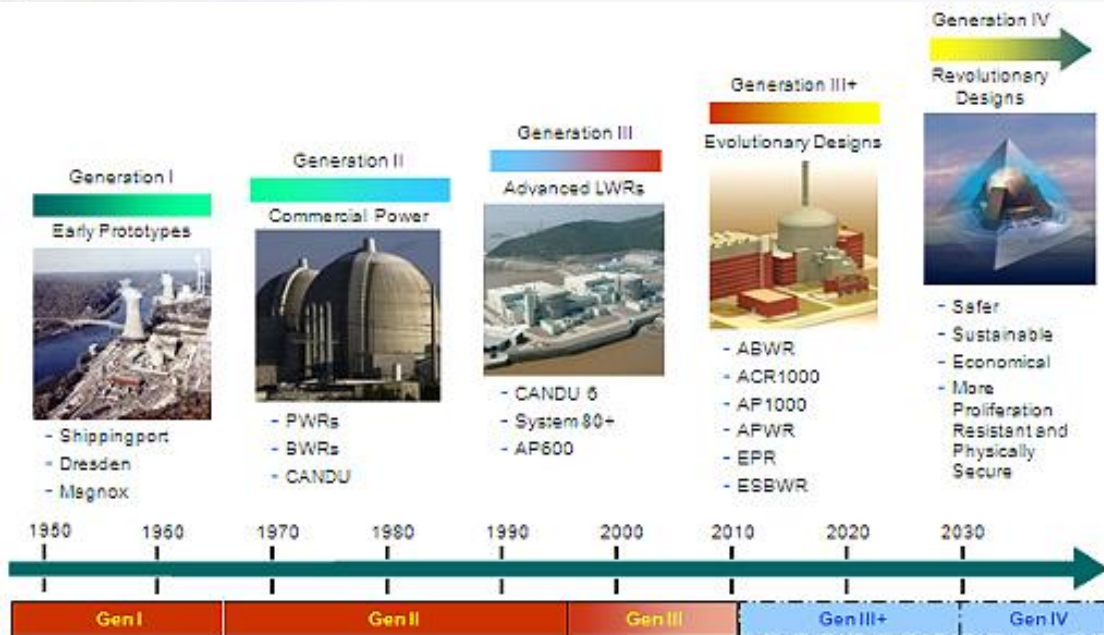


## Collaborators:

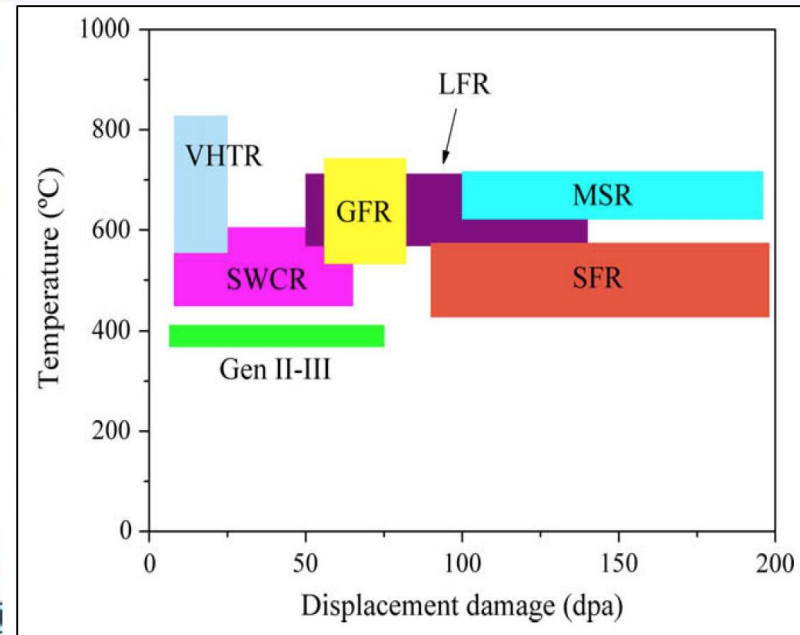
- IBL: C.M. Barr, S.A. Briggs, D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, M.T. Marshall, B. Muntifering, S.H. Pratt, & P. Price
- Sandia: M. Abere, B. Boyce, T.J. Boyle, R. Dingreville, R.F. Hess, A.C. Kilgo, B.E. Klamm, W.M. Mook, J.D. Puskar, J.A. Scott, & J.A. Sharon
- External: A. Aitkaliyeva, H. Bei, P.J. Ferreira, K.J. Ganesh, E.P. George, D. Gross, P. Hosemann, J. Kacher, S. Maloy, A. Minor, J. Qu, S. Rajasekhara, I.M. Robertson, D. Stauffer, & Hysitron Inc.

This work was partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

# Environmental Requirements for Current and Future Reactors



[ec.europa.eu/research/energy/euratom](http://ec.europa.eu/research/energy/euratom)



Lambert, T. and Nghiem, X.H., 2015. PAM Review: Energy Science & Technology, 1, pp.90-108.

- The environment in current reactors are already complex and harsh
- Future proposed reactors only require harsher conditions



# Investigating the **nm** Scale to Understand the **km** Scale



1 nm

1  $\mu$ m



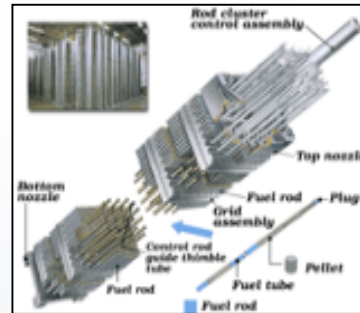
1 mm

1 m



1 km

10<sup>7</sup> m



*In situ* Ion Irradiation TEM (I<sup>3</sup>TEM)



Ion Beam Lab (IBL)



Sandia National Laboratories





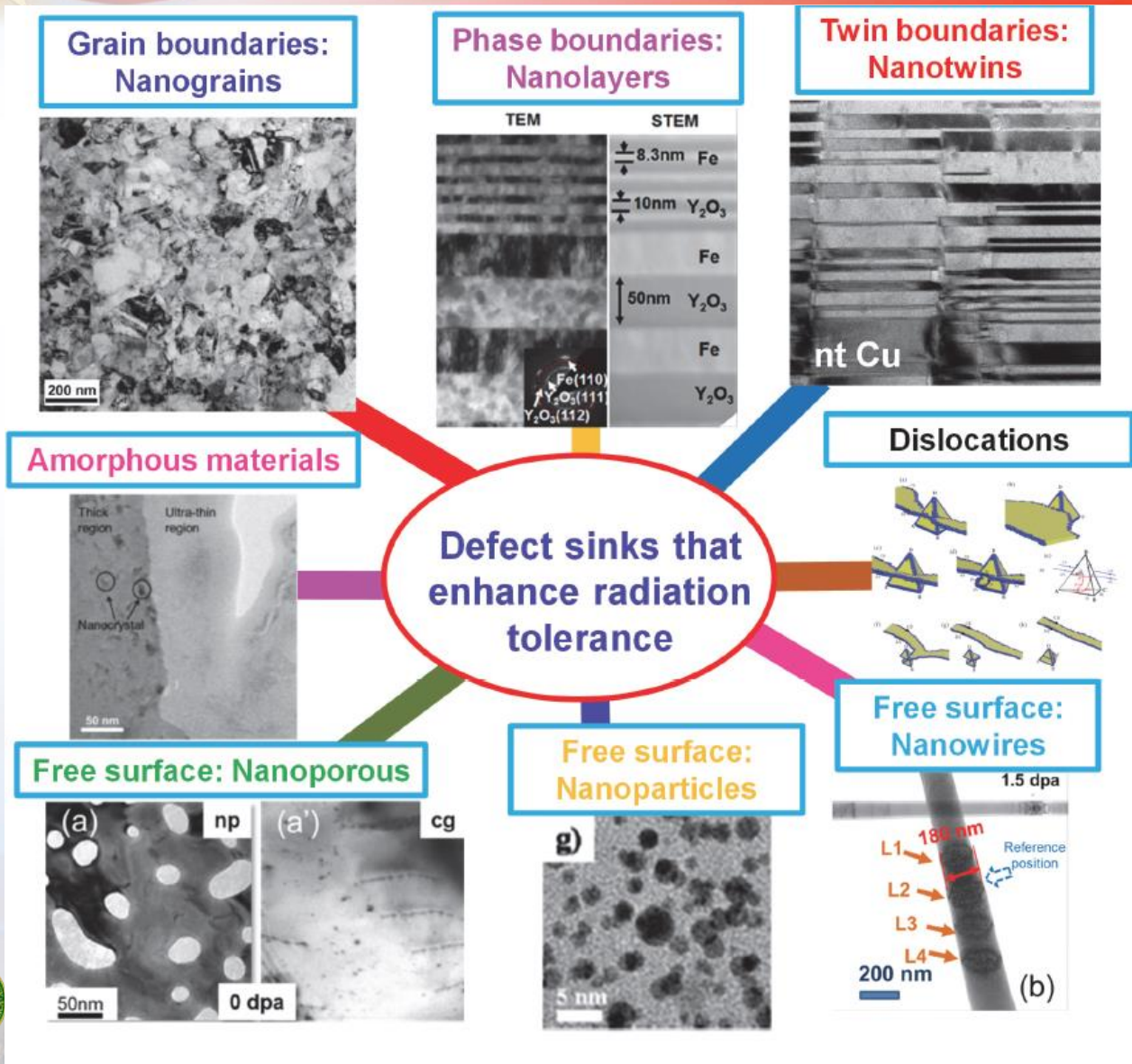
# Outline

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- 1. How Does Nanostructuring Effect a Materials Radiation Tolerances?**
- 2. Can We Tailor Boundaries/Interfaces to Control Radiation Tolerances?**
- 3. Can We Explore Other Aspects of the Complex Environment Present in Modern and Future Reactors Using *In situ* Techniques?**



# Nanomaterials Proposed for Improving Radiation Tolerance



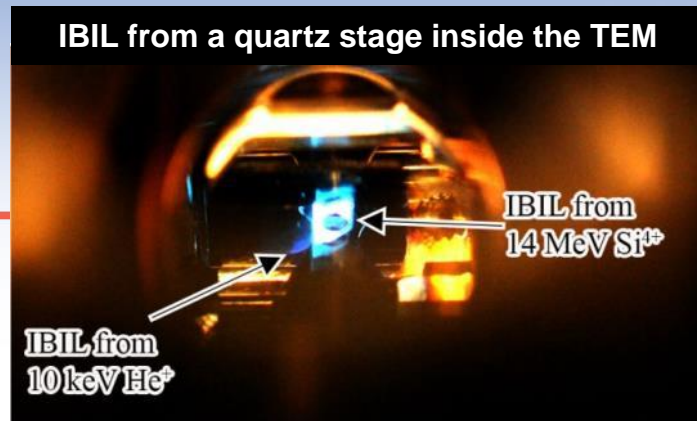
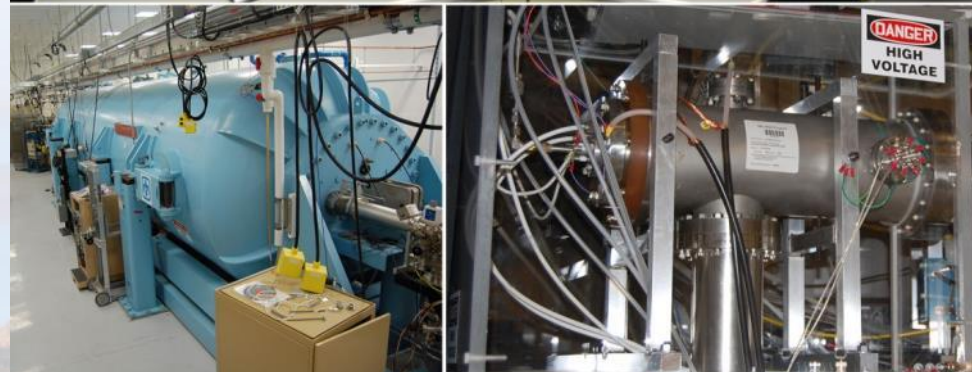
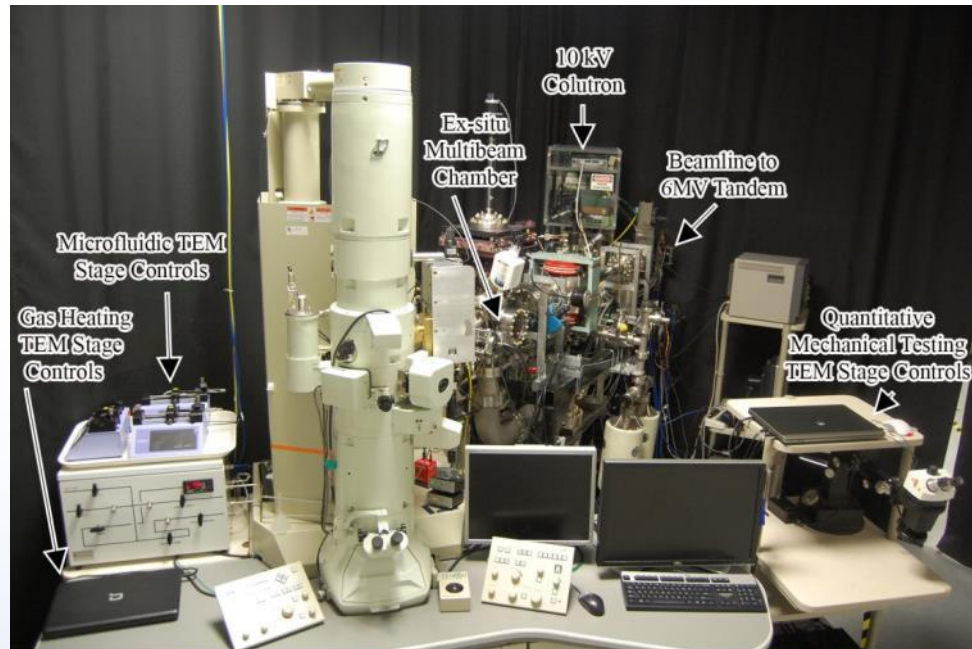
[Zhang et al., PMS 2017]



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

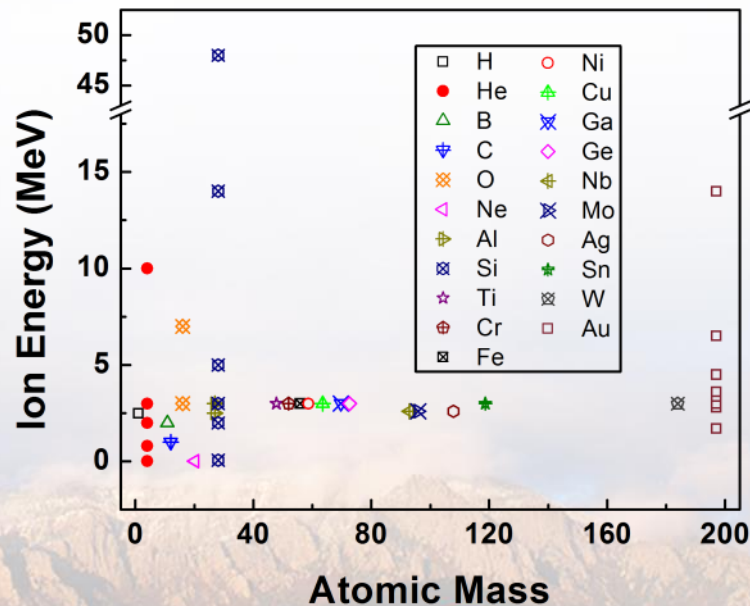
Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem



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

Ion species & energy introduced into the TEM



# 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

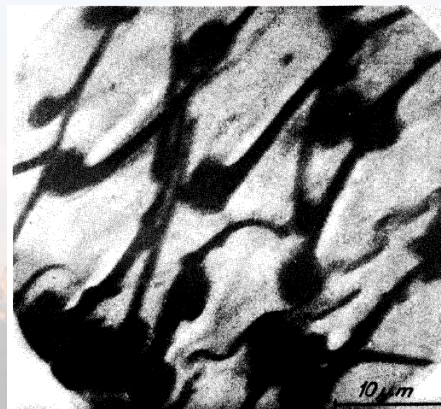


Fig. 6: Wing surface of the house fly.  
(First internal photograph,  $U = 40$  kV,  $M_x = 2200$ )  
(Dietz, E., and Müller, H.O.: Z. Wiss. Mikroskopie 52, 53-57 (1955))

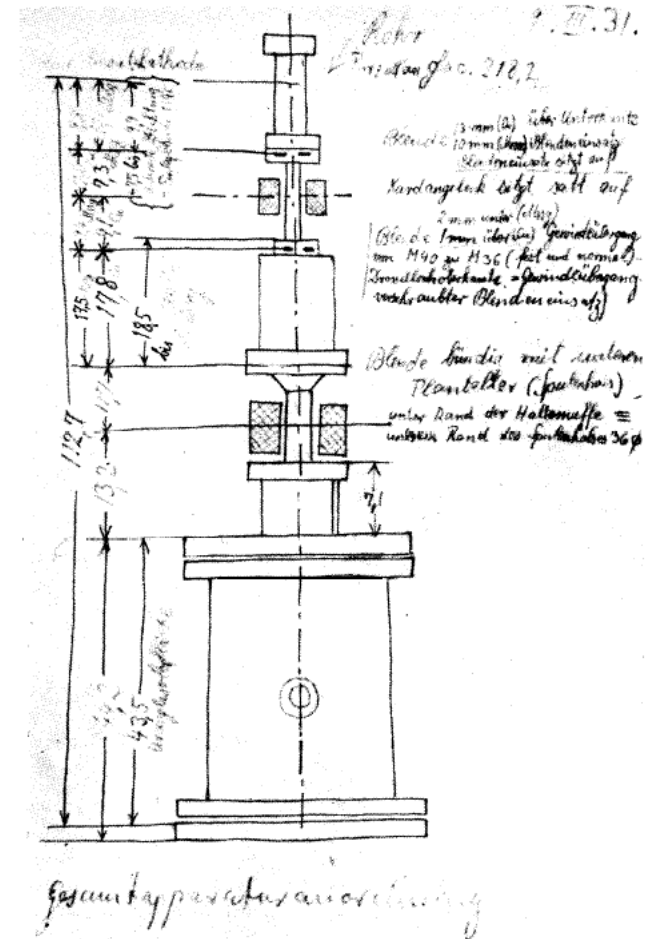


Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].





# Outline

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3. Can We Explore Other Aspects of the Complex Environment Present in Modern and Future Reactors Using *In situ* Techniques?

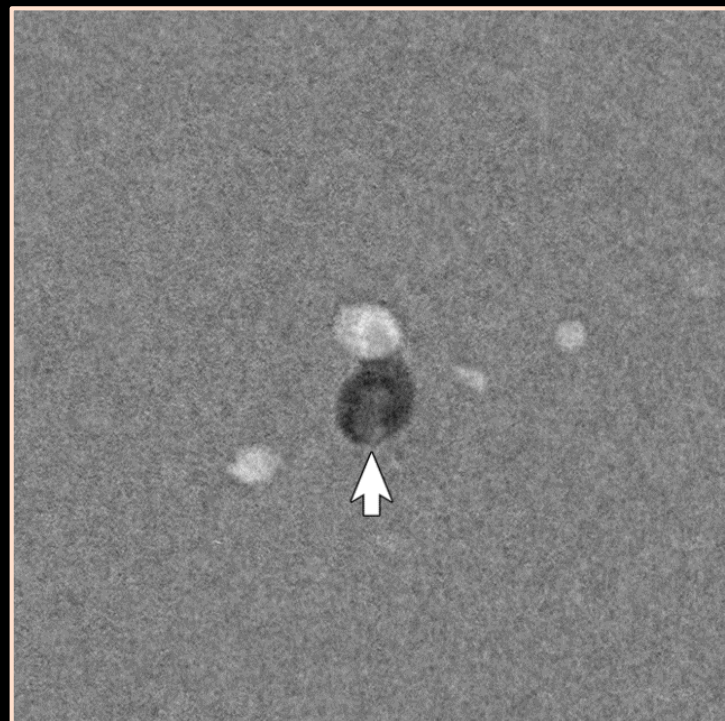
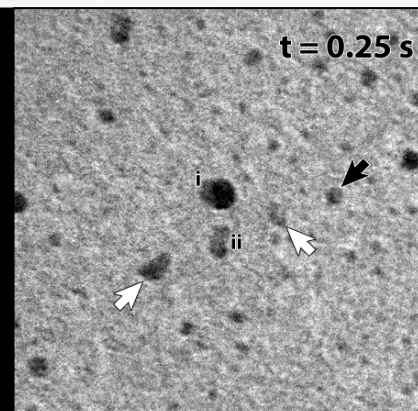
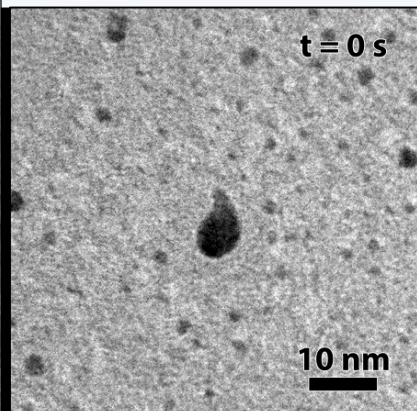
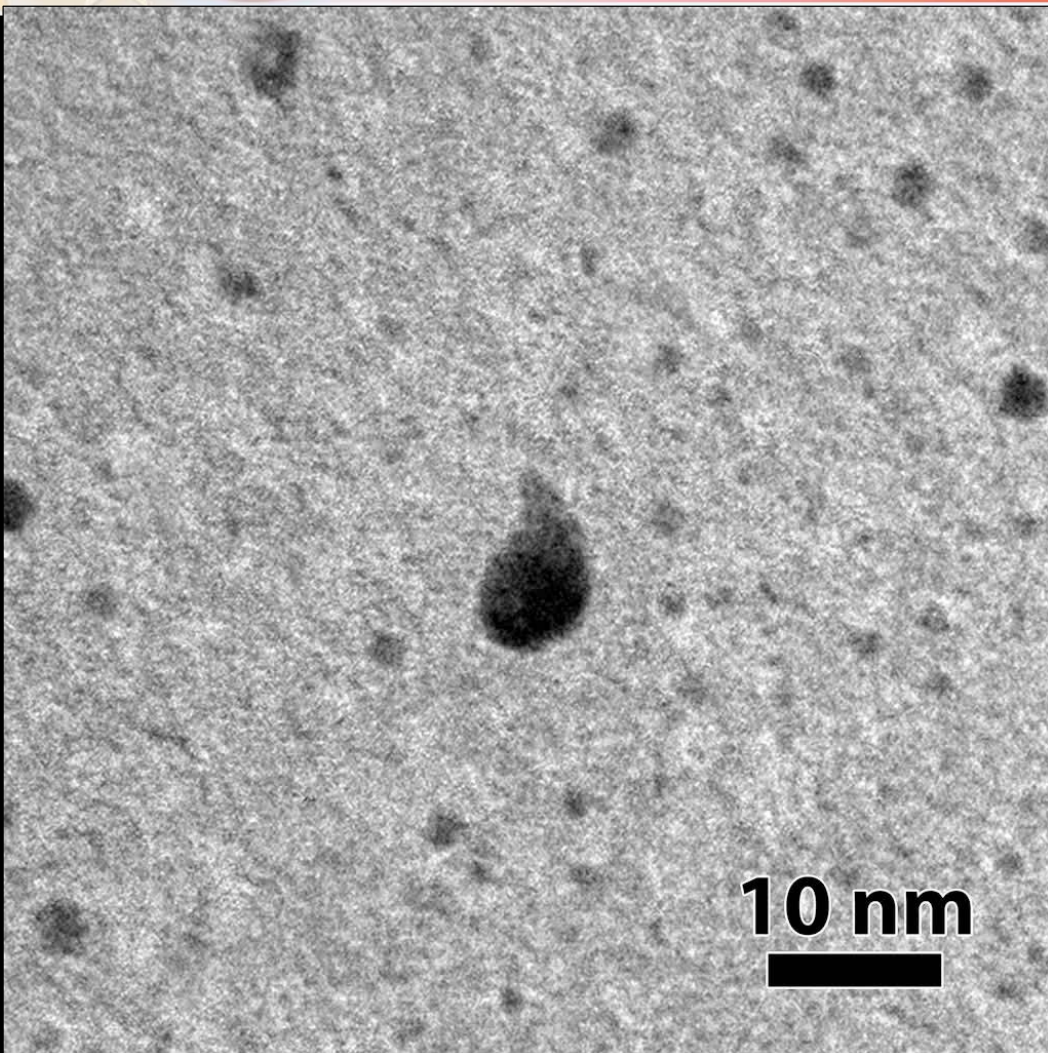




# Single Ion Strikes:

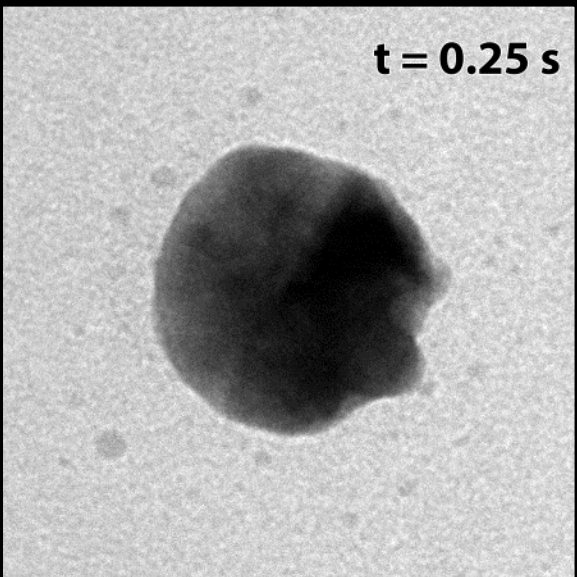
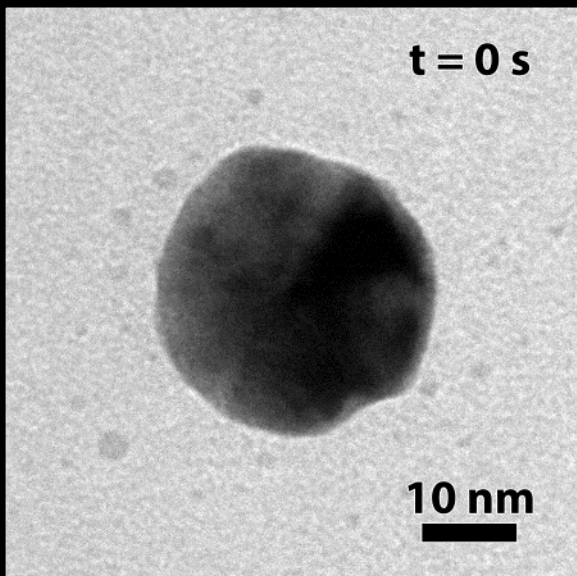
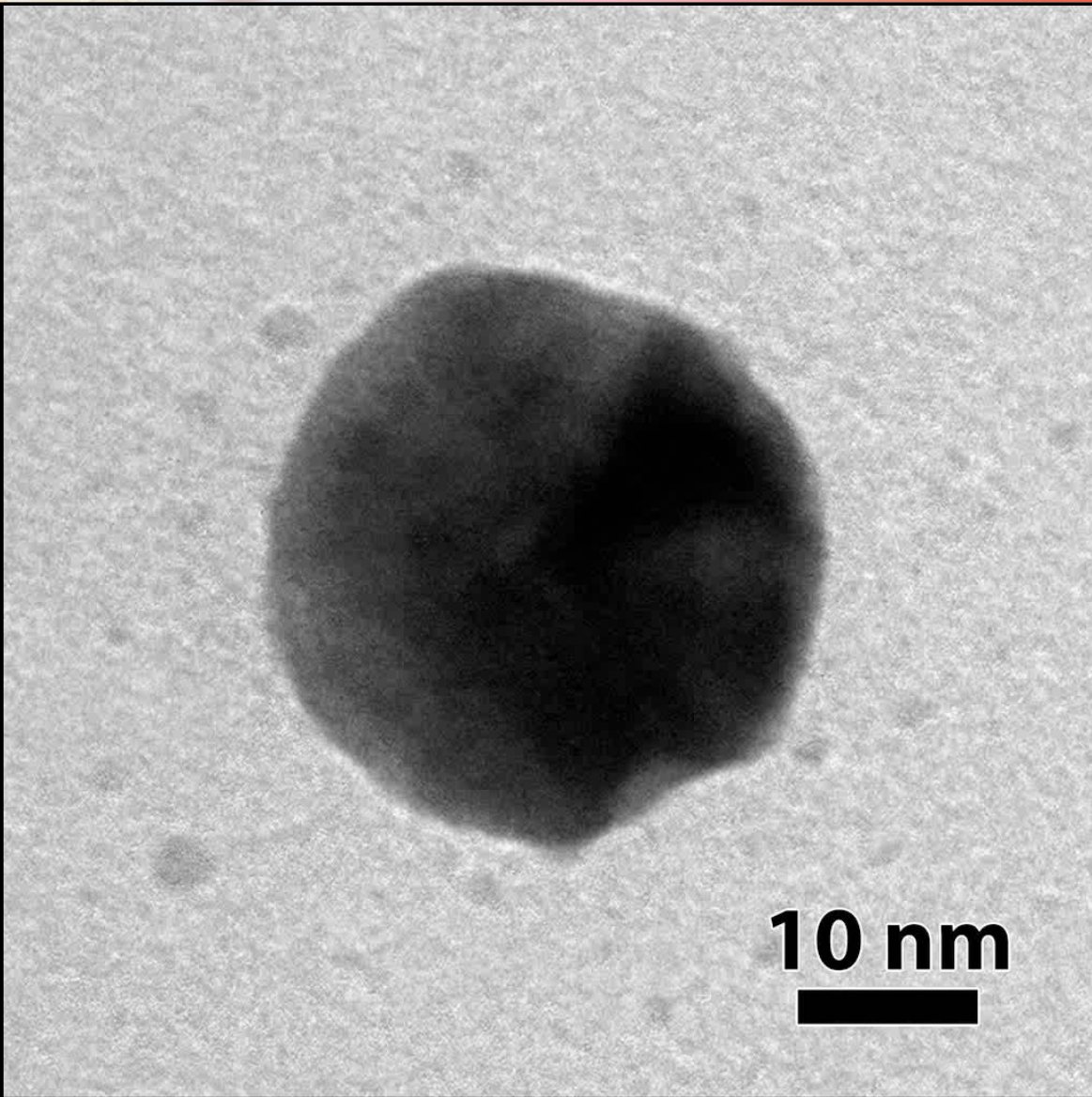
## 46 keV Au<sup>1+</sup> ions into 5 nm Au nanoparticles

Collaborator: D.C. Bufford



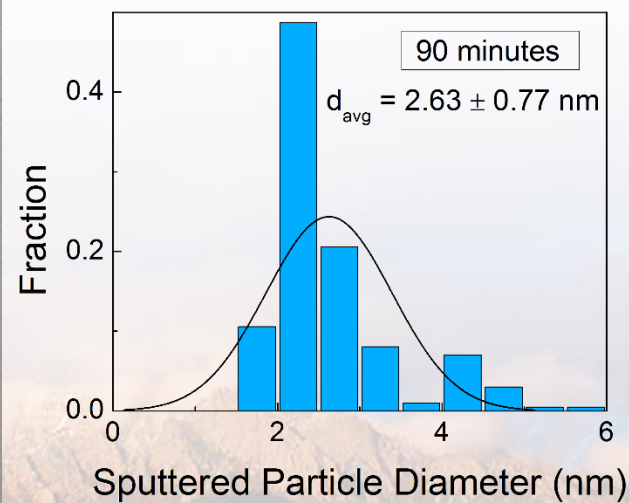
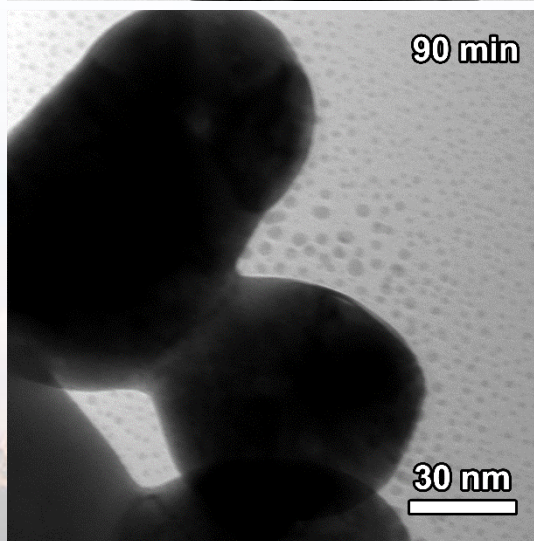
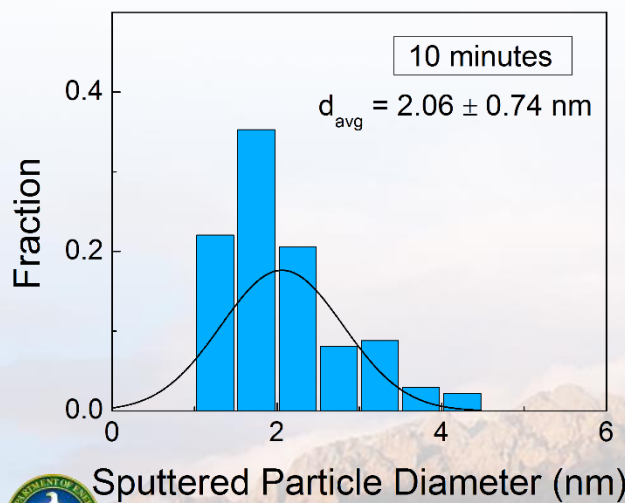
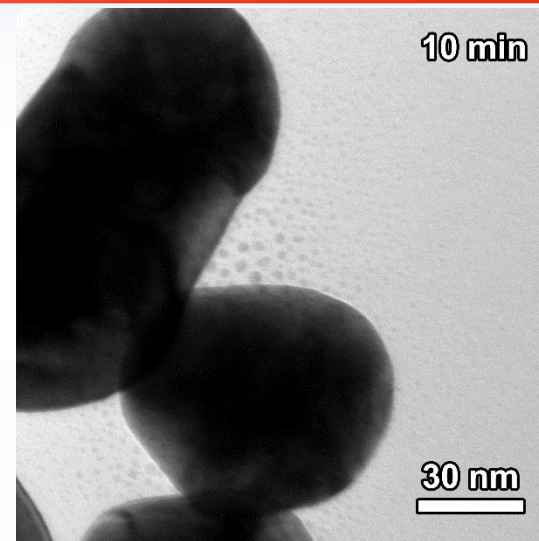
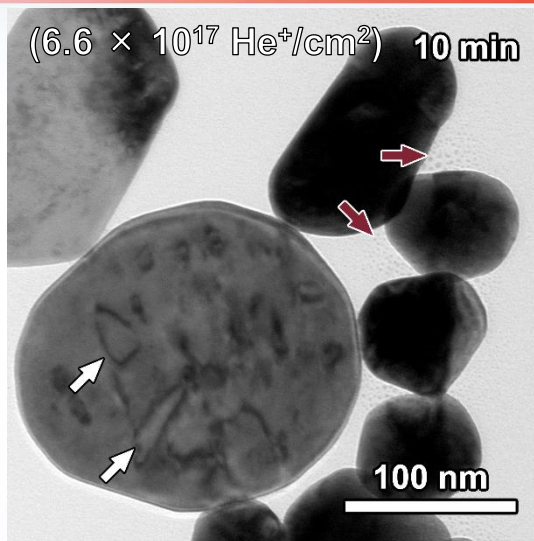
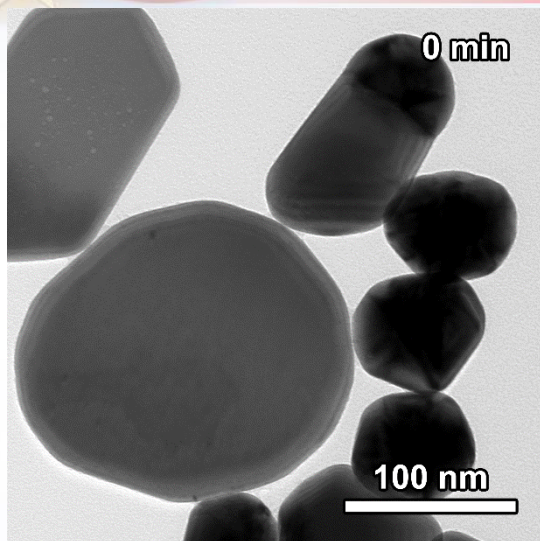
# Single Ion Effects with 46 keV Au<sup>1+</sup> ions: 20 nm

Collaborator: D.C. Bufford



# Formation of Dislocation Loops & Sputtered Particles due to He implantation

Collaborators: D.C. Bufford, S.H. Pratt & T.J. Boyle



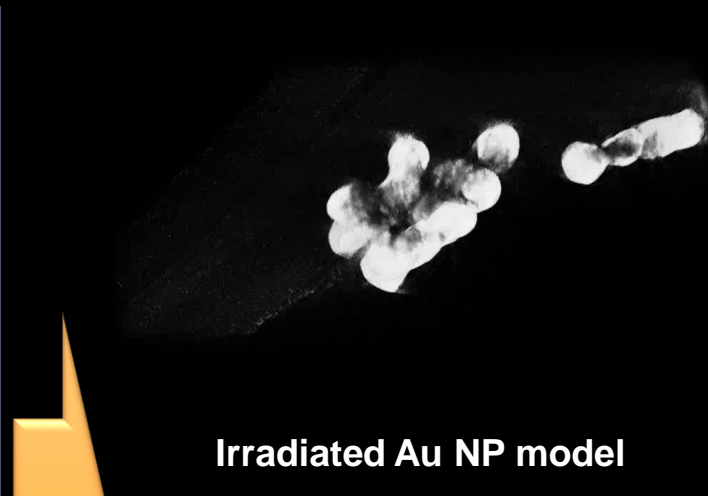
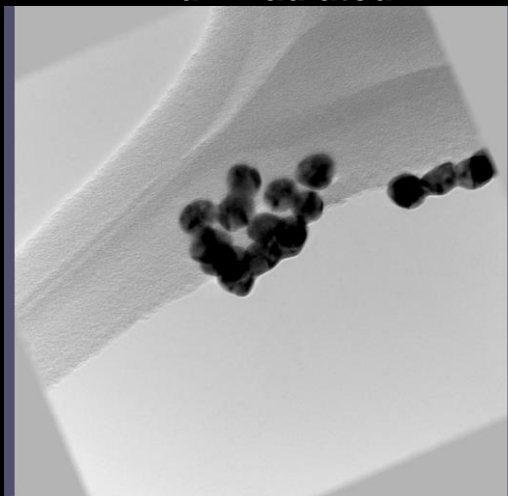
# Electron Tomography Provides 3D Insight

Collaborators: S.H. Pratt & T.J. Boyle

*In situ* Ion Irradiation TEM (I<sup>3</sup>TEM)

Aligned Au NP tilt series -  
unirradiated

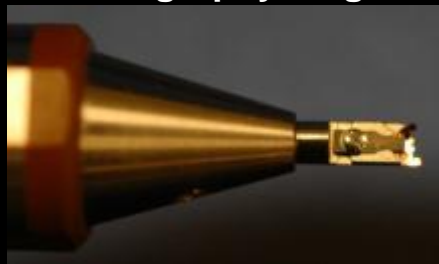
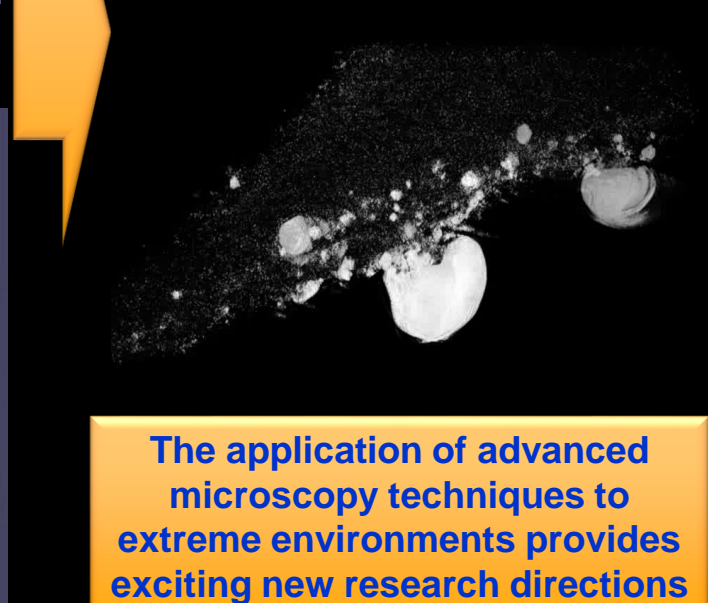
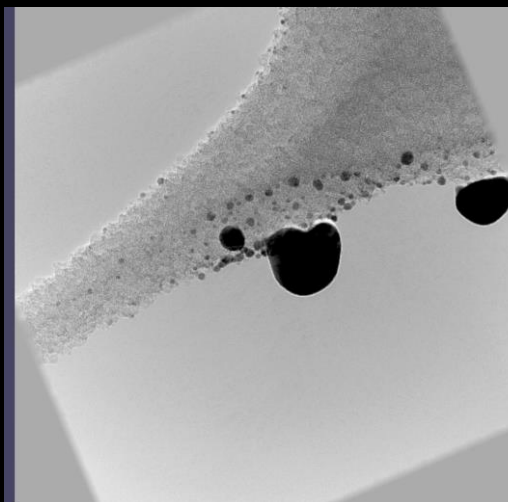
Unirradiated Au NP model



Hummingbird  
tomography stage

Aligned Au NP tilt series -  
irradiated

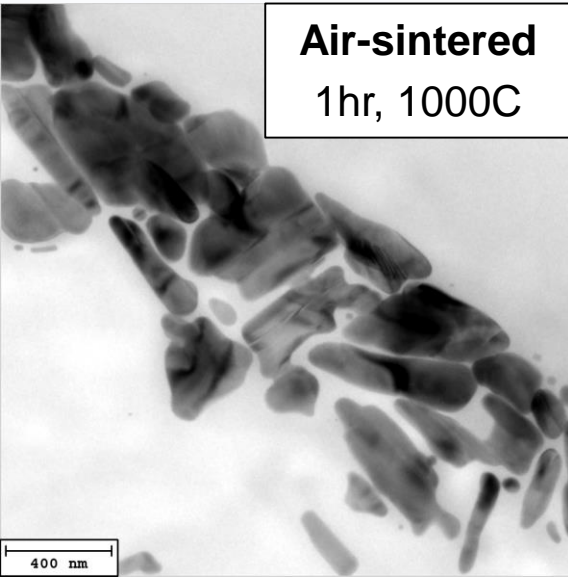
Irradiated Au NP model



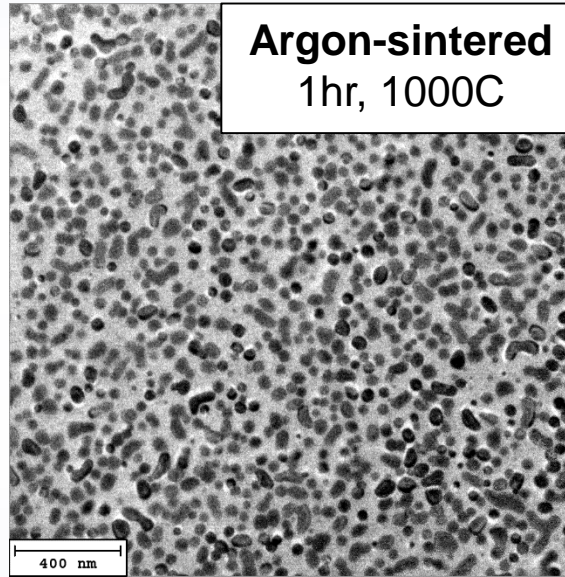
The application of advanced  
microscopy techniques to  
extreme environments provides  
exciting new research directions

# Uranium Oxide Formation in Different Sintering Environments

Collaborators: S.A. Briggs, R. Hess, and B. Klamm



**Air-sintered**  
1hr, 1000C

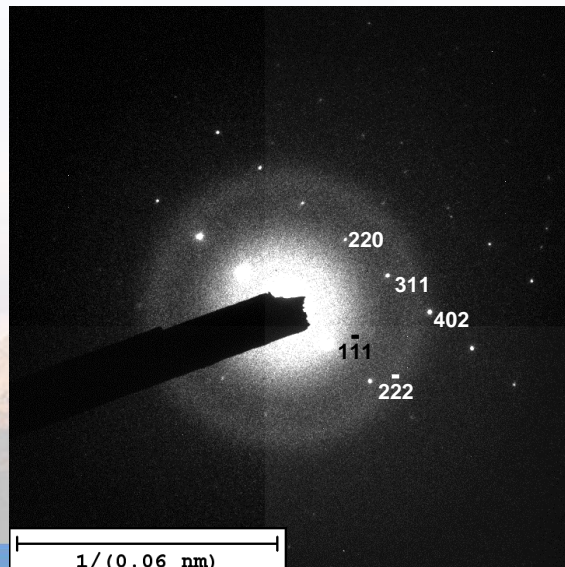
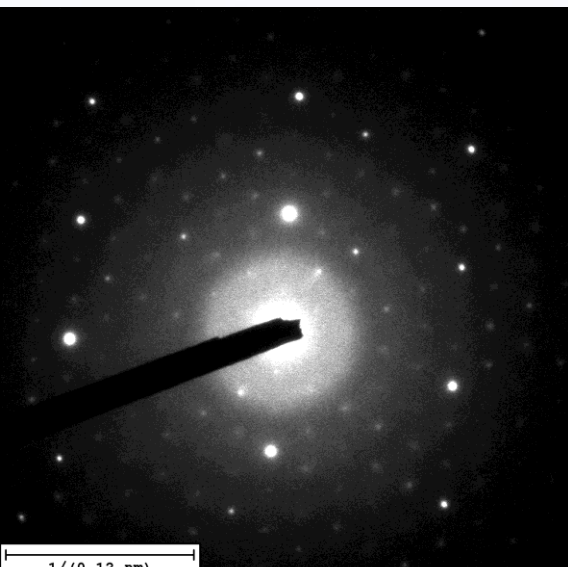


**Argon-sintered**  
1hr, 1000C

Nanoparticles prepared from solution of  $\text{UO}_2(\text{NO}_3)_2$ , PEI, and EDTA

Ar-sintered specimen phase diffraction patterns map to fluorite/FCC structure characteristic of  $\text{UO}_2$

- Larger lattice parameter suggests hypostoichiometric uranium-dioxide phase ( $\text{UO}_{2-x}$ )



Air-sintered specimens do not map to fluorite/FCC

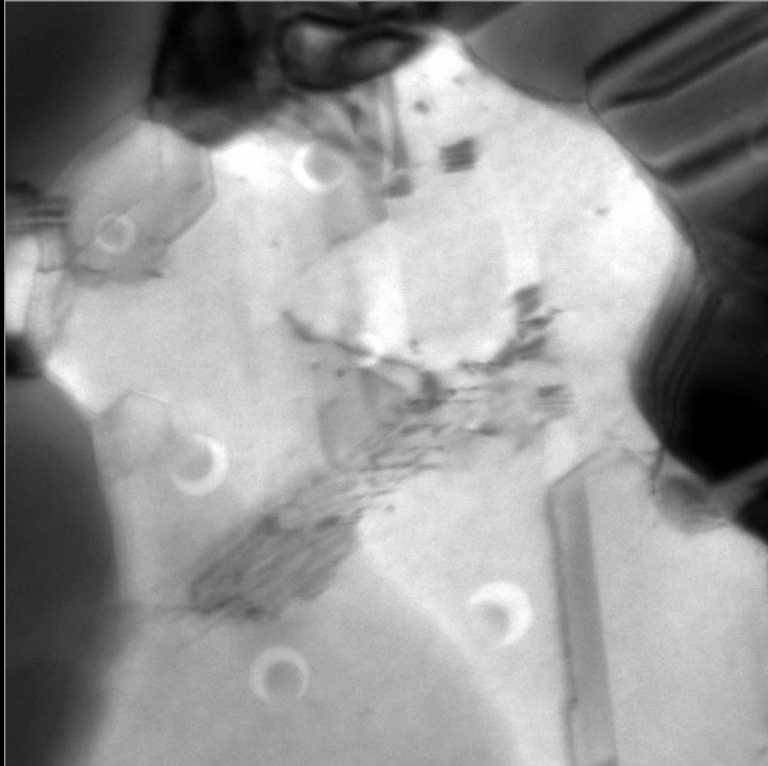
- Likely a hyperstoichiometric uranium-oxide phase ( $\text{U}_3\text{O}_8$ ,  $\text{U}_4\text{O}_9$ )

Characterization of ion-irradiation response is work-in-progress

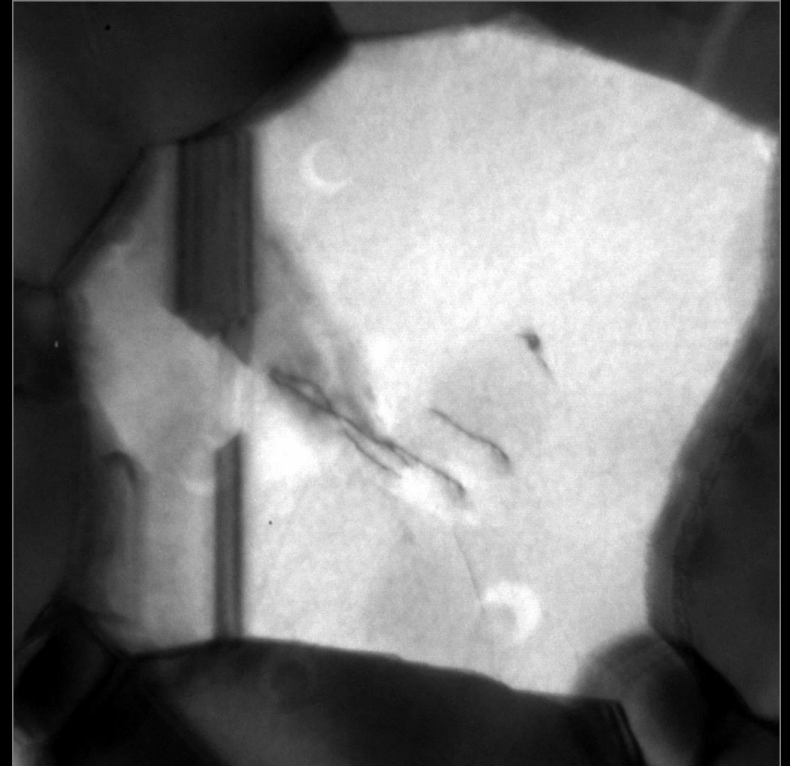
# Dose Rate Effects

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

$7.9 \times 10^9$  ions/cm<sup>2</sup>/s



$6.7 \times 10^7$  ions/cm<sup>2</sup>/s



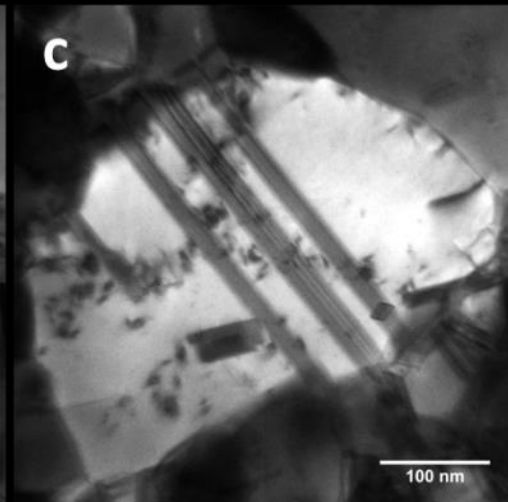
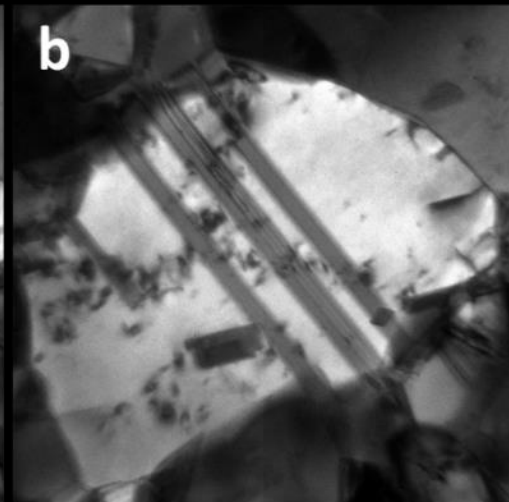
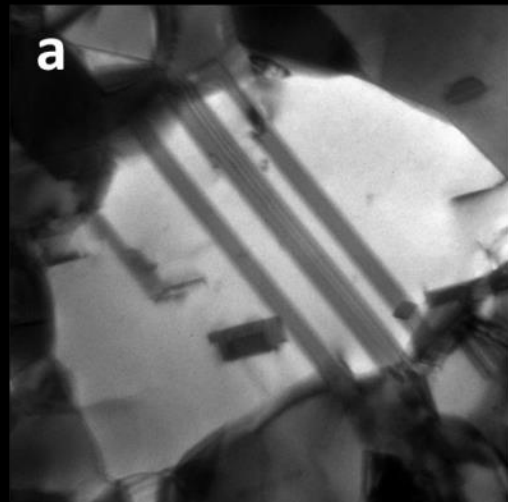
**VS**

Improved vibrational and ion beam stability permits us to work at 120kx or higher permitting imaging of single cascade events

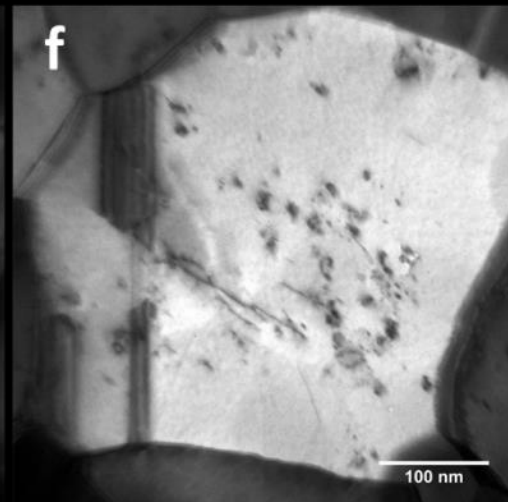
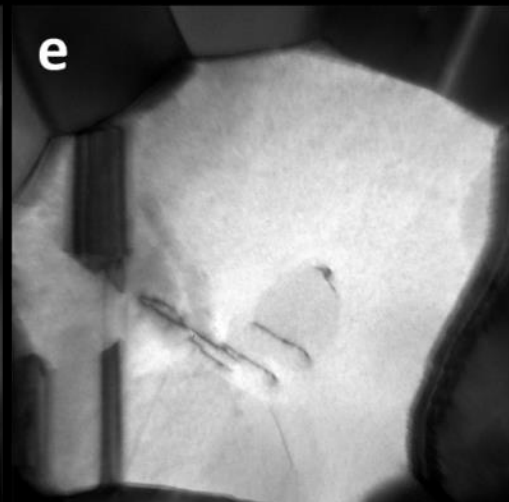
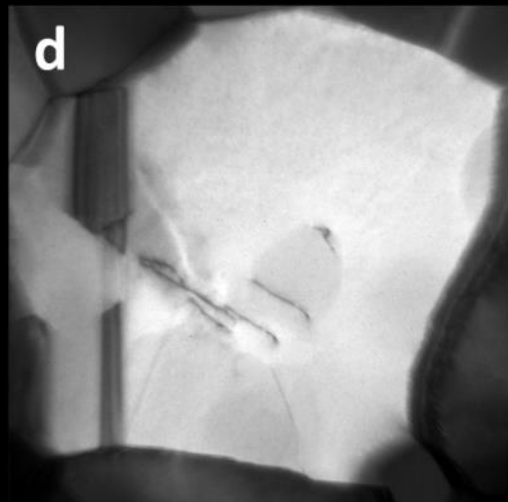
# *In situ* Successive Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor

Successive  $\text{Au}^{4+}$  then  $\text{He}^{1+}$



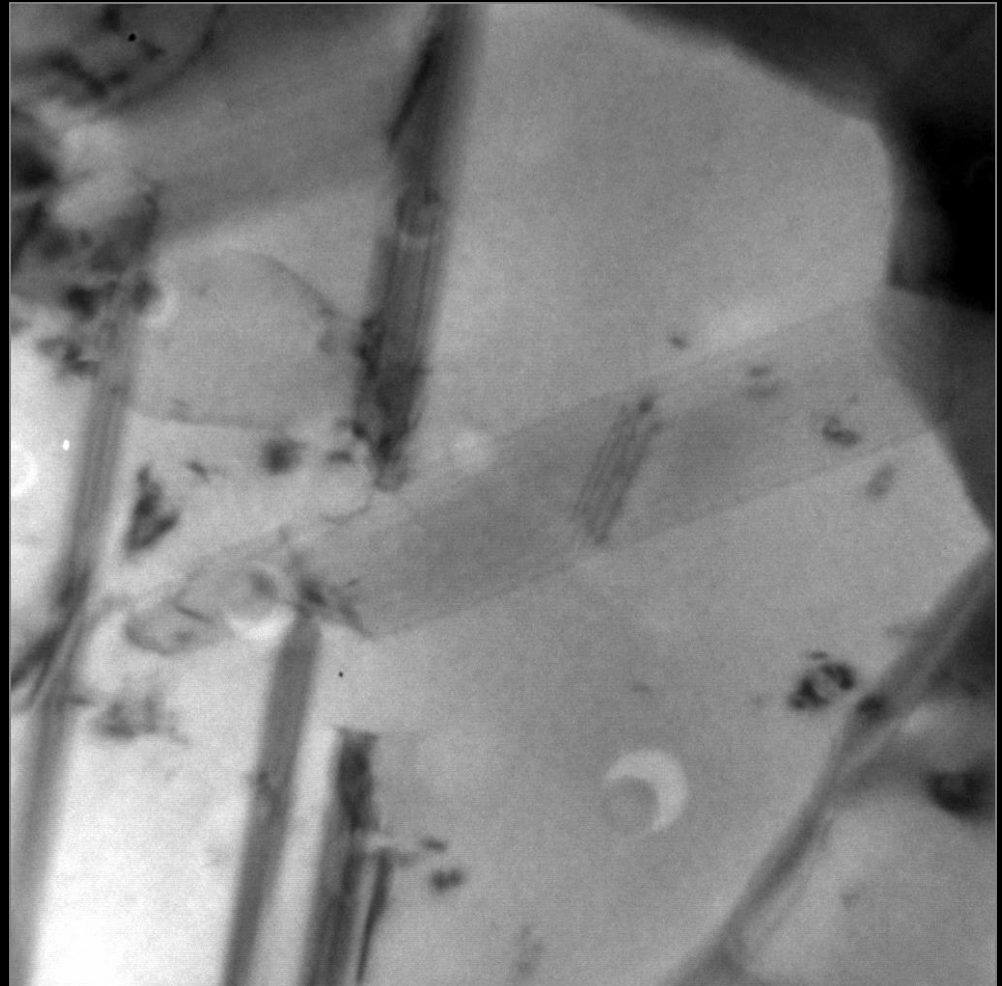
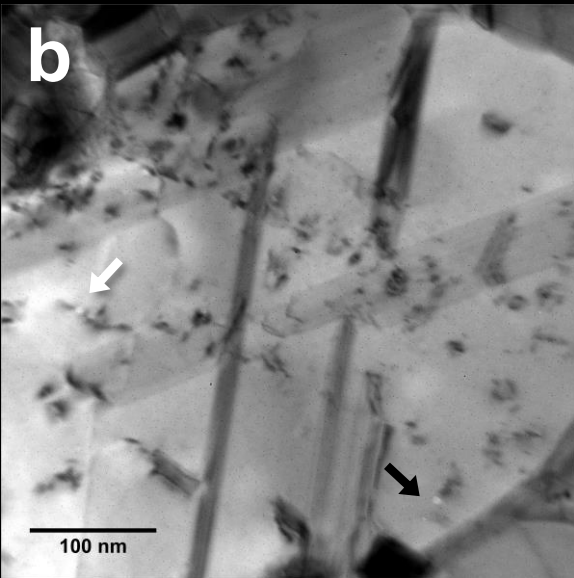
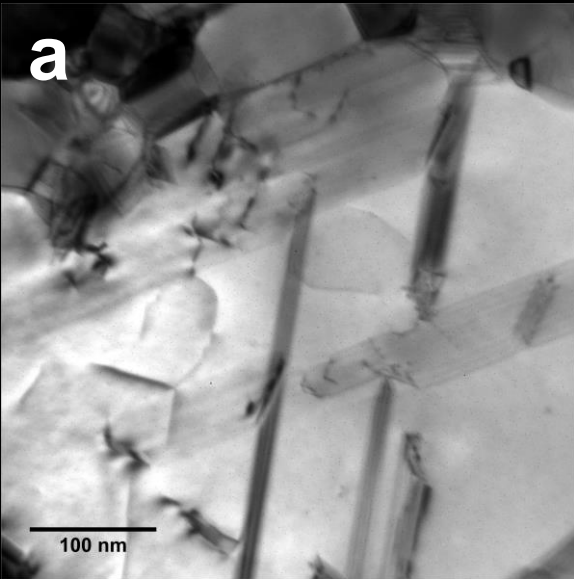
Successive  $\text{He}^{1+}$  then  $\text{Au}^{4+}$



# *In situ* Concurrent Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor

He<sup>1+</sup> implantation and Au<sup>4+</sup> irradiation  
of a gold thin film

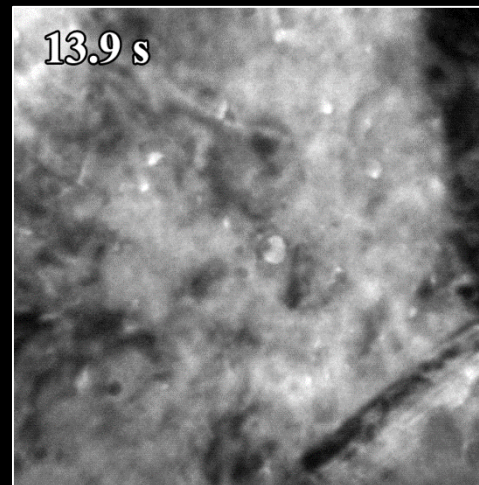
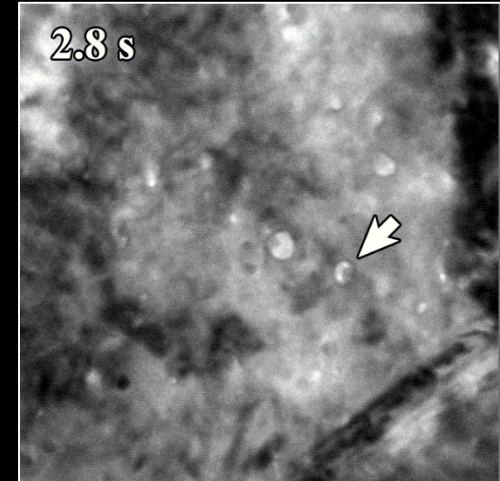
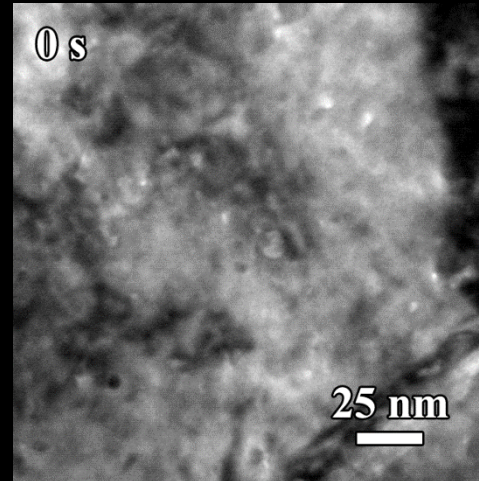
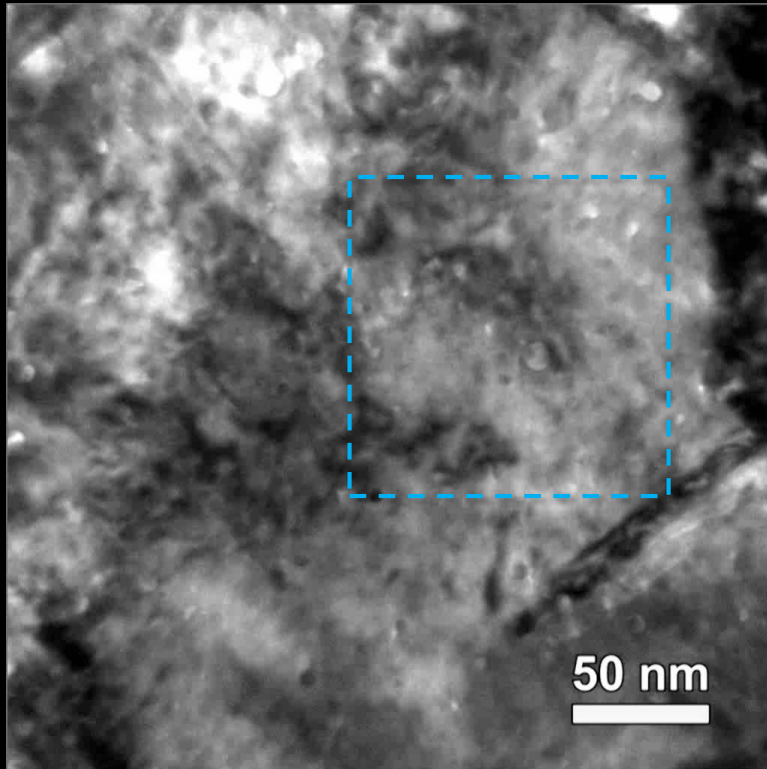


# Simultaneous *In situ* TEM Triple Beam:

## 2.8 MeV Au<sup>4+</sup> + 10 keV He<sup>+</sup> / D<sub>2</sub><sup>+</sup>

Collaborator: D.C. Bufford

Video playback speed x1.5.



In-situ triple beam He, D<sub>2</sub>, and Au beam irradiation has been demonstrated on Sandia's I<sup>3</sup>TEM!

Intensive work is still needed to understand the defect structure evolution that has been observed.

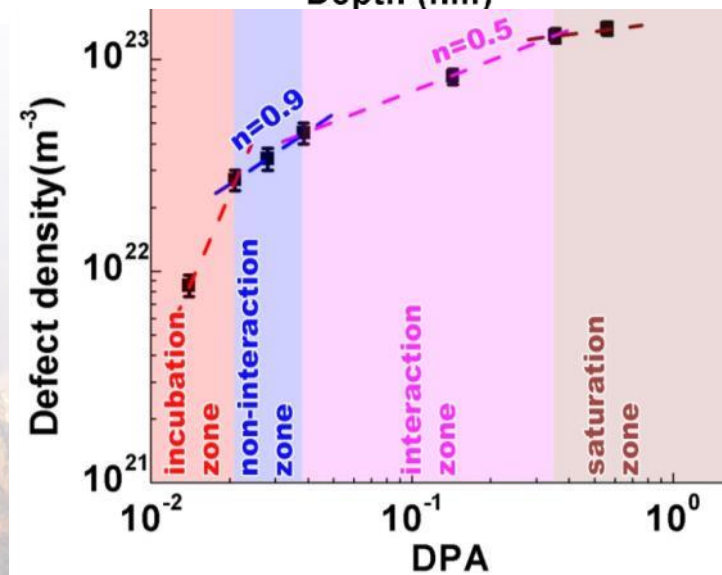
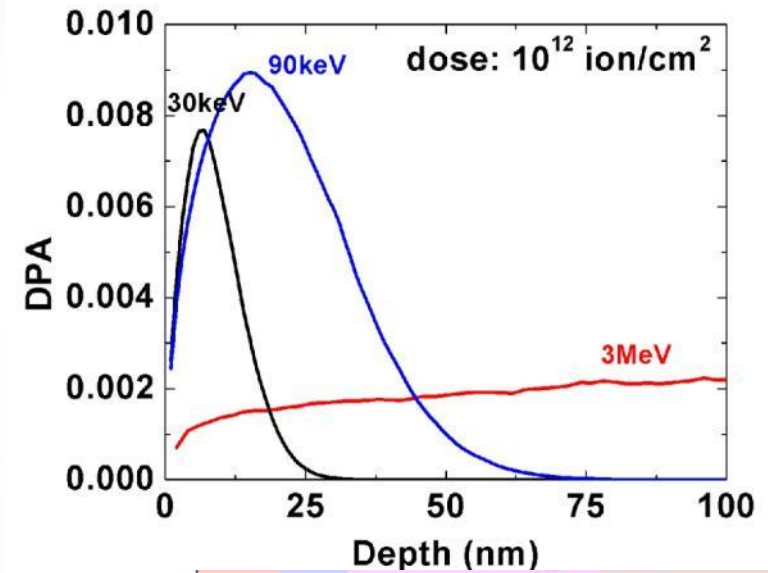
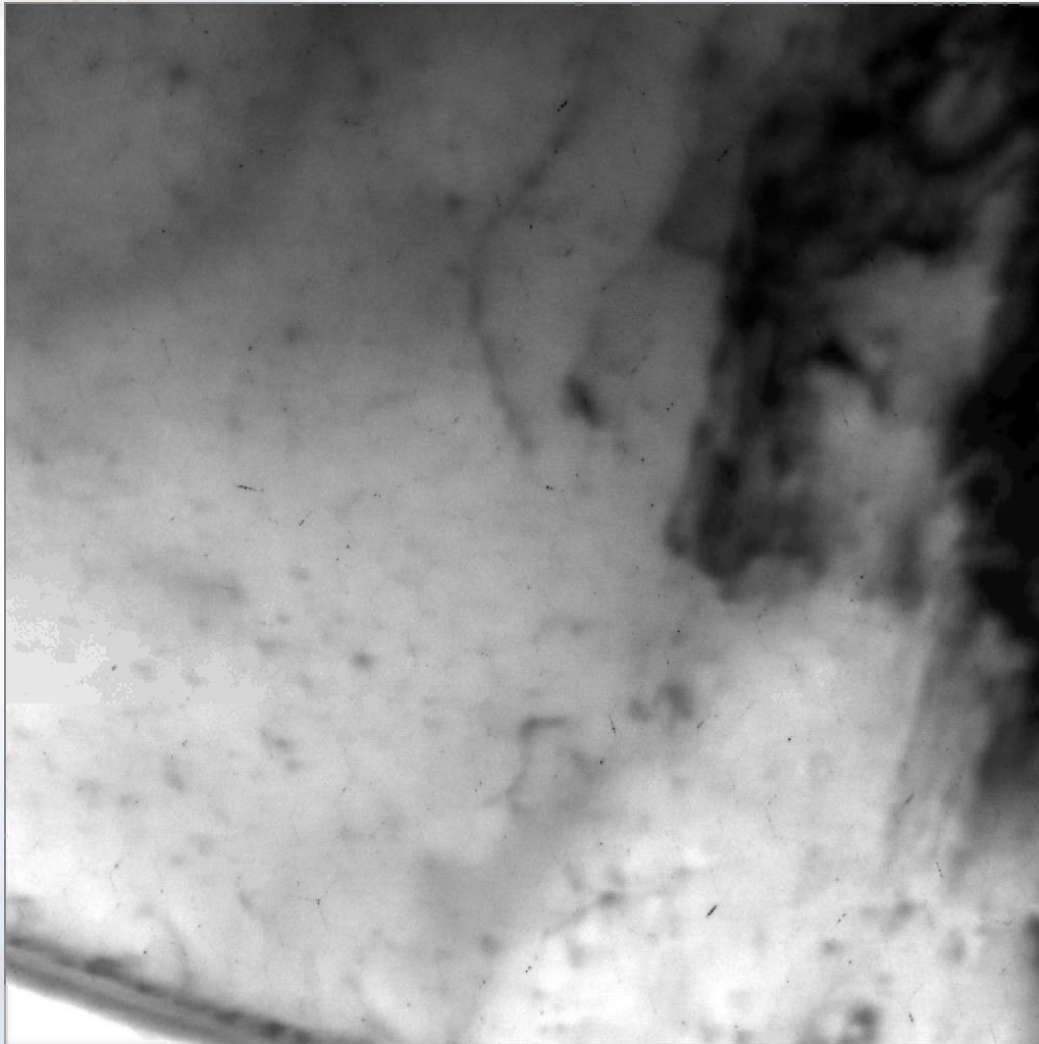
### ■ Approximate fluence:

- Au  $1.2 \times 10^{13}$  ions/cm<sup>2</sup>
- He  $1.3 \times 10^{15}$  ions/cm<sup>2</sup>
- D  $2.2 \times 10^{15}$  ions/cm<sup>2</sup>

### ■ Cavity nucleation and disappearance

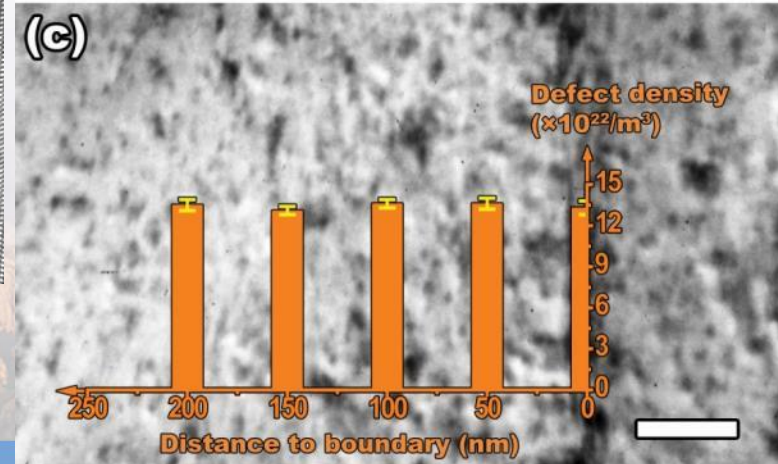
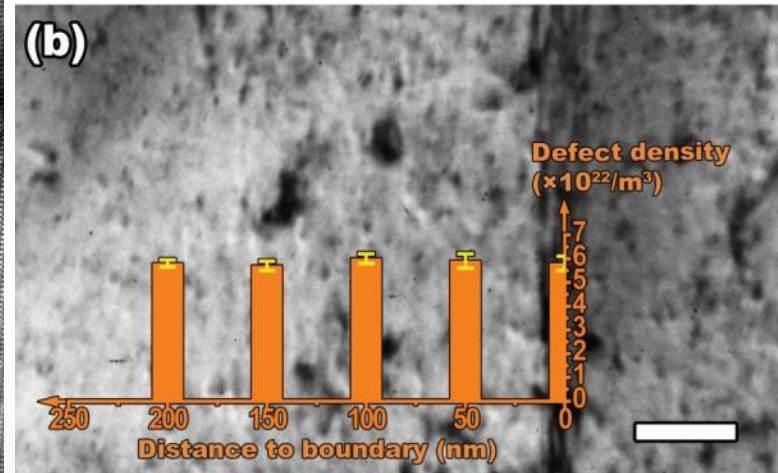
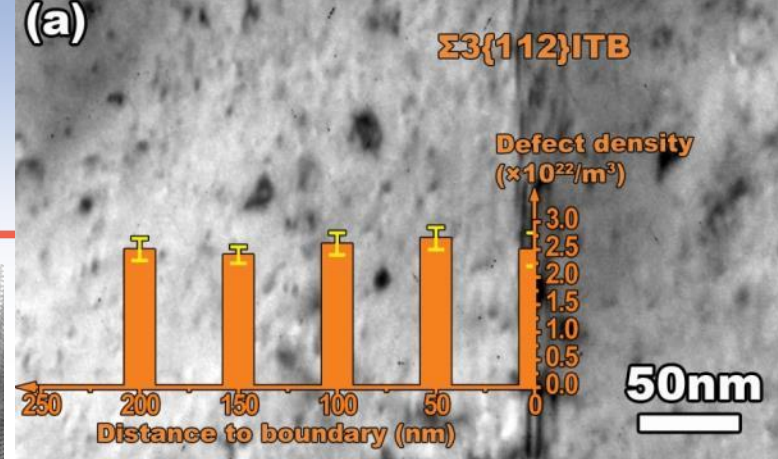
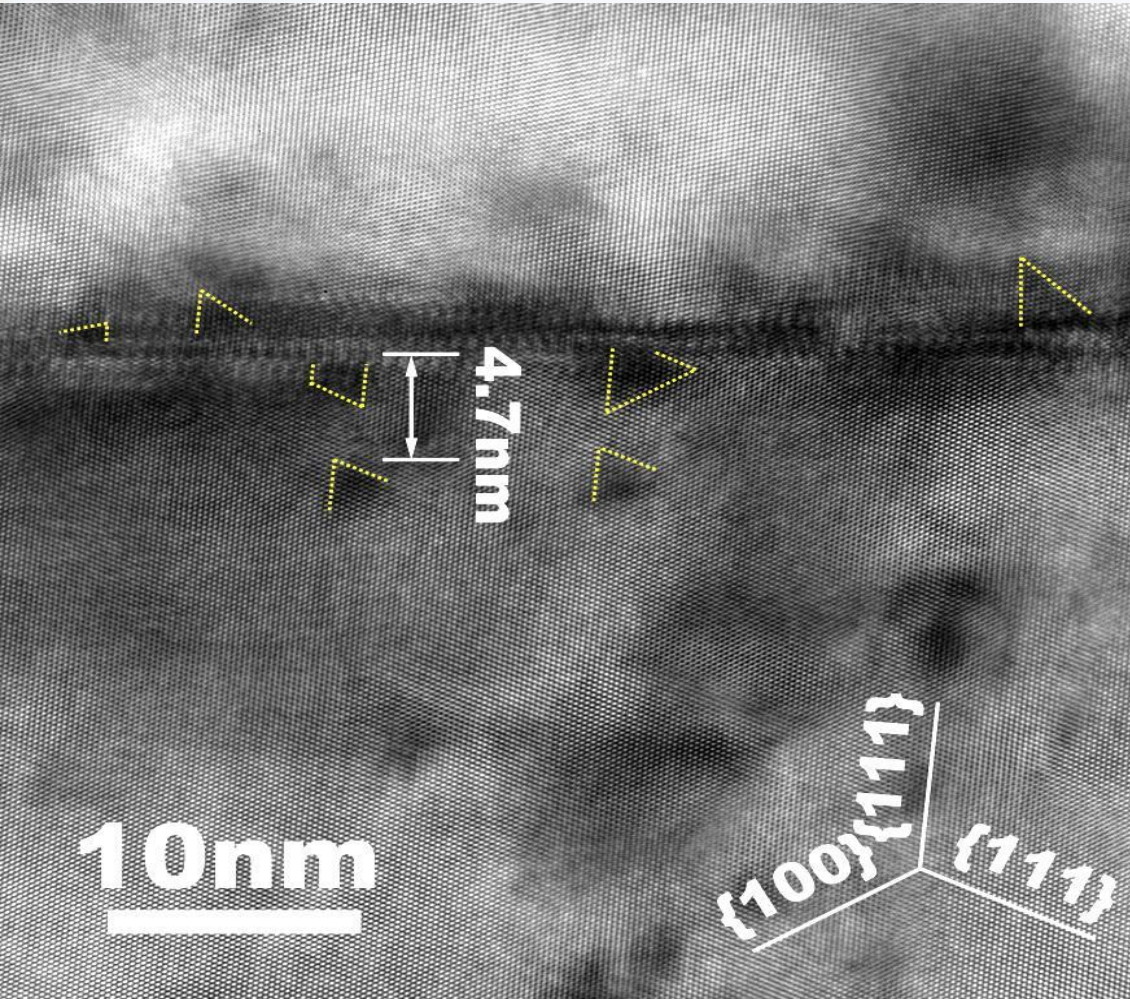
# Quantifying Defect Evolution

Collaborators: N. Li & A. Misra



# Defects are Altered Little by the Presence of Grain Boundaries

Collaborators: N. Li & A. Misra

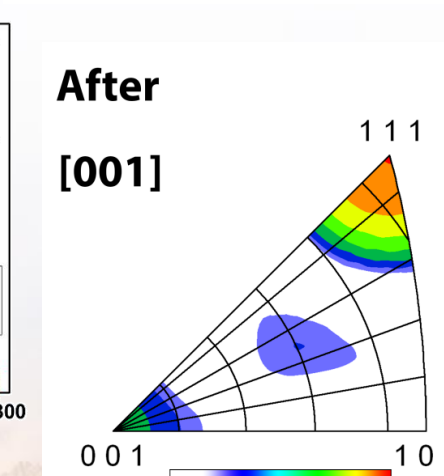
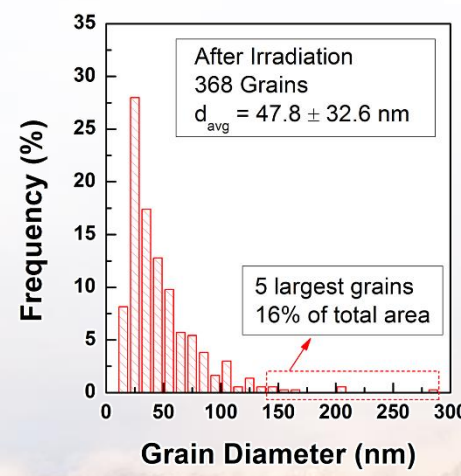
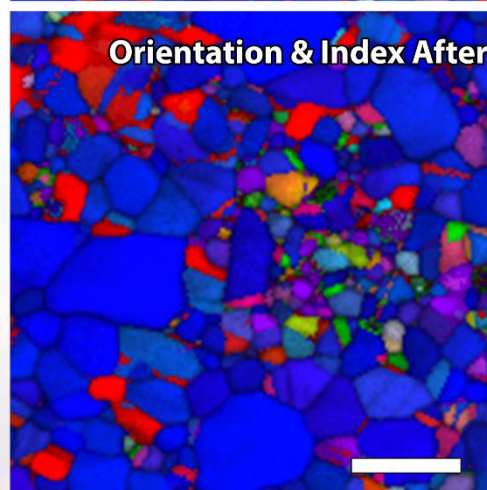
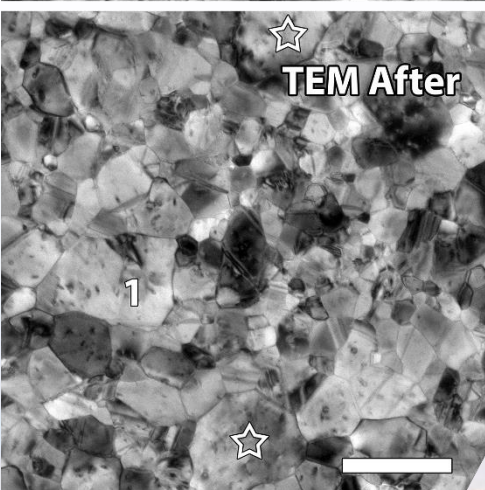
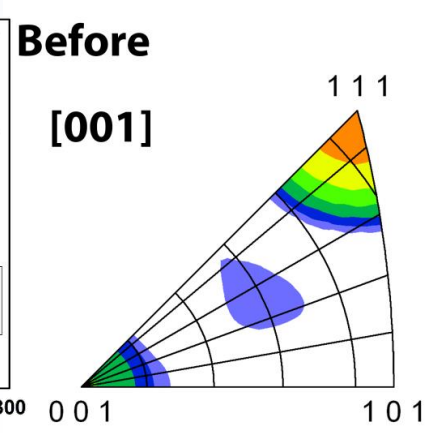
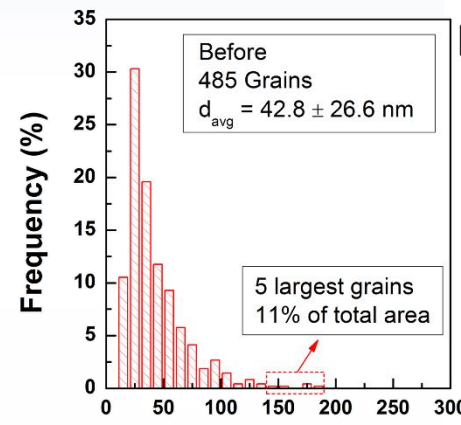
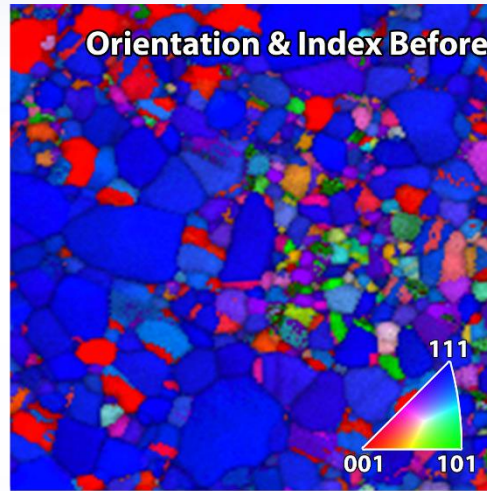
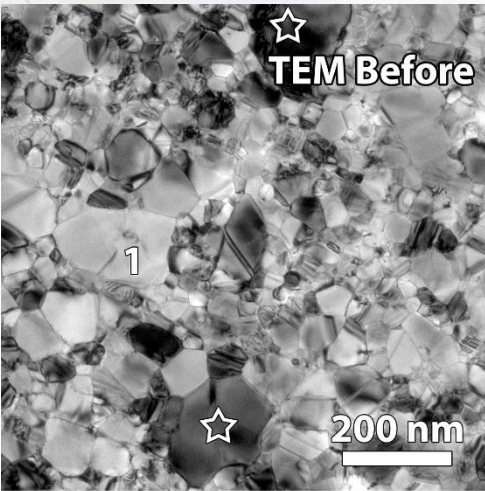


SFT appear to be directly at GB

No change in defect density is observed near GB

# Quantifying Stability of Nanocrystalline Au during 10 MeV Si Ion Irradiation

Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles

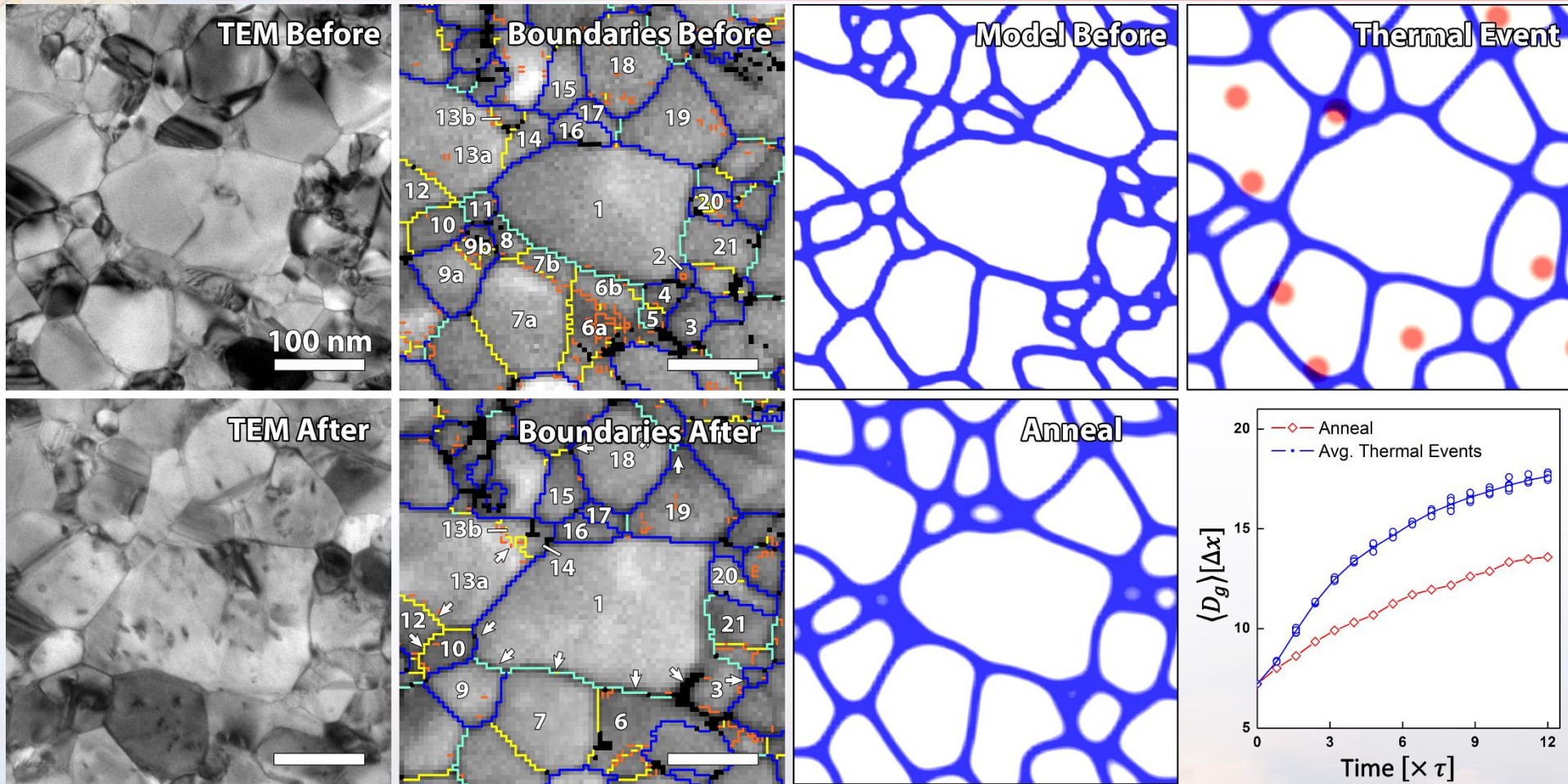


Any texture or grain boundary evolution can be directly observed and quantified



# Direct Comparison to Mesoscale Modeling

Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles

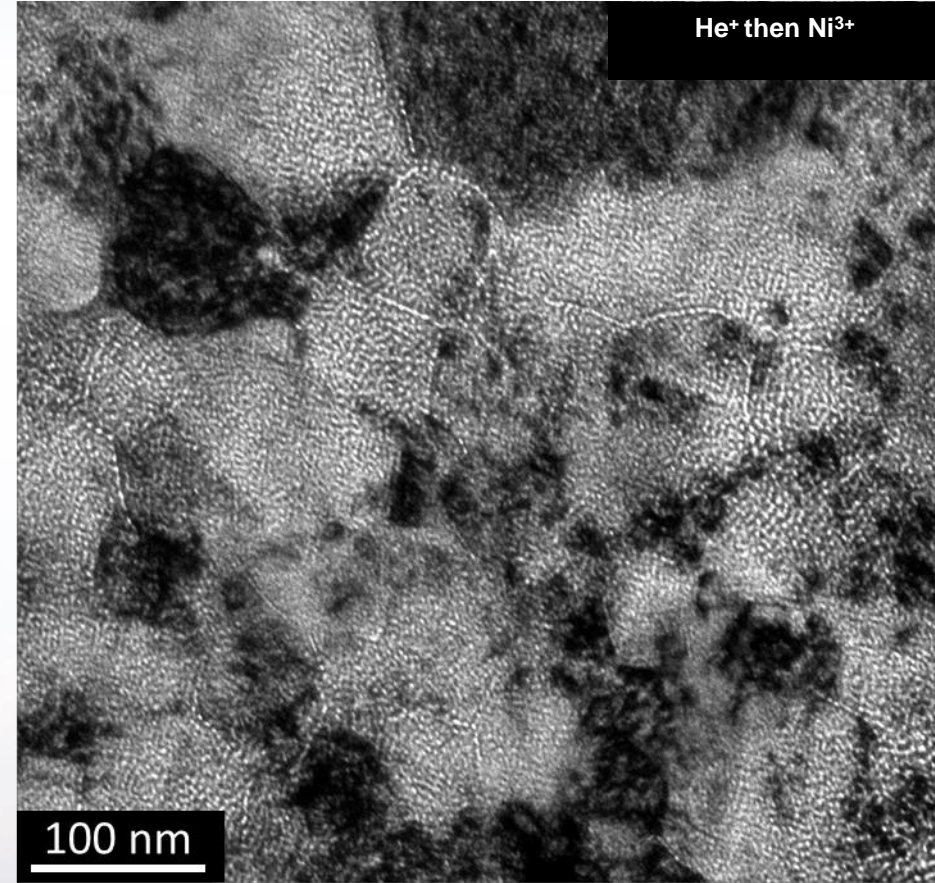
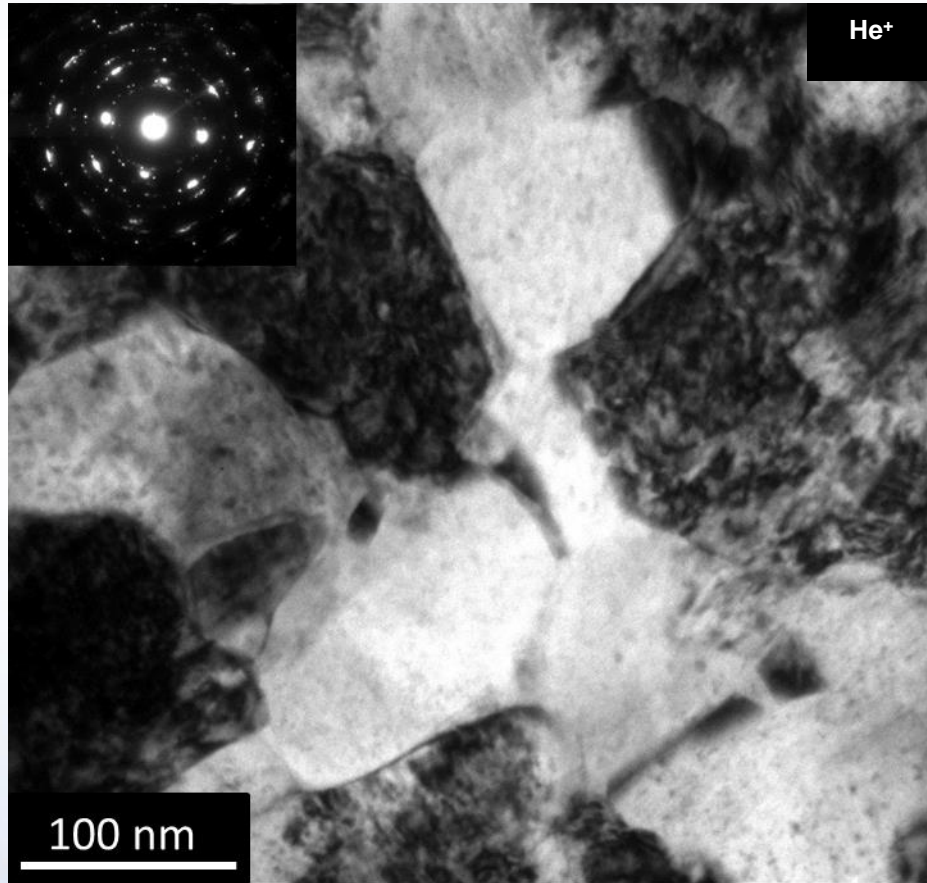


Because of the matching length scale, the initial microstructure can serve as direct input to either MD or mesoscale models & subsequent structural evolution can be directly compared.



# 10 keV He<sup>+</sup> Implantation followed by 3 MeV Ni<sup>3+</sup> Irradiation

Collaborator: B. Muntifering & J. Qu



10<sup>17</sup> He<sup>+</sup>/cm<sup>2</sup>

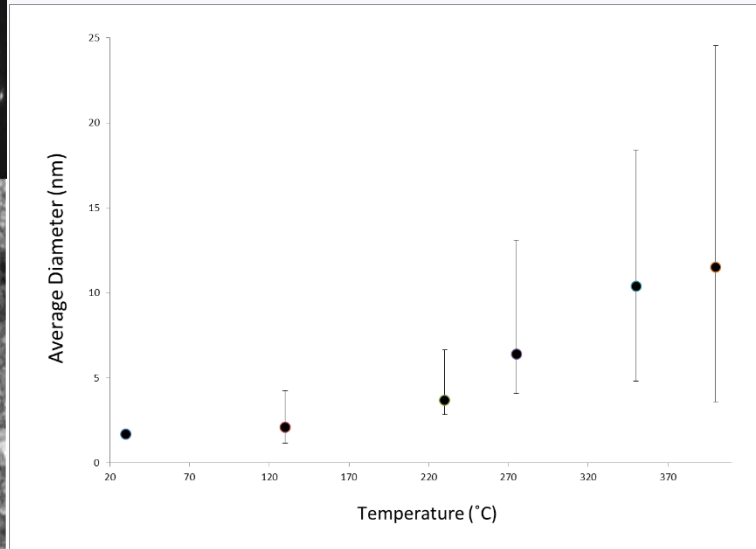
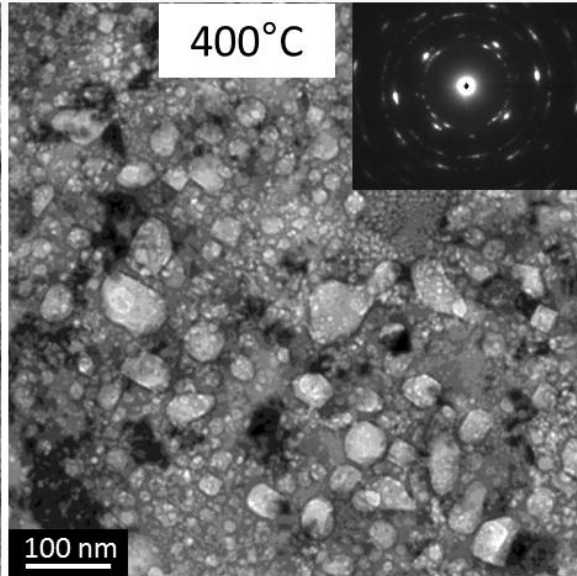
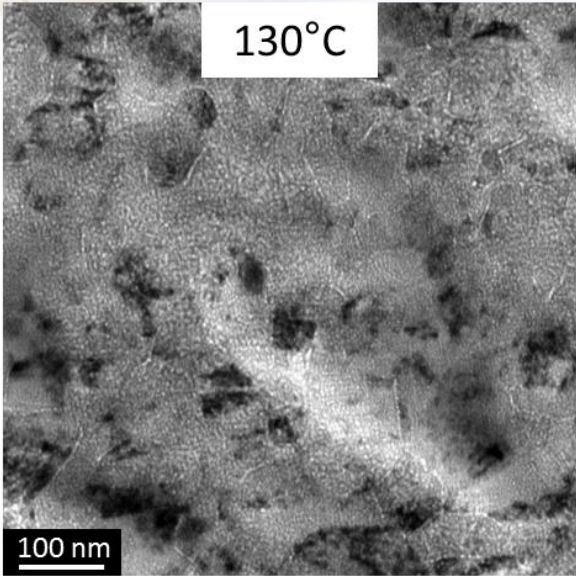
Visible damage to the sample

0.7 dpa Ni<sup>3+</sup> irradiation

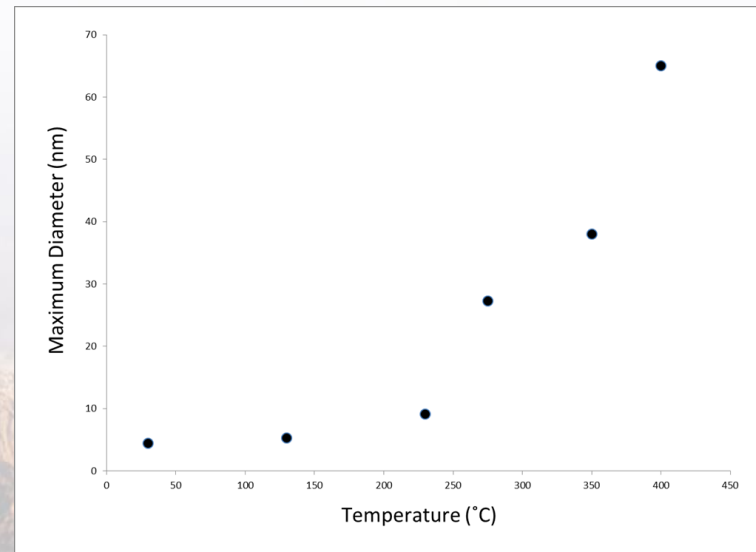
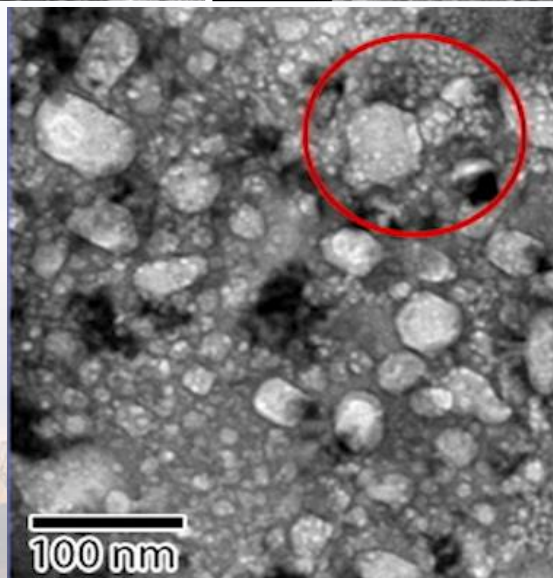
High concentration of cavities along  
grain boundaries



# Cavity Growth during In-situ Annealing of 10 keV He<sup>+</sup> Implanted and then 3 MeV Irradiated Ni<sup>3+</sup>

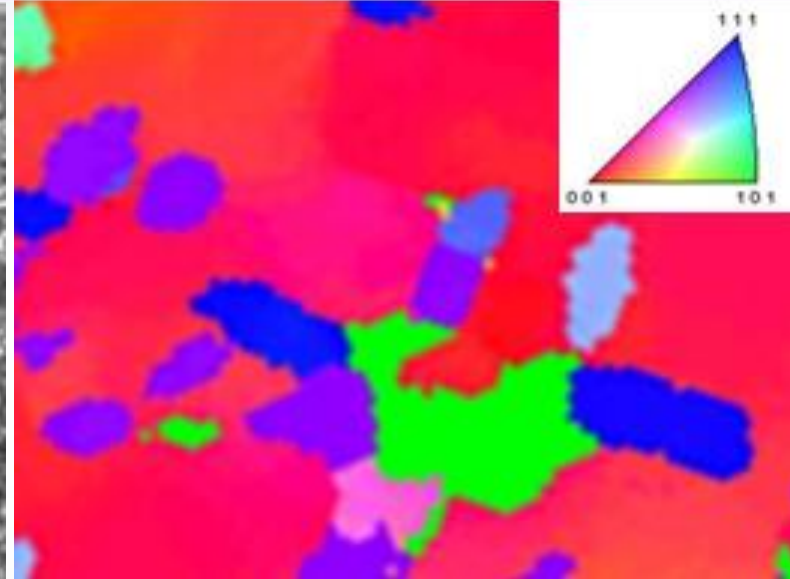
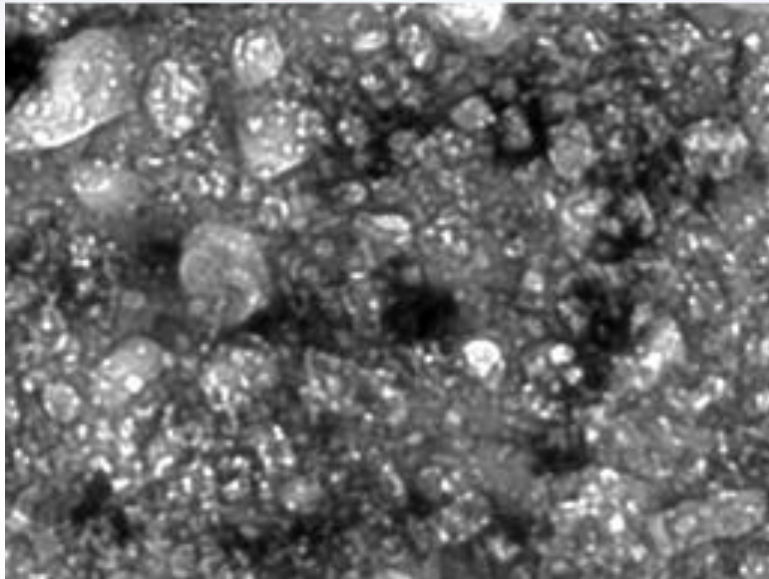


Bubble to cavity transition and cavity evolution can be directly studied

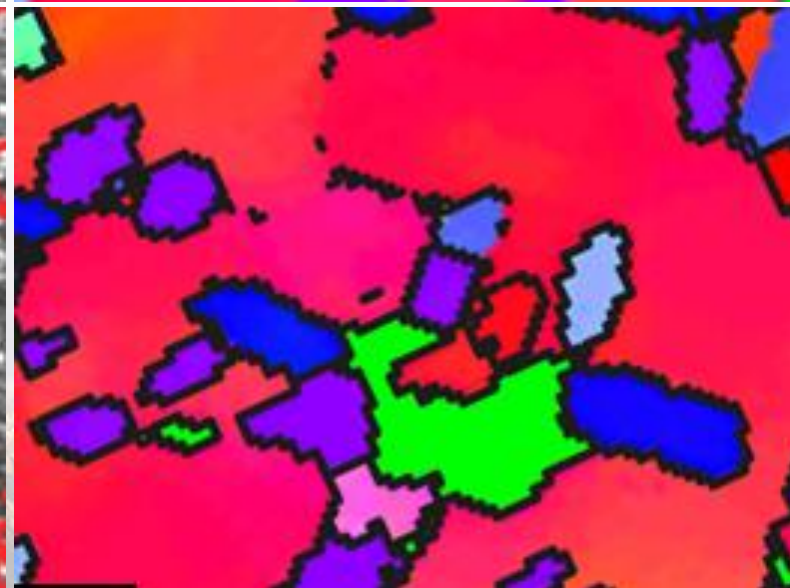
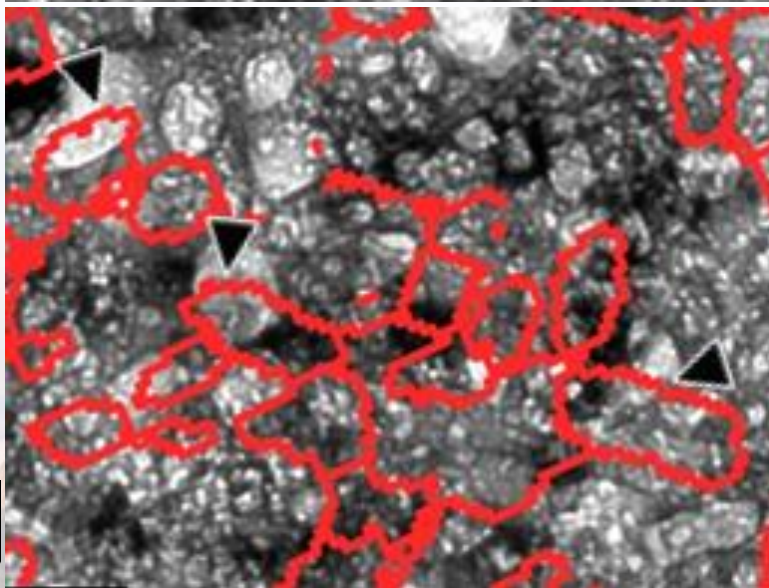


# Precession Electron Diffraction Reveals Hidden Grain Structure

Cavities in helium implanted, self-ion irradiated, nc nickel film annealed to 400 °C



Cavities span multiple grains at identified grain boundaries

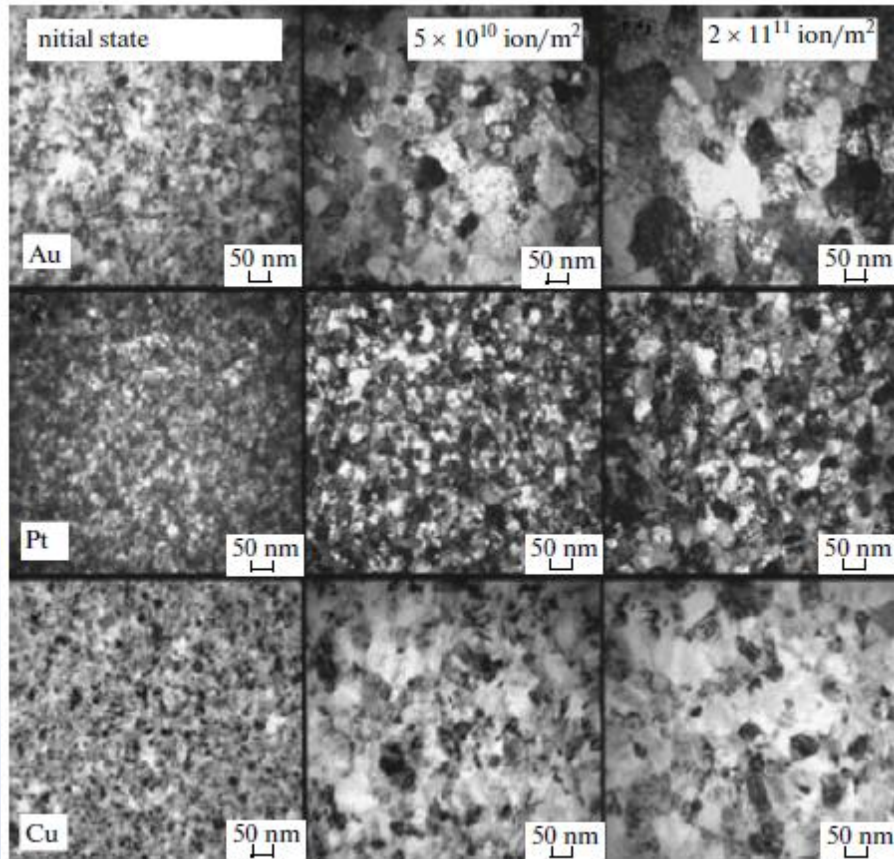


100 nm

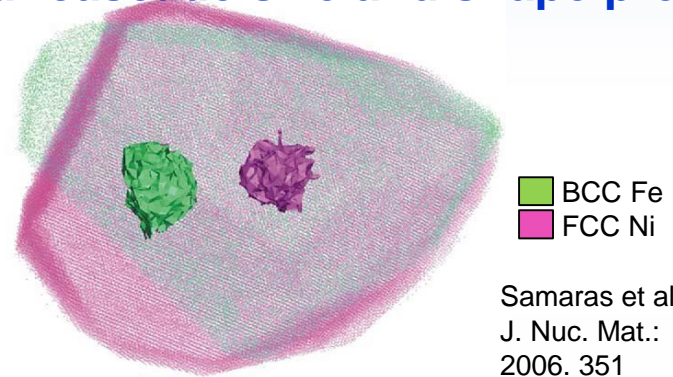
# Summary: Radiation Tolerance from Nanostructured Metals

## Variation in radiation tolerances

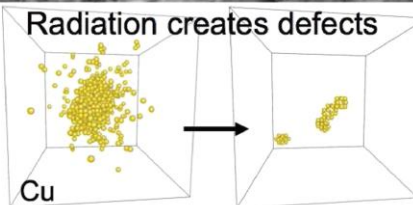
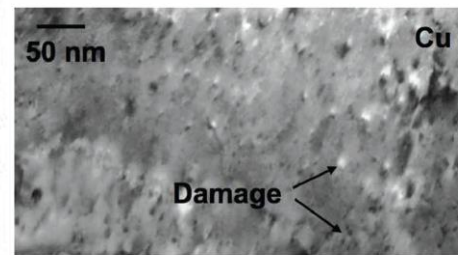
## Similar cascade size and shape predicted



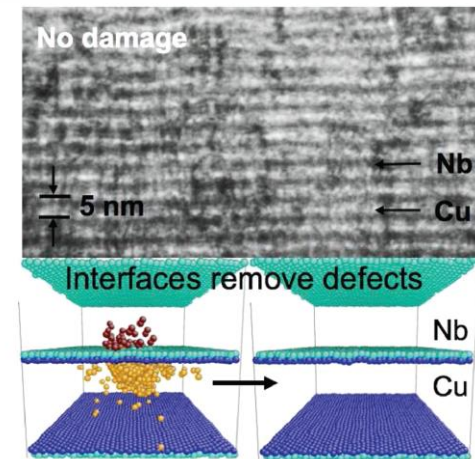
Kaomi et al., JAP: 2008. 104 073525



## Nanolamellars are radiation tolerant



Demkowicz et al., MRS Bulletin: 2010. 35



To a first order mean grain size comparison, these reports appear conflicting.

Not necessarily the case if initial microstructural details and associated properties are considered



This research was partially funded by the U.S. Department of Energy, Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering and by the Laboratory Directed Research and Development (LDRD) program at Sandia National Laboratories. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA-0003525. SAND2017-4611 A



Sandia National Laboratories



# Outline

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1. How Does Nanostructuring Effect a Materials Radiation Tolerances?

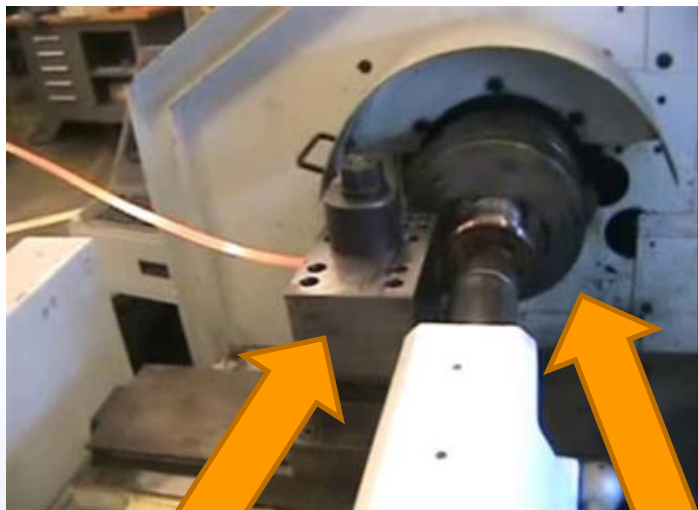
**2. Can We Tailor Boundaries/Interfaces to Control Radiation Tolerances?**

3. Can We Explore Other Aspects of the Complex Environment Present in Modern and Future Reactors Using *In situ* Techniques?



# Large Scale Production of Ultrafine Grained Tungsten

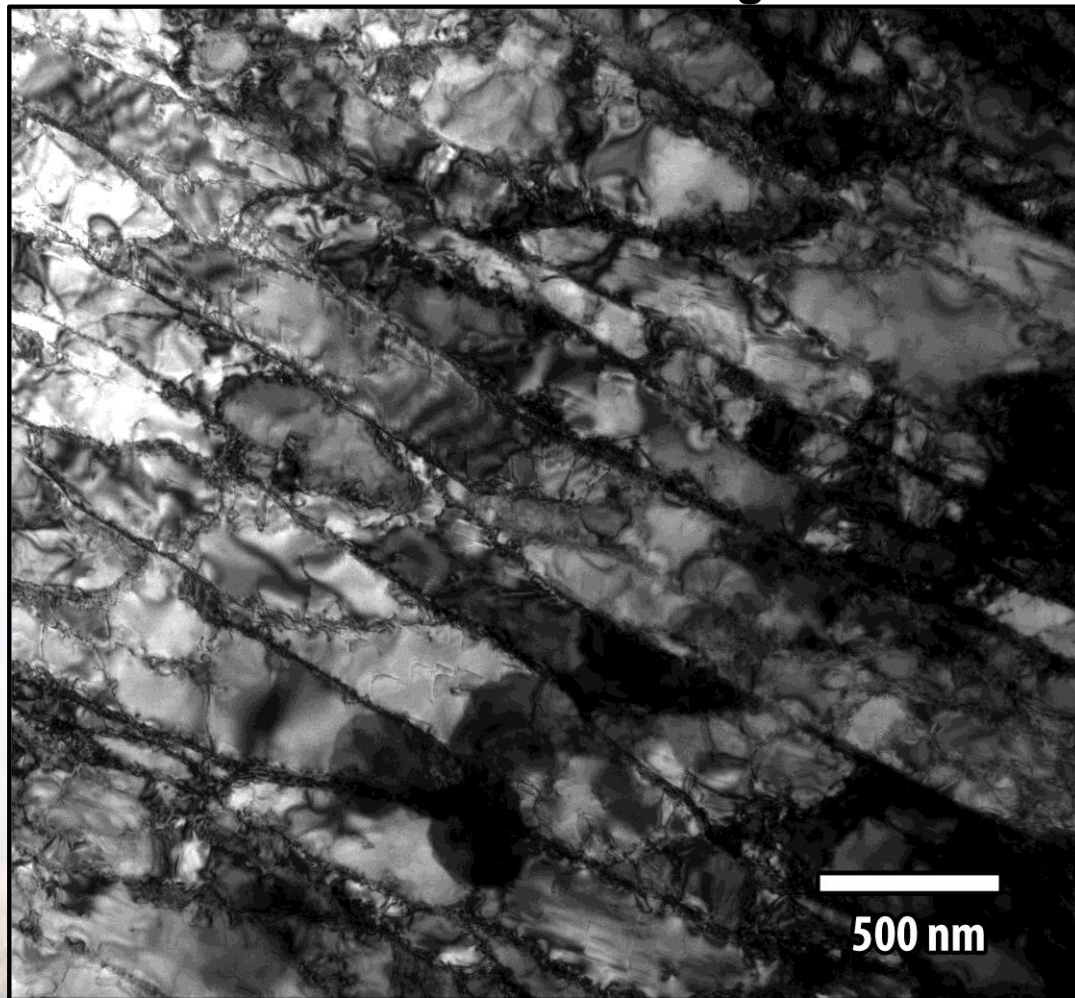
## Snapshot of the Process



Extrusion Machining tooling

Commercially available lathe

## Ultrafine Grained Tungsten



500 nm



# Displacement Damage Evolution at Nanometer Resolution

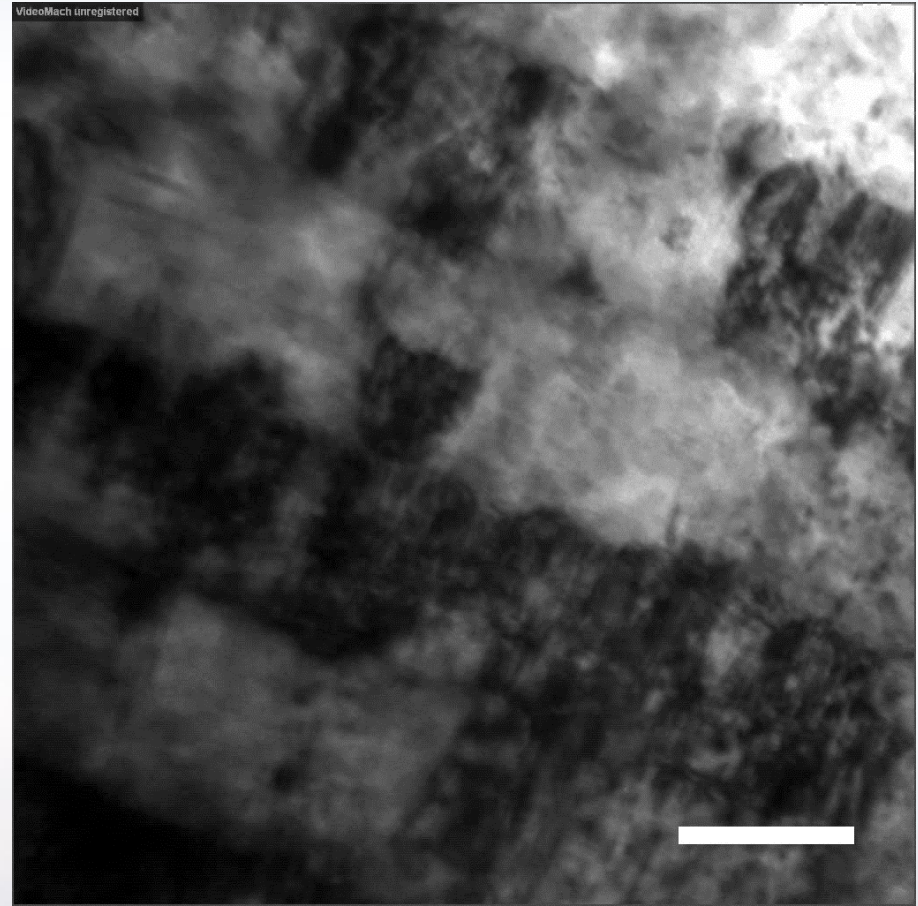
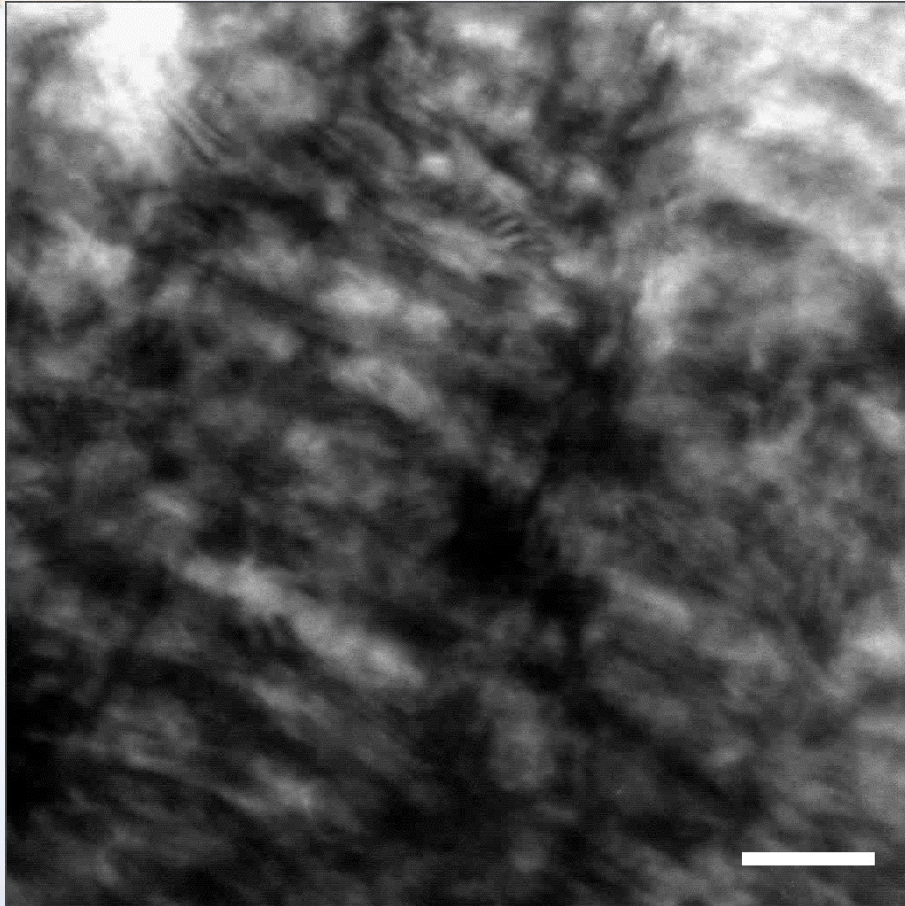
*In situ* TEM self-ion irradiation at 3 MeV of a samples from the same SPD tungsten lot used for the He implantation.

**I<sup>3</sup>TEM can providing insight into:**

- 1) Loop formation**
- 2) Loop stability & migration**
- 3) Rad & structural defect interactions**

200 nm

# Layer Deposition to Provide Defect Sinks

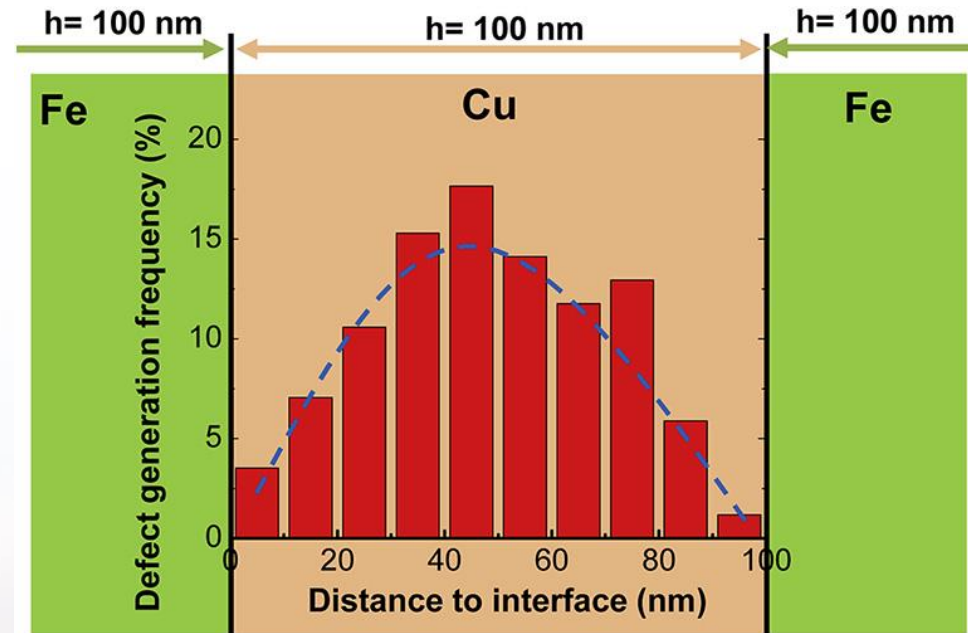
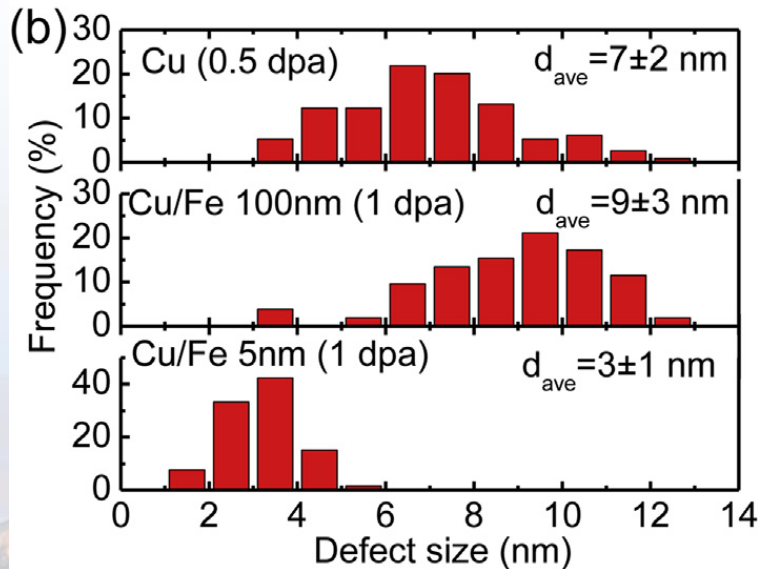
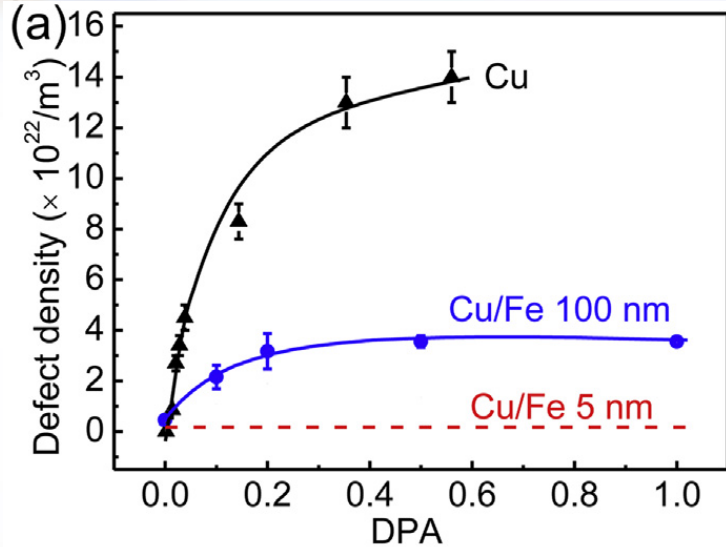


Film deposition of layered structures can also provide an opportunity for radiation defect sinks



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# Layer Deposition to Provide Defect Sinks

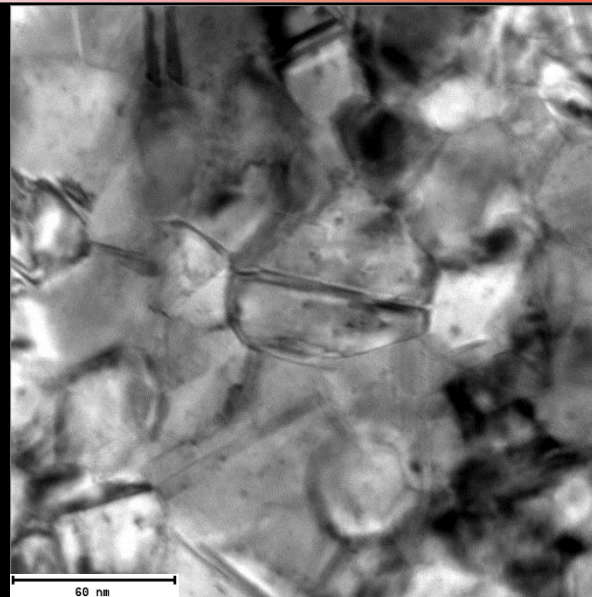
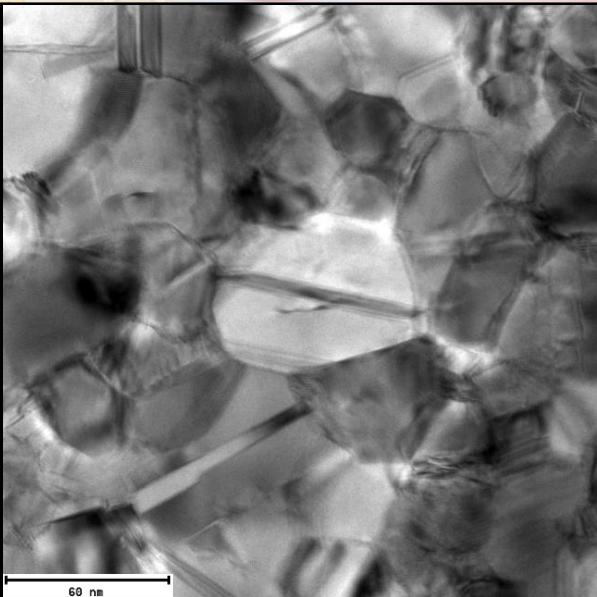


- Layered structure provides significant improvement over either pure system.
- The radiation tolerance improves with decreasing layer thickness.



# Nanocrystalline vs. Nanoporous Au results

Collaborators: N. Briot and T.J. Balk



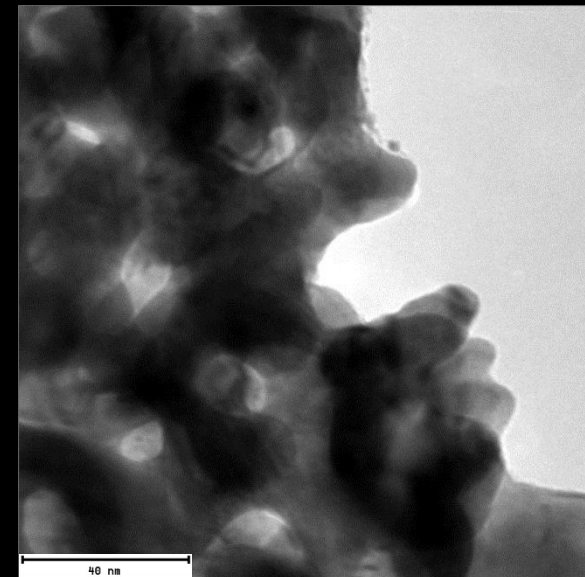
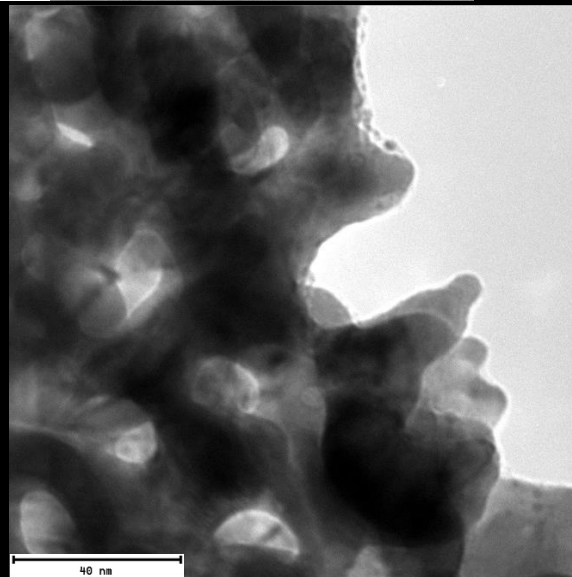
Nanoporous Au after ~ 6.6 dpa at  
46 keV:

Melting of the ligament

Nanocrystalline Au after ~ 0.5 dpa  
at 46 keV:

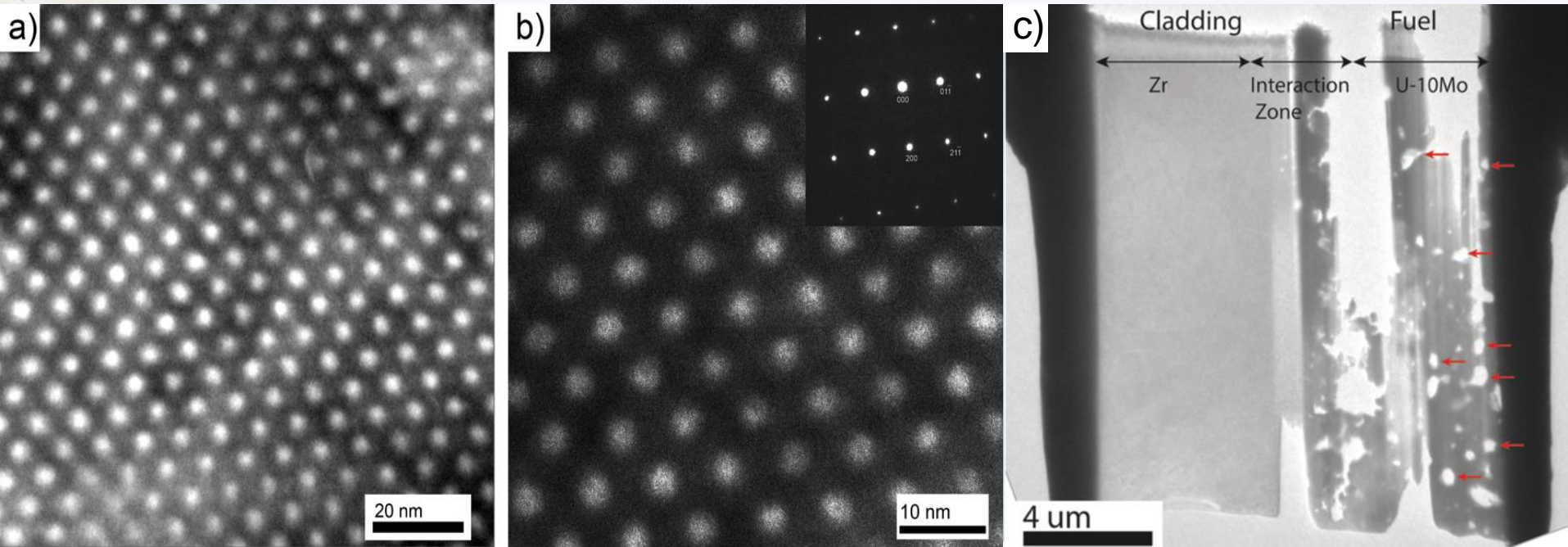
Lots of defects constantly created

Free surfaces provide the  
ideal sink, but nanoporous  
materials undergo surface  
modification and many other  
effects under radiation  
damage



# U-10Mo Depleted Samples from INL

Collaborators: C.M. Barr, A. Aitkaliyeva, R. Dingreville



- Local average fission density:  $4.42 \times 10^{21}$  (fiss/cm<sup>3</sup>)
- Samples from AFIP-6MKII Reduced Enrichment for Research and Test Reactors (RERTR) experiments at Idaho National Laboratory (INL)
- The average gas superlattice bubble diameter distribution:  $3.5 \pm 0.25$  nm diameter





# Outline

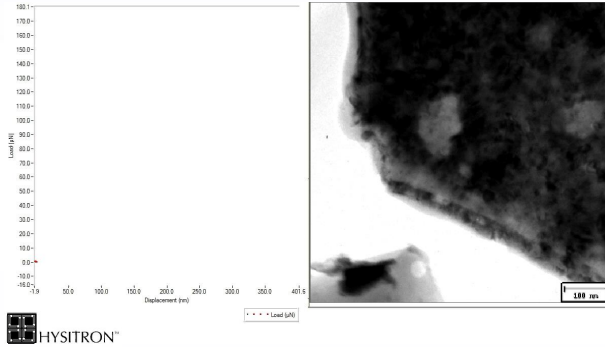
---

1. How Does Nanostructuring Effect a Materials Radiation Tolerances?
2. Can We Tailor Boundaries/Interfaces to Control Radiation Tolerances?
- 3. Can We Explore Other Aspects of the Complex Environment Present in Modern and Future Reactors Using *In situ* Techniques?**



# Potential Synergistic In situ Experiments

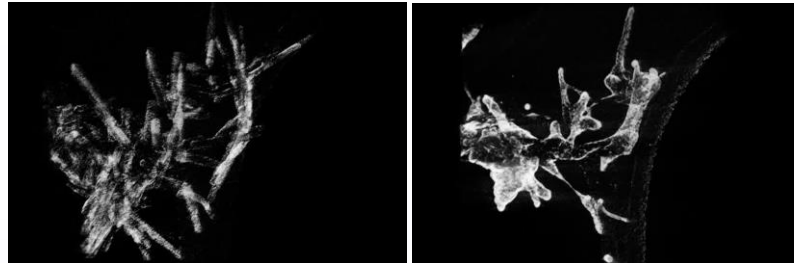
## Mechanical Effects



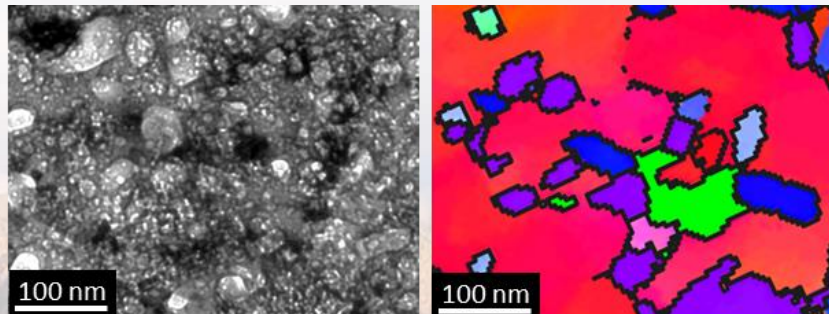
**Hysitron PI95 TEM Picoindenter**  
**Gatan 654 Straining Holder**  
Allows for direct correlation of dose and defect density with resulting changes in strength, ductility, and defect mobility

## Structural Effects

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

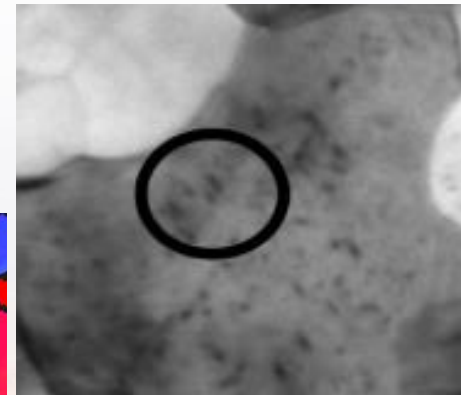


**Nanomegas ASTAR**  
Grain structure changes as a result of radiation and implantation



## Thermal Effects

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



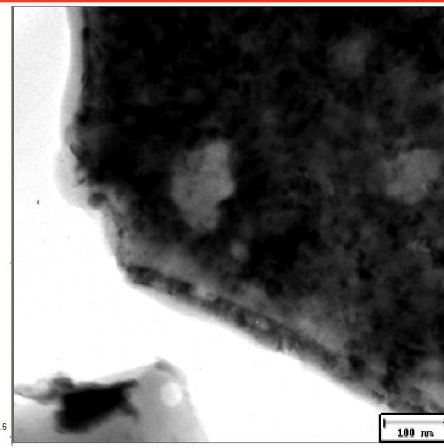
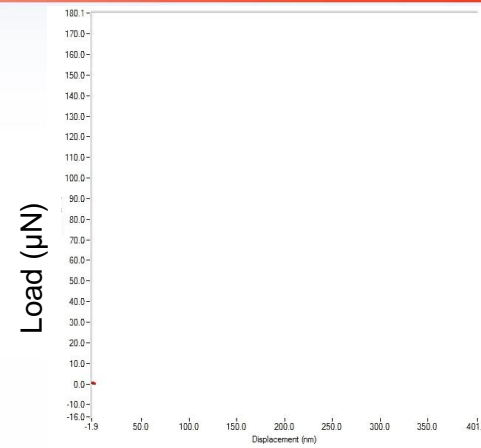
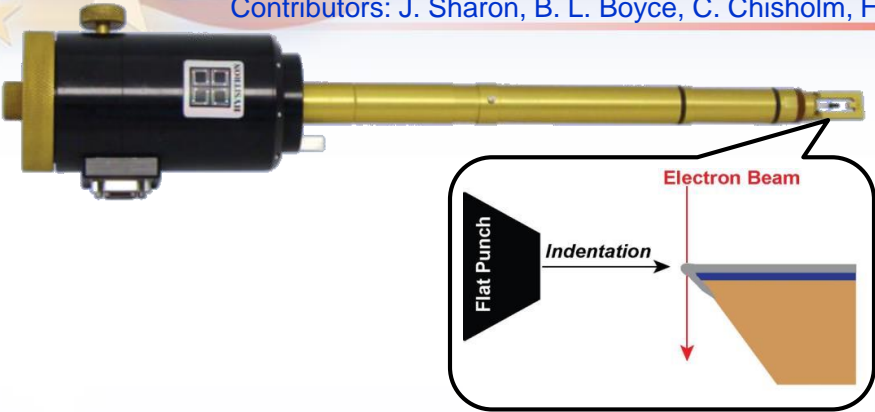
## Environmental Effects

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



# In situ TEM Quantitative Mechanical Testing

Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

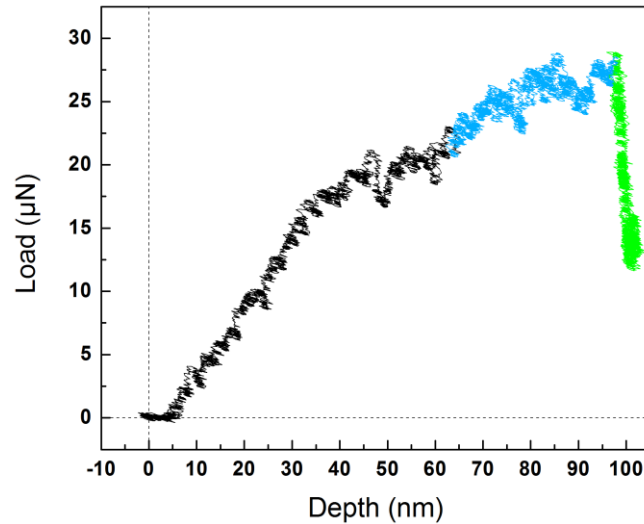
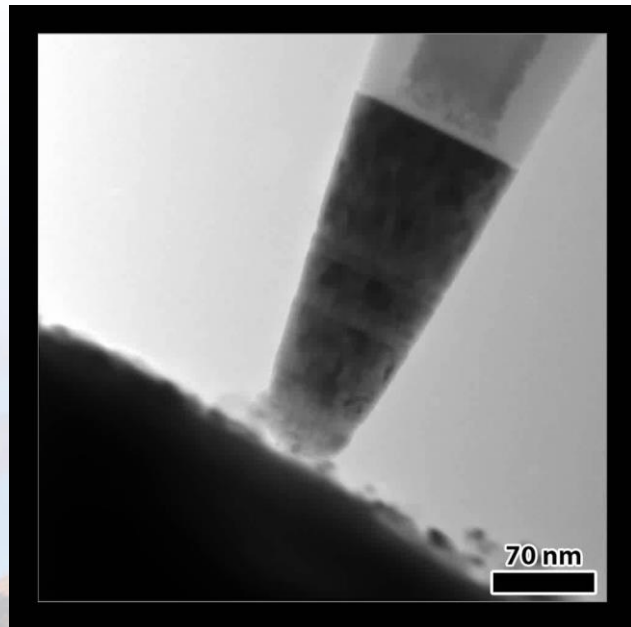


## Range of Mechanical Testing Techniques

- Indentation
- Compression
- Tension
- Wear
- Fatigue
- Creep

Displacement (nm)

## Fundamentals of Mechanical Properties

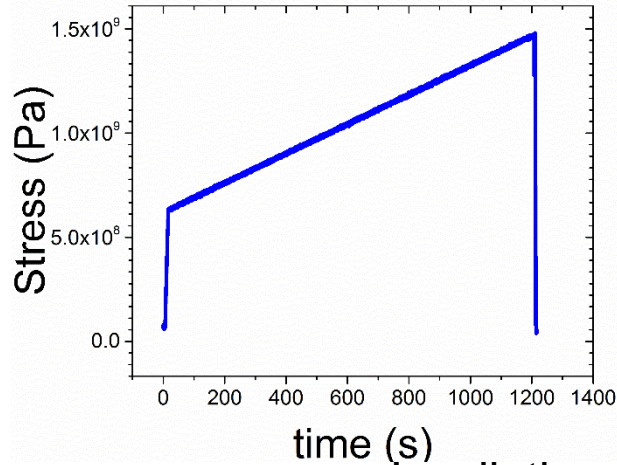


- 0.5 nm/s loading rate
- Trapezoid load function
- 60s load/60s hold/60s unload

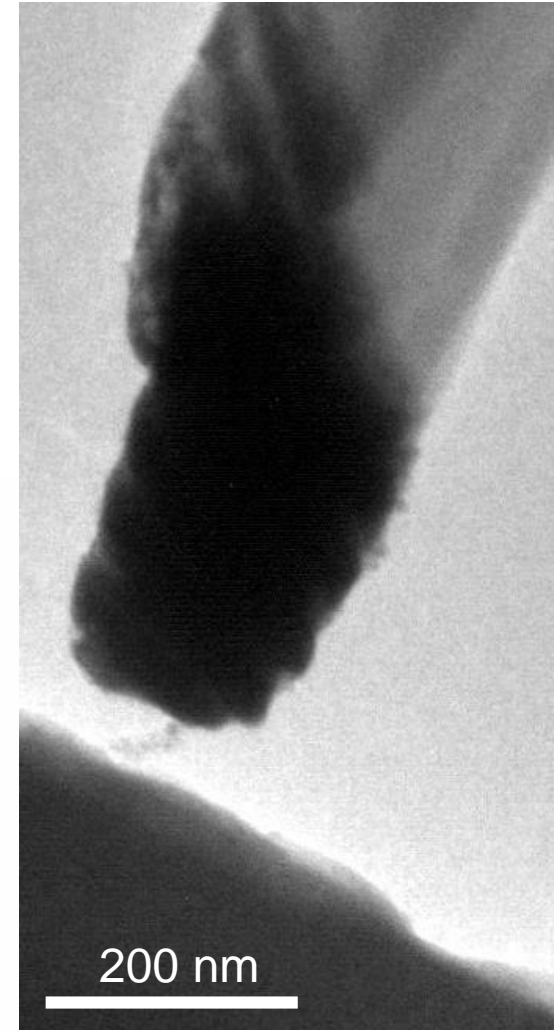
# Irradiation Creep (4 MeV Cu<sup>3+</sup> 10<sup>-2</sup> DPA/s)

Contributors: S. Dillon & R.S. Averback

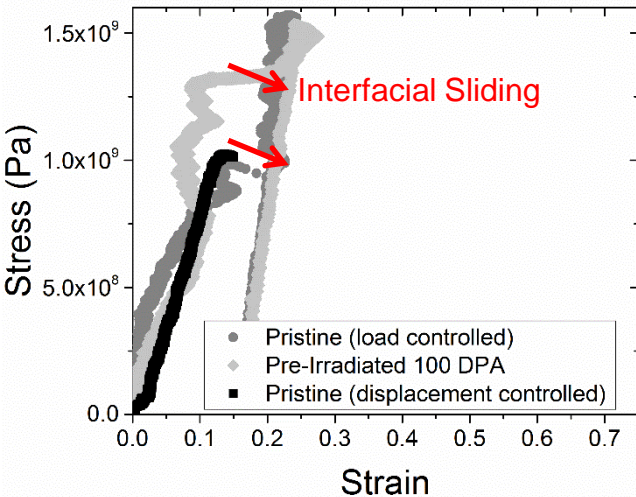
## Controlled Loading Rate Experiments



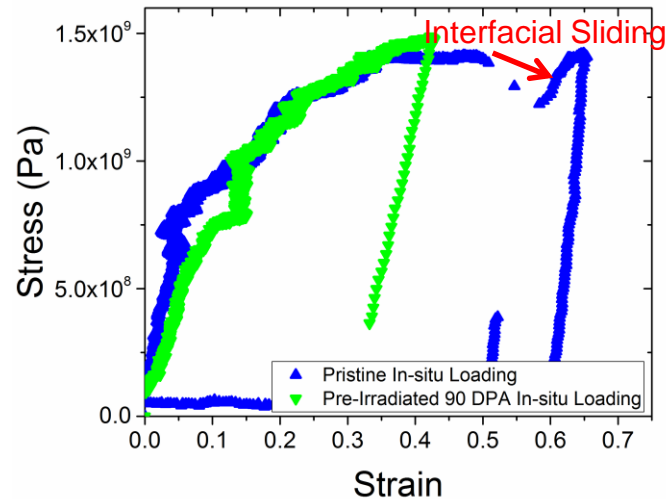
50 nm Cu-W multilayer  
20 Min



No Irradiation  
(Loading rate 0.6 Mpa s<sup>-1</sup>)



Irradiation Creep  
(Loading rate 0.6 Mpa s<sup>-1</sup>)



# Laser Initiation of Multilayer Reactive Foils

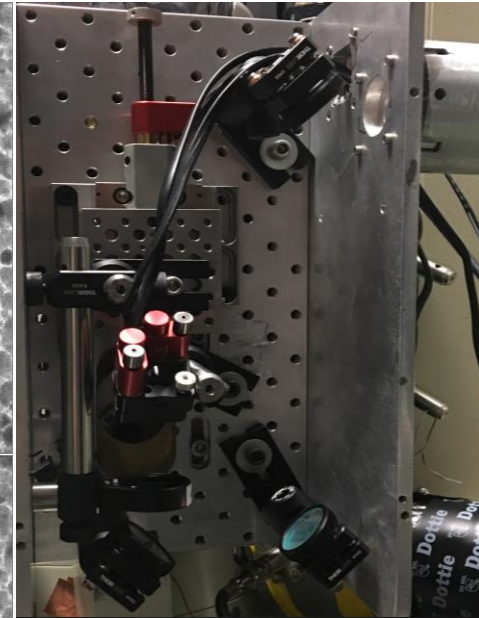
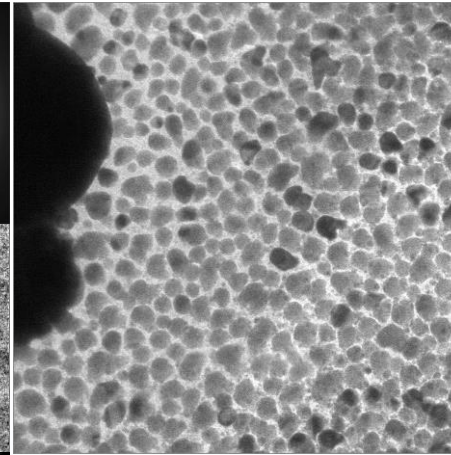
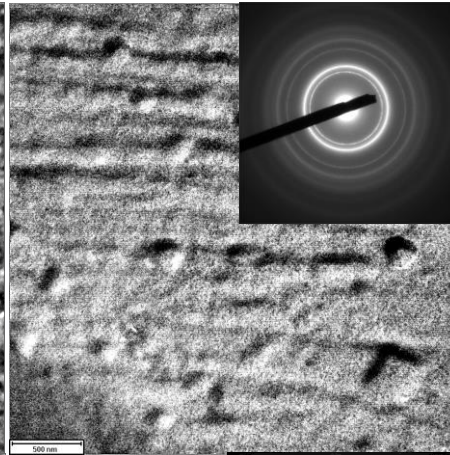
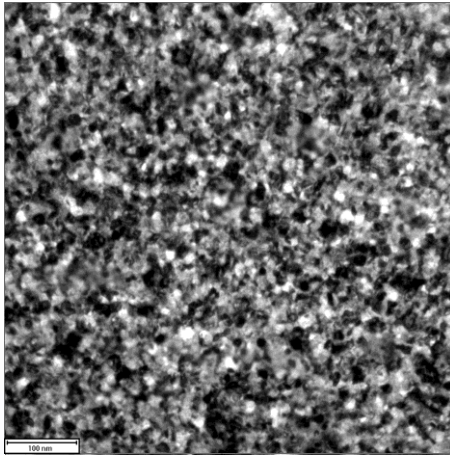
Collaborator: P. Price, C.M. Barr, D. Adams, M. Abere

Pt Grain Growth

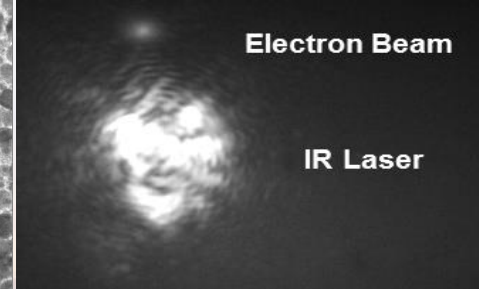
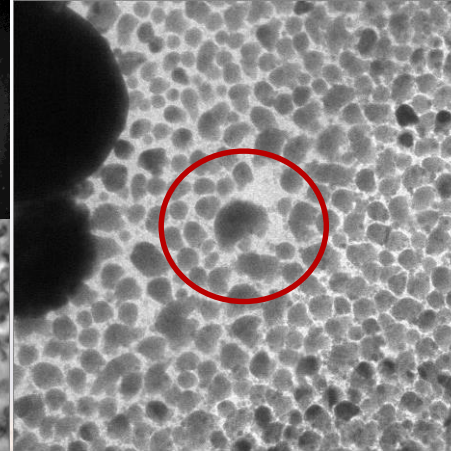
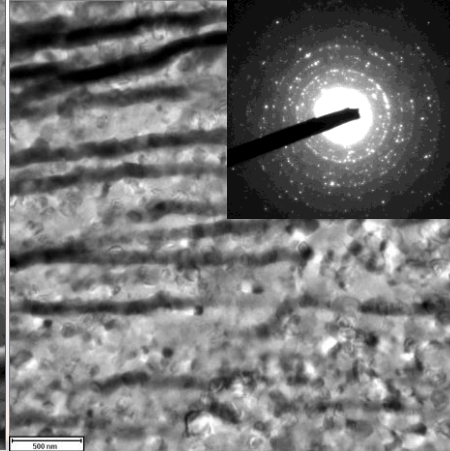
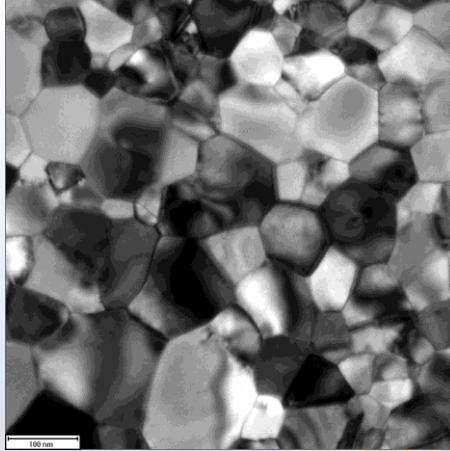
Reactive Multilayer Films

Nanoparticle Sintering

Before



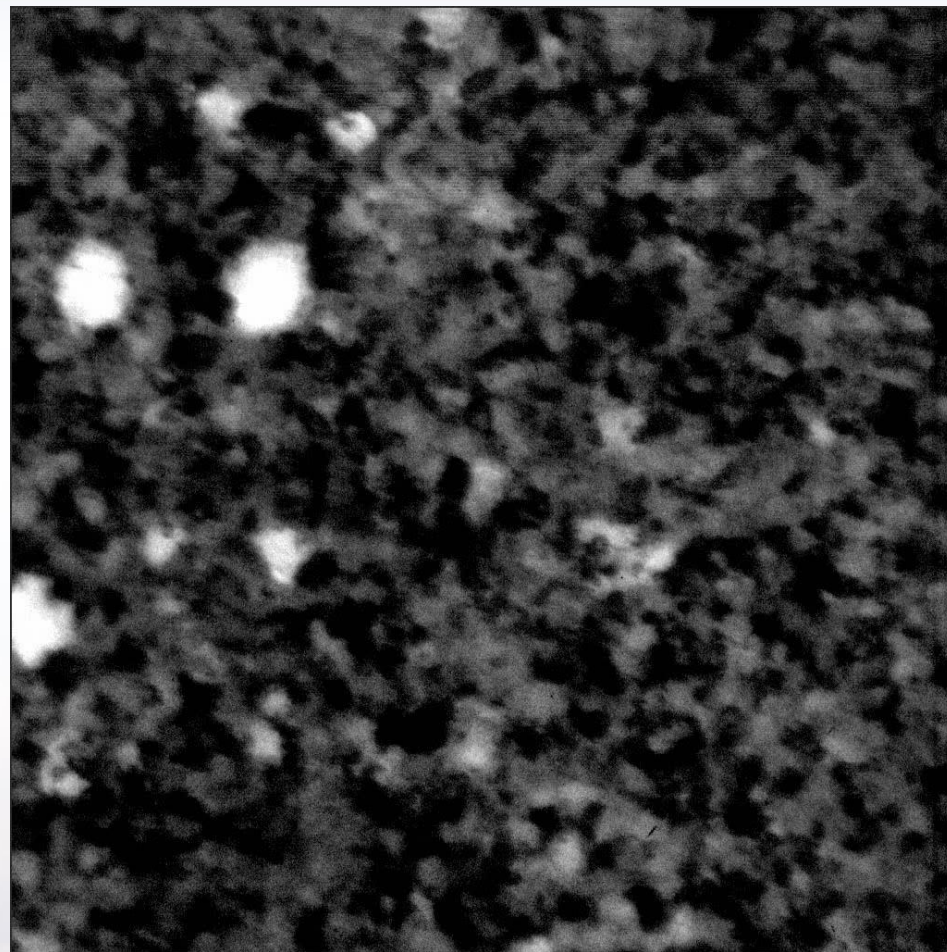
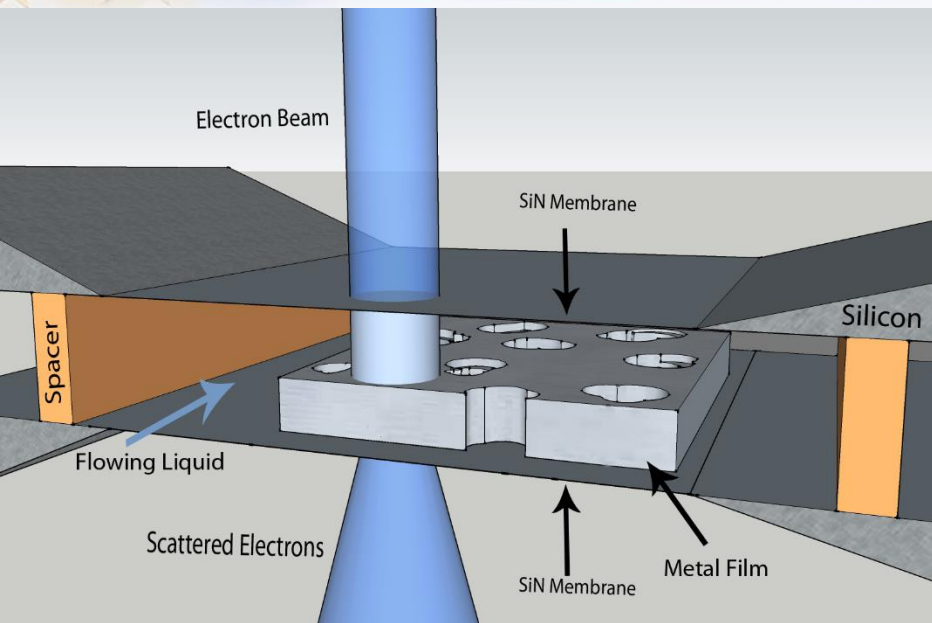
After



Understanding materials in extreme thermal environments as well as additive manufacturing processes

# *In situ* TEM Corrosion

Contributors: D. Gross, J. Kacher, & I.M. Robertson



## Microfluidic Stage

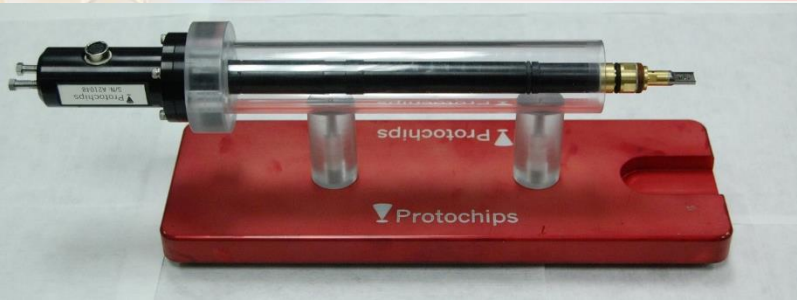
- Mixing of two or more channels
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Films can be directly deposited on the electron transparent SiN membrane

Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.



# Feasibility of Studying Zircaloy 2 at Nominally 1 atm

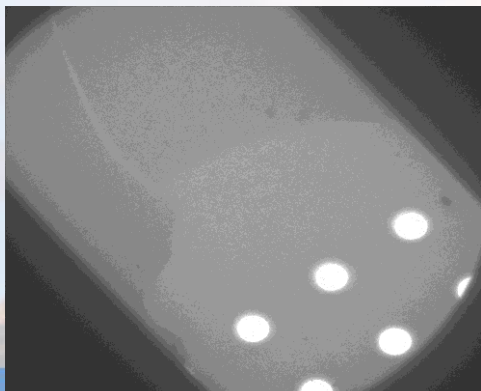
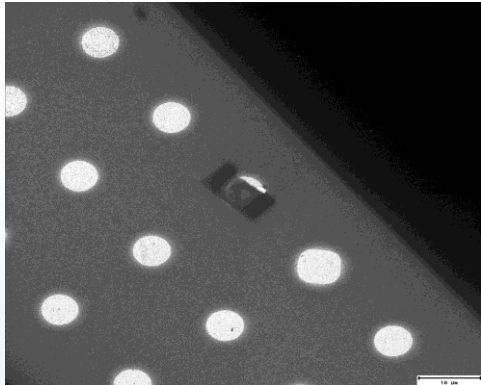
Collaborators: S. Rajasekhara and B.G. Clark



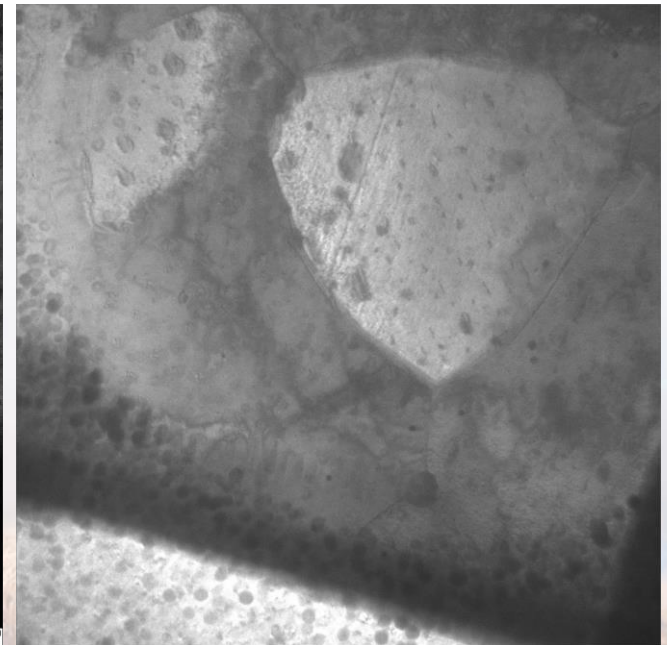
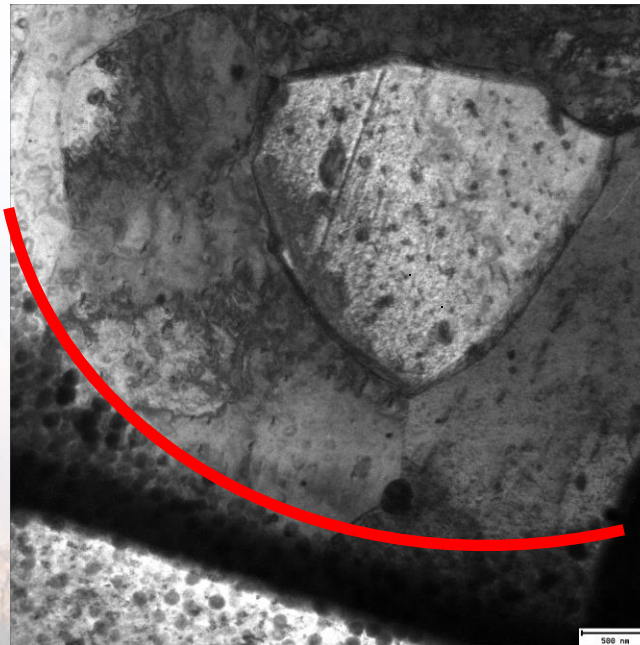
## Vapor-Phase Heating TEM Stage

- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools

## Vacuum & Single Window



## Nominally 1 atm H<sub>2</sub> & Two Windows

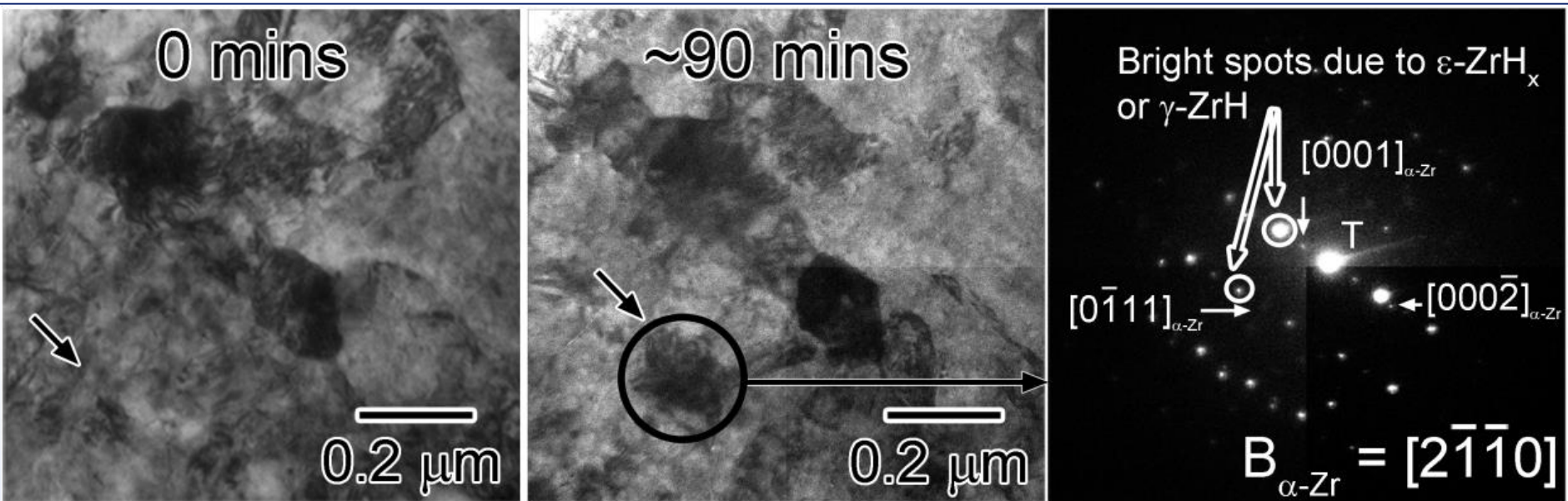


Most features are observed in both despite the decreased resolution resulting from the additional SiN window and 5 μm of air

# In situ Observation of Hydride Formation in Zirlo

Collaborators: S. Rajasekhara and B.G. Clark

Absolute hydrogen pressure: 327 torr (~ 0.5 atm),  
Ramp rate: 1 °C/s, Final temperature: ~ 400 C, Dwell time: ~ 90 mins



Hydride formation shown, for the first time by use of a novel TEM gas-cell stage, at elevated temperature and hydrogen pressure



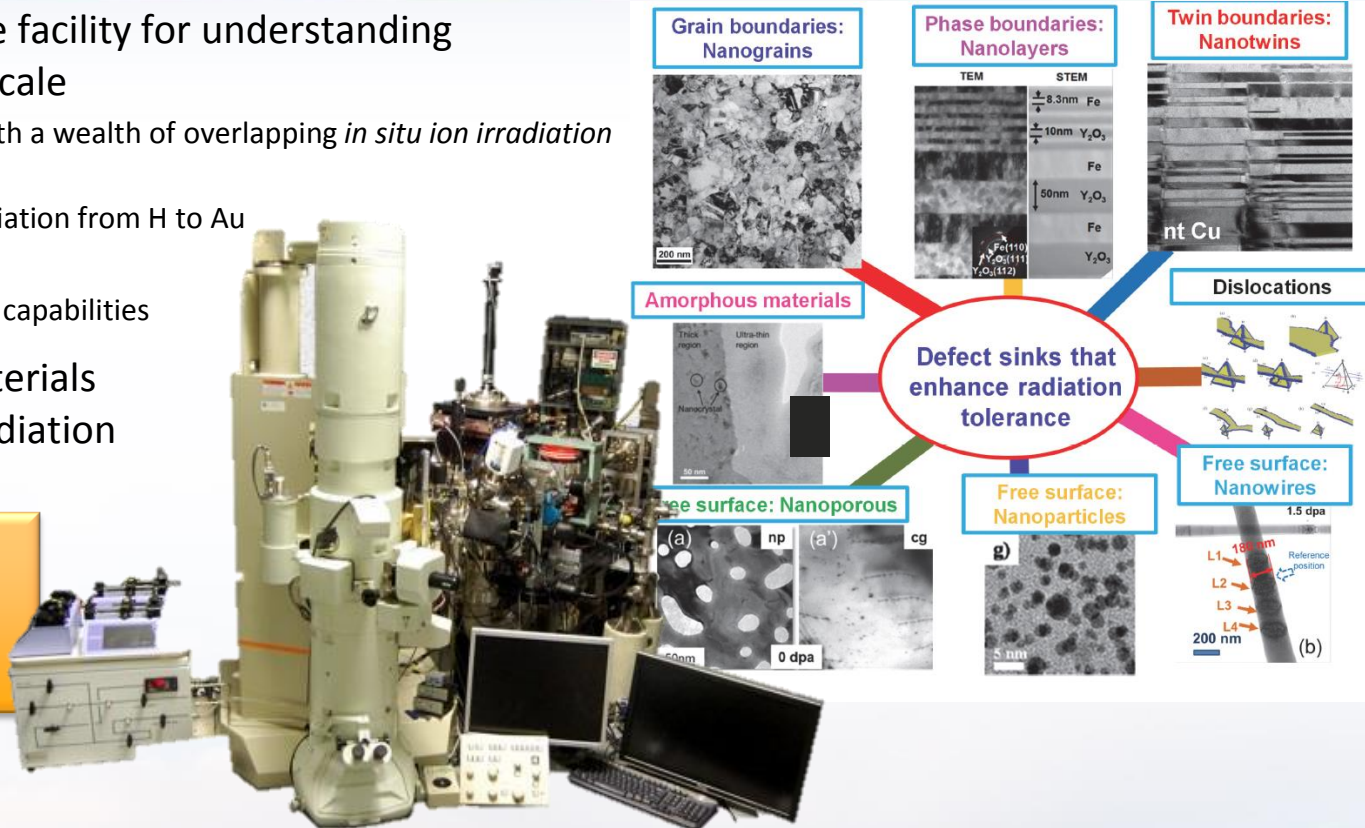


# Summary



- Sandia's I<sup>3</sup>TEM is a unique facility for understanding mechanisms at the nanoscale
  - Only facility in the world with a wealth of overlapping *in situ* ion irradiation capabilities
  - *In situ* high energy ion irradiation from H to Au
  - *In situ* gas implantation
  - 11 TEM stages with various capabilities
- Some nanostructured materials demonstrate improved radiation tolerance, but not all!

Sandia's I<sup>3</sup>TEM although still under development is providing a wealth of interesting initial observations



Collaborators:

- IBL: C.M. Barr, S.A. Briggs, D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, M.T. Marshall, B. Muntifering, S.H. Pratt, & P. Price
- Sandia: M. Abere, B. Boyce, T.J. Boyle, R. Dingreville, R.F. Hess, A.C. Kilgo, B.E. Klamm, W.M. Mook, J.D. Puskar, J.A. Scott, & J.A. Sharon
- External: A. Aitkaliyeva, H. Bei, P.J. Ferreira, K.J. Ganesh, E.P. George, D. Gross, P. Hosemann, J. Kacher, S. Maloy, A. Minor, J. Qu, S. Rajesekhara, I.M. Robertson, D. Stauffer, & Hysitron Inc.

This work was partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.



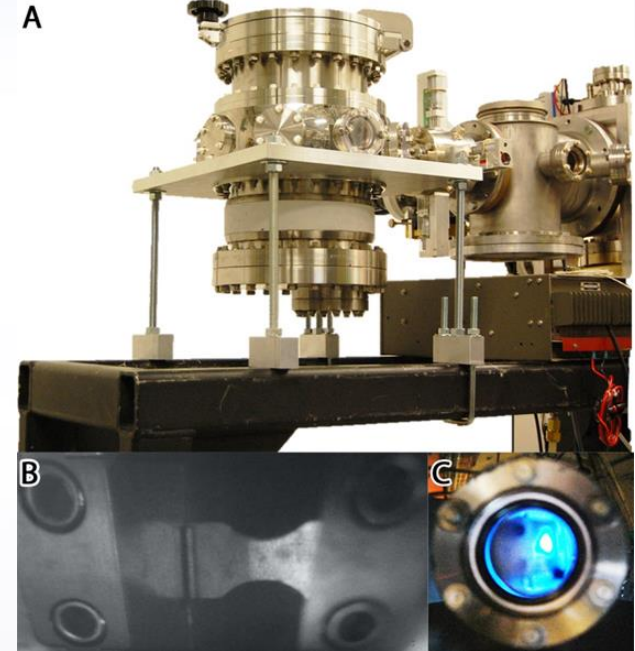
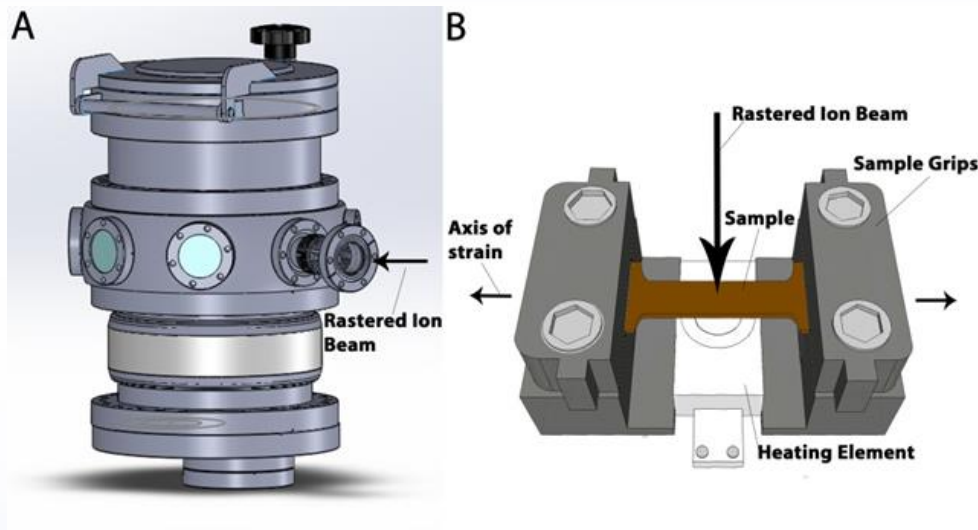


# Back-up Slides



# Mechanical Testing End Station

Collaborator: M. Steckbeck, B. Boyce, T. Furnish, D. Bufford, D. Buller, C. Barr

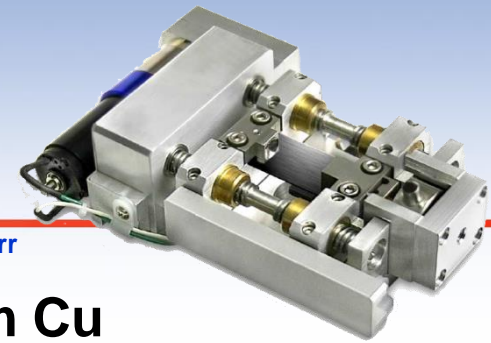


End-station developed consists of a two possible micro-mechanical test frames situated to receive a variety of ion species at energies up to 88 MeV from the 6 MV Van de Graff Tandem accelerator:

1. Commercial MTI/Fullham Multi-use Mechanical Stage (4000 N max): tensile, three point bend, creep, stress relaxation, others
2. Fatigue (custom design) for thin (5 to 10 micron samples)



# Irradiation and Stress Relaxation

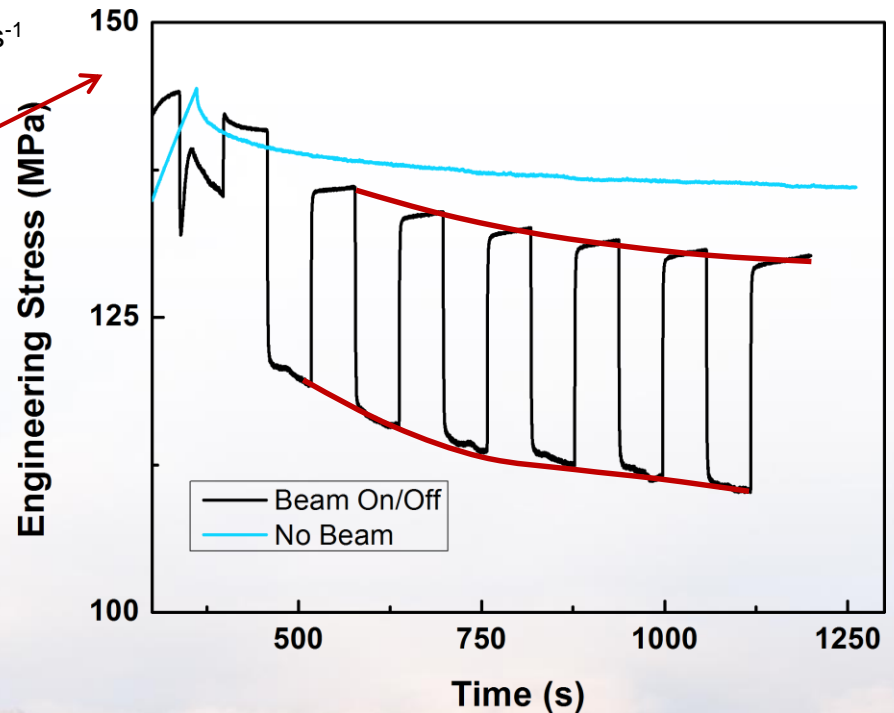
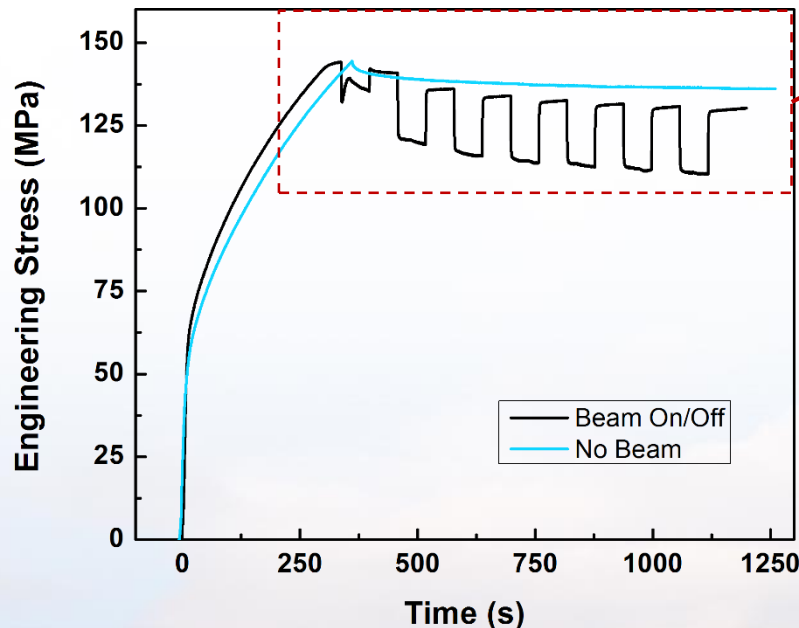


Collaborator: M. Steckbeck, B. Boyce, T. Furnish, D. Bufford, D. Buller, C. Barr

## ■ 0.25mm/min elongation rate to 22.5 N load in 50 $\mu\text{m}$ Cu

- Approximately 75% of typical ultimate tensile load
- 900 s hold at constant position
- Beam cycled on/off at 60 s intervals
- Beam conditions: 4.5 MeV H<sup>+</sup>:  $2.1 \times 10^{11}$  ions cm<sup>-2</sup>s<sup>-1</sup>

MTI/Fullham SEMTester

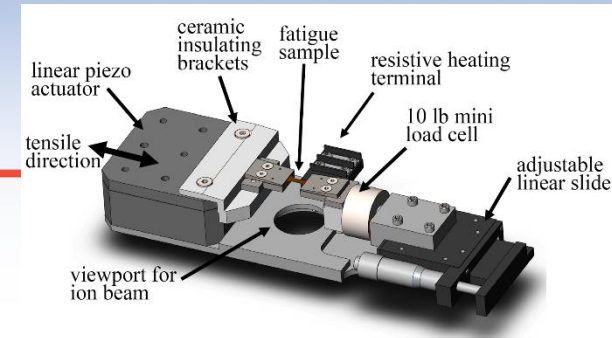


- Offset likely an artifact
- Different relaxation rates with beam on and off

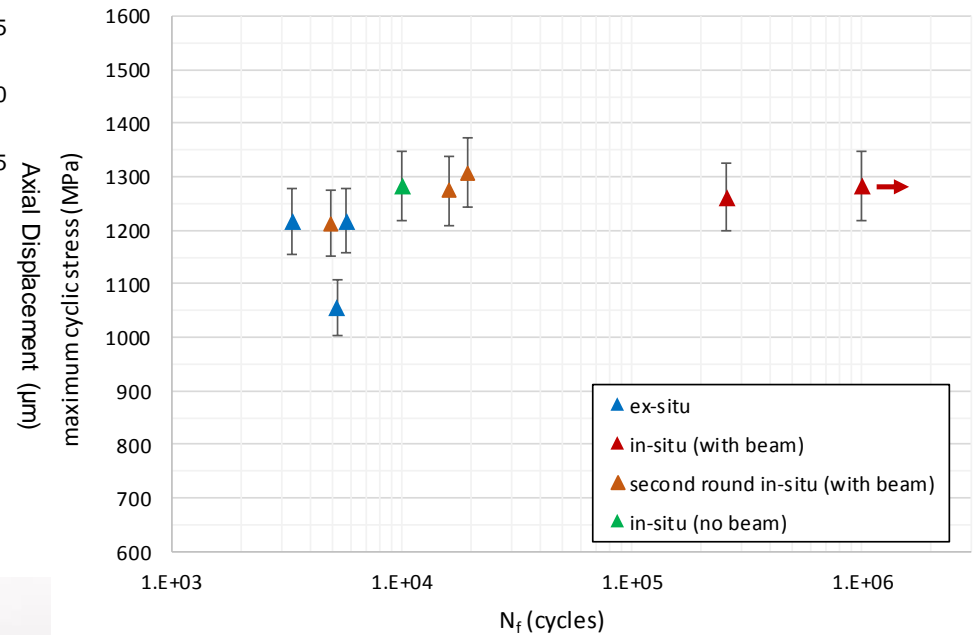
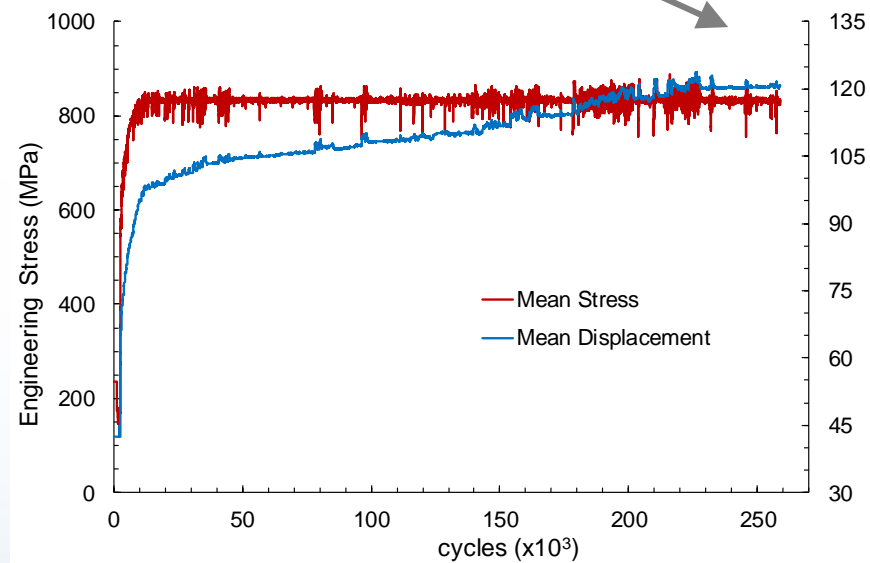


# Irradiation and Fatigue

Collaborator: B. Boyce, T. Furnish, D. Buller



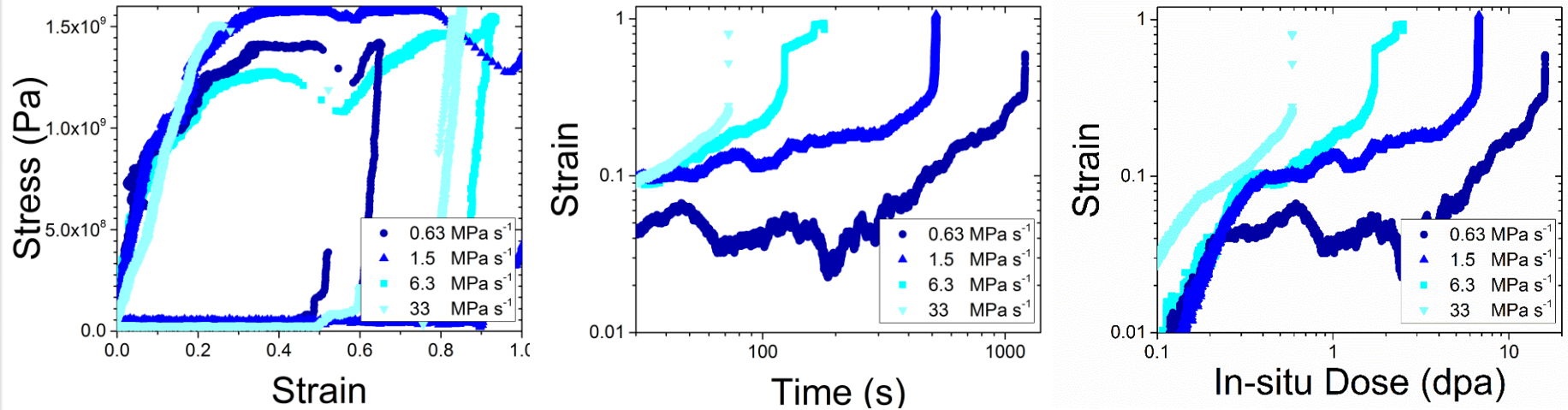
Fatigue Failure after 259k cycles



- Preliminary fatigue results in Ni-Fe alloy with both irradiation beam on sample (10 MeV He<sup>+</sup>) and beam off sample.
- Custom mechanical stage designed for thin samples (5 to 10  $\mu\text{m}$  thickness: suitable for allowing entire thickness to be irradiated with raster or defocused beam)

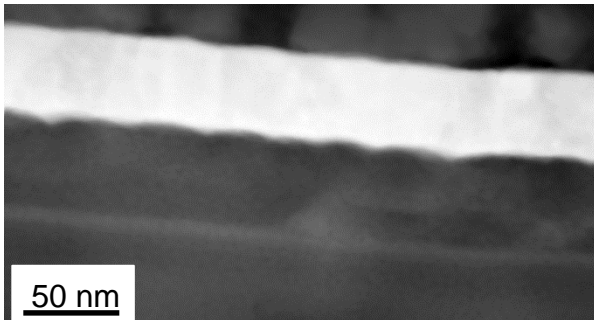


# Creep Response at Different Loading Rates

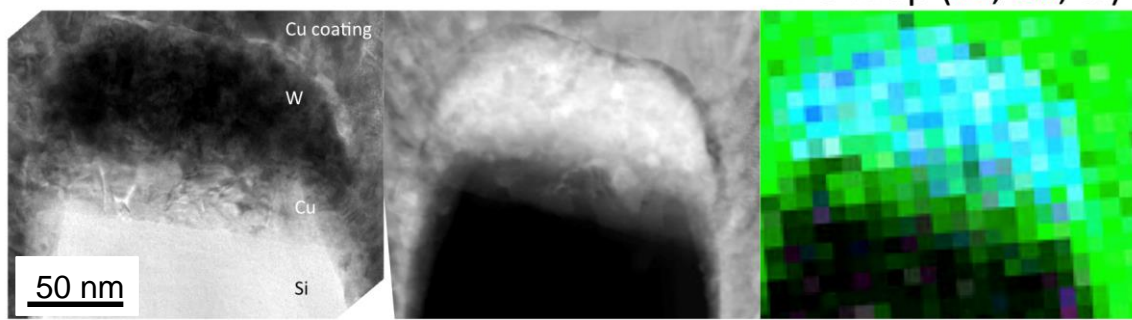


*Significant creep observed at a fraction of the bulk yield strength*

As-deposited Sample  
ADF-STEM



Post Creep Characterization  
BF-TEM ADF-STEM EDS Map (W, Cu, Si)



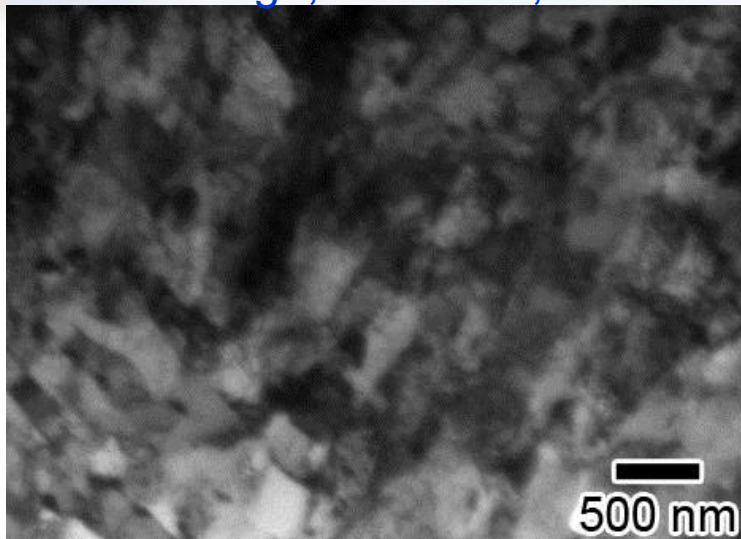
*Compression (creep) only observed in Cu layer*

# *In situ* TEM Ion Irradiation

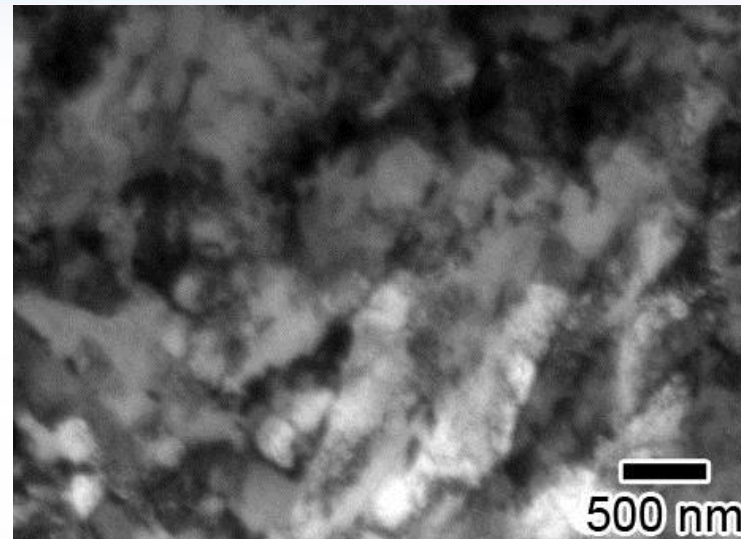
HT9 3 MeV Cu<sup>3+</sup> at ~10 nA RT

Collaborators: A. Kilgo, J. Puskar, and S. Maloy

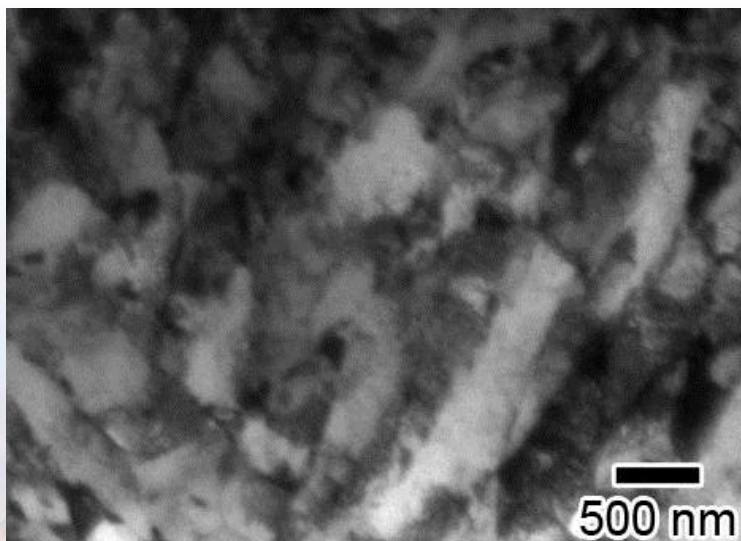
Initial



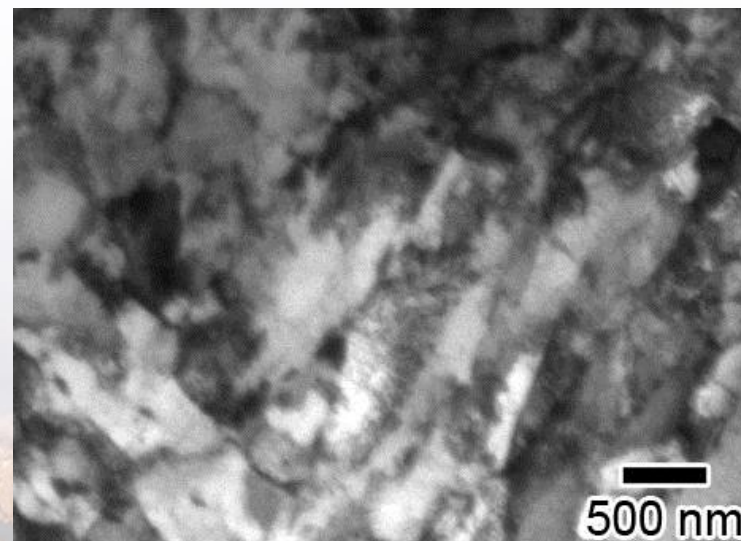
2 hrs



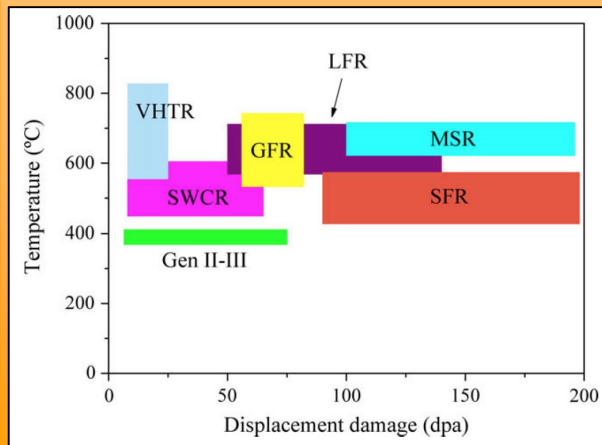
3 hrs



6 hrs



# Radiation management in materials



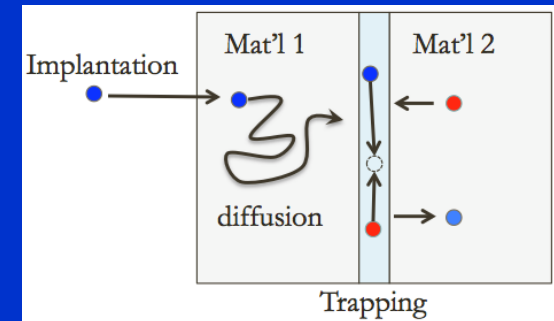
High temperature  
+  
Intense radiation  
+  
Unfailing mechanical  
integrity

**Coarse grain materials:**  
Thermally stable, but  
weak to rad effects (e.g. embrittlement)

**Nanocrystalline materials:**  
Improved rad tolerance but  
thermally unstable (coarsening)

## → Solution:

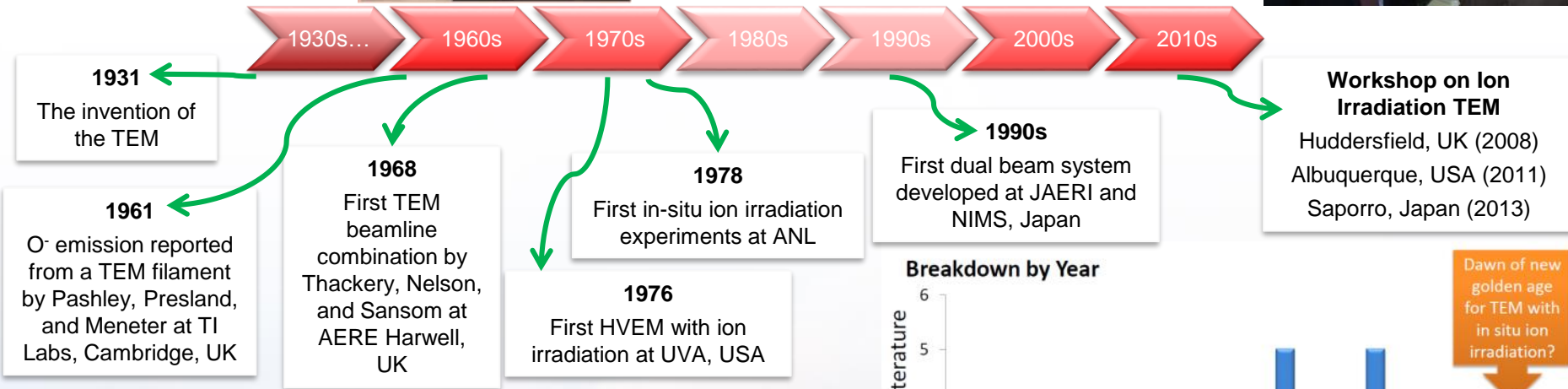
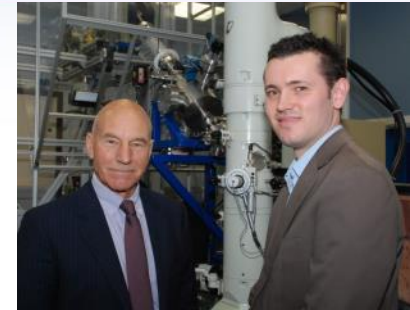
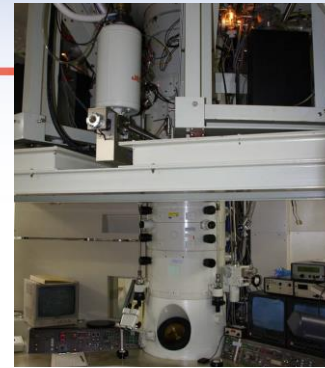
- Radiation-induced defect management:
  - **High density of trapping sites:** free surface vs. interfaces
  - **Sink efficiency:** ability of promoting recombination and annihilation



# History of *In situ* Ion Irradiation TEM



Courtesy of: J. Hinks

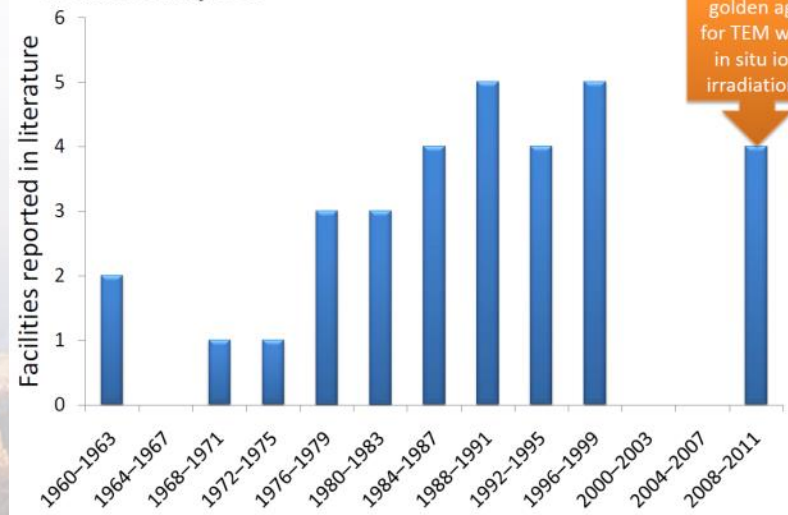


**“The direct observation of ion damage in the electron microscope thus represents a powerful means of studying radiation damage”**



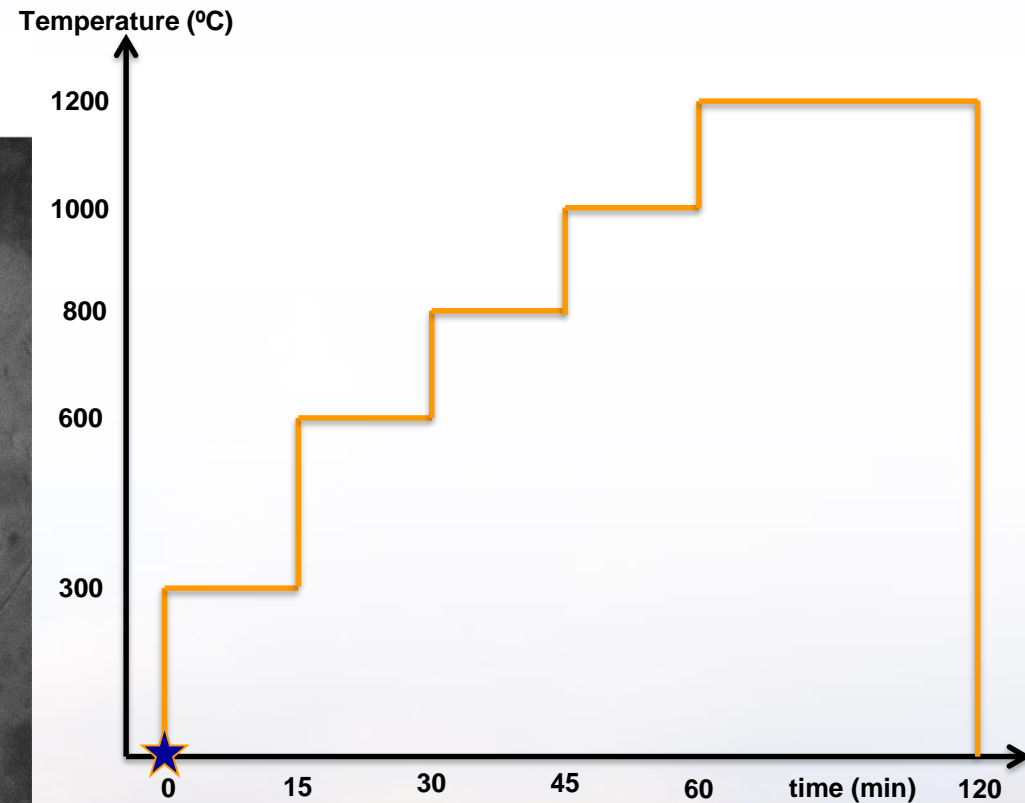
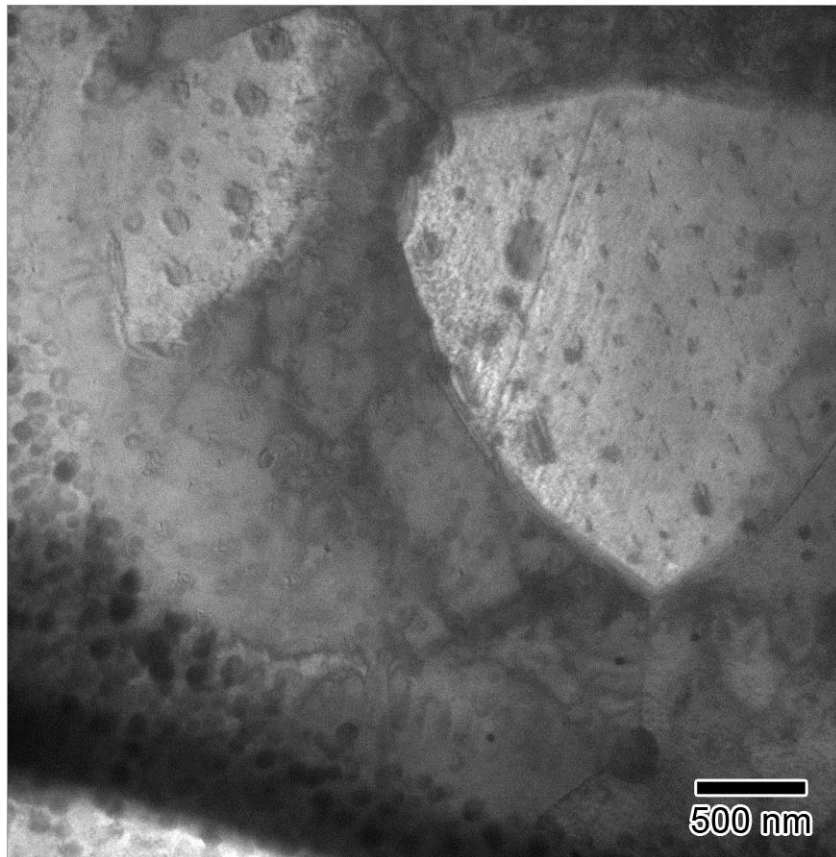
D.W. Pashley and A.E.B. Presland *Phil Mag.* 6(68) 1961 p. 1003

**Breakdown by Year**



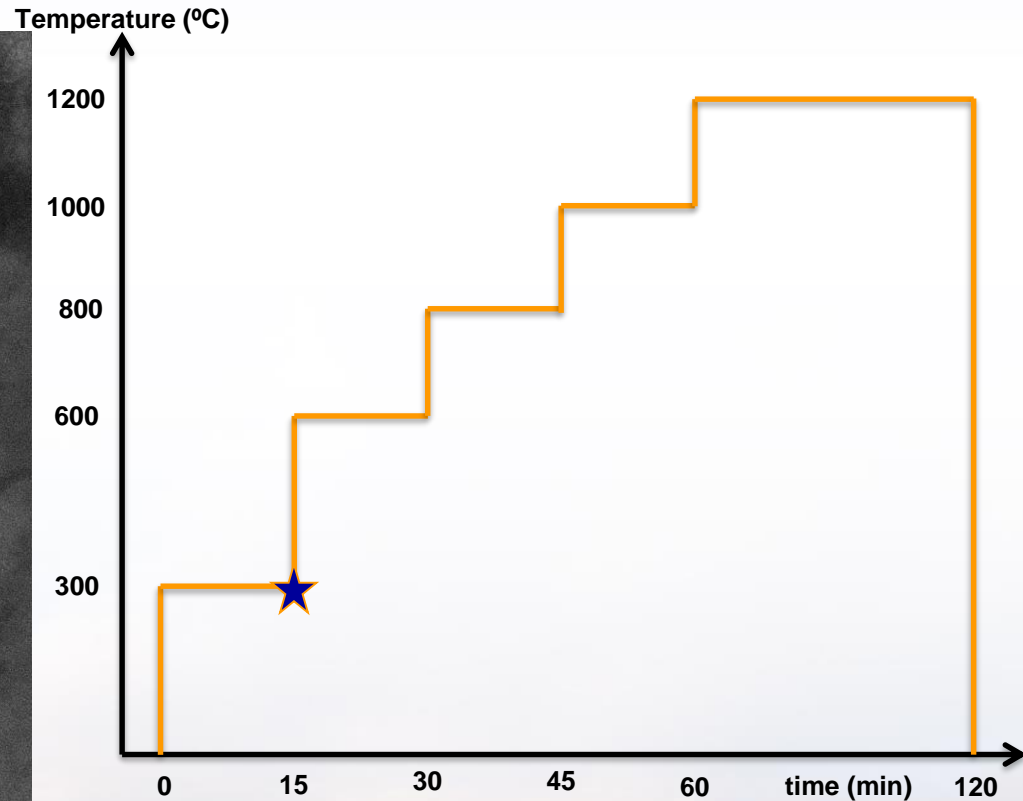
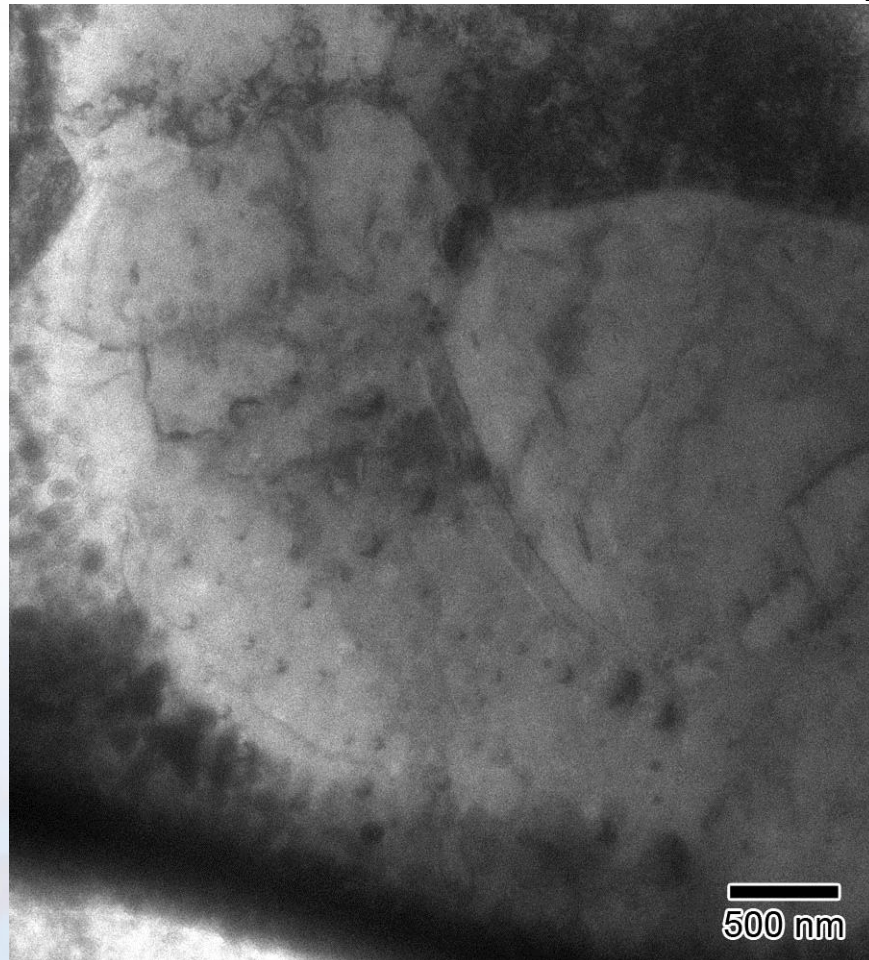
# *In situ* TEM Heating of Zircaloy

Collaborators: S. Rajasekhara and B.G. Clark



# *In situ* TEM Heating of Zircaloy

Collaborators: S. Rajasekhara and B.G. Clark

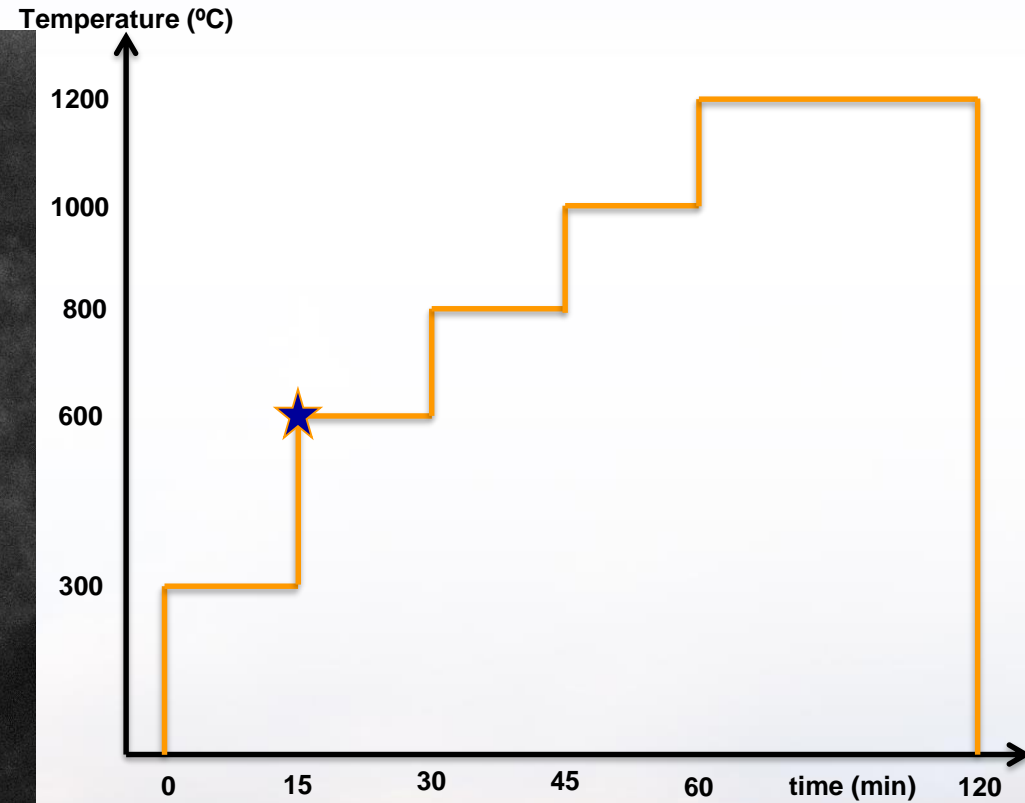
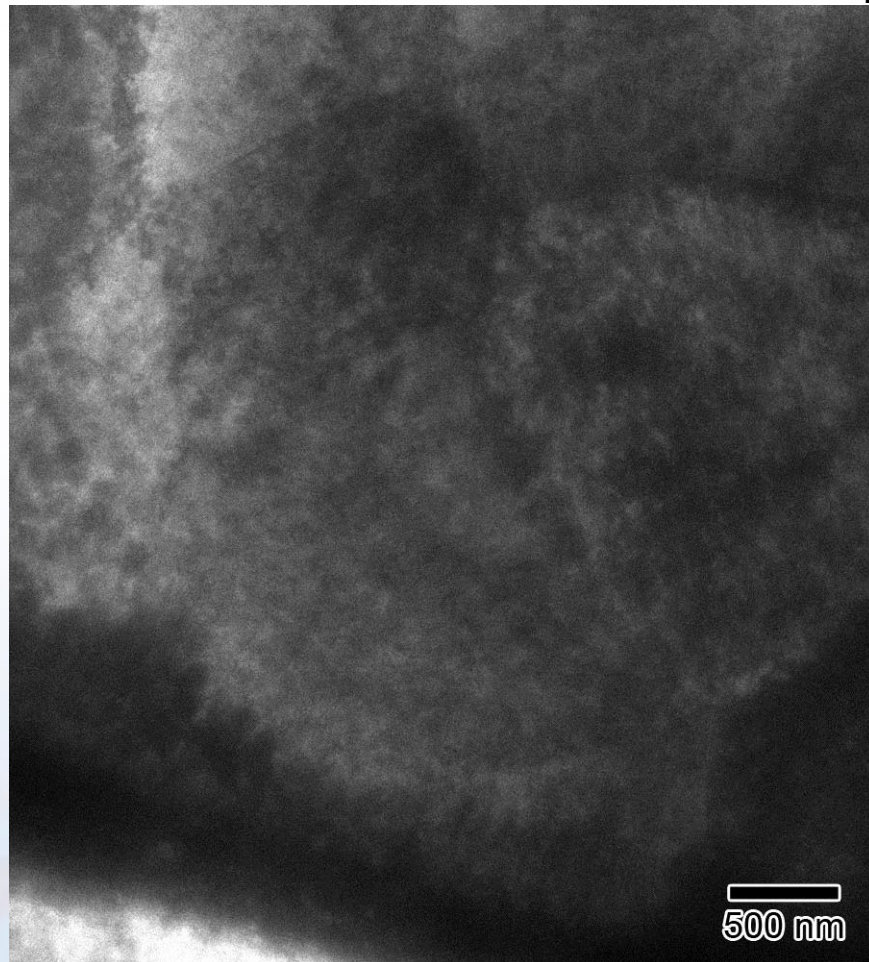


Some of the contamination appears to lessen



# *In situ* TEM Heating of Zircaloy

Collaborators: S. Rajasekhara and B.G. Clark

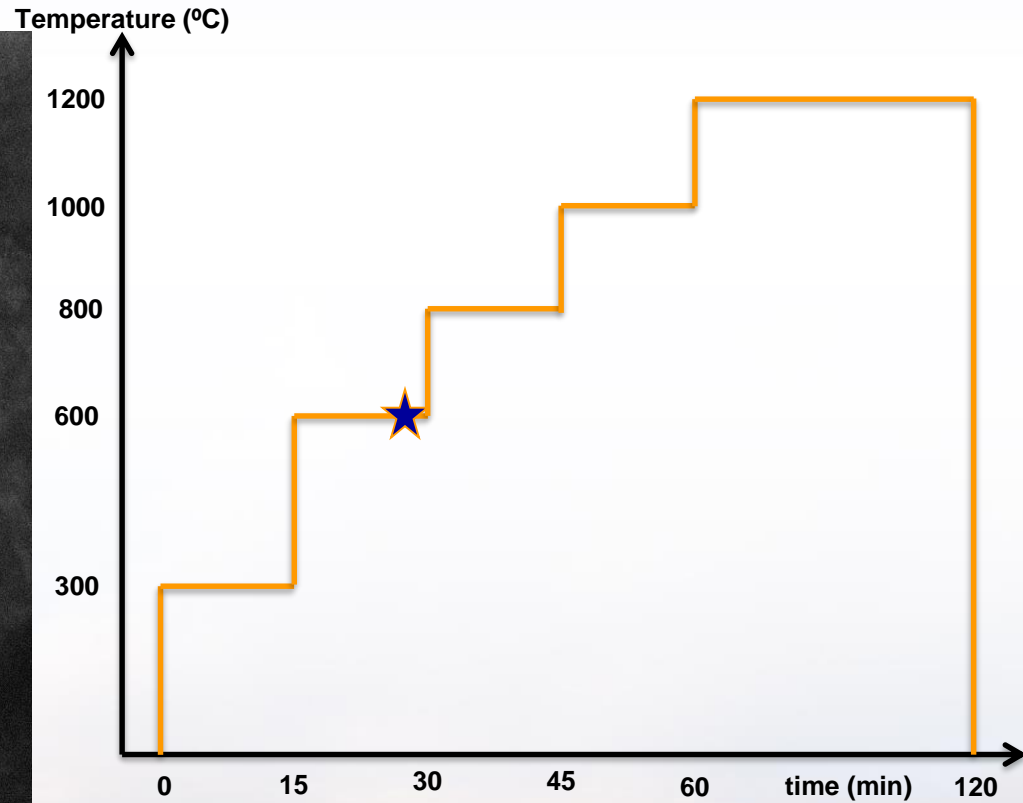
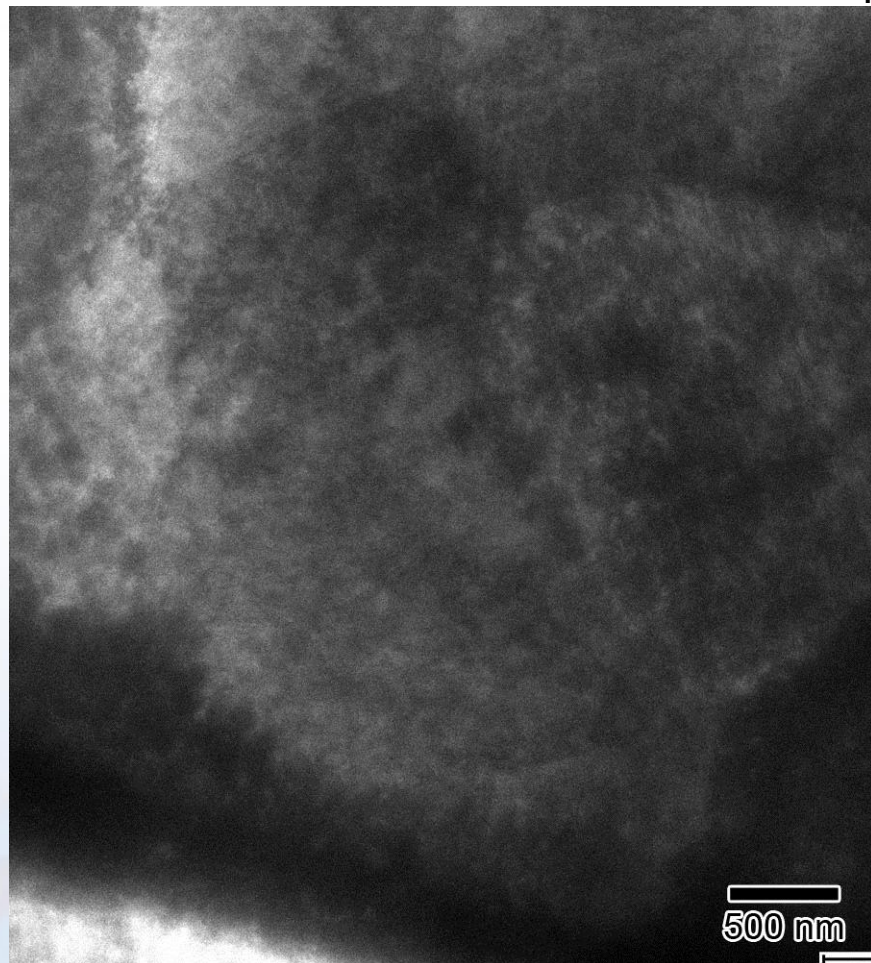


**Drastic microstructural evolution  
due to exponential oxidation**



# *In situ* TEM Heating of Zircaloy

Collaborators: S. Rajasekhara and B.G. Clark

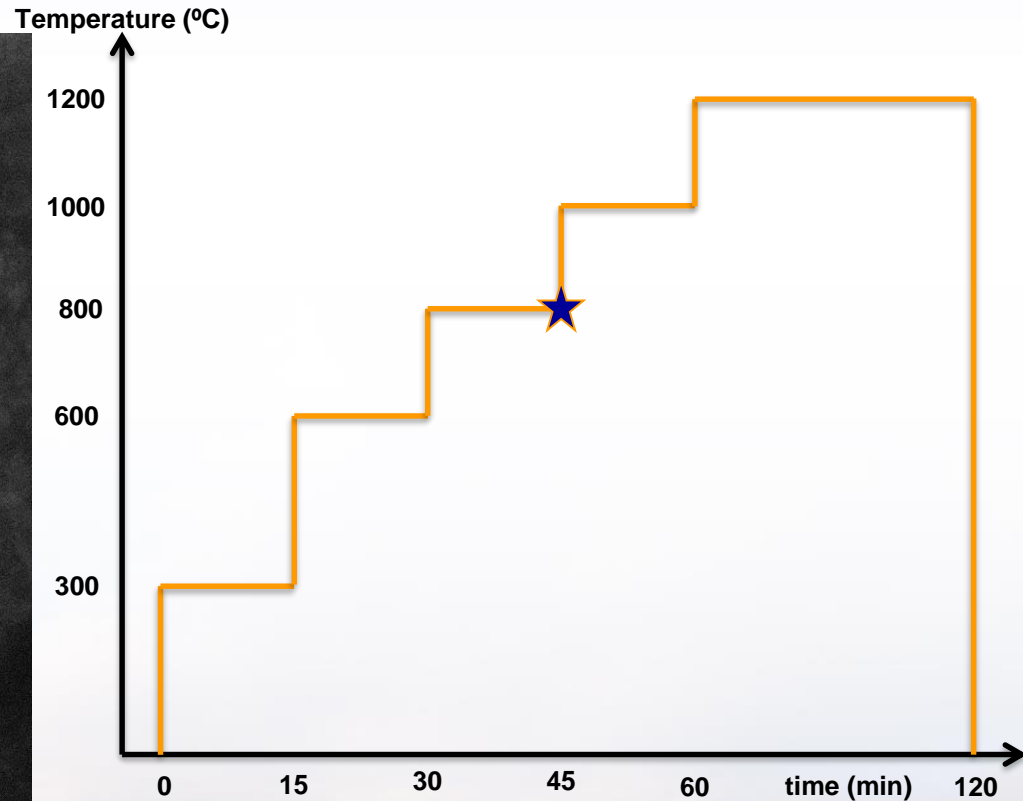
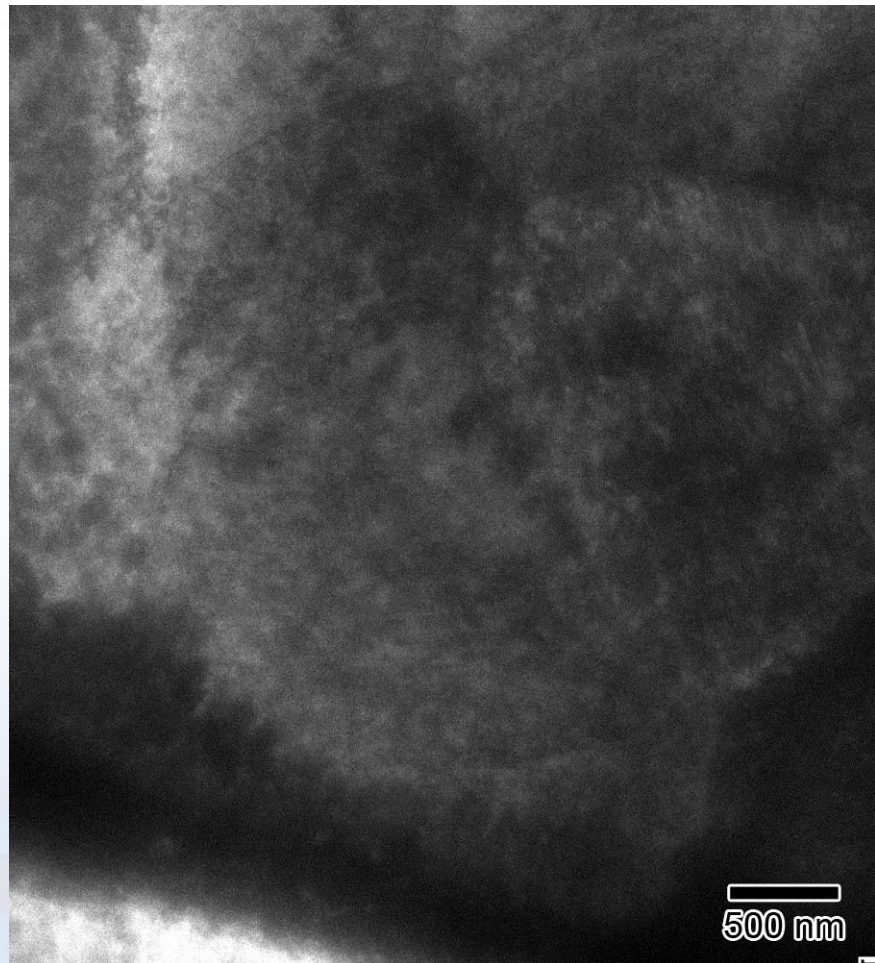


Minimal change in the microstructure



# *In situ* TEM Heating of Zircaloy

Collaborators: S. Rajasekhara and B.G. Clark

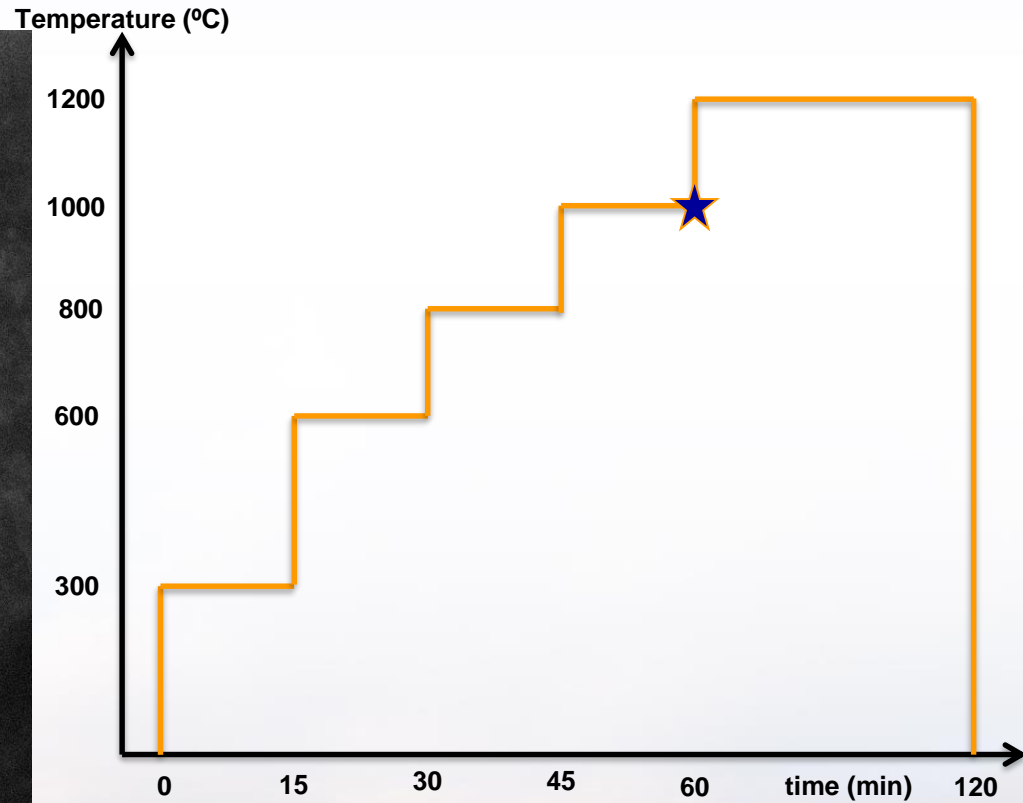
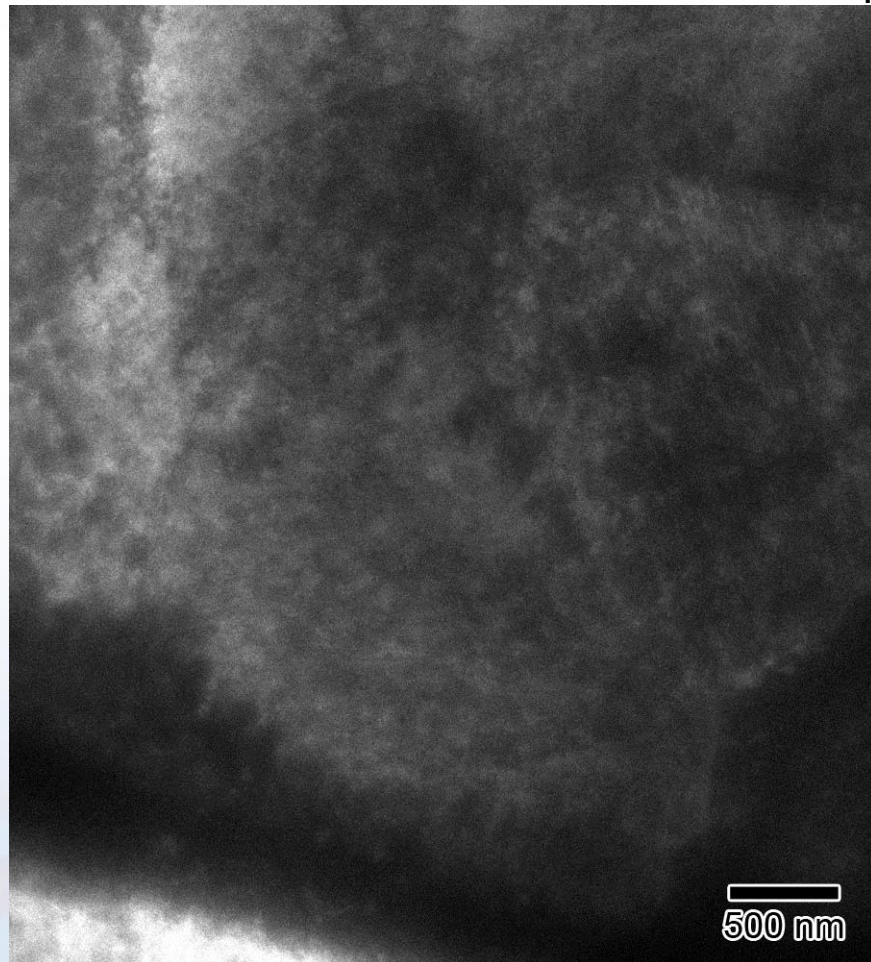


Should have undergone rapid oxidation during this temperature increase



# *In situ* TEM Heating of Zircaloy

Collaborators: S. Rajasekhara and B.G. Clark

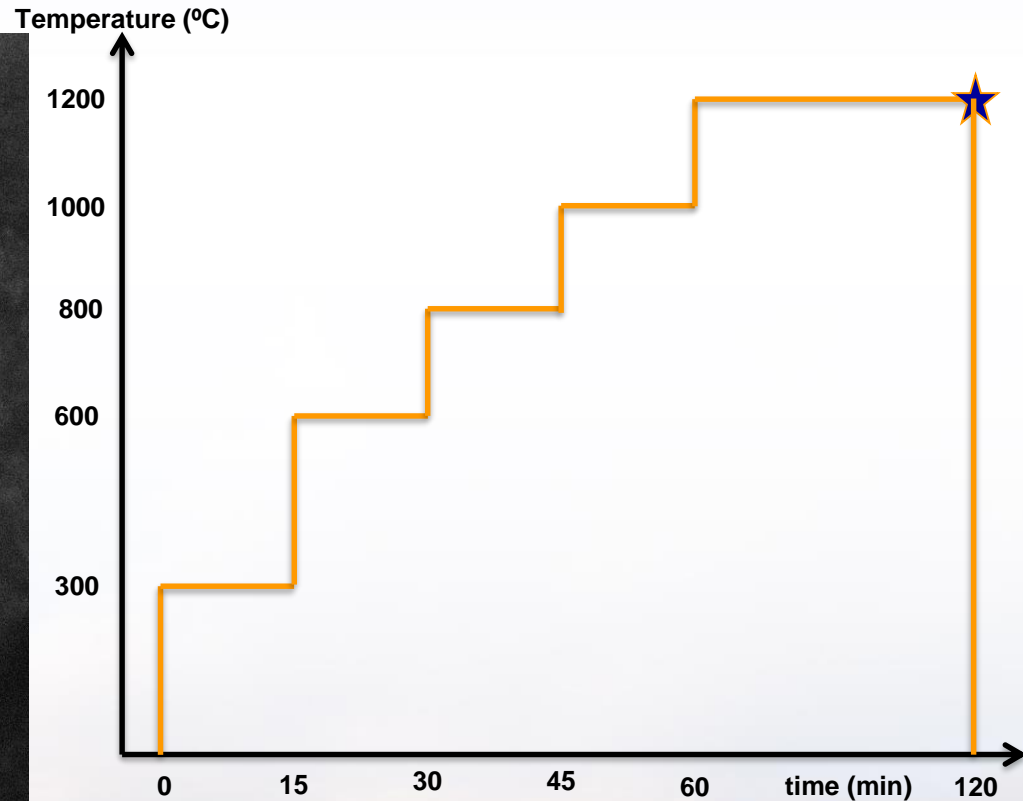
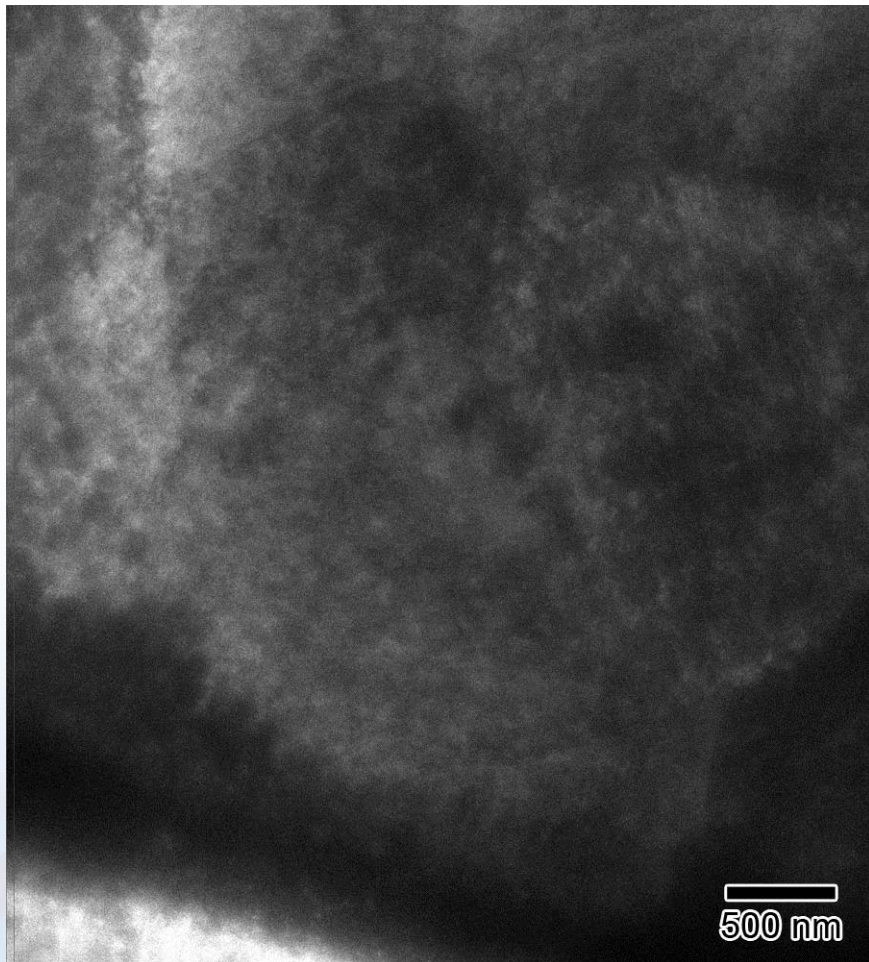


Limited microstructural change even at 1,000° C



# *In situ* TEM Heating of Zircaloy

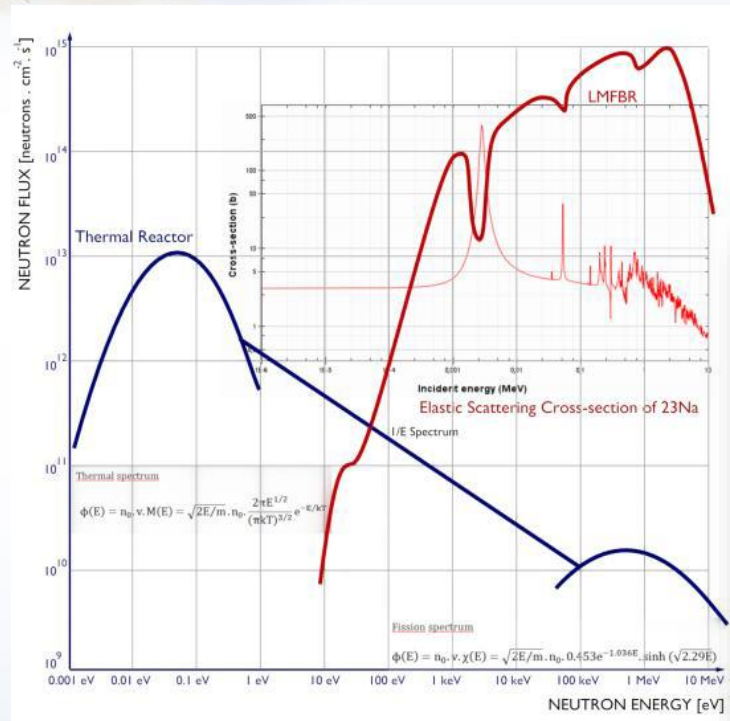
Collaborators: S. Rajasekhara and B.G. Clark



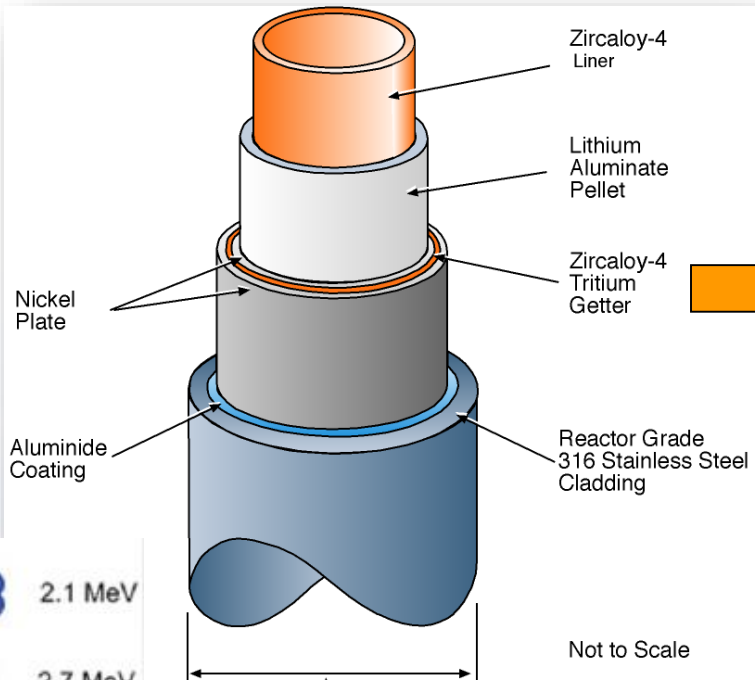
**Both stage and FIB foil are stable  
for 1 hour at 1,200 °C**



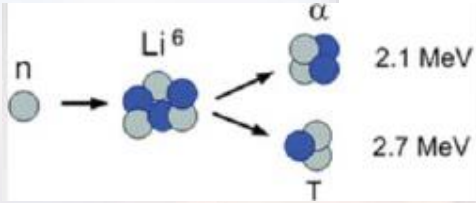
# Applying the Triple Beam Irradiation to Deconvolute Reactor Environments



## Tritium Producing Burnable Absorber Rod (TPBAR)

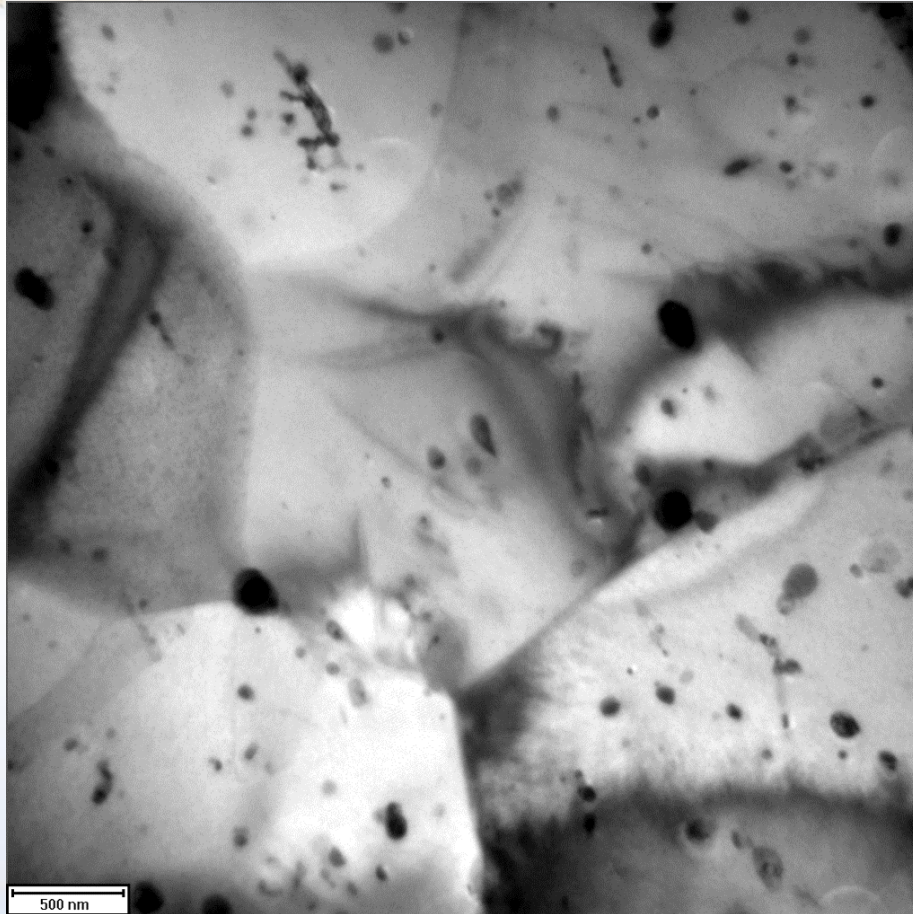


- Displacement Damage
- Helium Implantation
- Tritium Implantation
- Elevated Temperatures

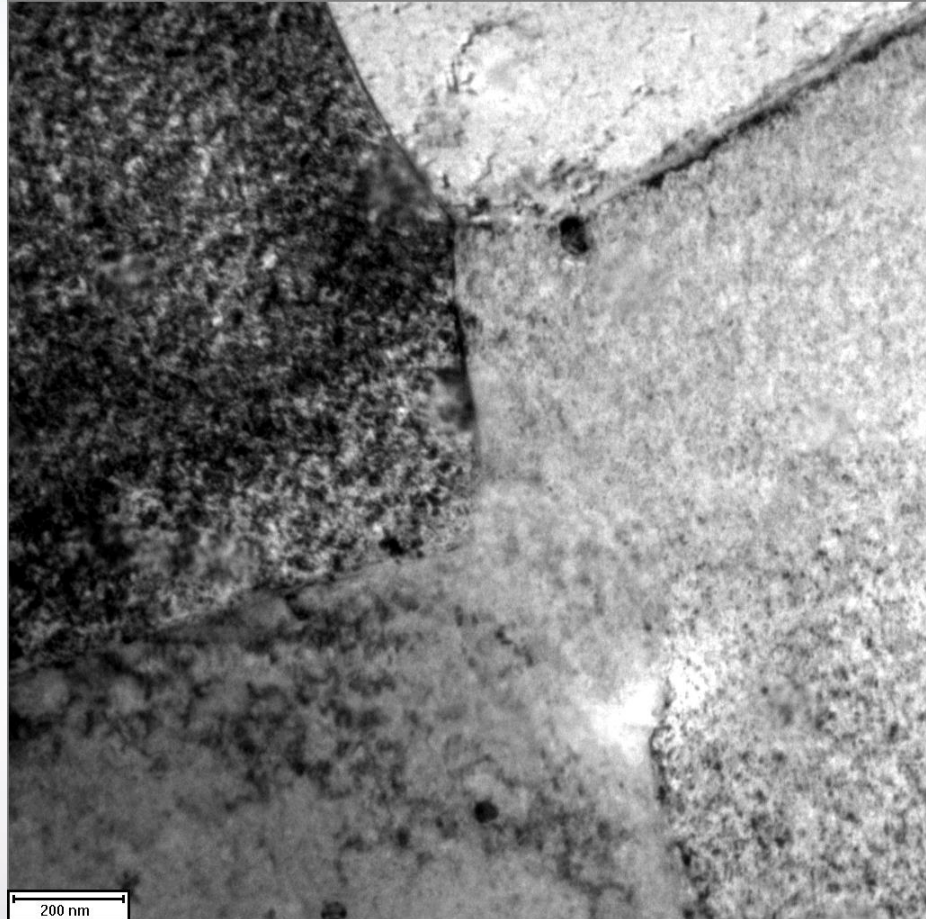


Simulating neutron irradiation in a reactor is complicated, and TPBAR adds the additional complication of <sup>3</sup>H production

# 3 MeV Self Ion Irradiation into Zr-4 at 310 C



Before Irradiation



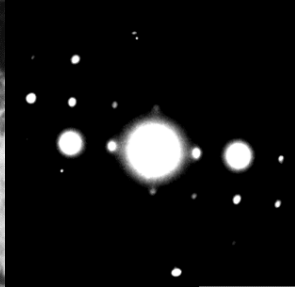
After Irradiation  
 $\approx 7$  DPA



# 10 keV He<sup>+</sup> Implantation

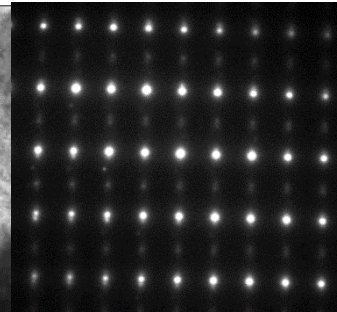
## Then 3 MeV Self Ion Irradiation into Zr-4 at 310 C

Two Beam  
g = 0002



On Axis

Two Beam  
g = 01-12

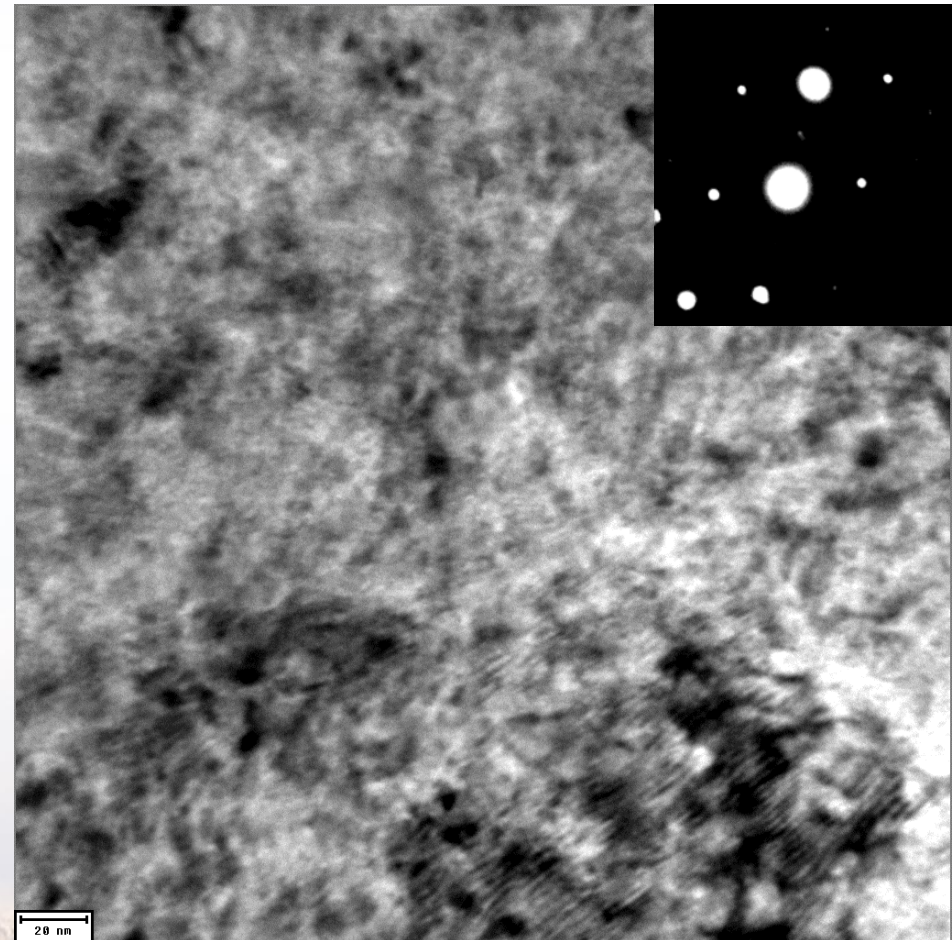
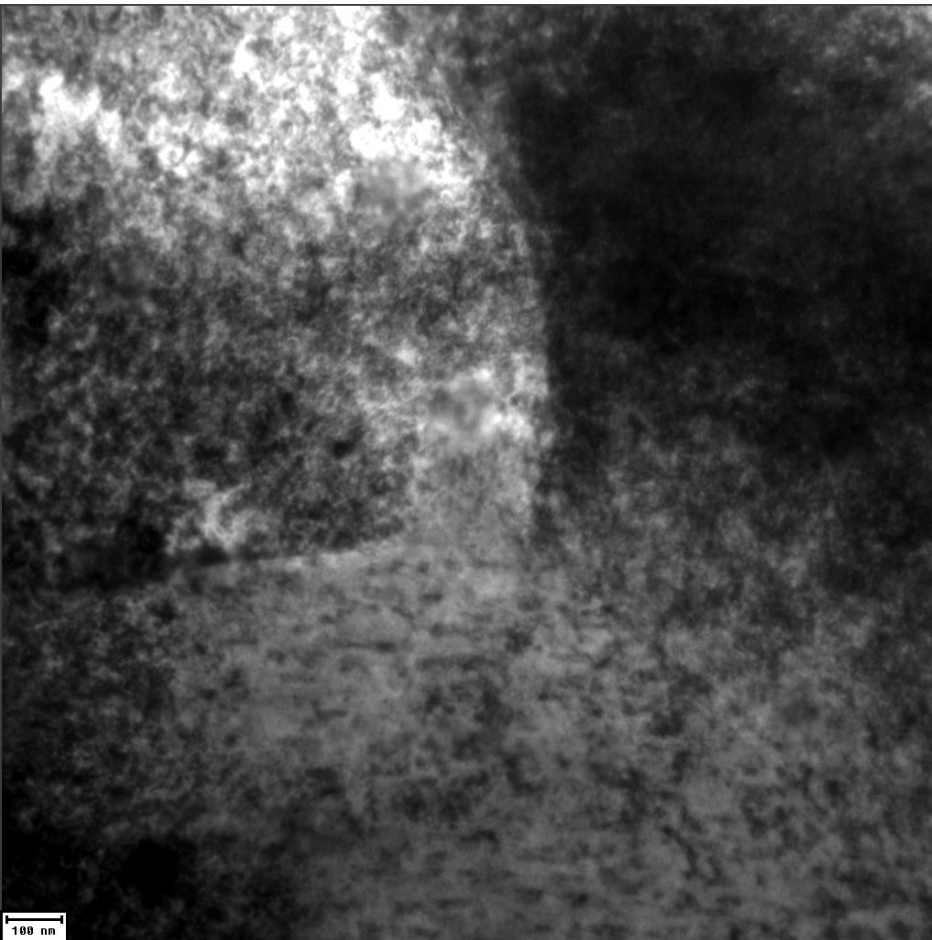


Sequence matters, as  
well as imaging  
condition!



200 nm

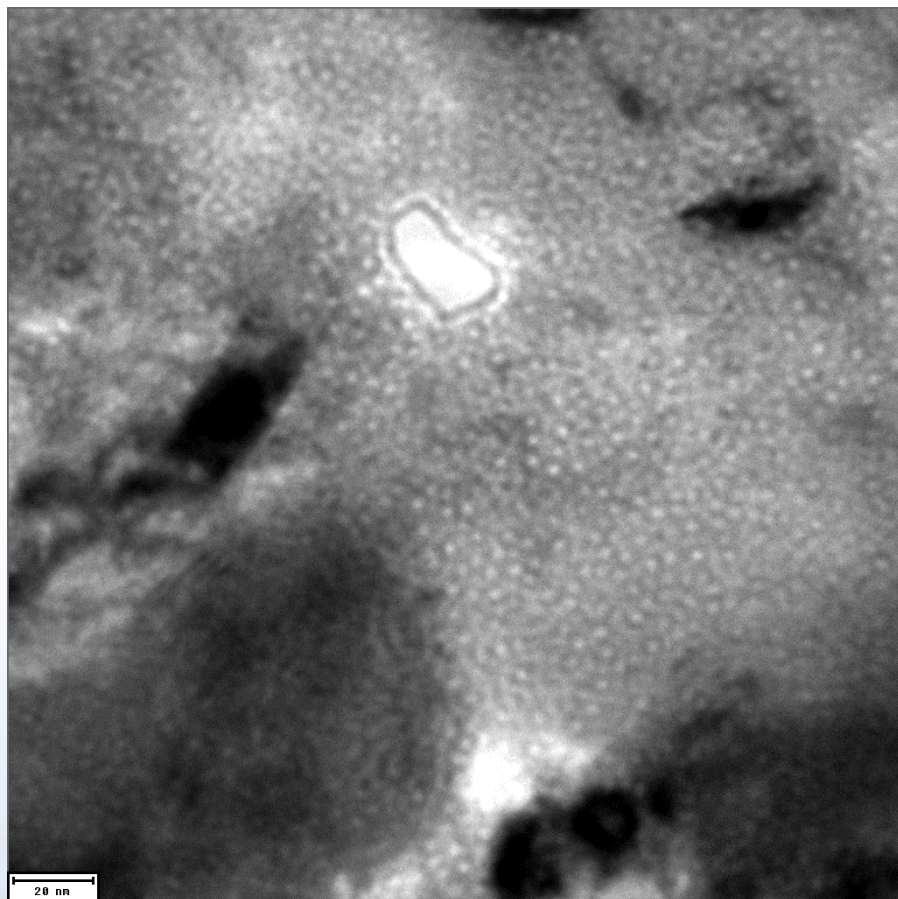
# Concurrent D & He Implantation & Zr Irradiation into Zr-4 at 310 C



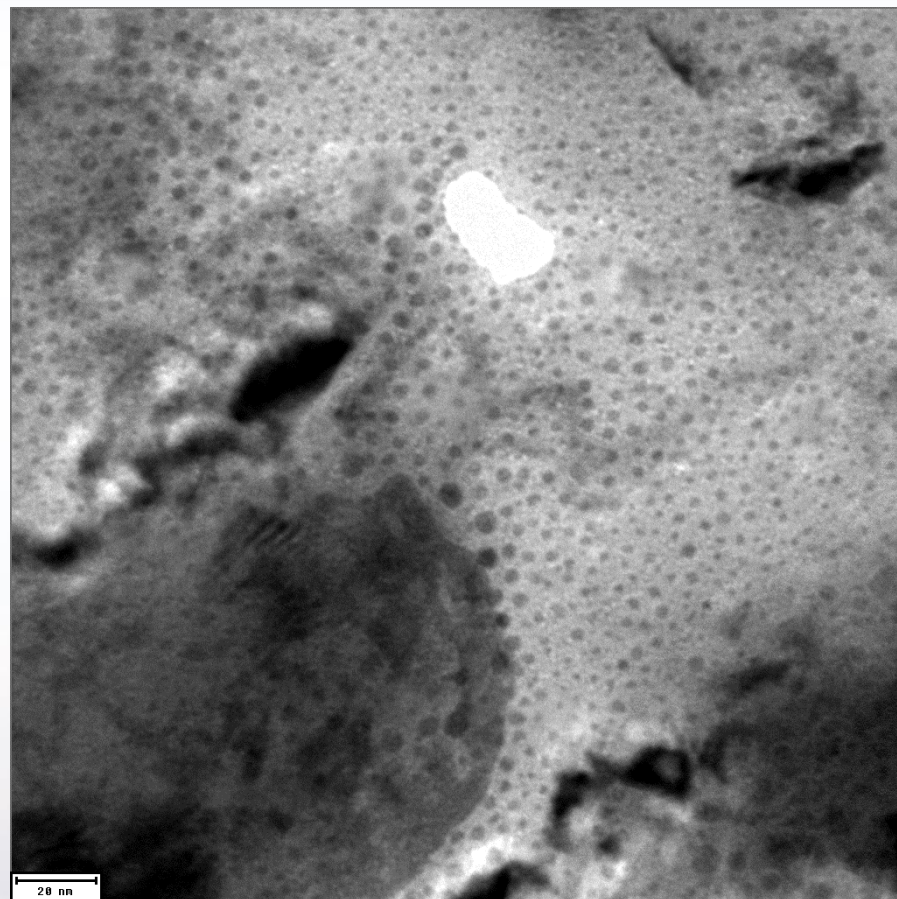
After Implantation/Irradiation  
Significant Damage, but No Cavities

Two Beam  $g = 1\bar{1}01$

# PIE: Through Focus Images 30 Days After the *In situ* Experiment



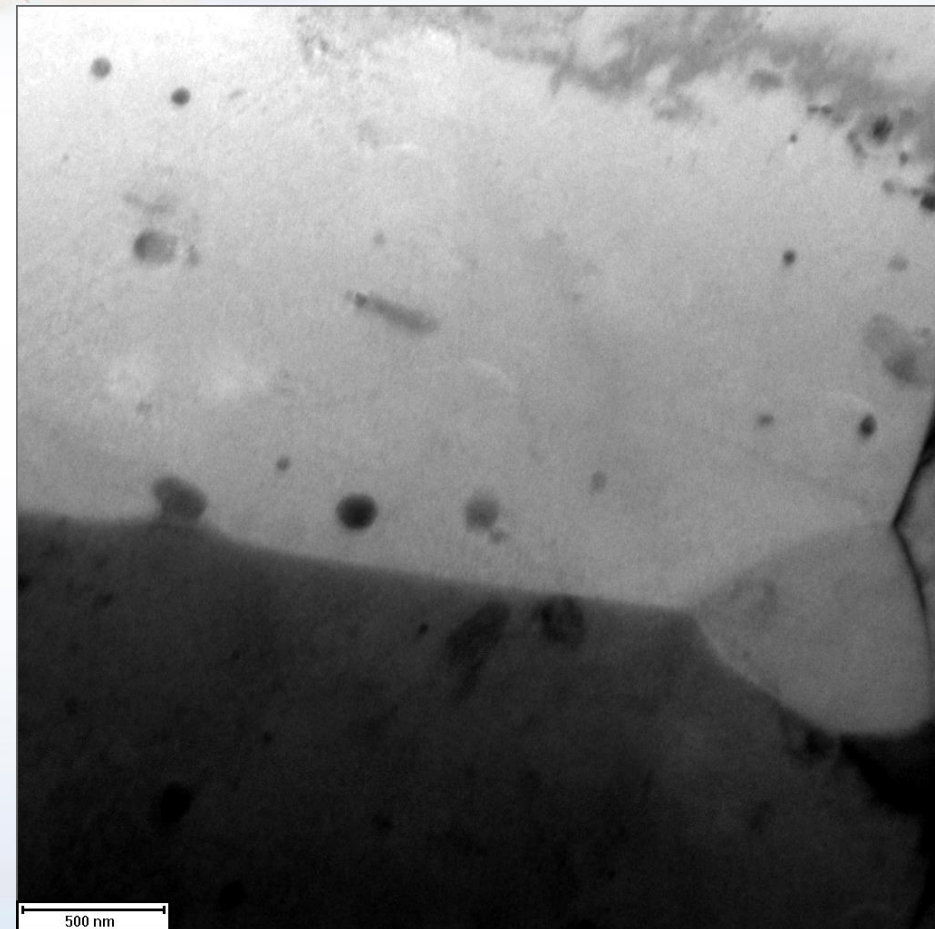
Under Focus



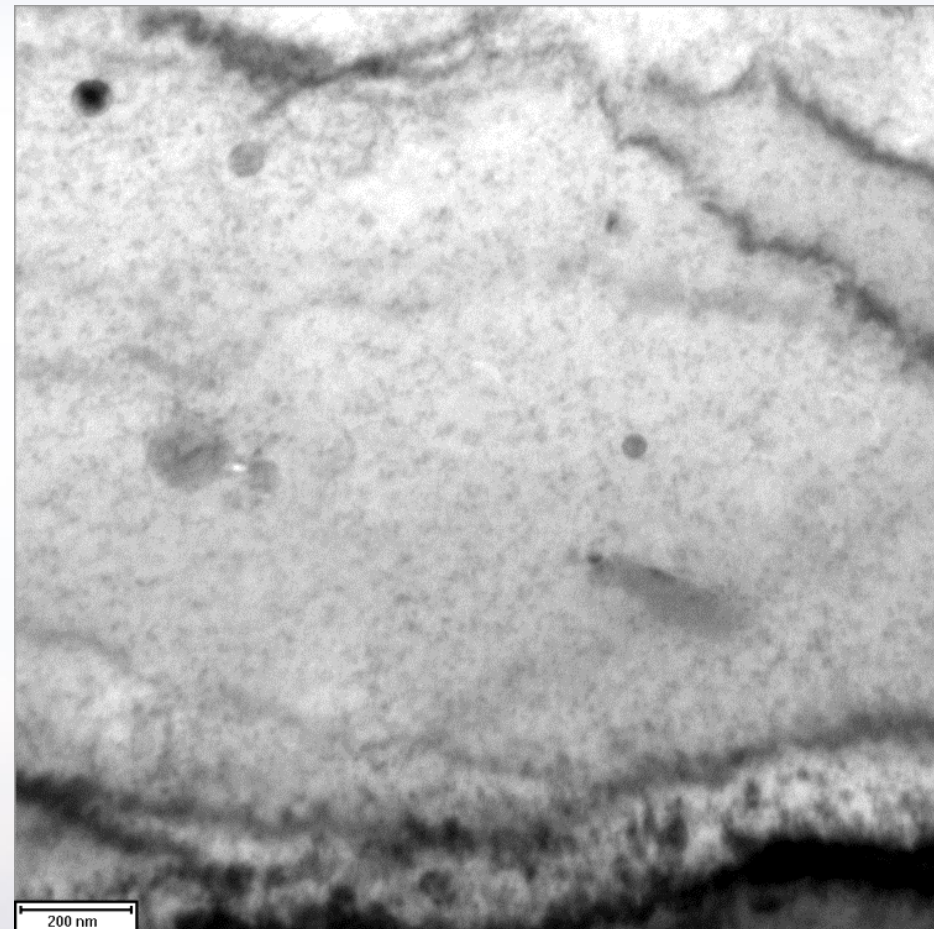
Over Focus



# 10 keV He<sup>+</sup> Implantation into Zr-4 at 310 C



Before Implantation

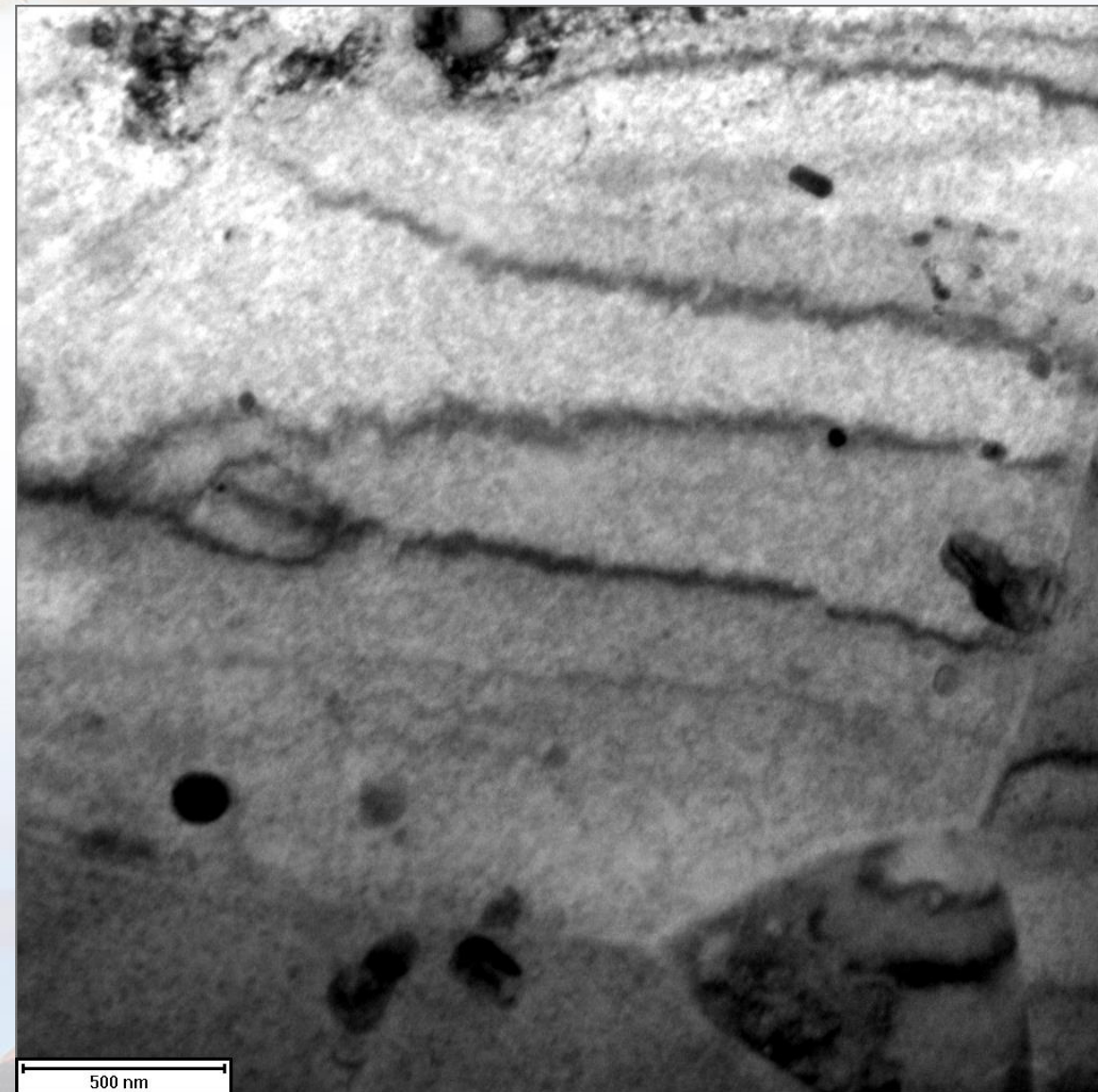


After Implantation  
Damage, No Cavities



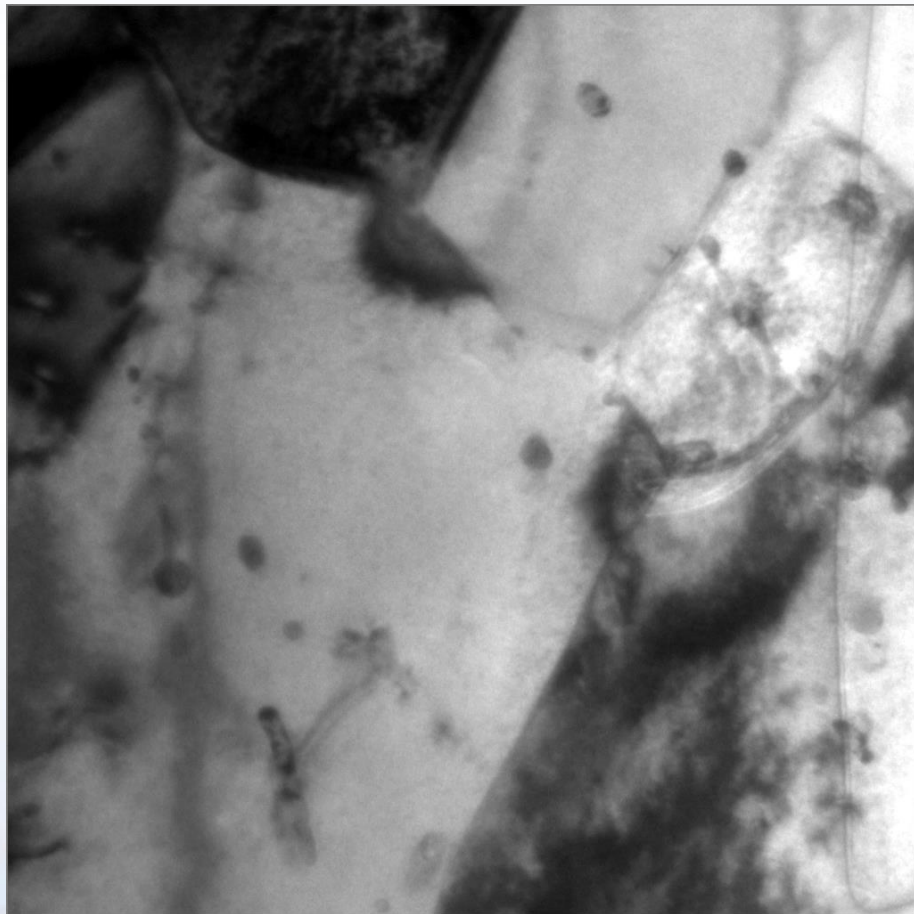
## 3 MeV Self Ion Irradiation

Then 10 keV He<sup>+</sup> Implantation into Zr-4 at 310 C

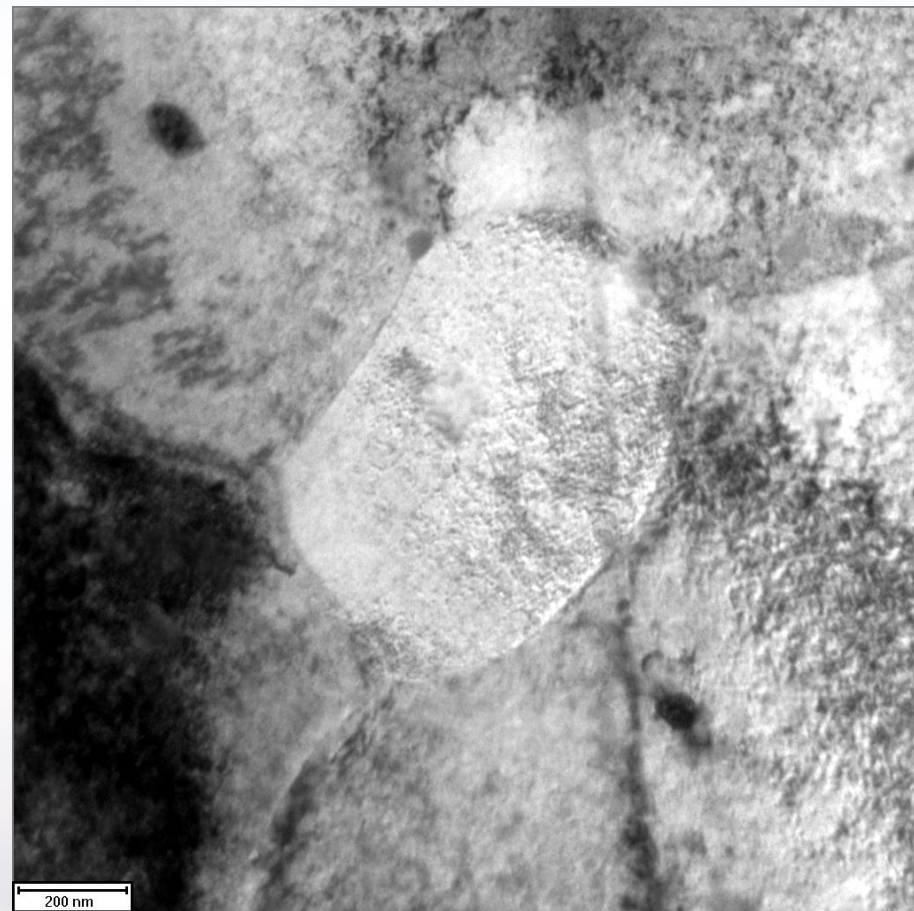


High Density of Defects but  
No Cavities

# Concurrent 3 MeV Self Ion Irradiation & 10 keV He<sup>+</sup> Implantation into Zr-4 at 310 C



Before Implantation/Irradiation



After Implantation/Irradiation  
Damage, No Cavities

