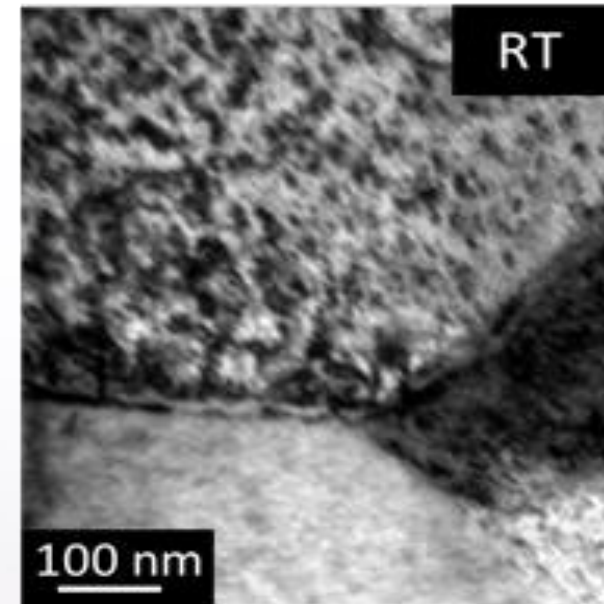
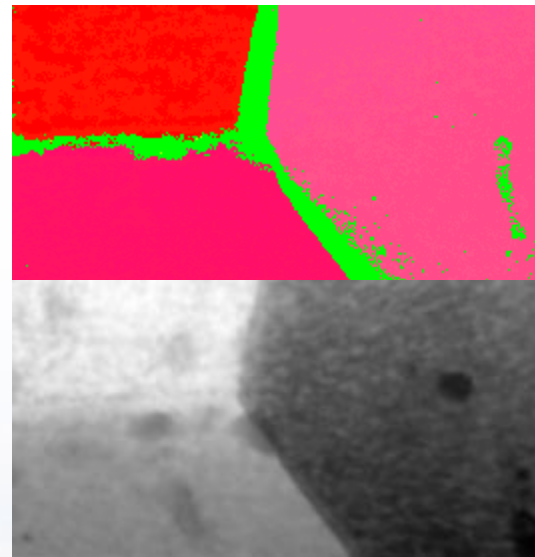
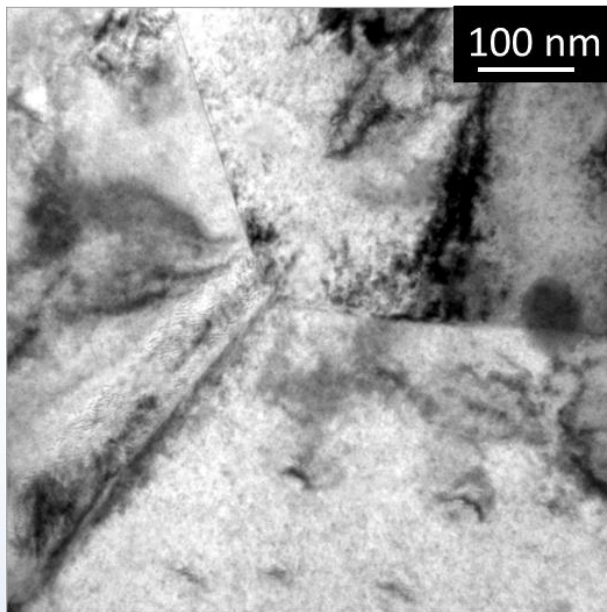


# Exploring the Stability of TPBAR Liner with *In situ* Triple Beam Ion Irradiation TEM



Brittany Muntifering, Caitlin Taylor,  
Clark Snow, Dave Senior, & Khalid Hattar

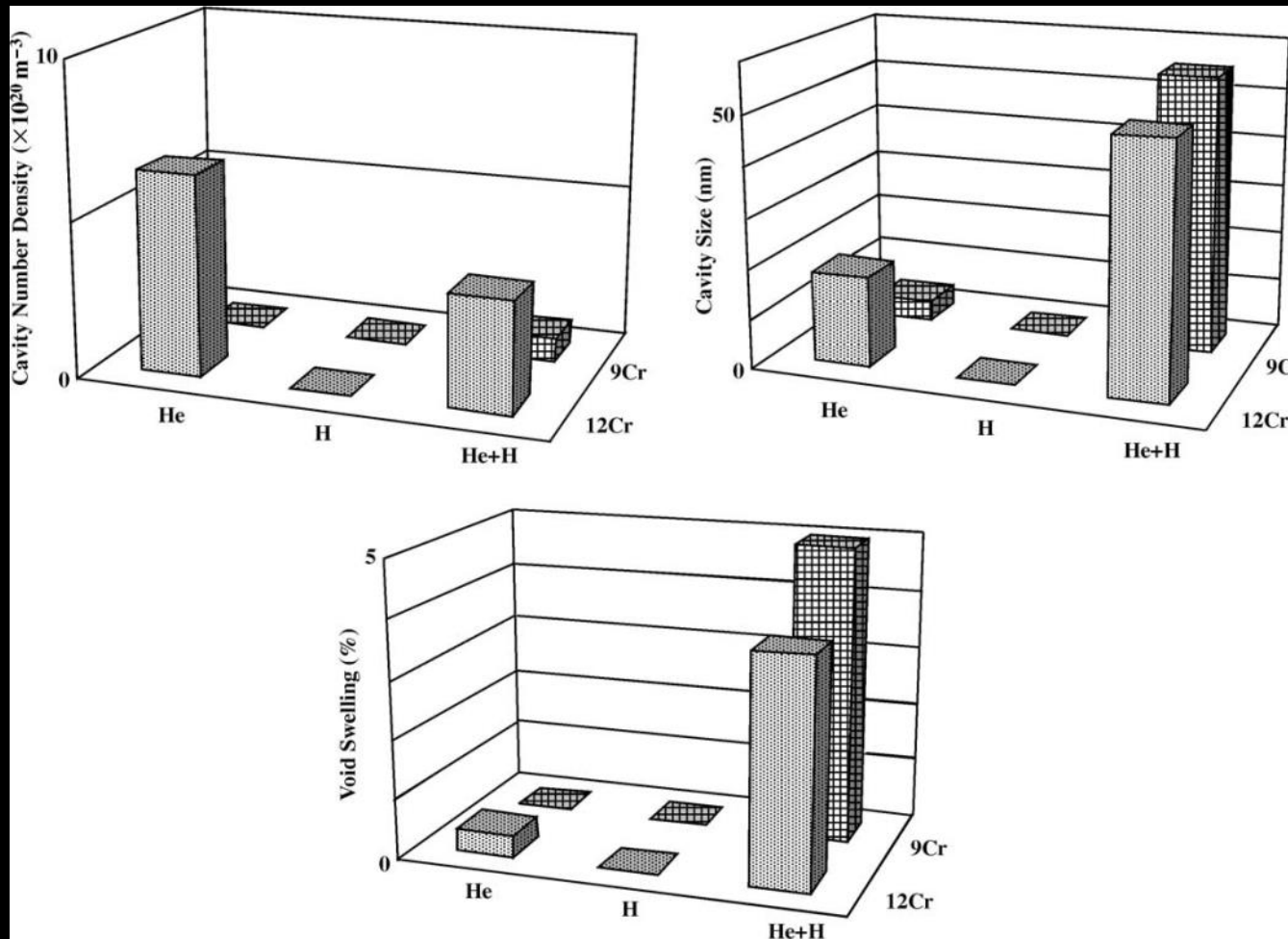


Sandia National Laboratories is a multi-mission laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.



Sandia National Laboratories

# H, He, and Displacement Damage Synergy



## 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

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

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

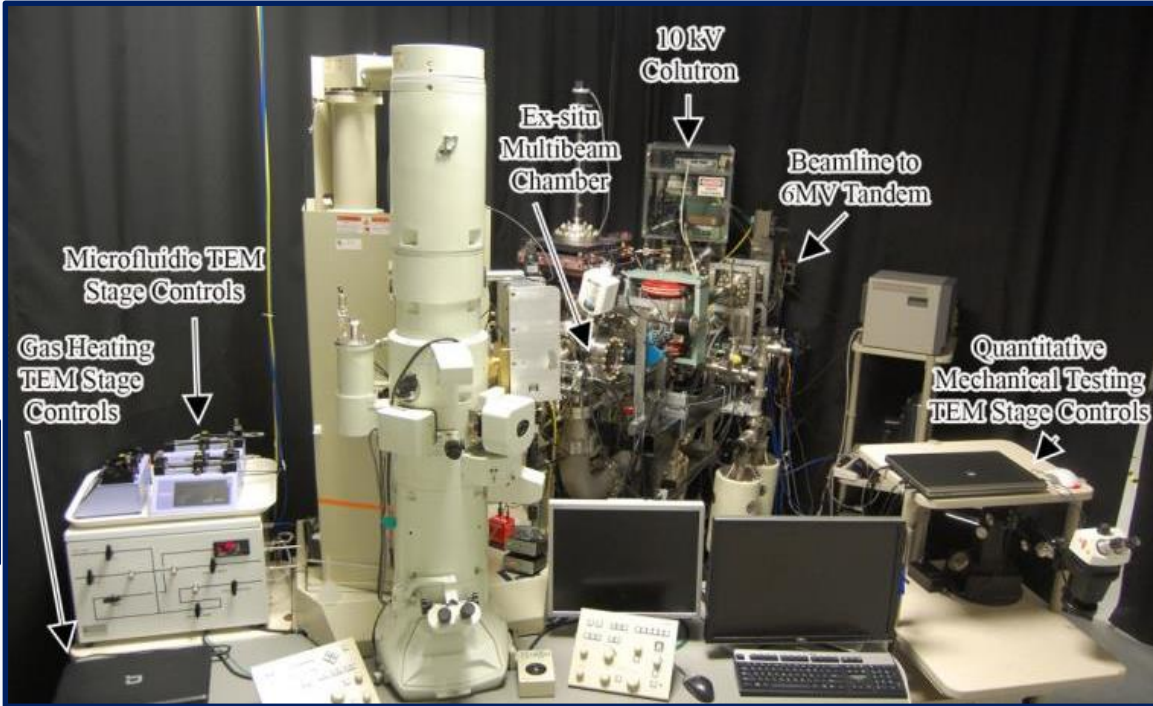
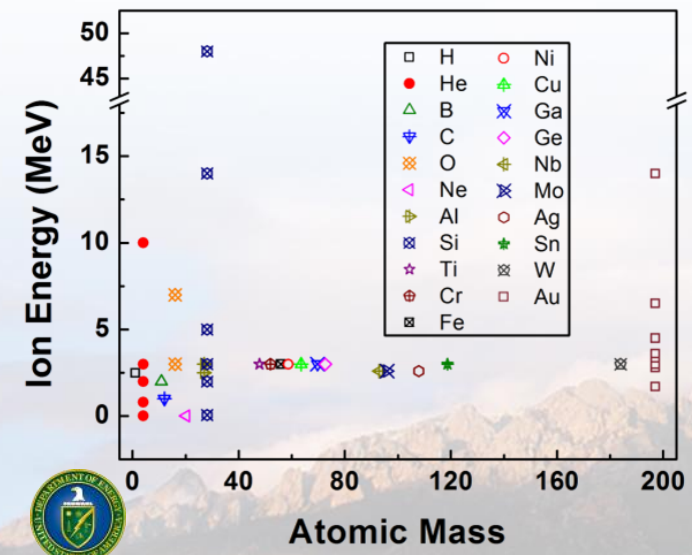


6 MV Tandem



10 kV Colutron

**Heavy Ion Irradiation + Gaseous Implantation**  
 Control ratio of dpa and gas species implantation and characterize coupling effects



200 kV JEOL 2100 TEM

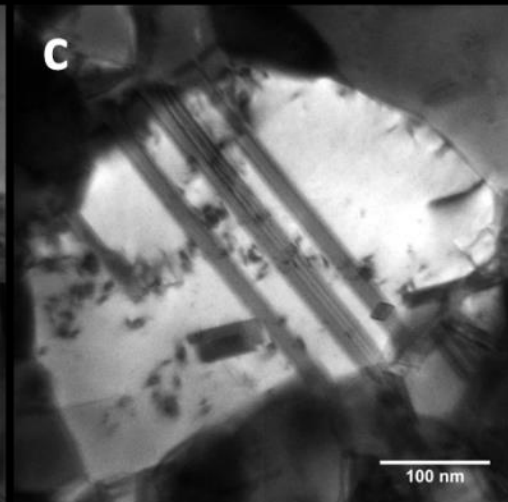
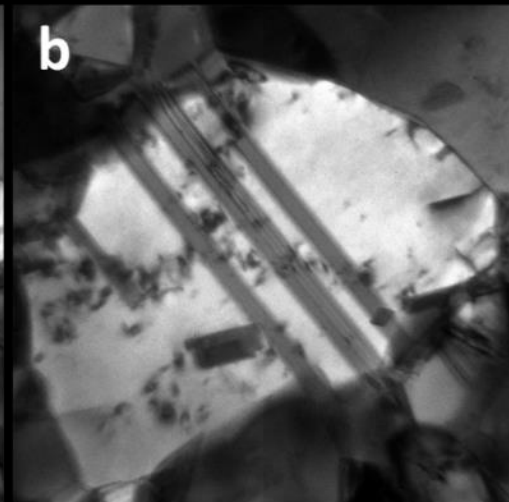
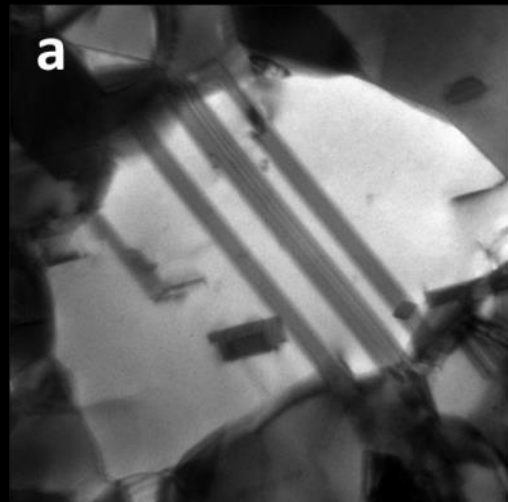
**Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution**



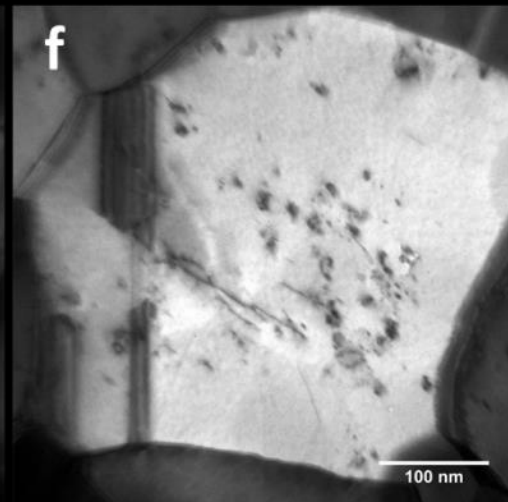
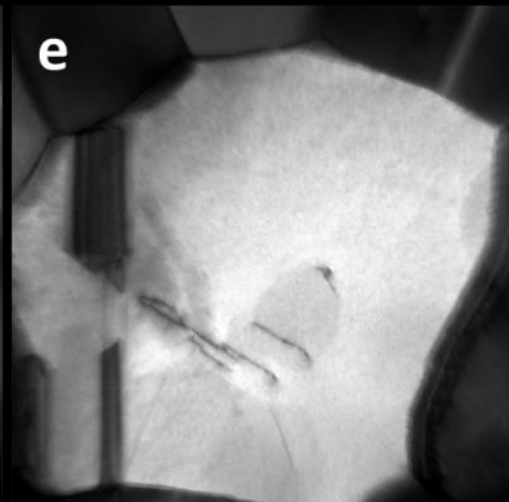
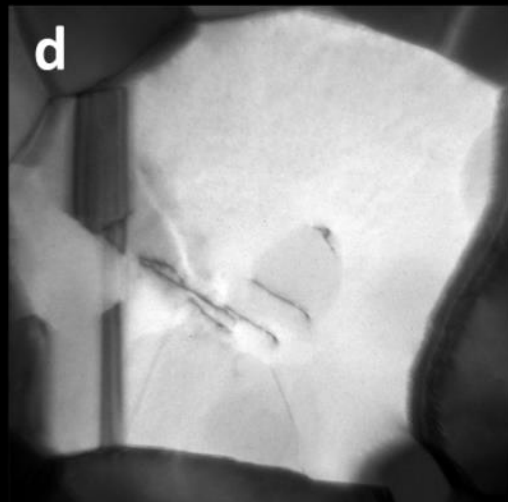
# *In situ* Successive Implantation & Irradiation

Collaborators: C. Chisholm & A. Minor

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



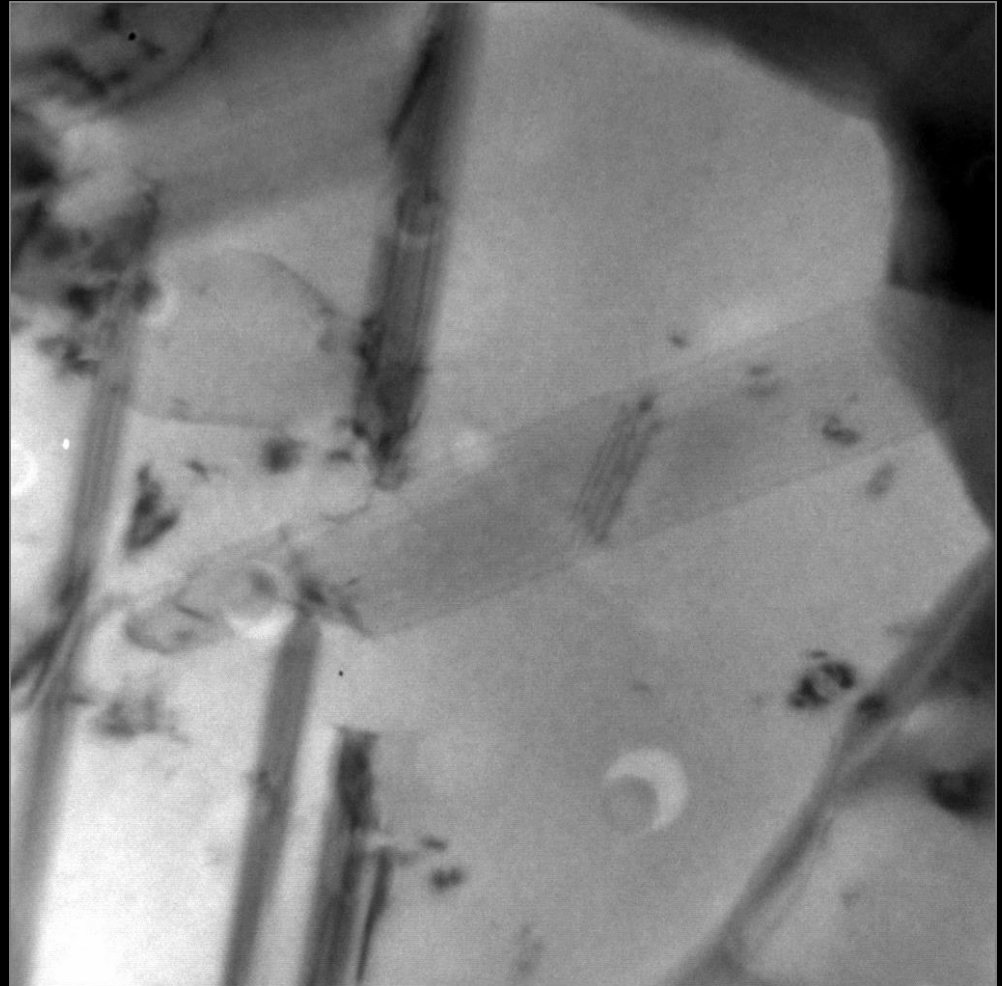
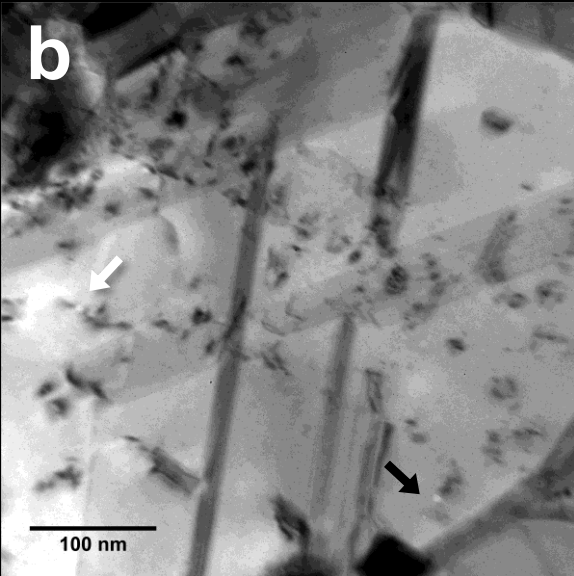
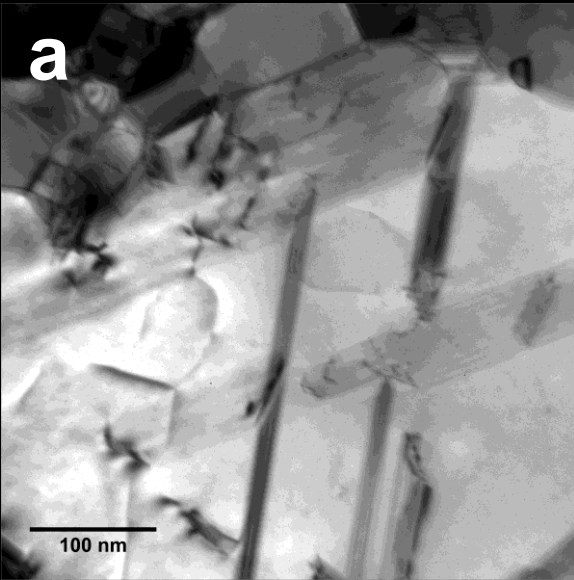
Successive  $\text{He}^{1+}$  then  $\text{Au}^{4+}$



# *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

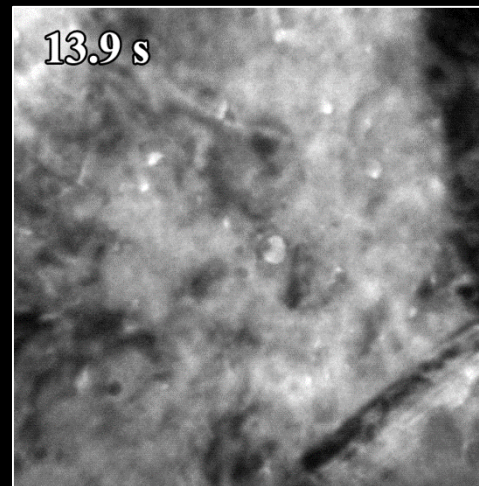
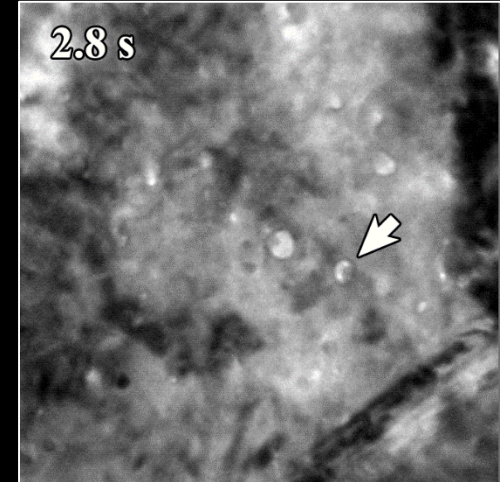
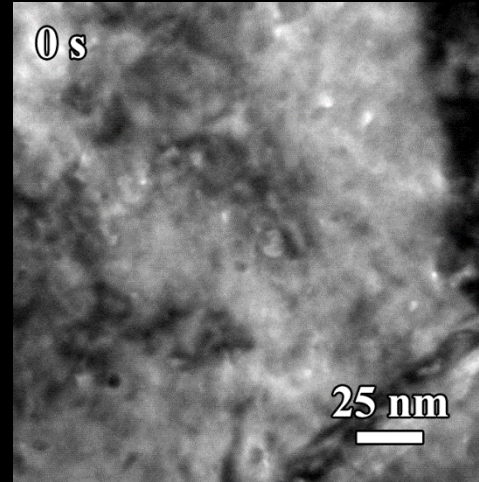
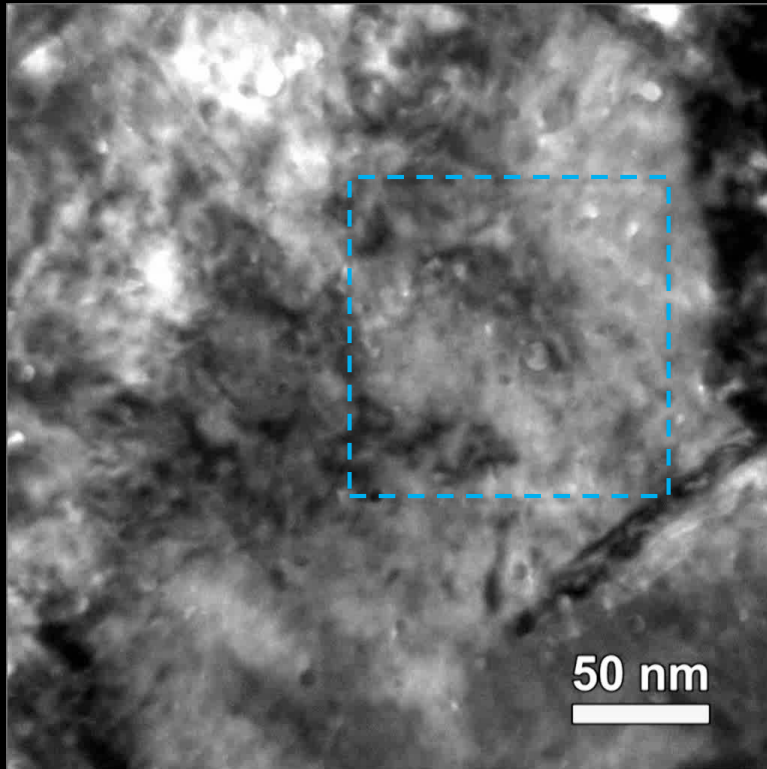


# Simultaneous *In situ* TEM Triple Beam:

## 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!

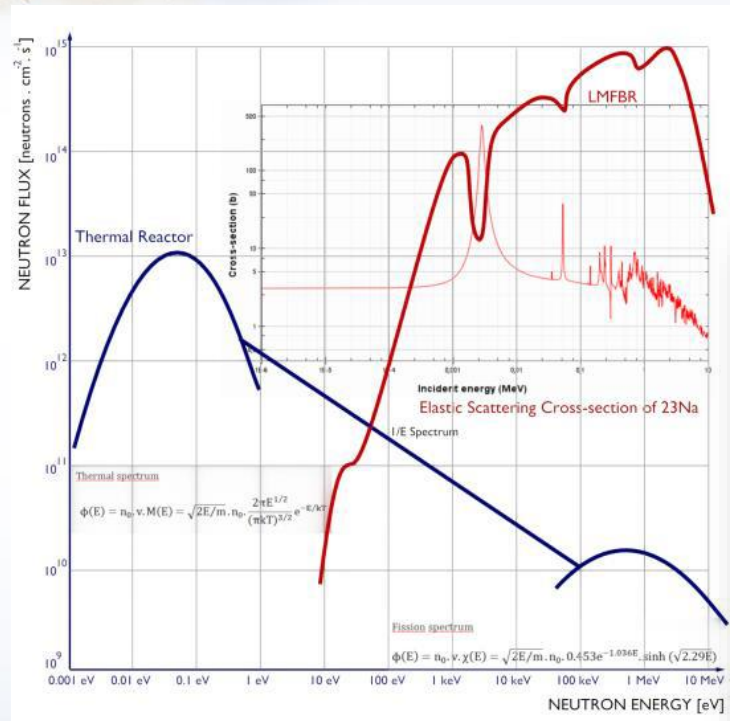
Intensive work is still needed to understand the defect structure evolution that has been observed.

### ■ Approximate fluence:

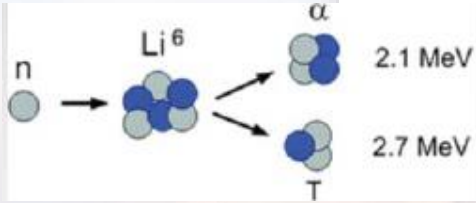
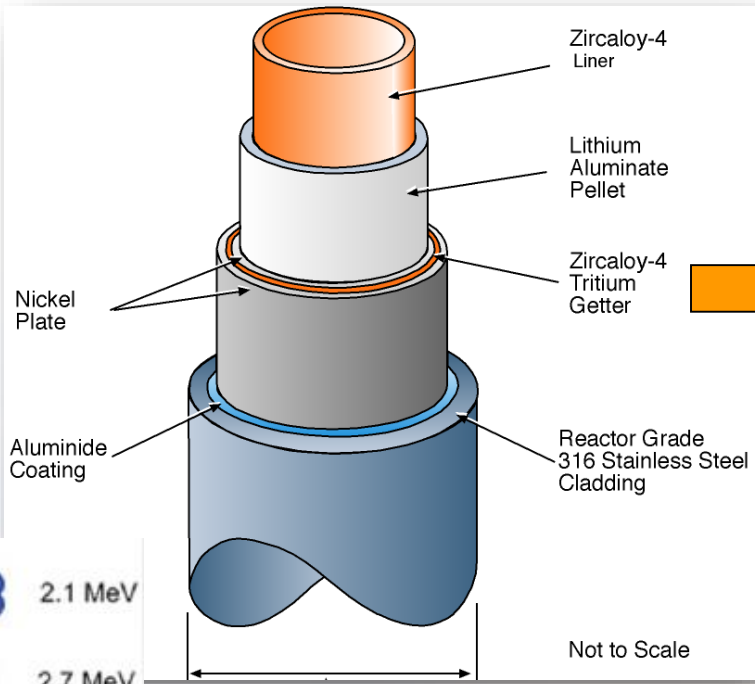
- Au  $1.2 \times 10^{13}$  ions/cm<sup>2</sup>
- He  $1.3 \times 10^{15}$  ions/cm<sup>2</sup>
- D  $2.2 \times 10^{15}$  ions/cm<sup>2</sup>

### ■ Cavity nucleation and disappearance

# Applying the Triple Beam Irradiation to Deconvolute Reactor Environments



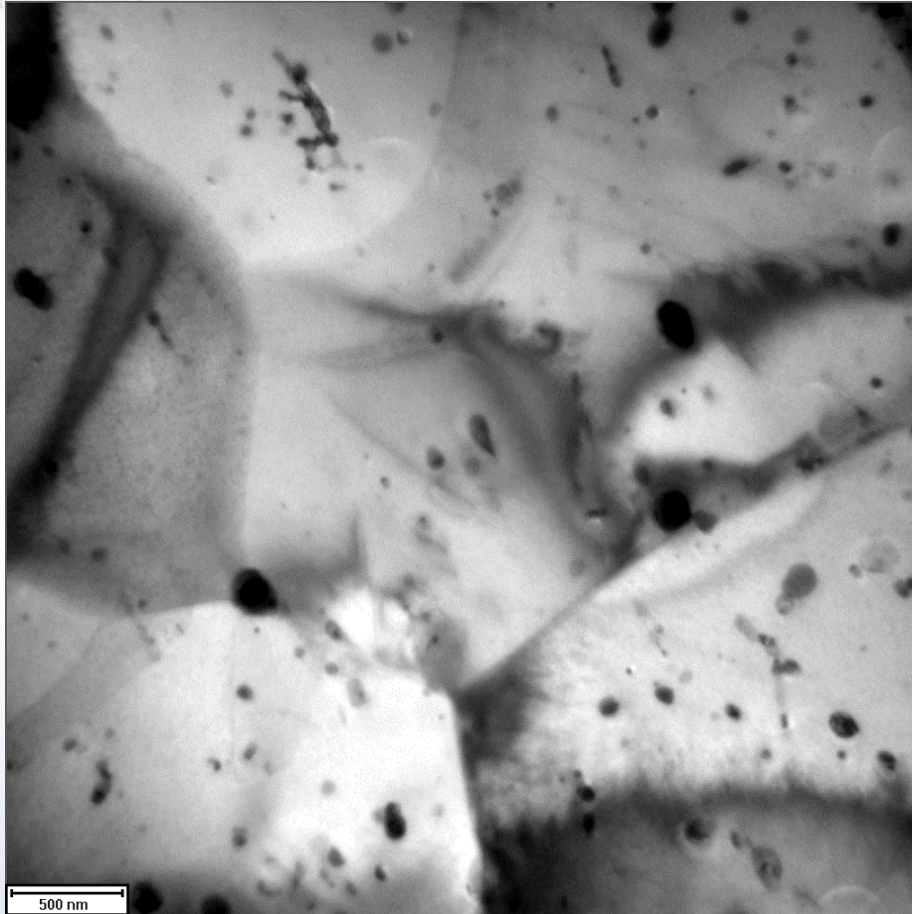
## Tritium Producing Burnable Absorber Rod (TPBAR)



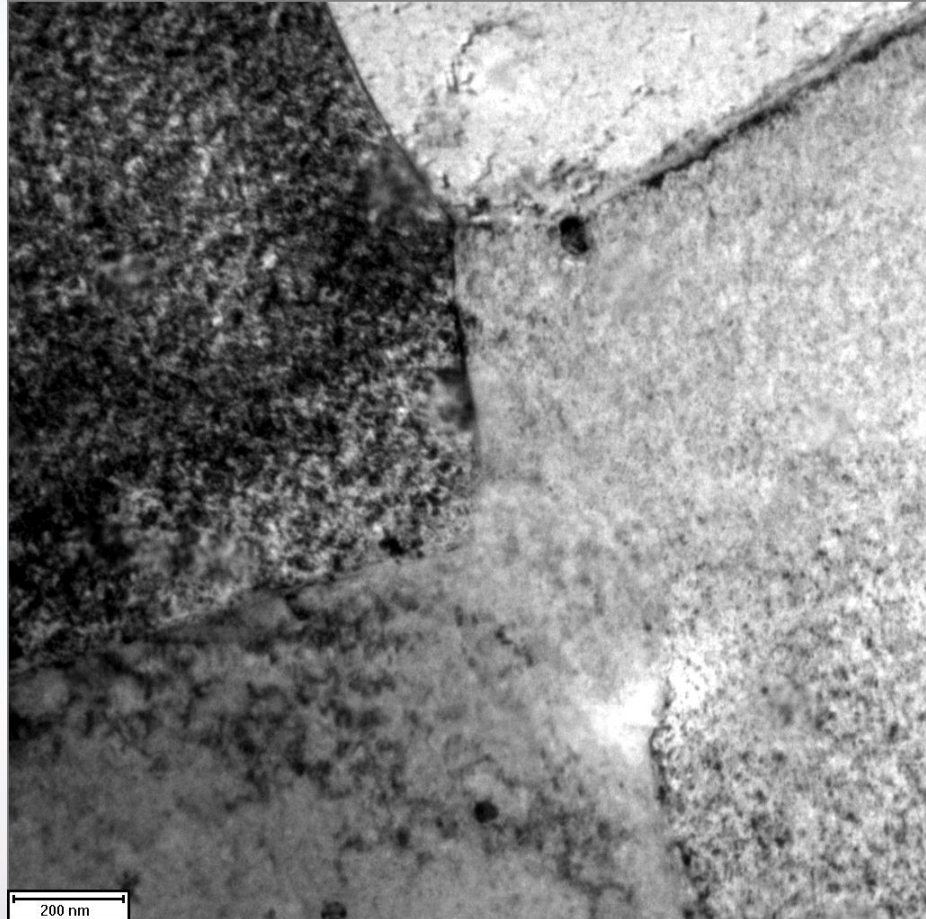
- Displacement Damage
- Helium Implantation
- Tritium Implantation
- Elevated Temperatures

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



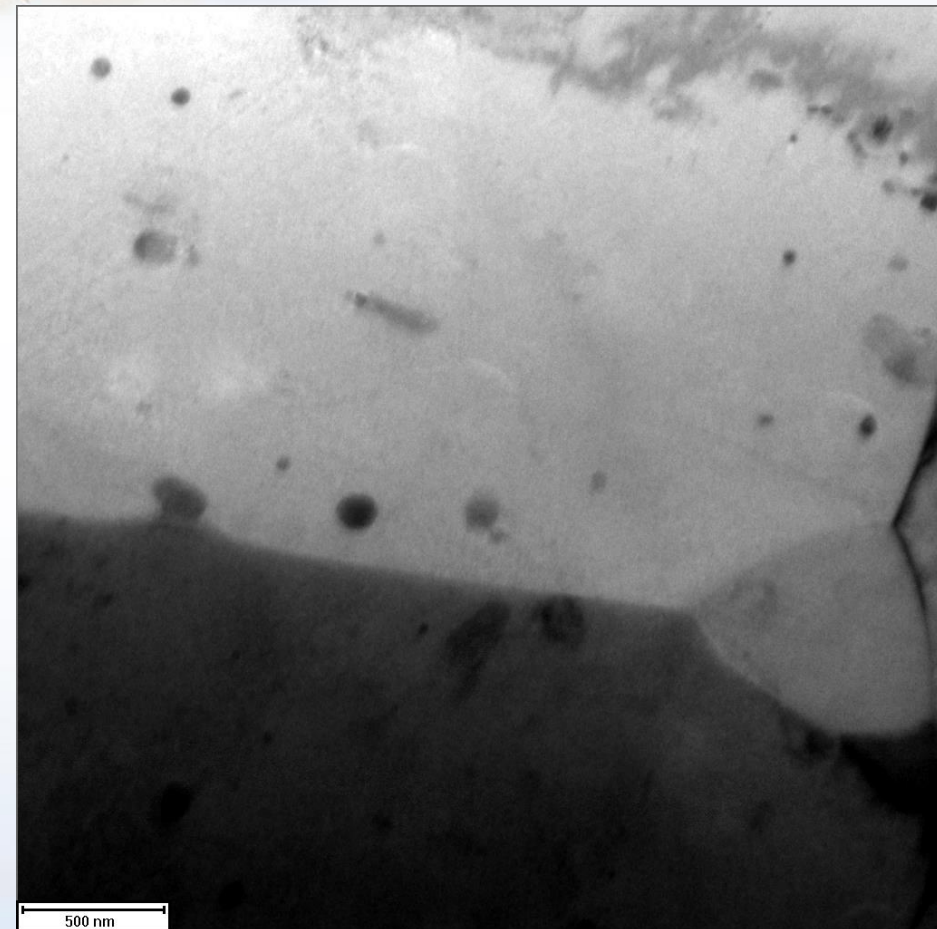
Before Irradiation



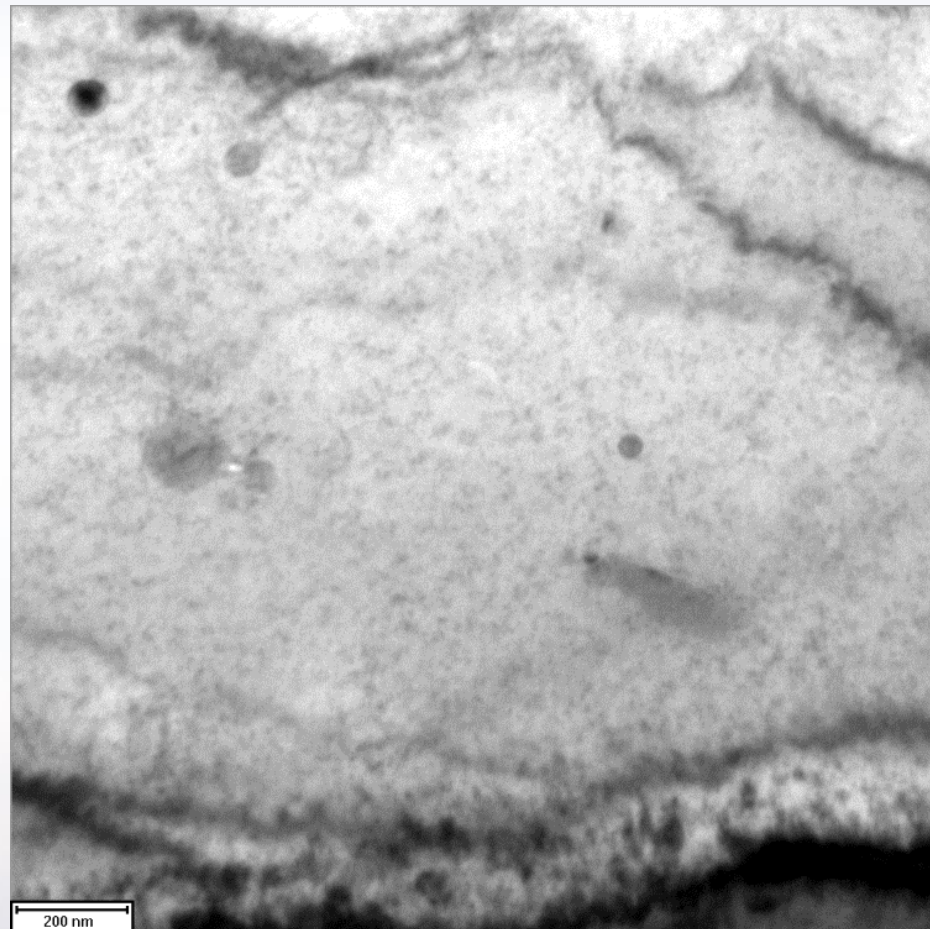
After Irradiation  
 $\approx 7$  DPA



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



Before Implantation

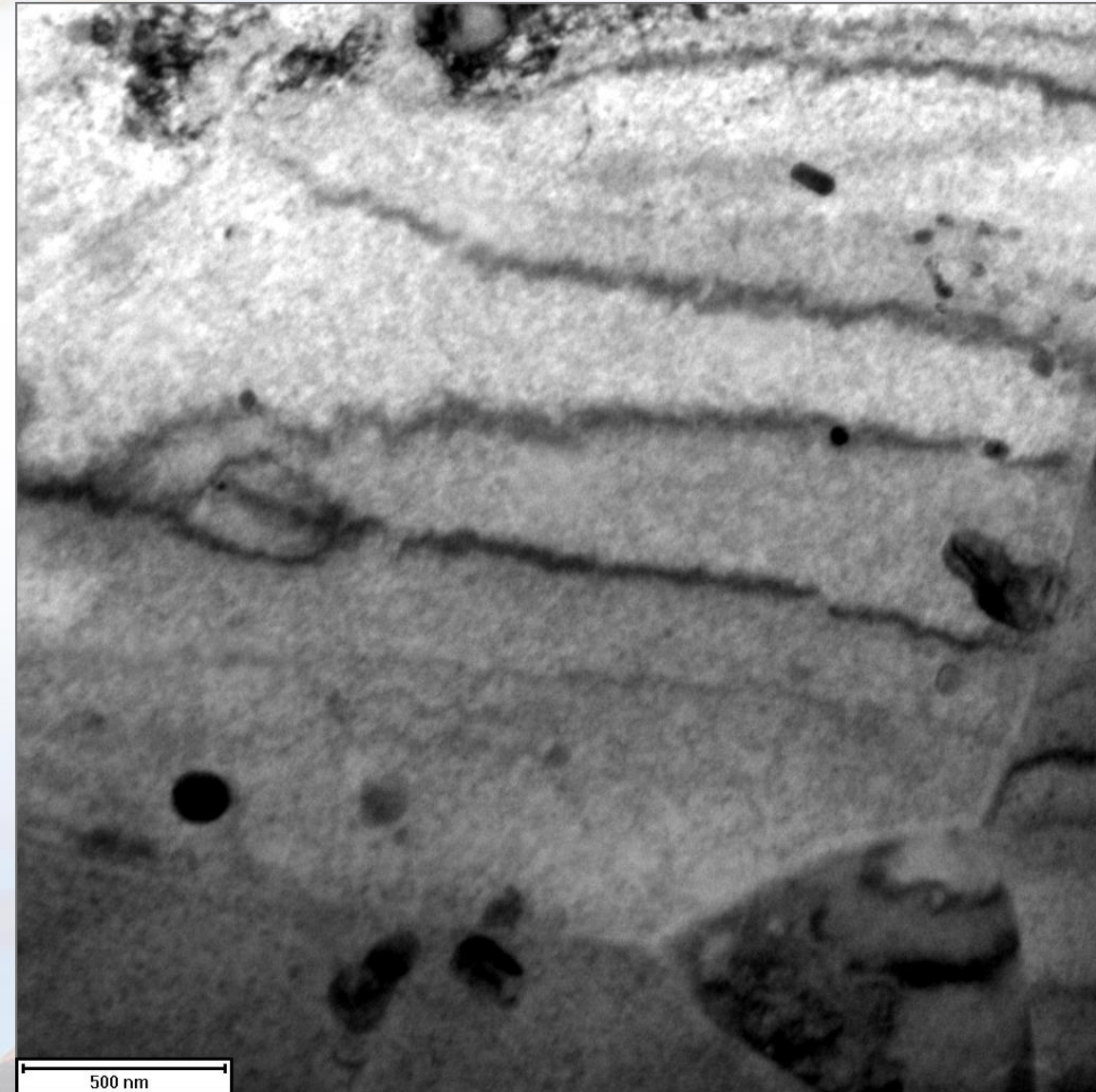


After Implantation  
Damage, No Cavities



# 3 MeV Self Ion Irradiation

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

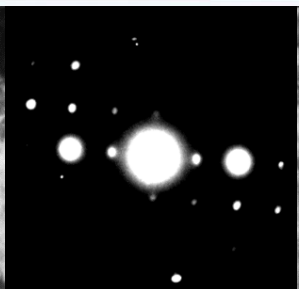


High Density of Defects but  
No Cavities

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

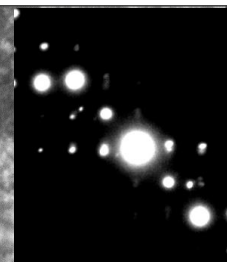
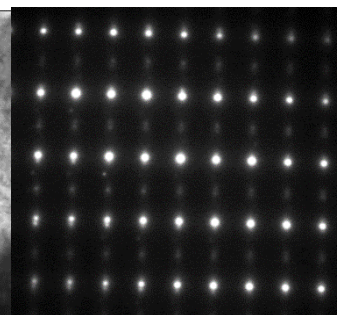
## Then 3 MeV Self Ion Irradiation into Zr-4 at 310 C

Two Beam  
g = 0002



On Axis

Two Beam  
g = 01-12

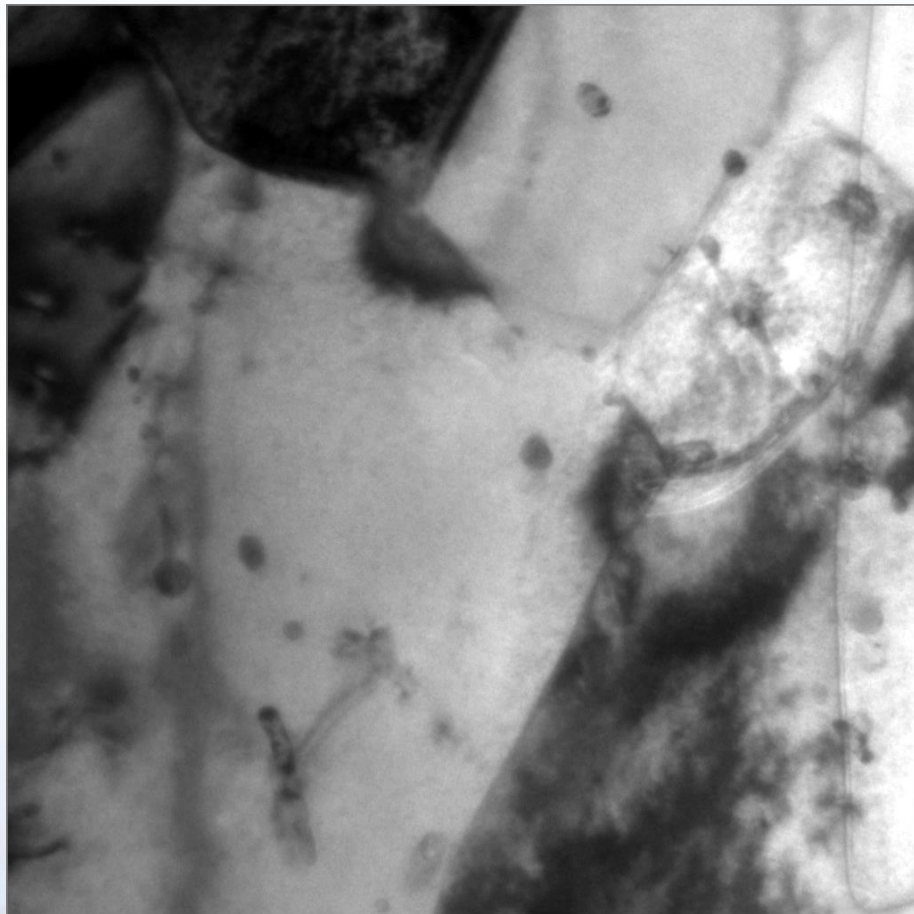


Sequence matters, as  
well as imaging  
condition!

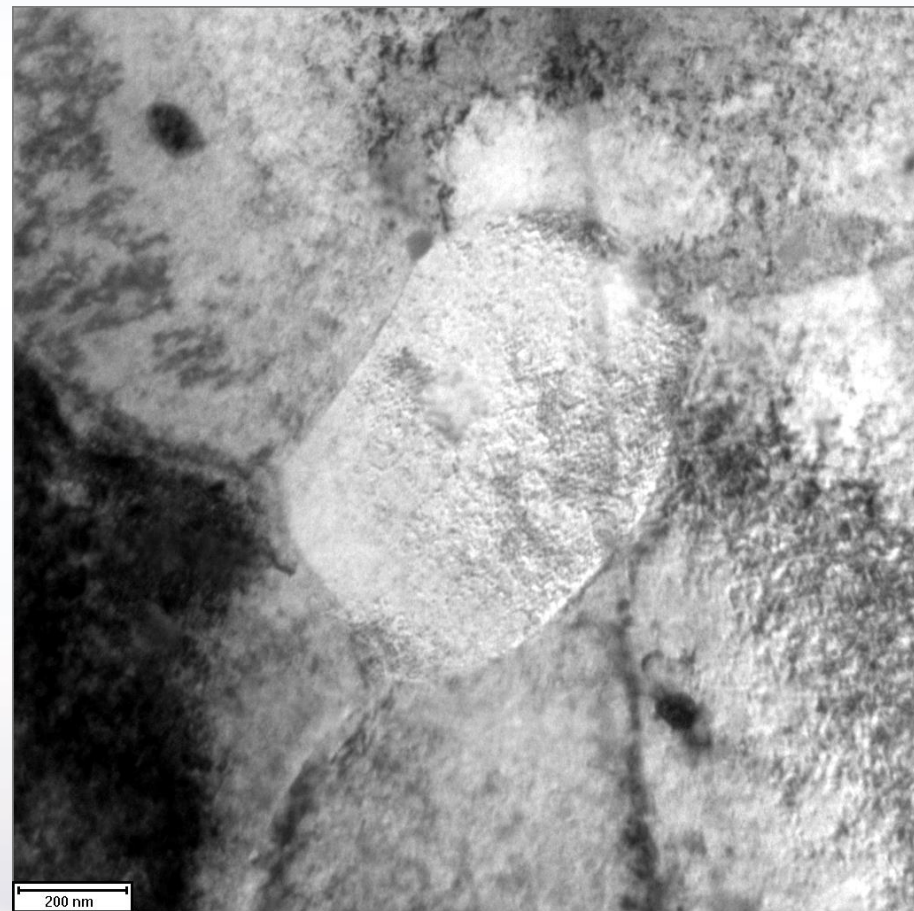


200 nm

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



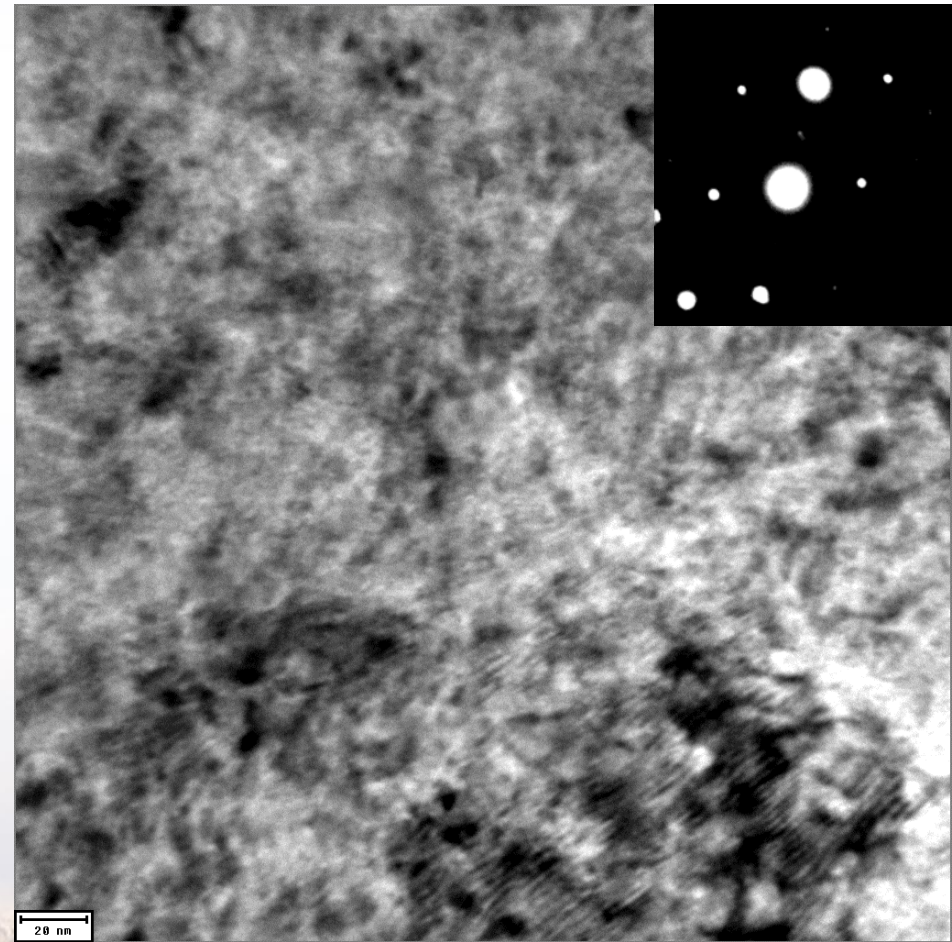
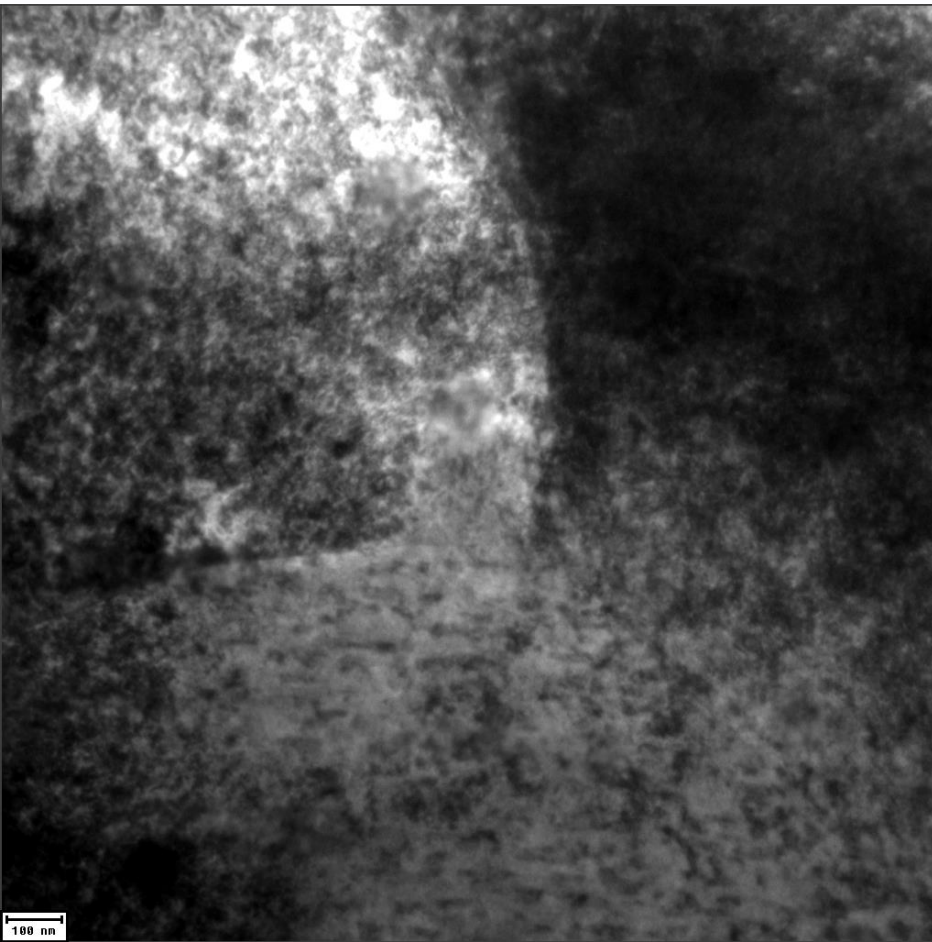
Before Implantation/Irradiation



After Implantation/Irradiation  
Damage, No Cavities



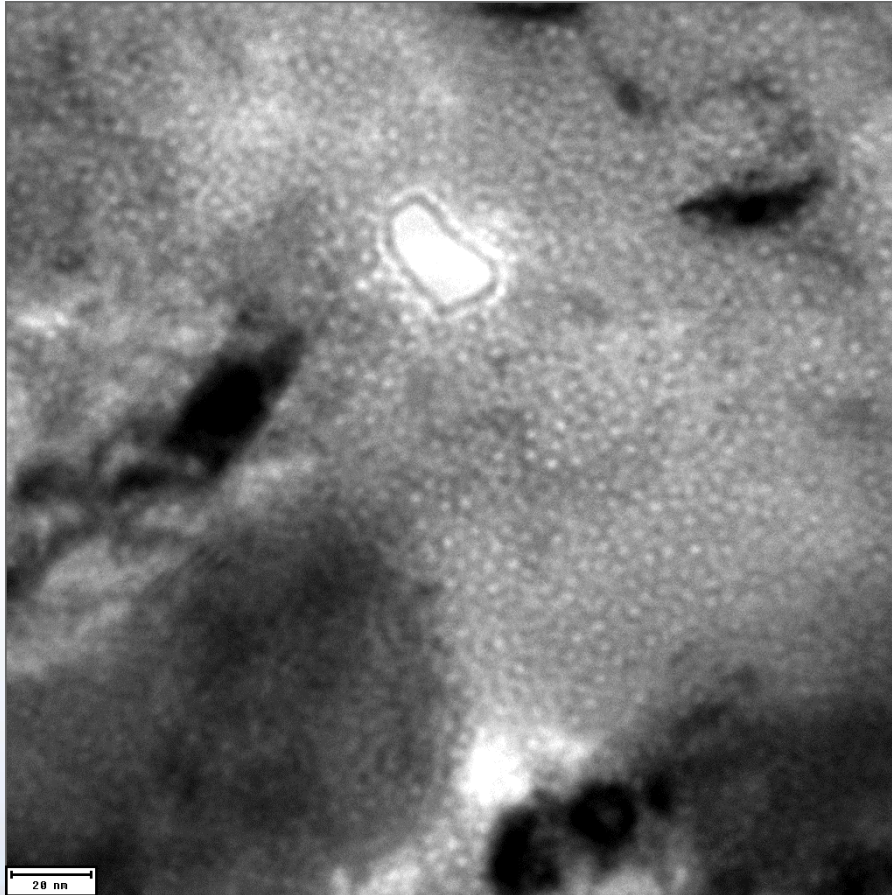
# Concurrent D & He Implantation & Zr Irradiation into Zr-4 at 310 C



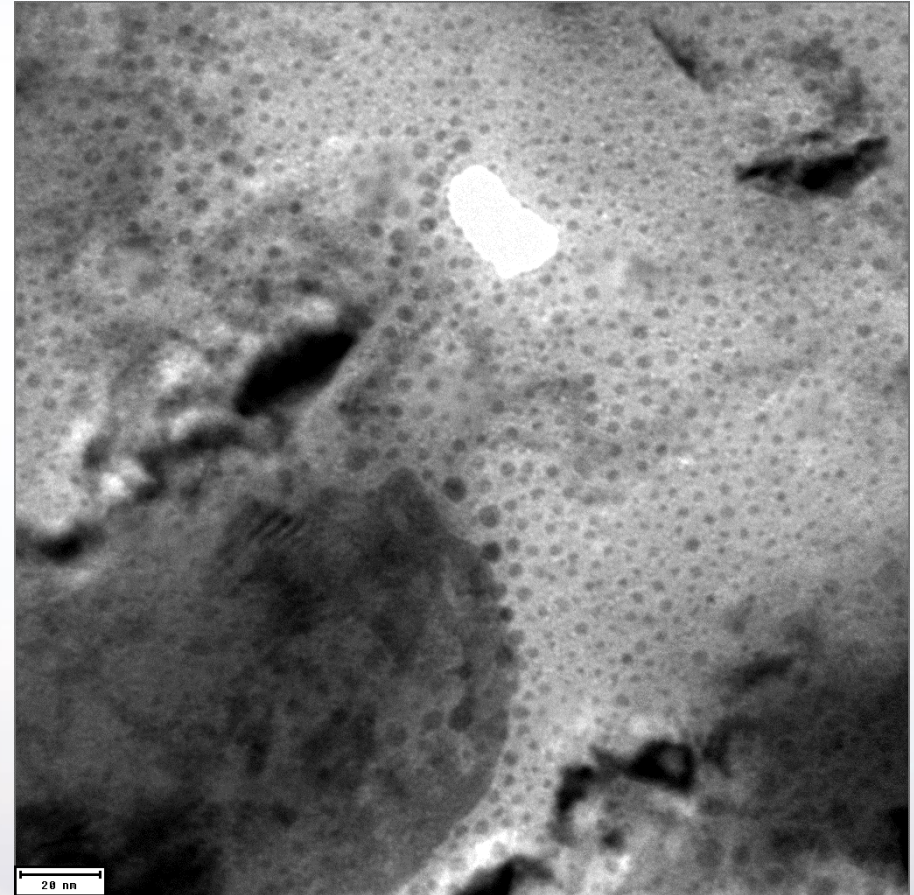
After Implantation/Irradiation  
Significant Damage, but No Cavities

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

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



Under Focus

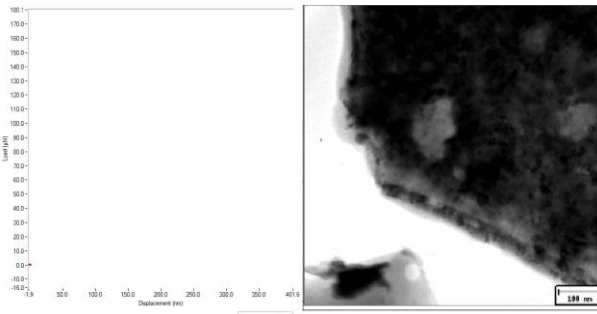


Over Focus



# Future Potential Synergistic *In situ* Triple Beam Experiments

## Mechanical Effects

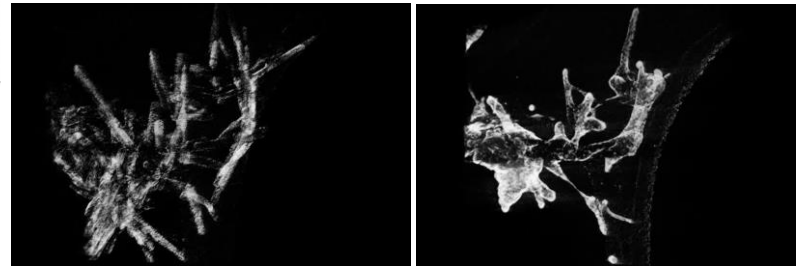


### Hysitron PI95 TEM Picoindenter Gatan 654 Straining Holder

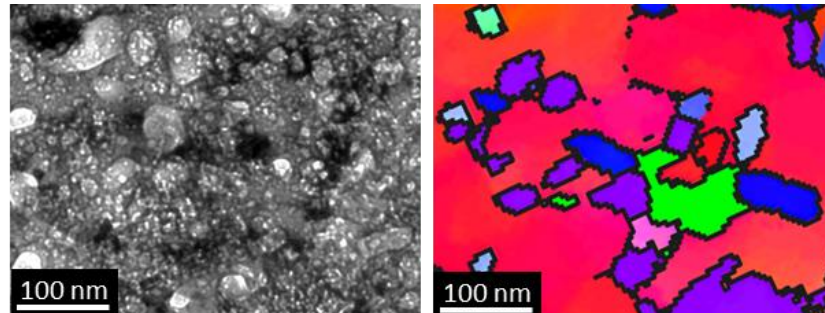
Allows for direct correlation of dose and defect density with resulting changes in strength, ductility, and defect mobility

## Structural Effects

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

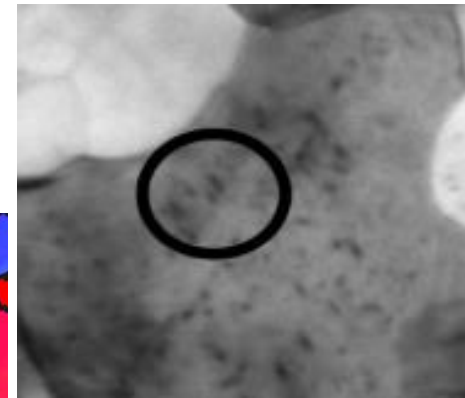


**Nanomegas ASTAR**  
Grain structure changes as a result of radiation and implantation



## 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)

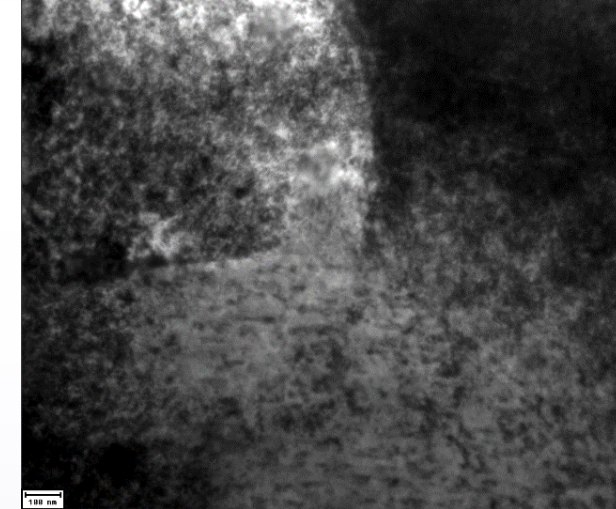
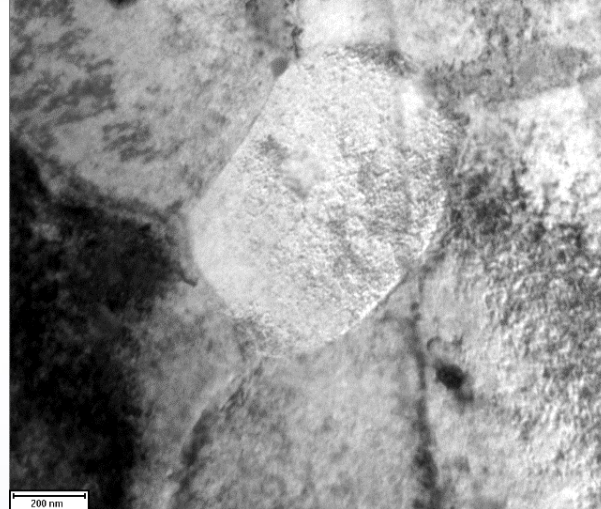
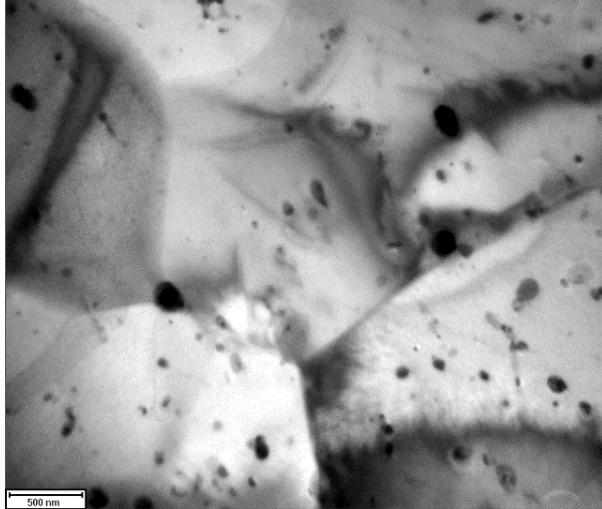
Helium Implantation – Deuterium Implantation –  
Self Ion Irradiation – Elevated Temperatures

# Summary

Single Ion Damage →

Damage + He →

Damage + He +  $^3\text{H}$



All at reactor operating temperature

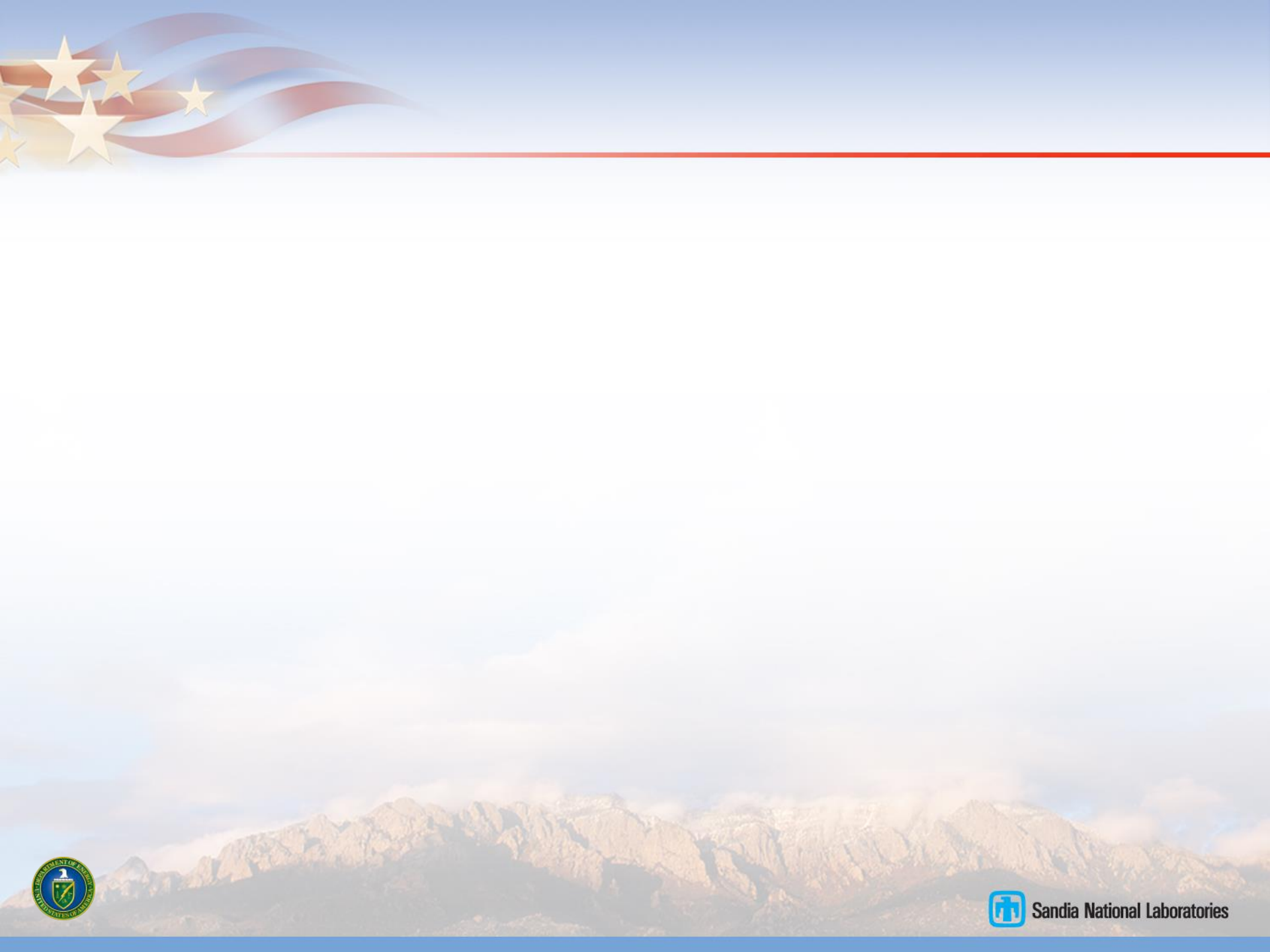
This work demonstrates that the I<sup>3</sup>TEM is capable of simulating the synergistic effects of damage, gas accumulation, and high temperature occurring in reactor-like conditions, *in situ*



Sandia National Laboratories is a multi-mission laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

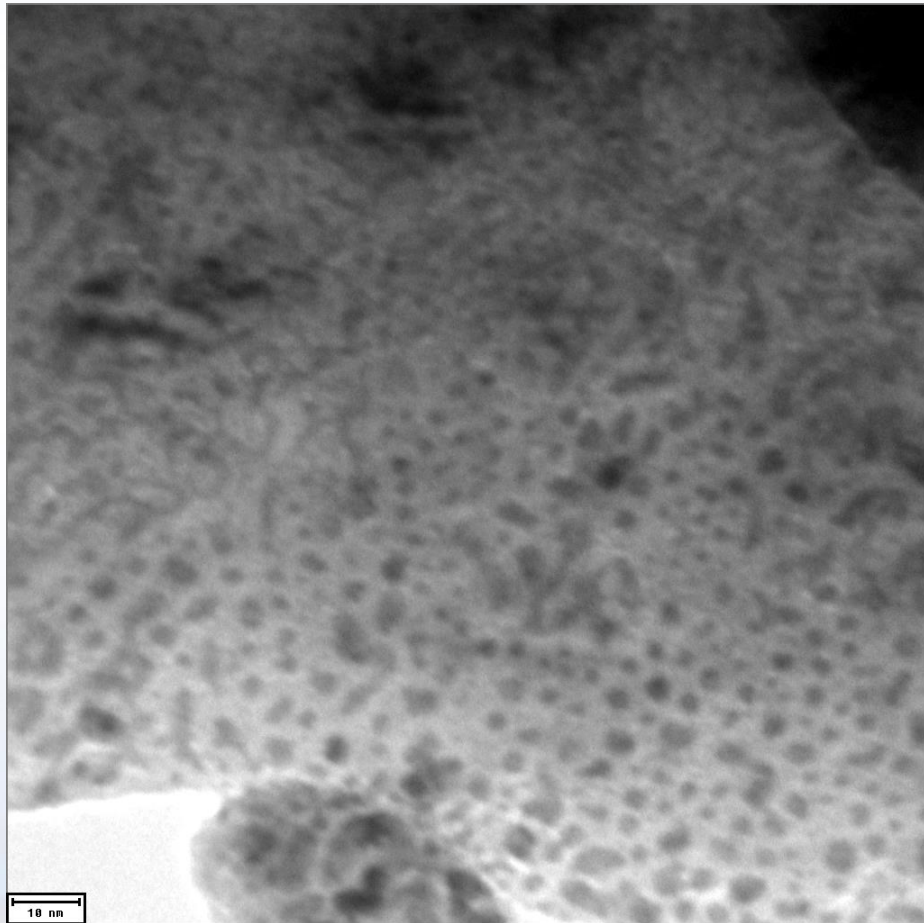


Sandia National Laboratories

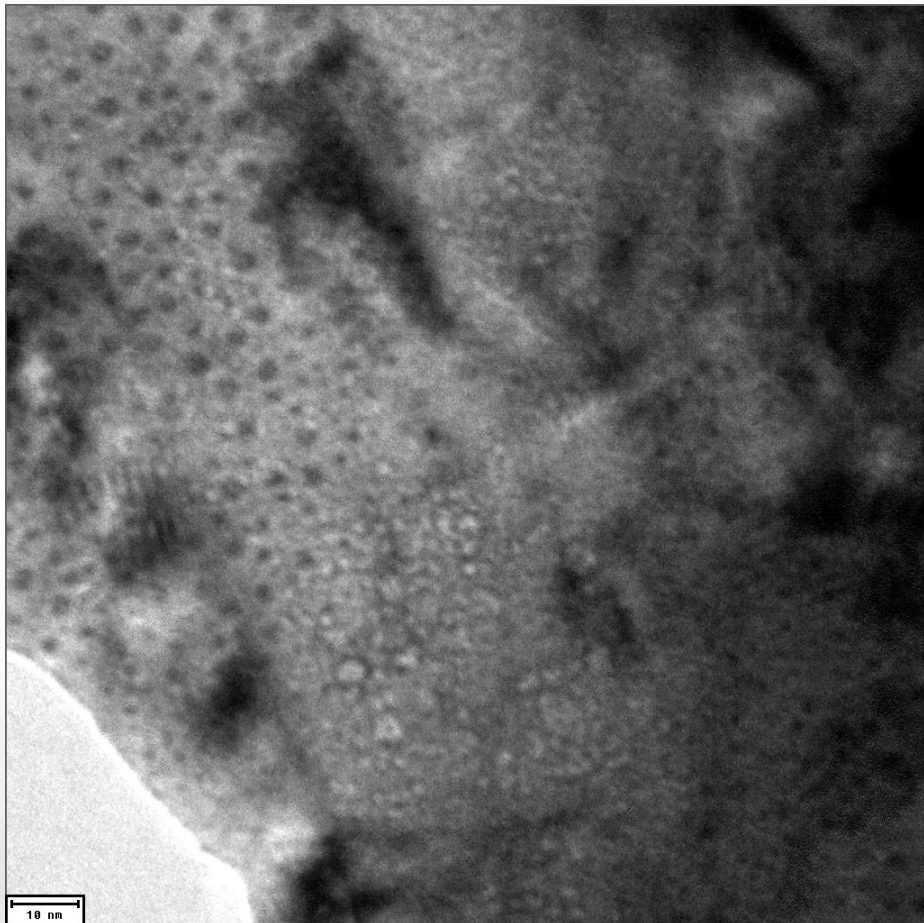




# Surface Effects?



Under Focus



Under Focus



# Future Planned TPBAR Work: $\text{LiAlO}_2$ Pellet

Drop-Cast Powders of As-Synthesized  $\text{LiAlO}_2$

Powders contain good regions for *in-situ*

