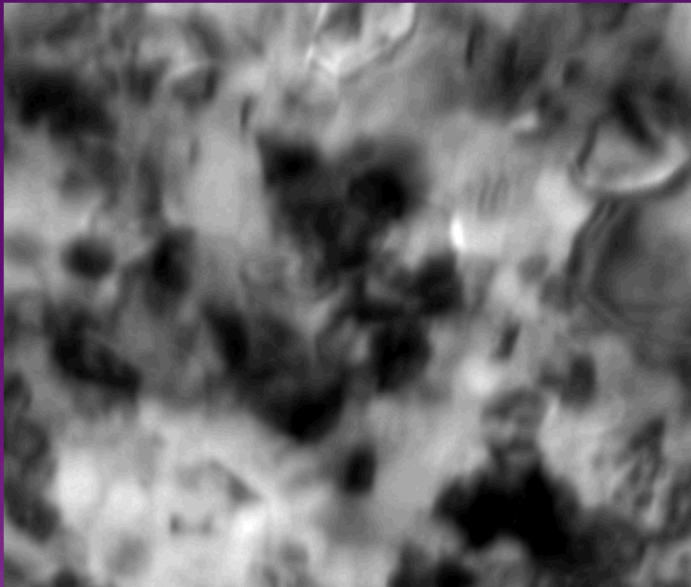


# In-Situ He<sup>+</sup> Implantation and Thermal Aging of Nanocrystalline Iron

SAND2015-6215C

Brittany Muntifering, Sarah Blair, Youwu Fang, Aaron Dunn,  
Remi Dingreville, Jianmin Qu, Khalid Hattar



NORTHWESTERN  
UNIVERSITY



# Sandia's I<sup>3</sup>TEM Facility



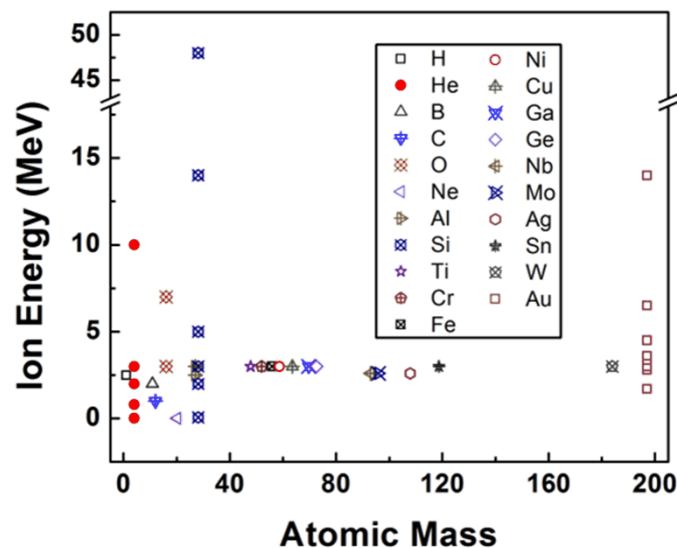
6 MV Tandem



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

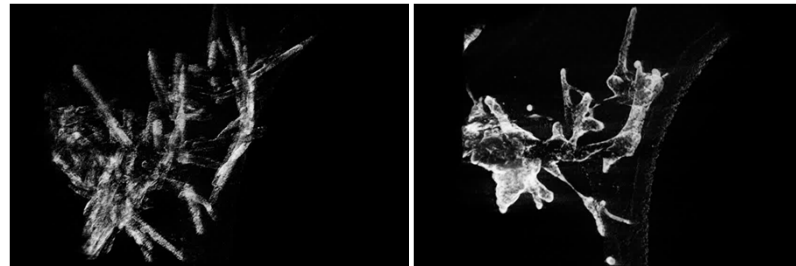
# Radiation & Synergistic In- Situ Capabilities

## Structural Effects

### Hummingbird Tomography Stage

#### Gatan 925 Double Tilt Rotate

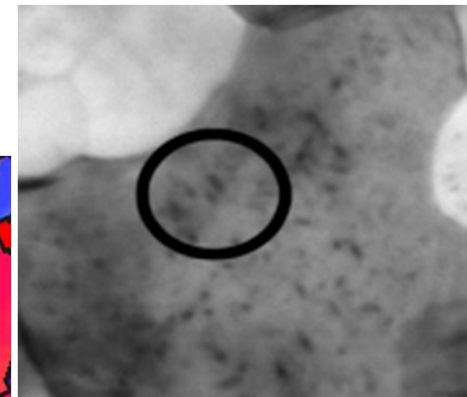
Morphology changes as a result of radiation damage



## Thermal Effects

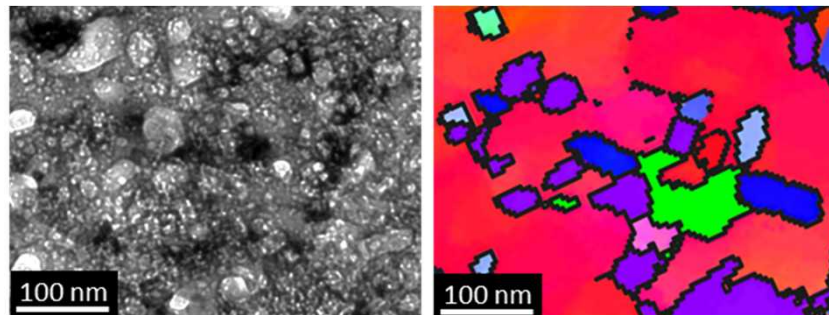
### Hummingbird Heating Stage

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

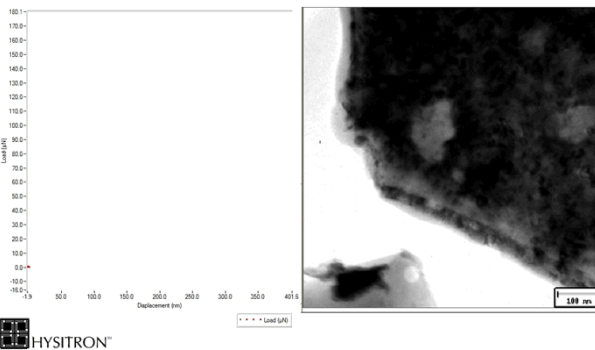


### Nanomegas ASTAR

Grain structure changes as a result of radiation and implantation



## Mechanical Effects



### Hysitron PI95 TEM Picoindenter

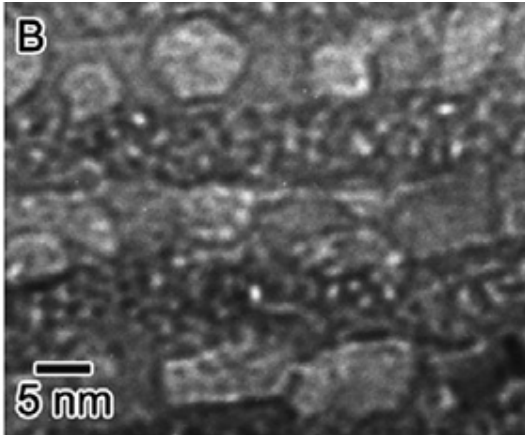
#### Gatan 654 Straining Holder

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

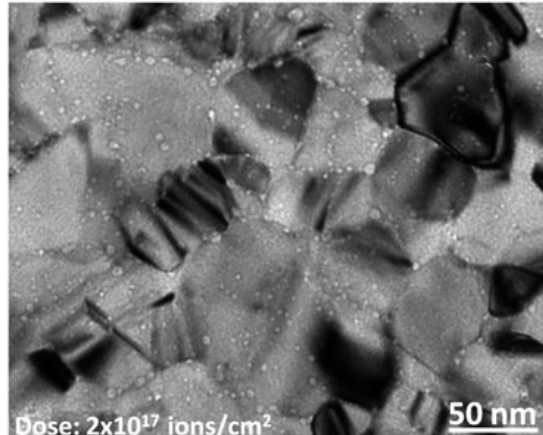
The application of advanced microscopy techniques to characterize synergistic effects in a variety of extreme environments

# Helium Interaction with Boundaries and Interfaces

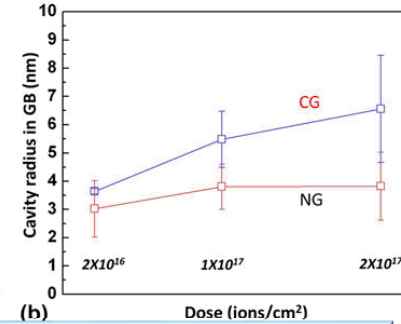
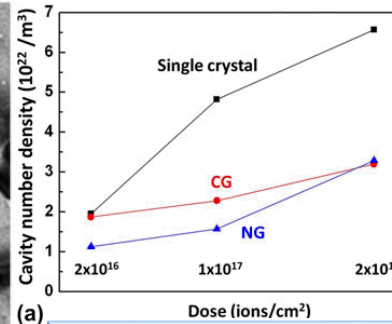
Helium effects embrittlement, void swelling, creep rupture, etc.



K. Hattar, et al. Arrest of He bubble growth in Cu-Nb multilayer nanocomposites. Scripta Materialia (2008)



Weizhong Hana, et al. Irradiation damage of single crystal, coarse-grained, and nanograin copper under helium bombardment at 450 °C. J. Mater. Res. 2013

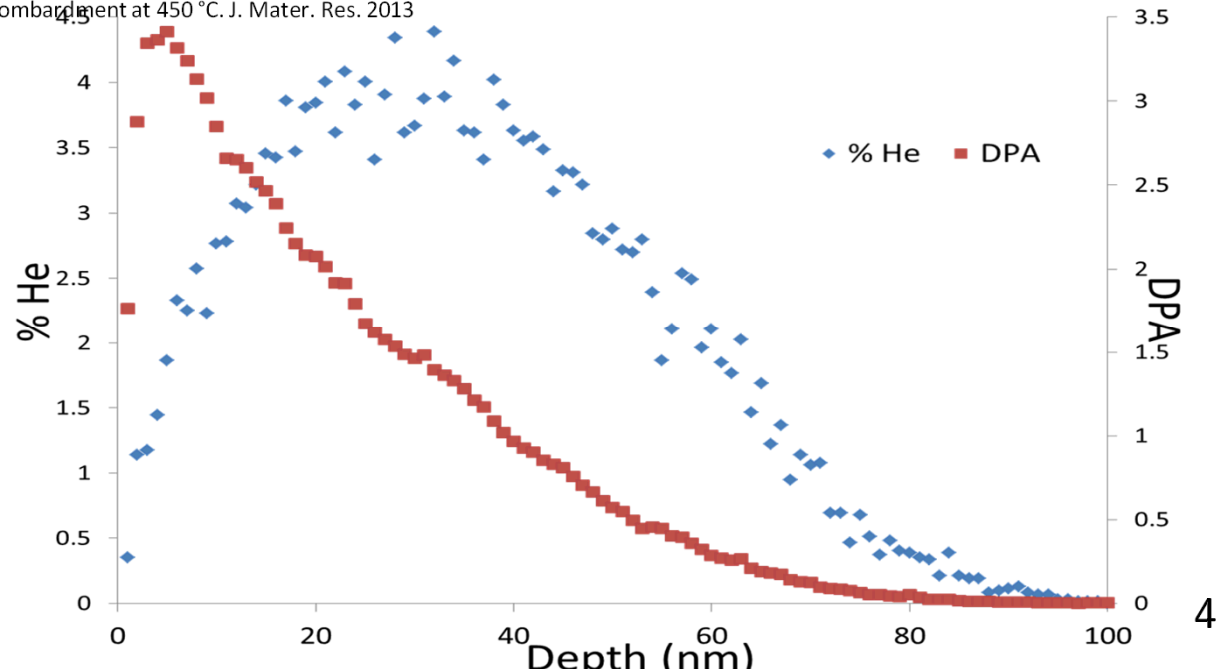


- Defects attracted to interfaces where they can annihilate
- Interfaces may limit cavity growth

10 keV He<sup>+</sup> into  
Nanocrystalline Iron

$10^{13}$  He<sup>+</sup>/cm<sup>2</sup>s

$2.6 \times 10^{16}$  He<sup>+</sup>/cm<sup>2</sup>  $\approx$  2% He

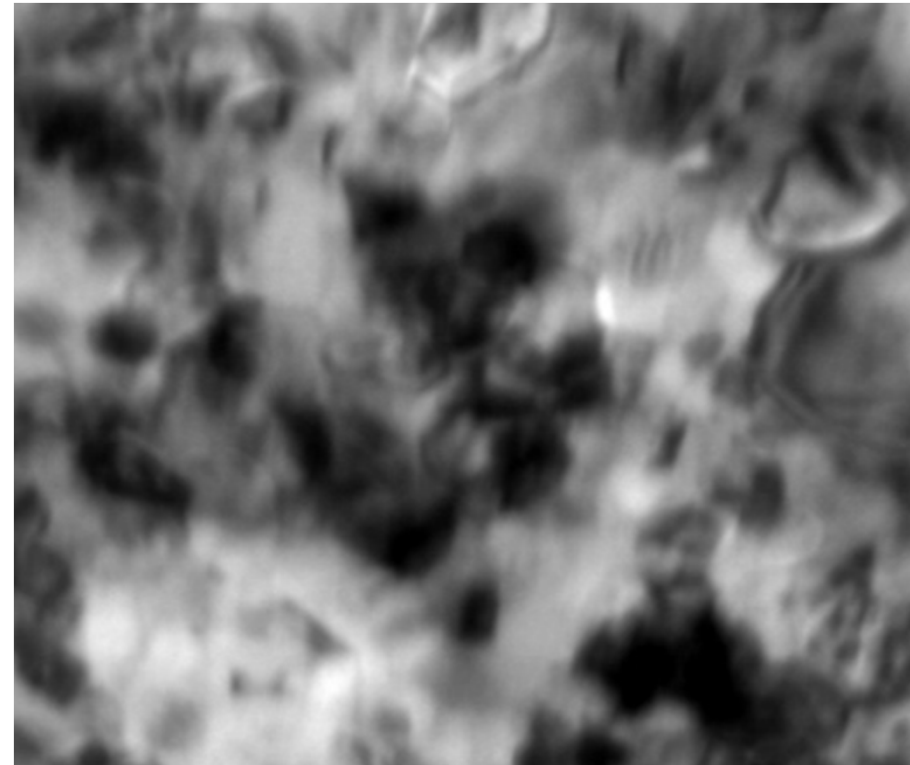
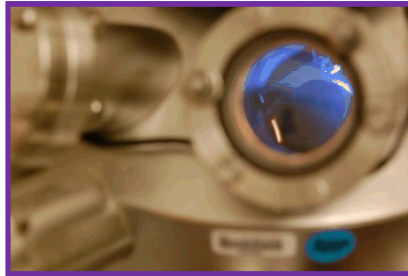




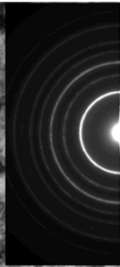
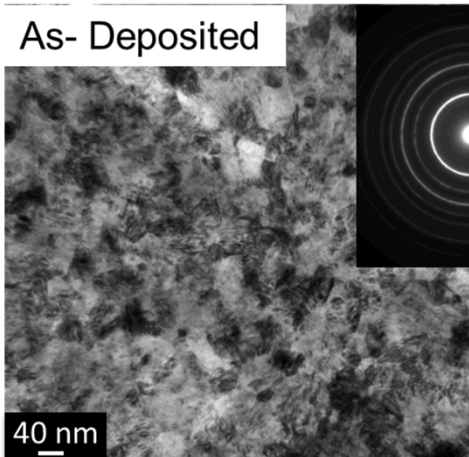
# Deposition & Grain Size Tailoring of Iron Films



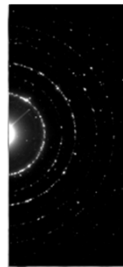
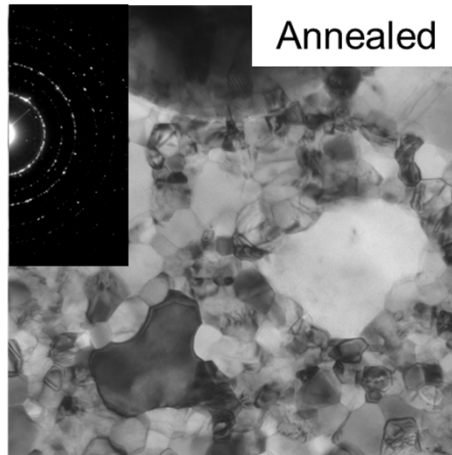
- KrF Excimer laser
- 35 Hz
- $4.5 \times 10^{-5}$  Pa



As- Deposited



Annealed

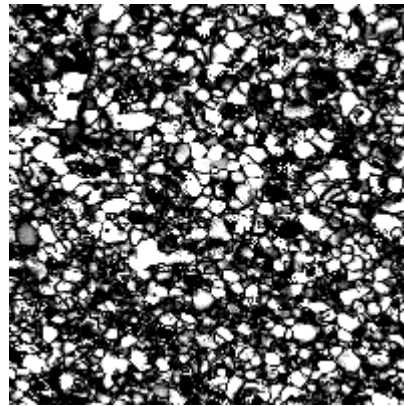
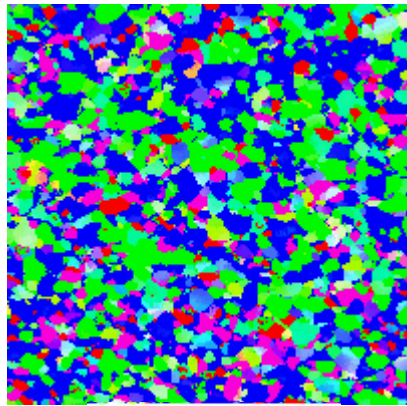


Control grain size within the nanometer regime

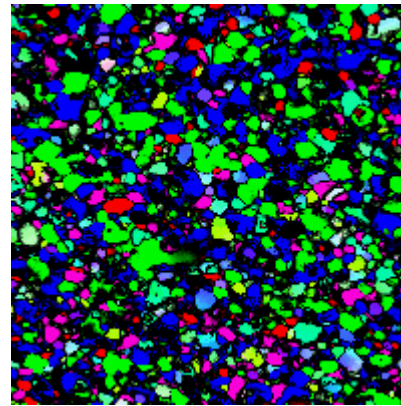
# Grain Size and Texture Modification of Iron Films

Annealing results in a stronger texture and loss of (001) oriented grains

## As Deposited

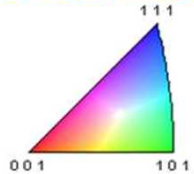
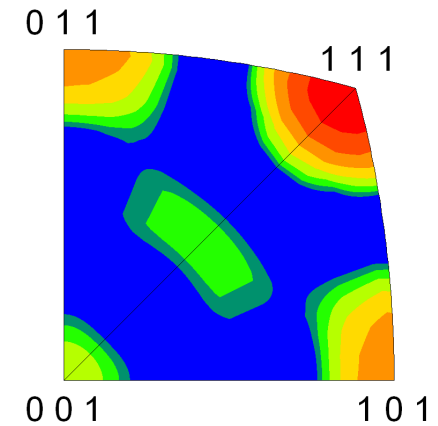


Reliability = 10-30

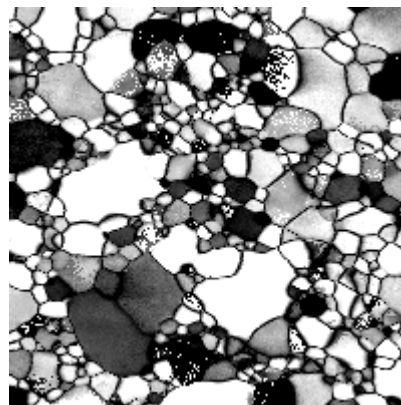
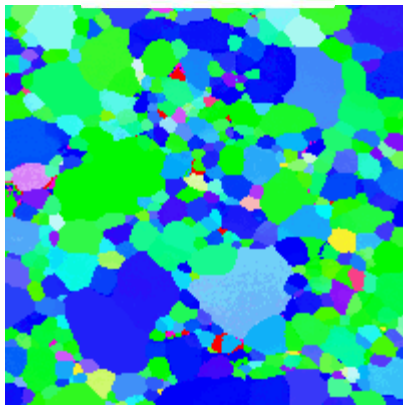


Combination

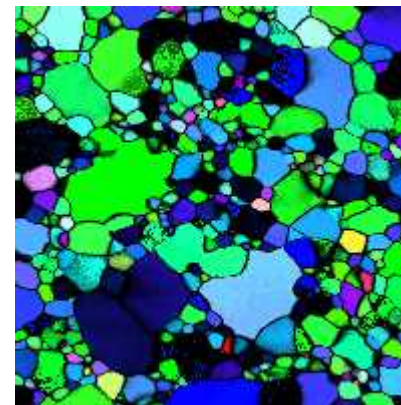
[001]



## Annealed

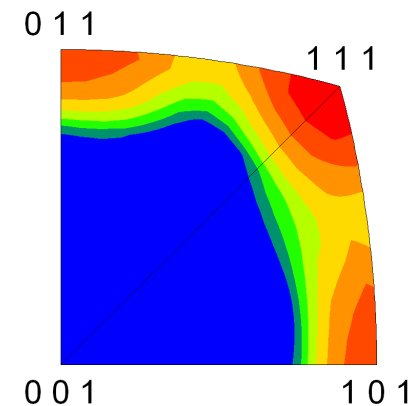


Reliability = 10-50



Combination

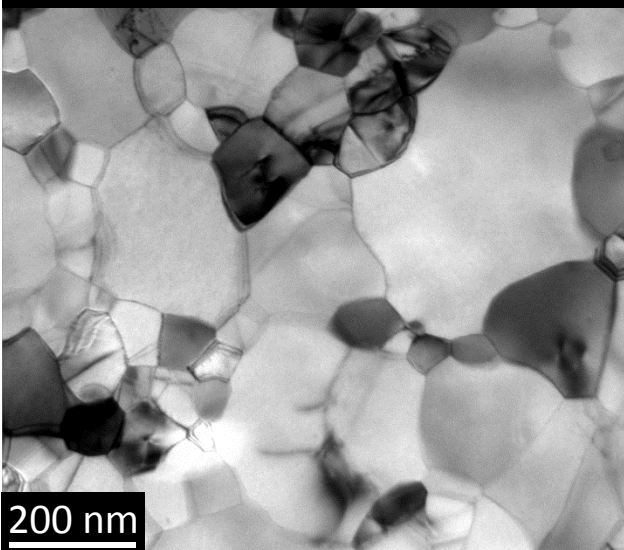
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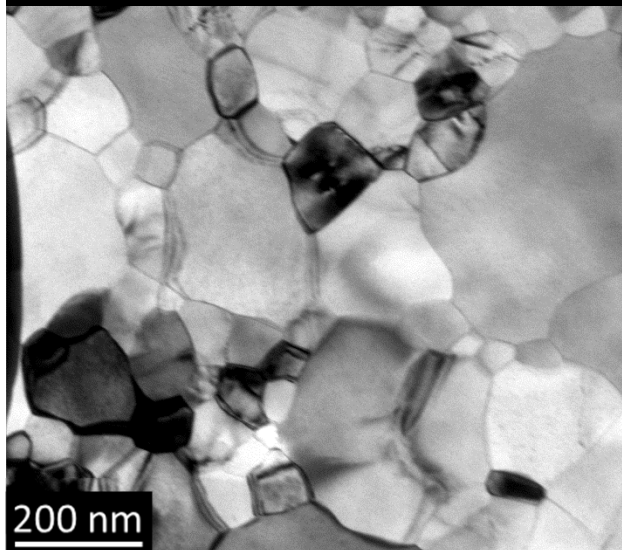


# Helium Implantation at Room Temperature

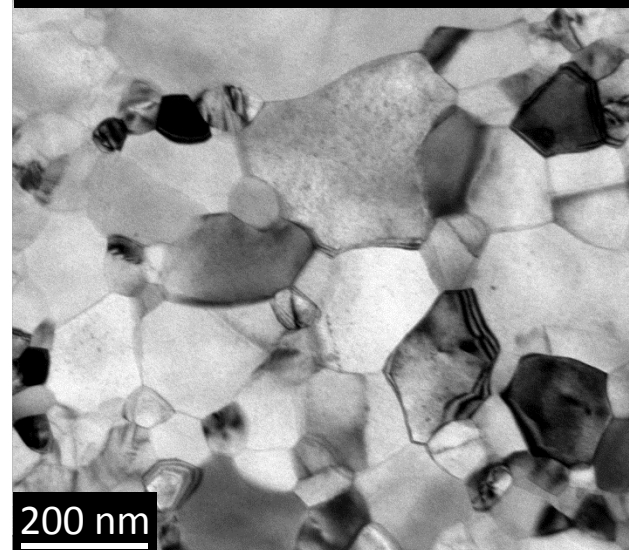
Pre-Implantation



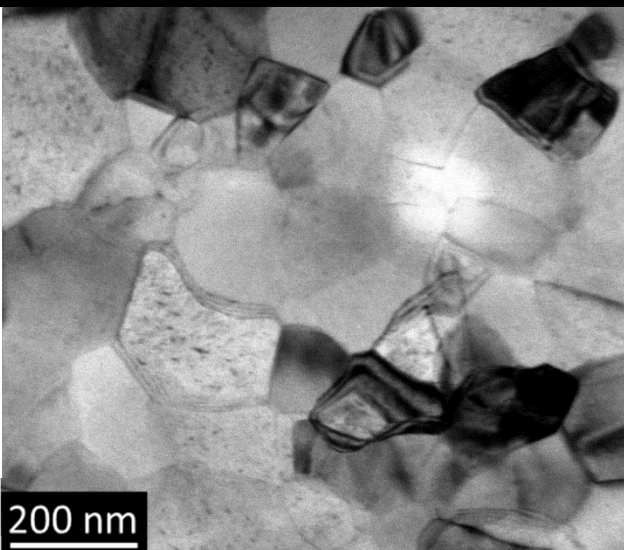
Post-Implantation, 25 °C



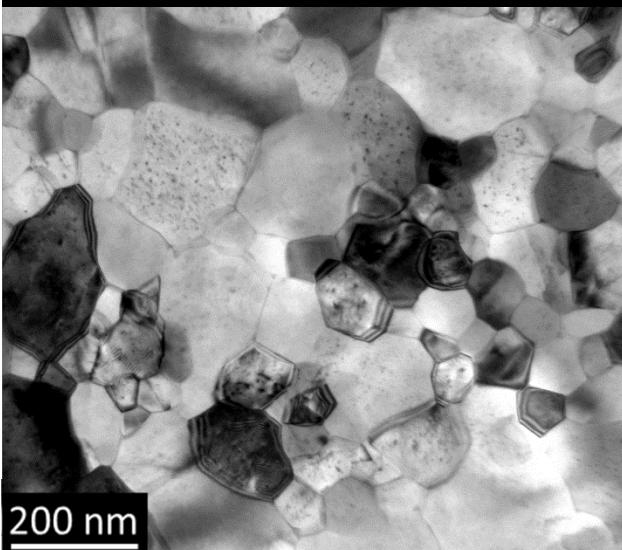
200 °C



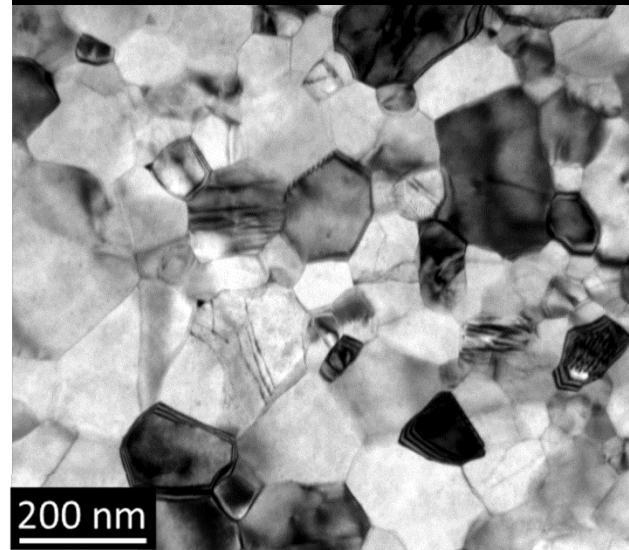
400 °C



500 °C

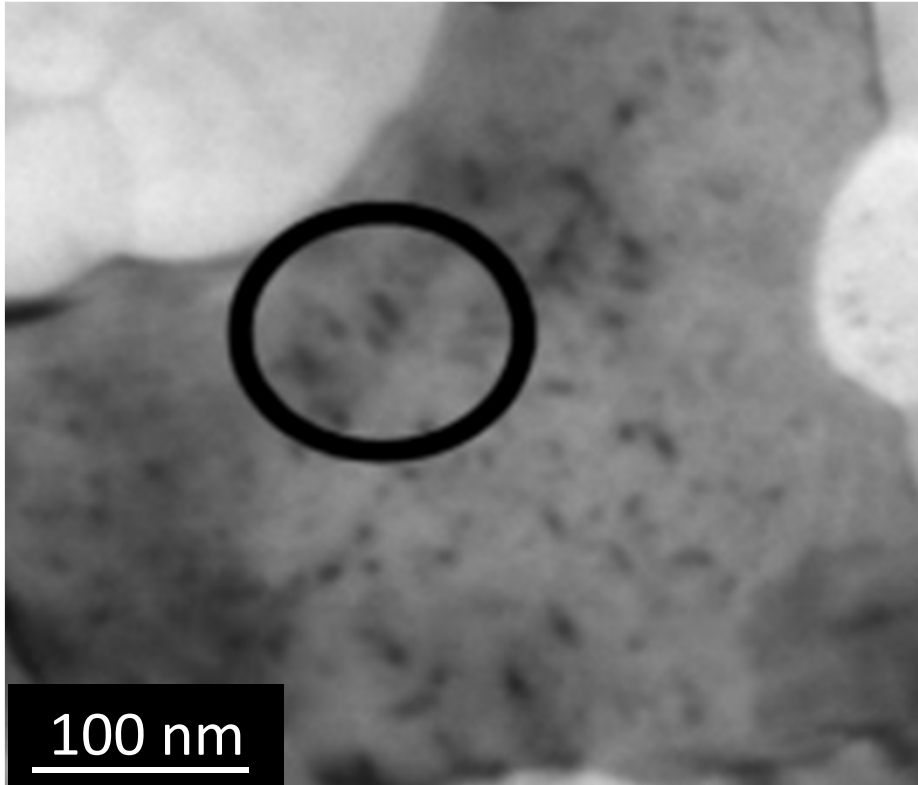


600 °C

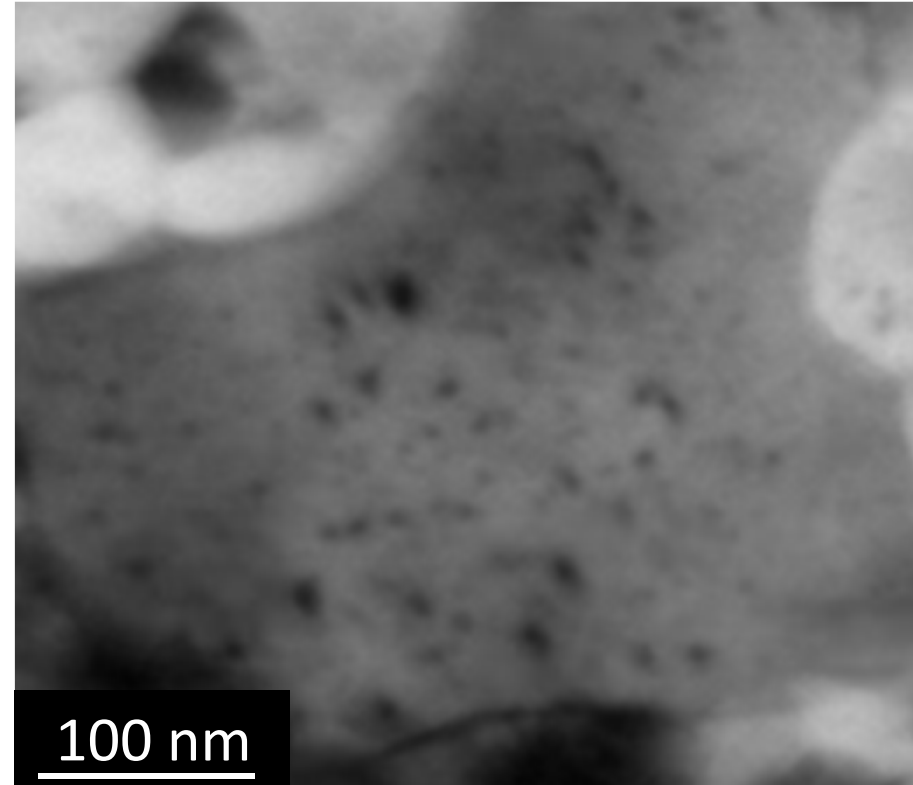


# Dislocation Loop Mobility 500 °C to 600°C

Dislocation Loop Coalescence

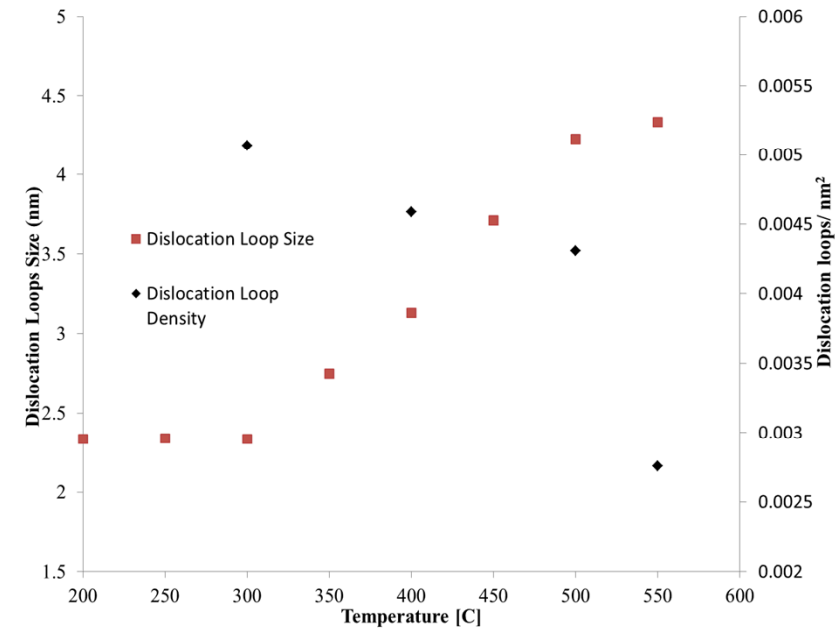
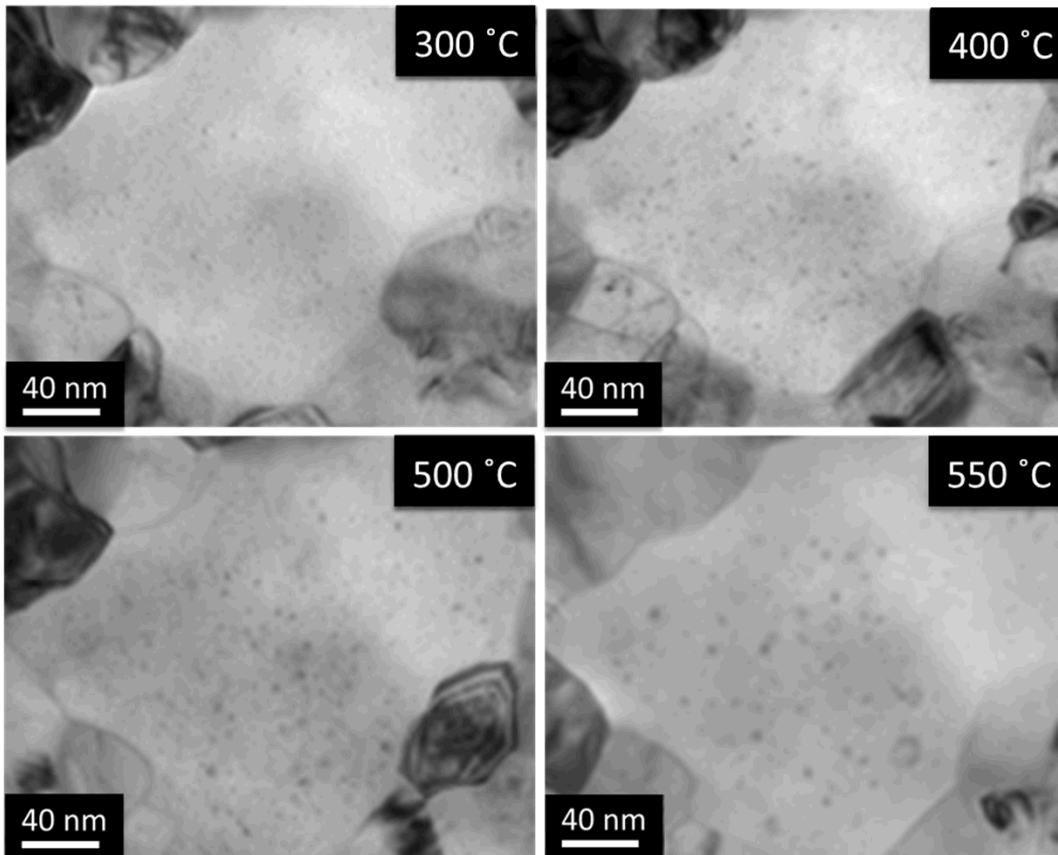


Dislocation Loop Migration to Surface



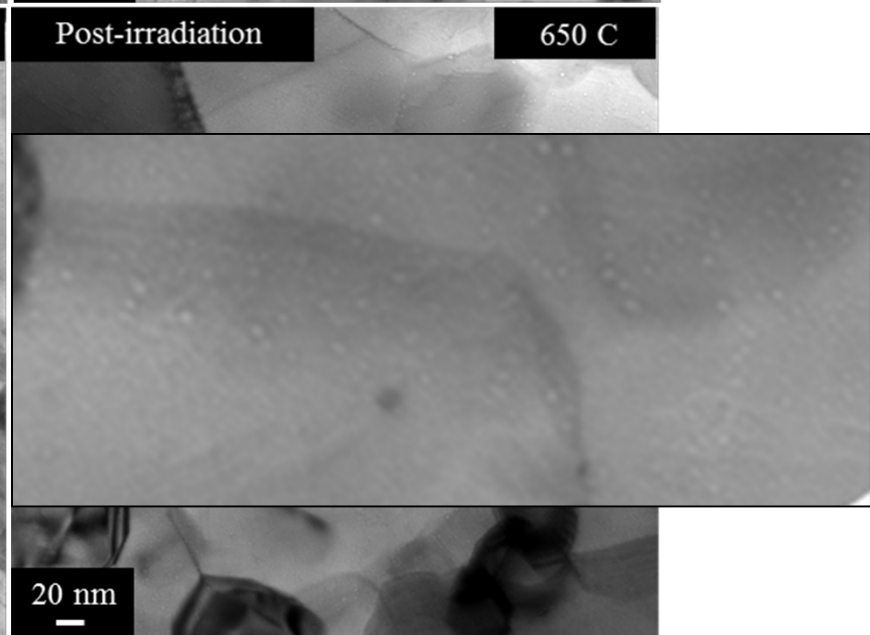
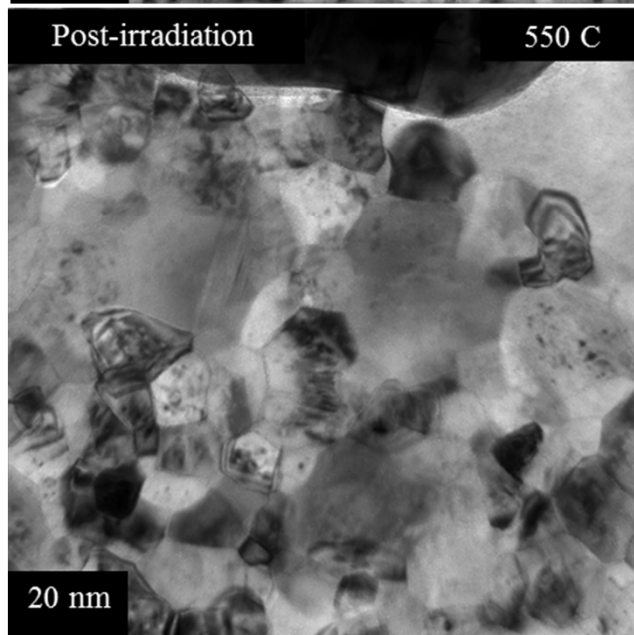
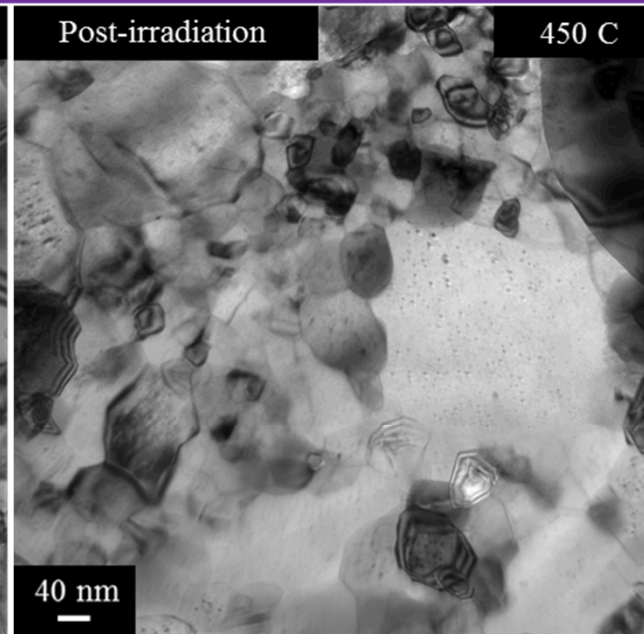
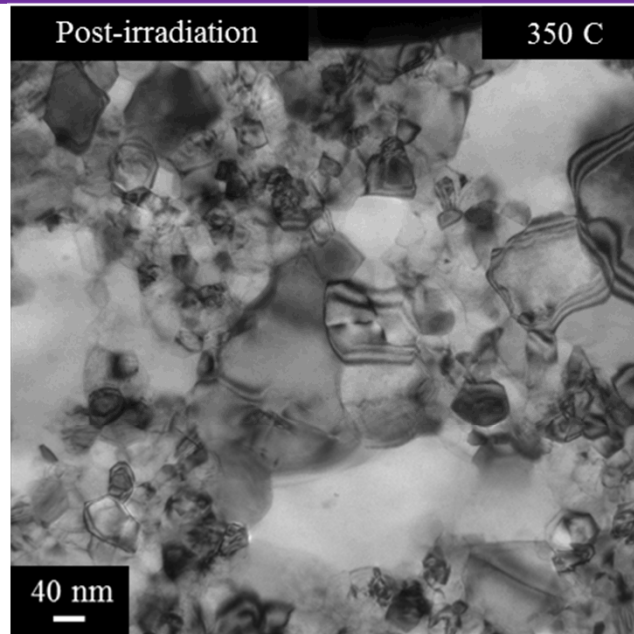


# Defect Size & Density Evolution with Annealing



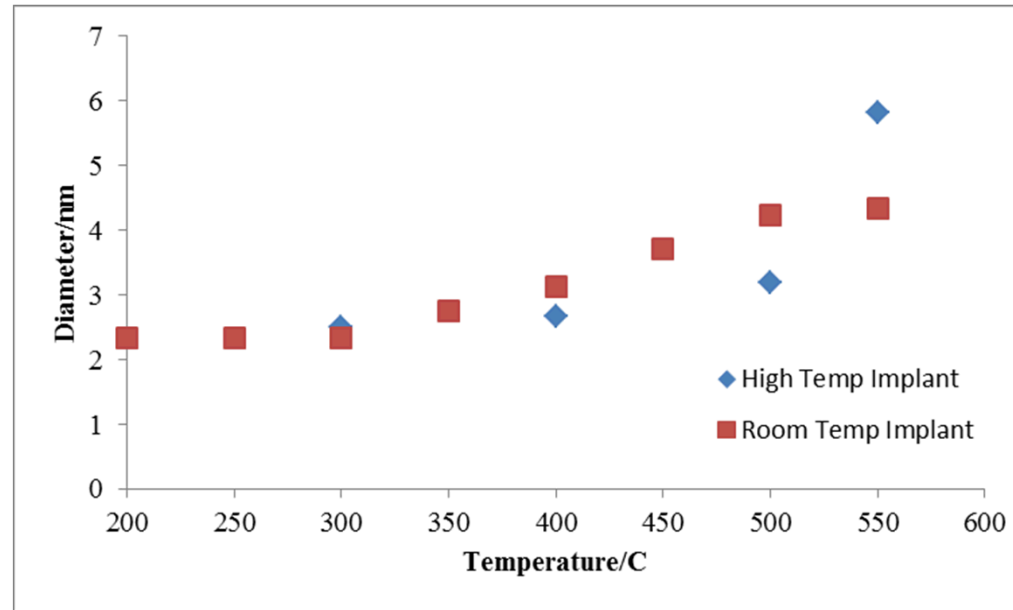
Defect Size Increases, Density Decreases  
No visible Cavities

# Elevated Temperature Helium Implantation



# Summary

- PLD used to create nano-grained Iron films
- Grain size can be controlled
- Annealing results in textured samples
- 10 keV He<sup>+</sup> implantation at Sandia's I<sup>3</sup>TEM
- Thermal effects studied
  - Room-temperature implantation then Annealing
  - High temperature implantation
- Similar size defects in both thermal conditions
- Cavities only appear during high-temp implantation



## Acknowledgments

US Department of Energy's Nuclear Energy University Program (DE-NE0000678)  
Cajer Gong, Daniel Bufford, Dan Buller