

Exploring the Effects of

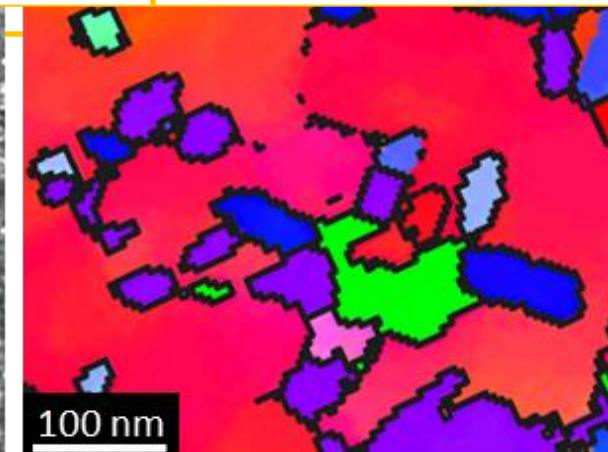
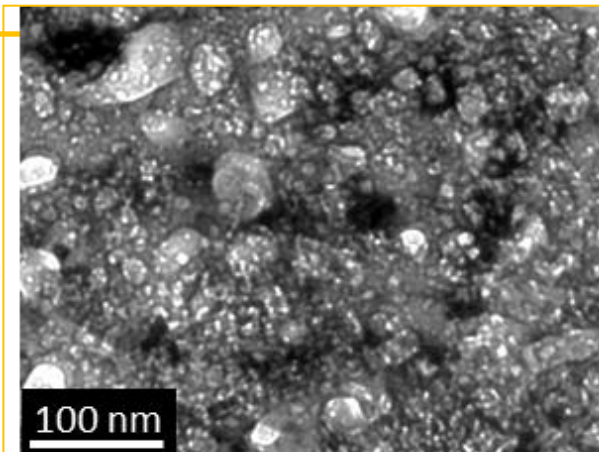
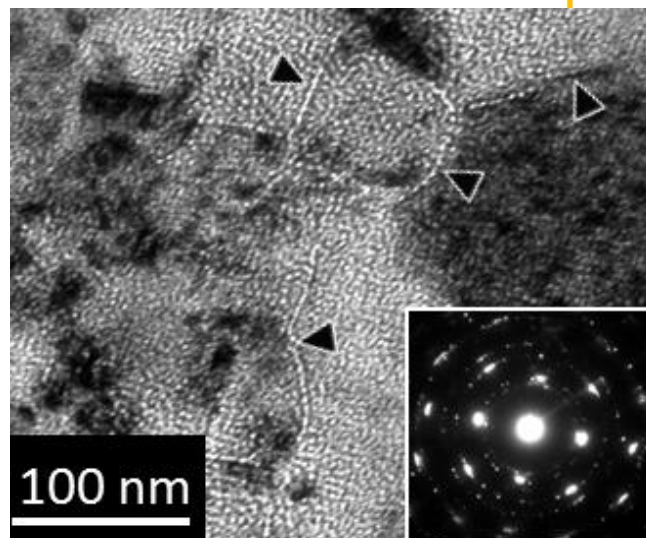
SAND2016-2329PE

Ion Irradiation: Single Ion Strikes to Macroscopic Property Changes

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Khalid Hattar

March 11, 2016



My Research Experience



RUHR
UNIVERSITÄT
BOCHUM



NORTHWESTERN
UNIVERSITY



Outline







- Sandia Information
- Radiation-Solid Interactions
- Physical Effects of Radiation: “Radiation Damage”
 - Dislocation loops
 - Stacking Fault Tetrahedra
 - Voids and Bubbles
 - Radiation Induced Segregation
- Macroscopic effects
 - Mechanical Effects
 - Magnetic Effects

National Laboratories



Sandia has two main locations

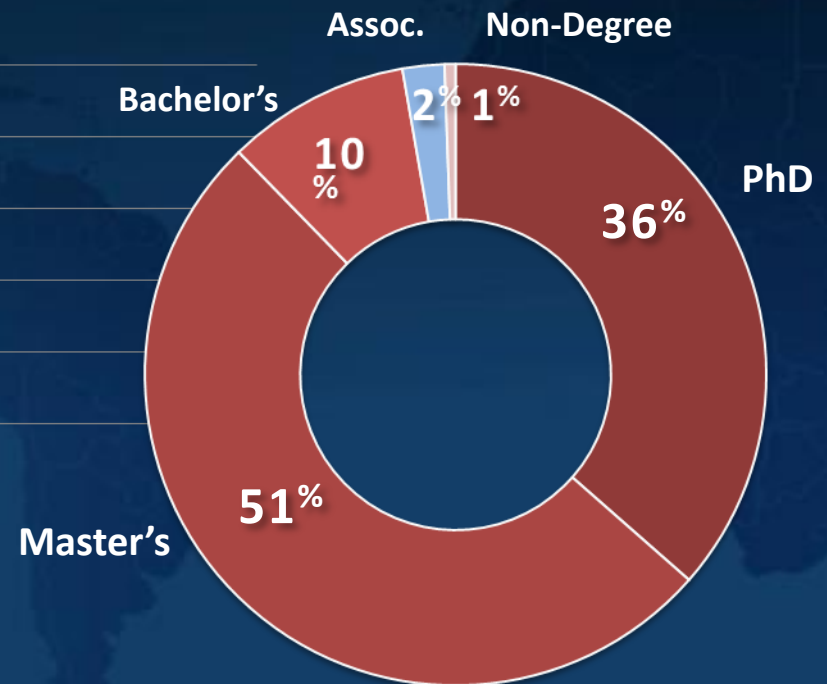
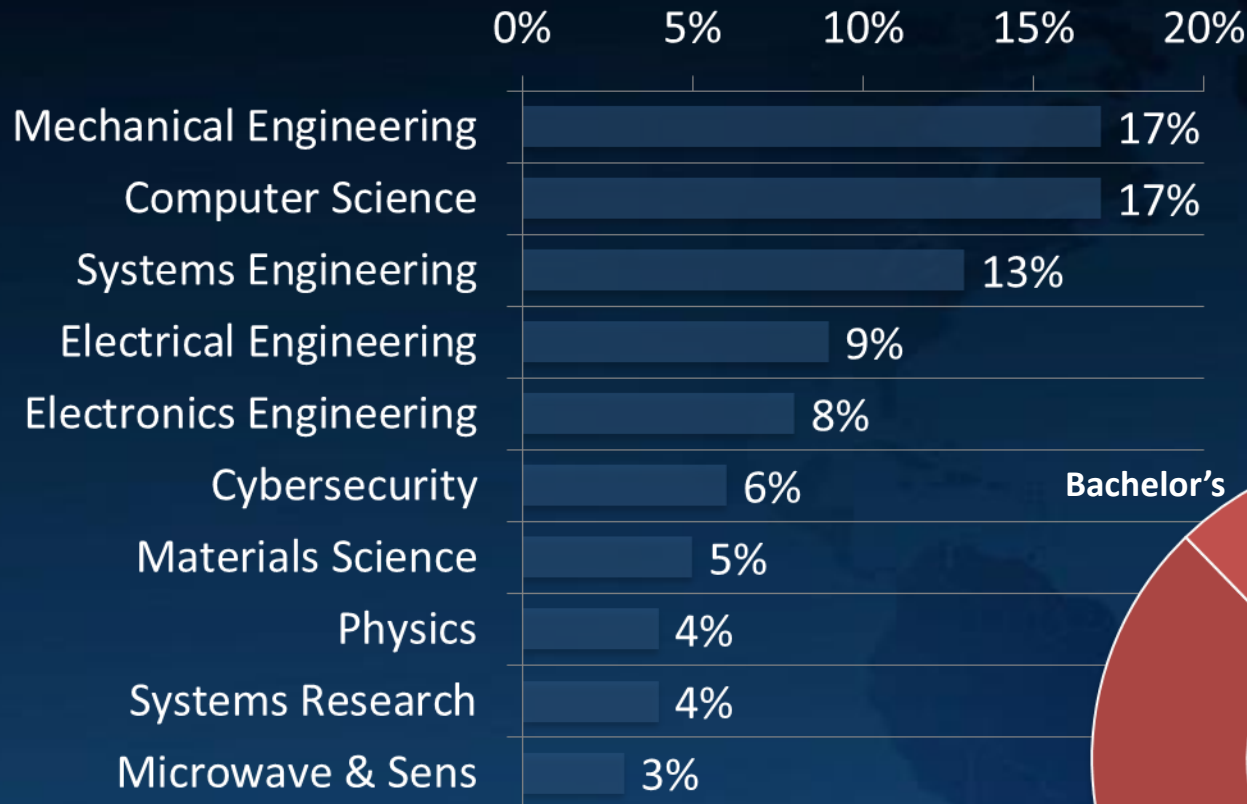


-  National Nuclear Security Administration labs
-  Science labs
-  Nuclear energy lab
-  Environmental management lab
-  Fossil energy lab
-  Energy efficiency and renewable energy lab

Sandia's Impact Video



R&D by Discipline & Degree



Top 10 job descriptions shown, Regular exempt non-management employees only

Living in Albuquerque



Life in Albuquerque

- Albuquerque is the largest city in New Mexico with a population of over 500,000
- Affordable housing, reasonable cost of living
- Minimal traffic congestion

Watch:

[Sandia New Mexico Video](#)

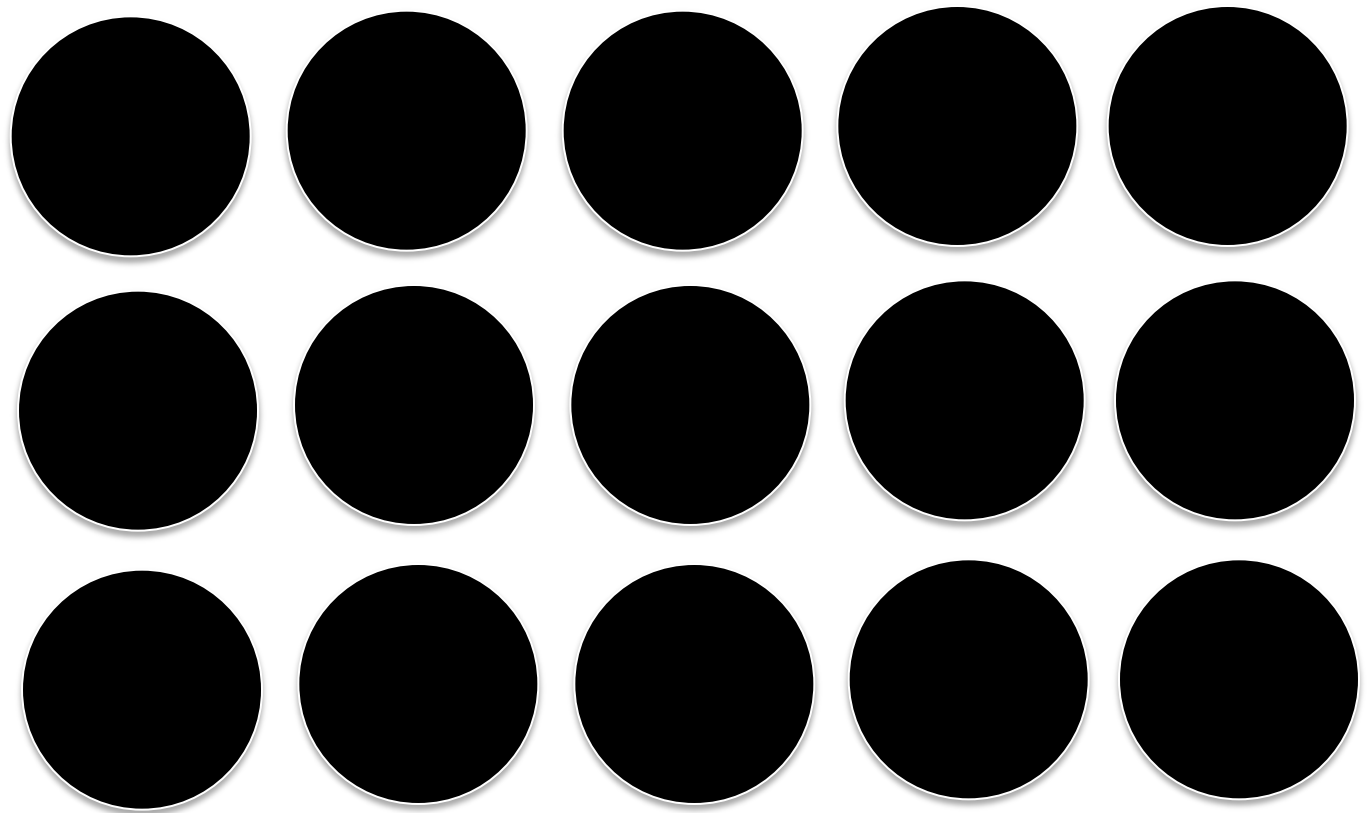
Albuquerque Environment

- High desert climate with 278 annual days of sunshine
- Average temperatures between 78° and 40°
- Wide-open spaces

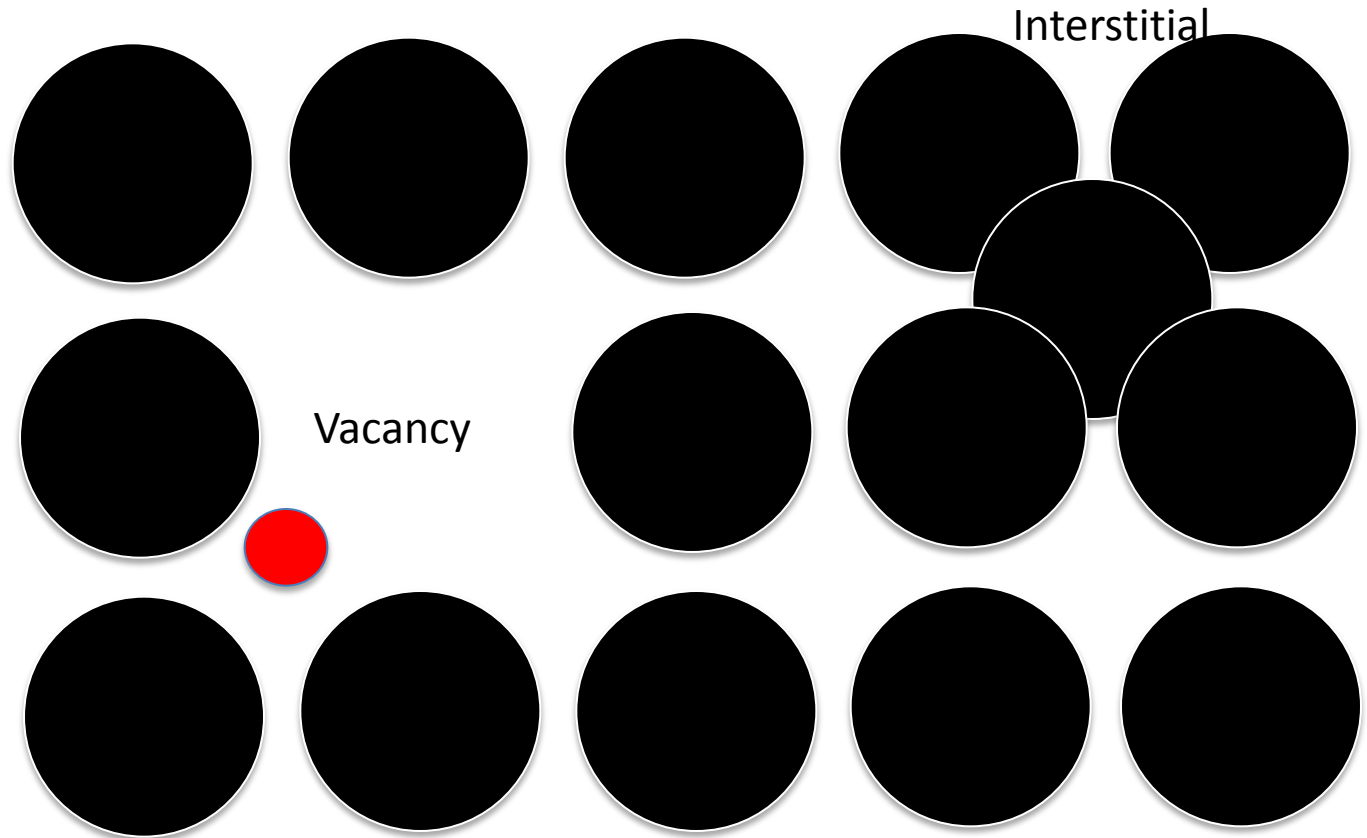
Things to Do

- Outdoor recreation - Ski, snowboard, hike, etc.
- Santa Fe – rich culture
- International Balloon Festival
- Explore Indian pueblos and our Hispanic heritage
- Green chile – NM Cuisine
- Museums, Parks, Sports

Radiation-Solid Interactions

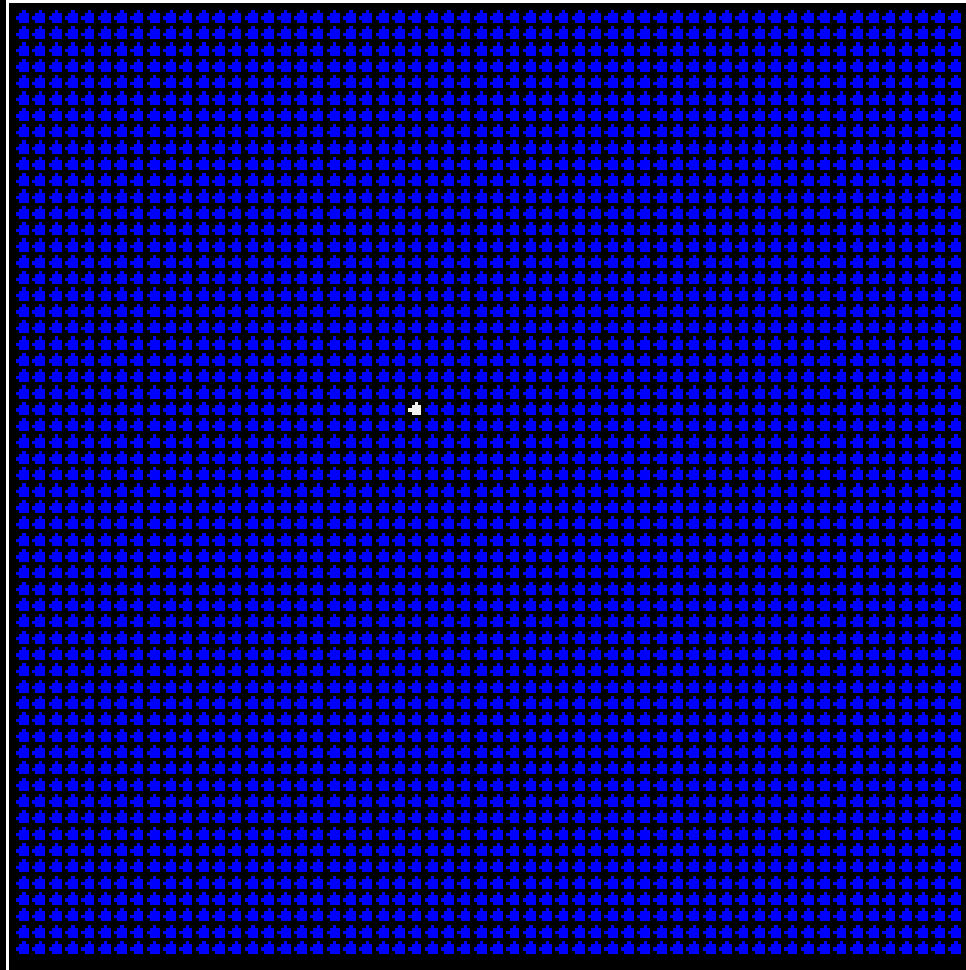


Radiation-Solid Interactions



Radiation-Solid Interactions

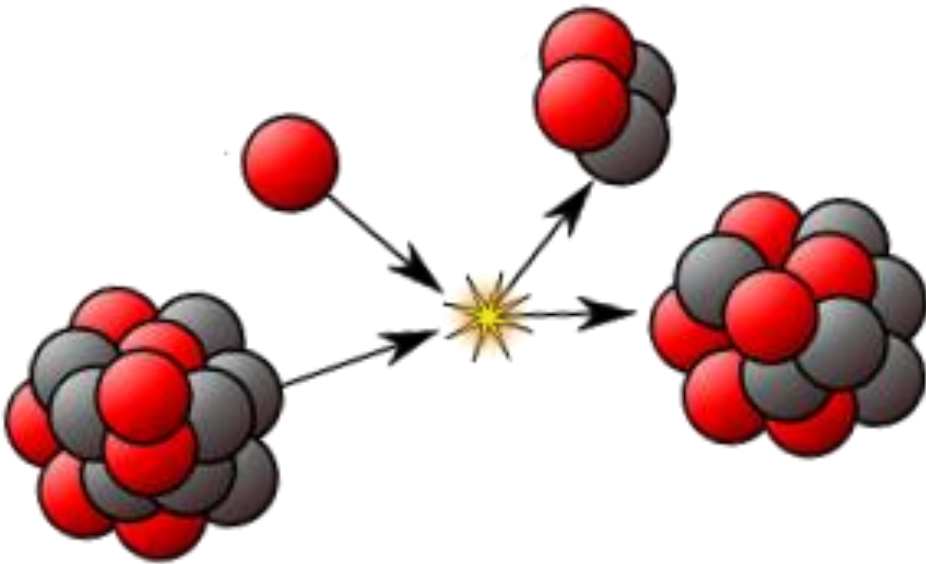
time 0.0001 ps



Kai Nordlund (2008)

Collision cascades may result in stable defects

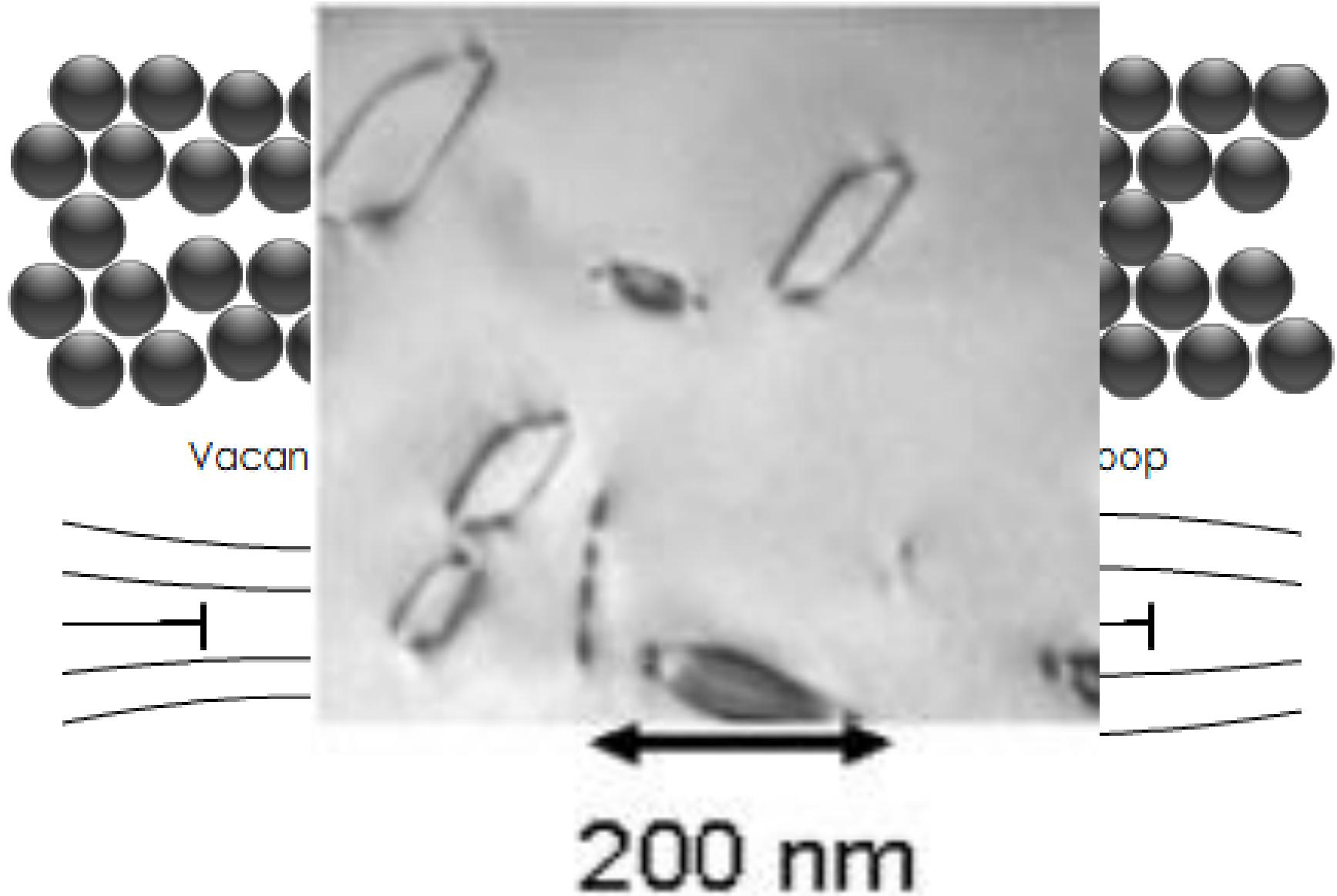
Nuclear Transmutation



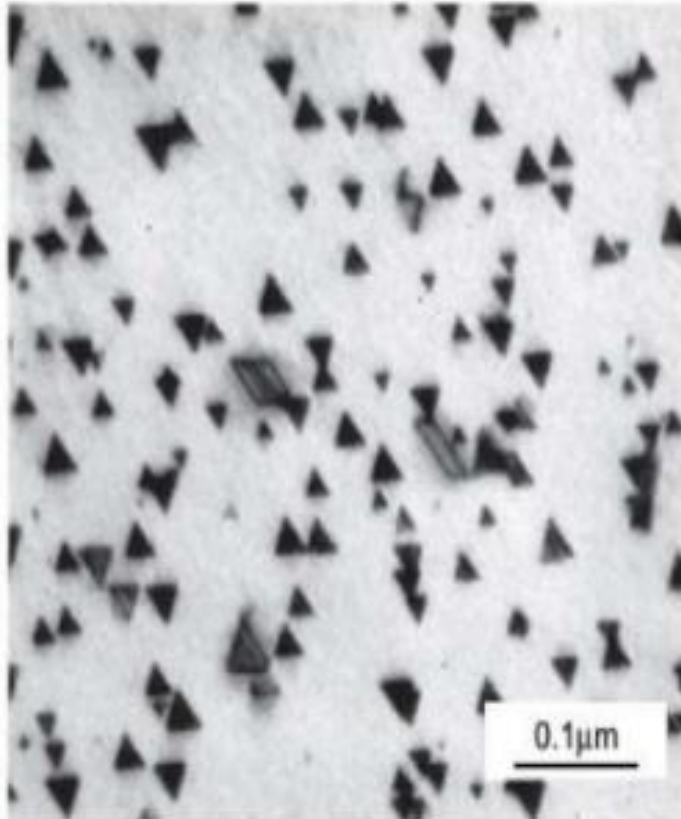
- (n, p)
- (n, α)

The conversion of one chemical element or an isotope into another

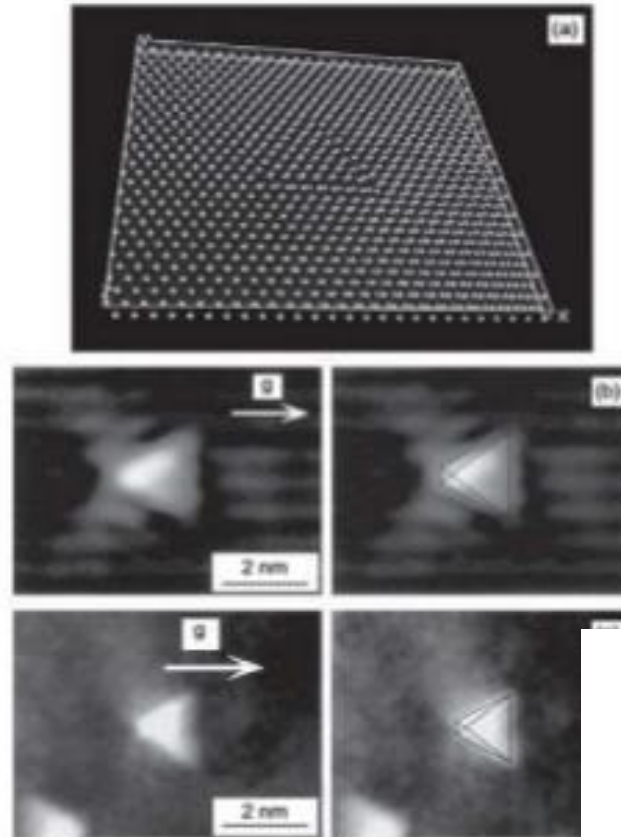
Microstructural Defects: Dislocation Loops



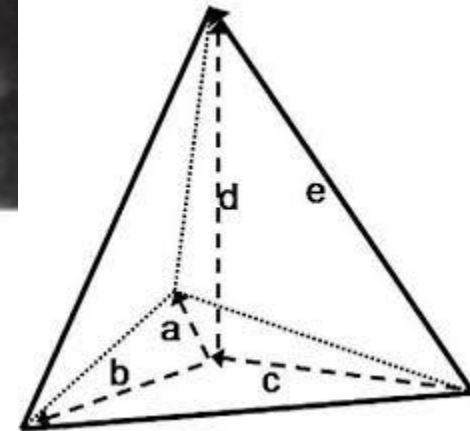
Microstructural Defects: Stacking Fault Tetrahedra



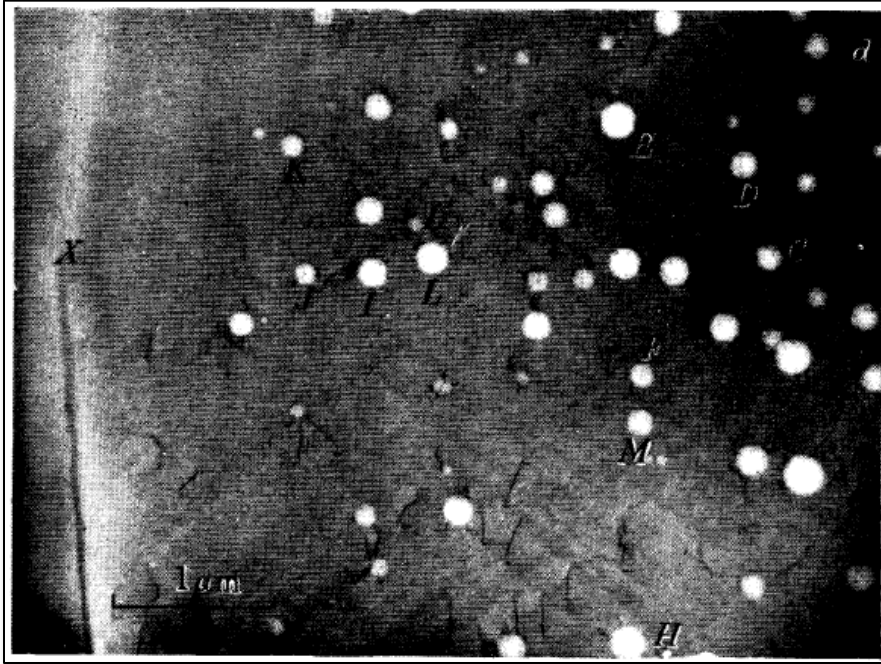
Transmission electron micrograph of tetrahedral defects in quenched gold.



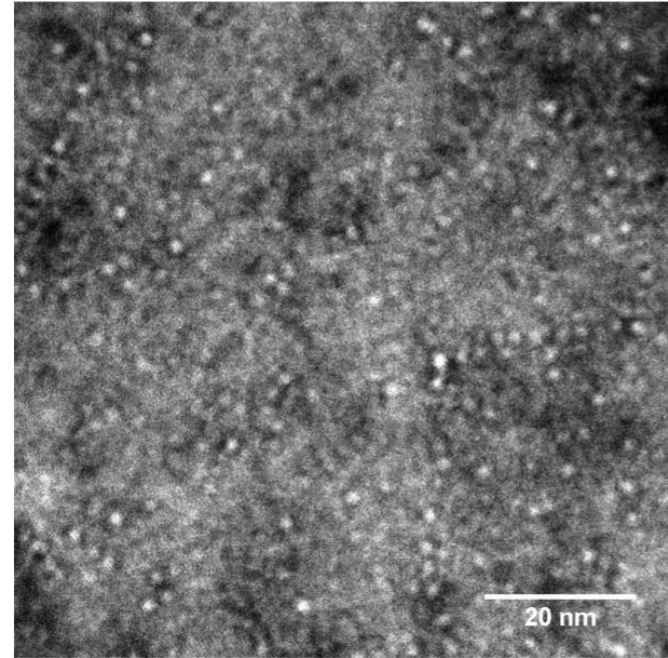
Stacking-fault tetrahedron in irradiated copper.



Microstructural Defects: Cavities

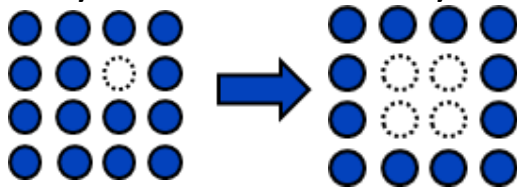


Voids



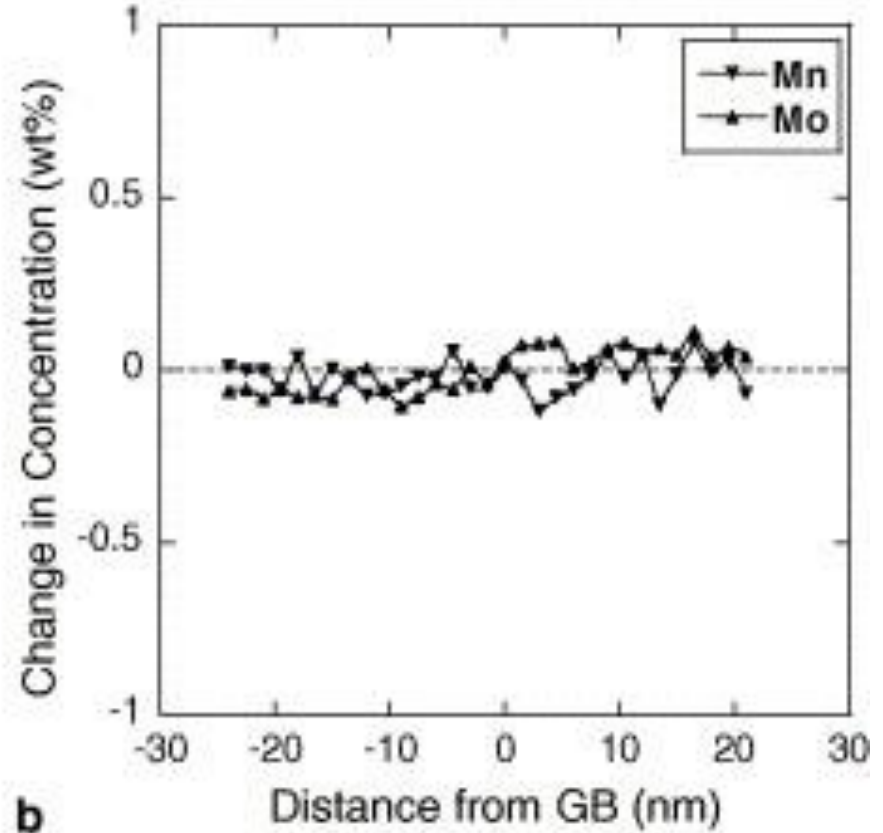
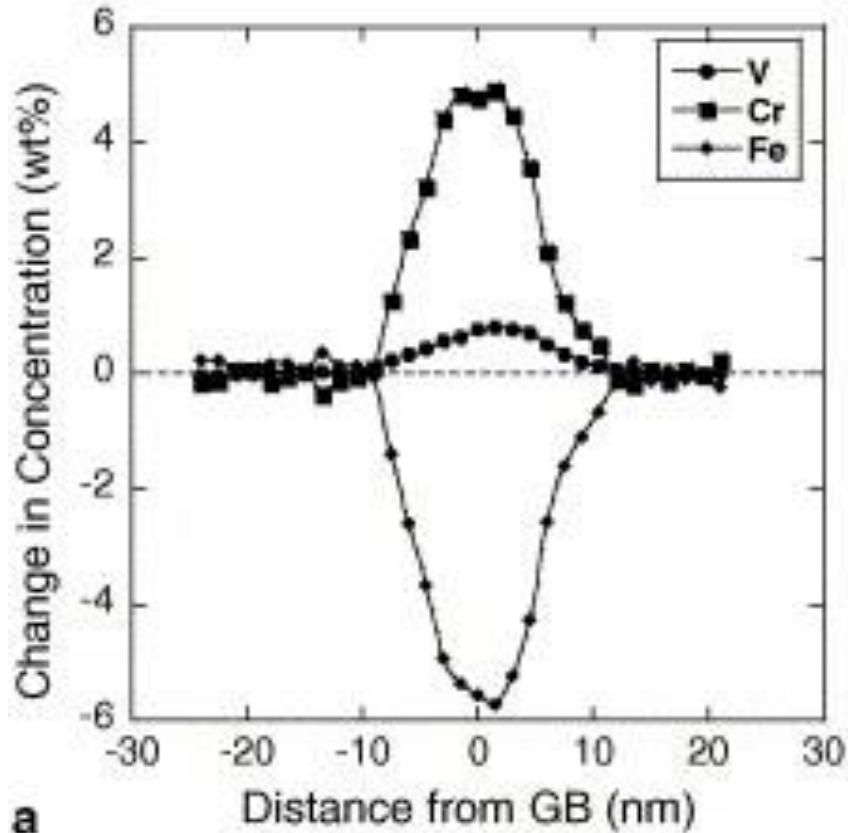
Helium Bubbles

Vacancy Diffusion Vacancy Clusters



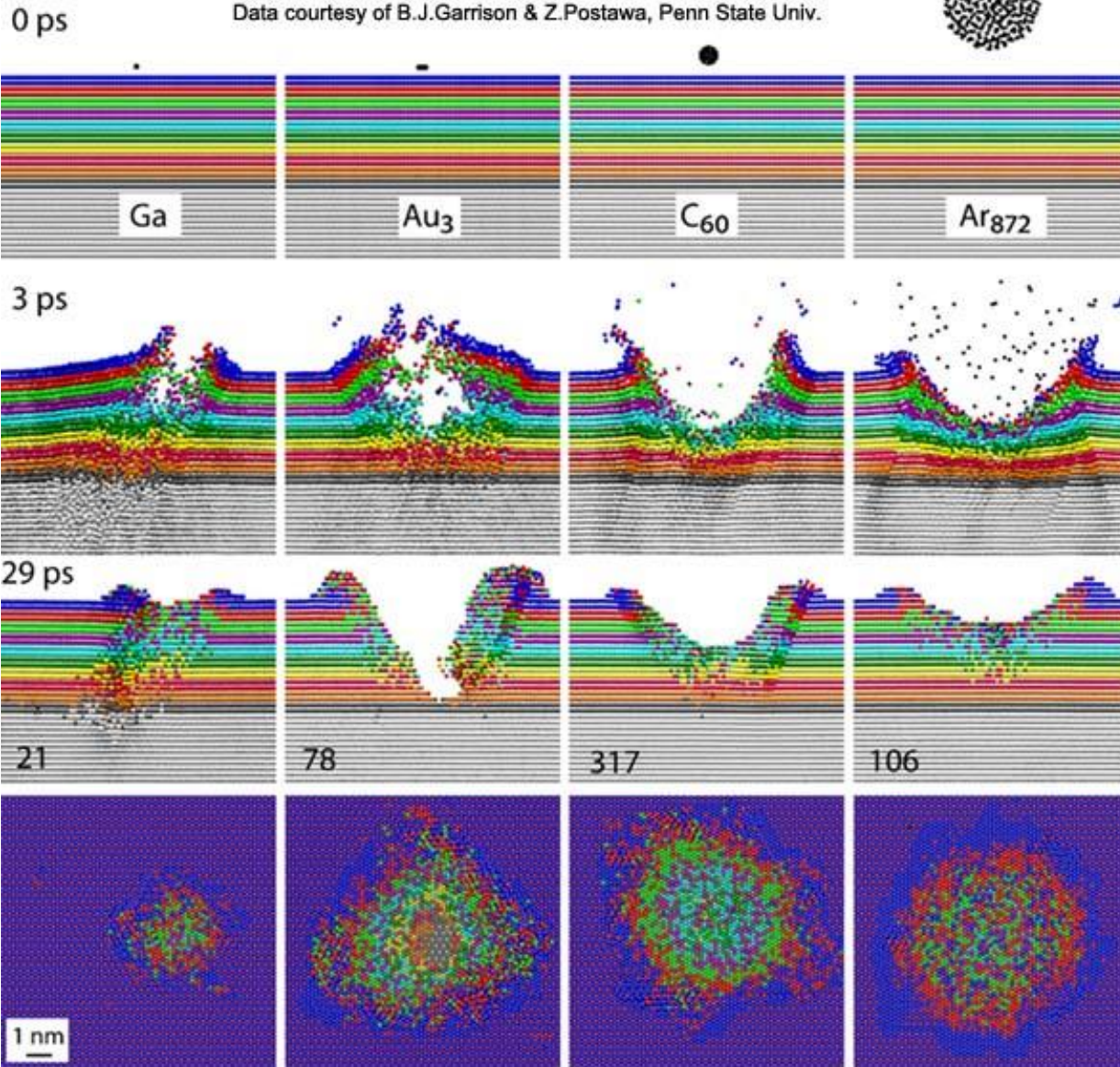
Helium and hydrogen
enhance cavity nucleation

Radiation Induced Segregation



Change in concentrations with distance from the GB for T91 irradiated to 10 dpa at 450 °C with 2.0 MeV protons. (a) Fe, Cr, and V, and (b) Mn and Mo.

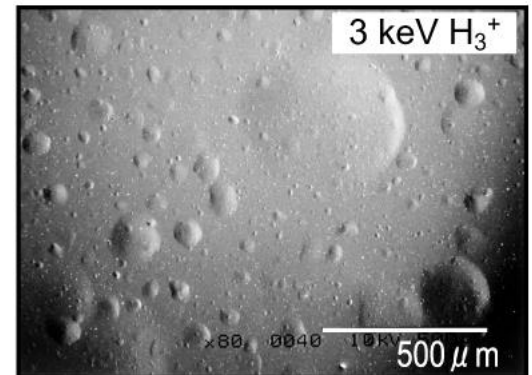
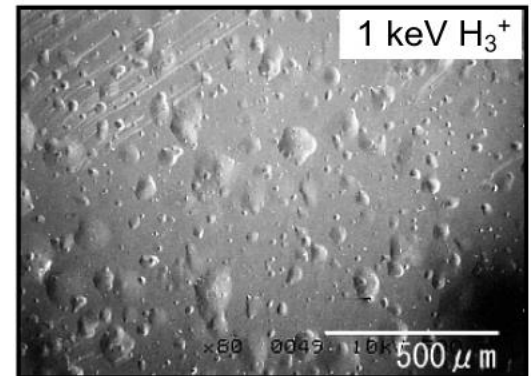
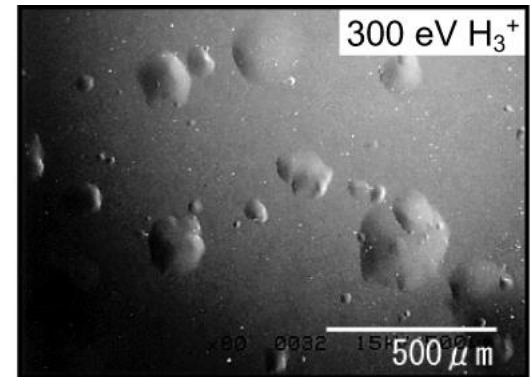
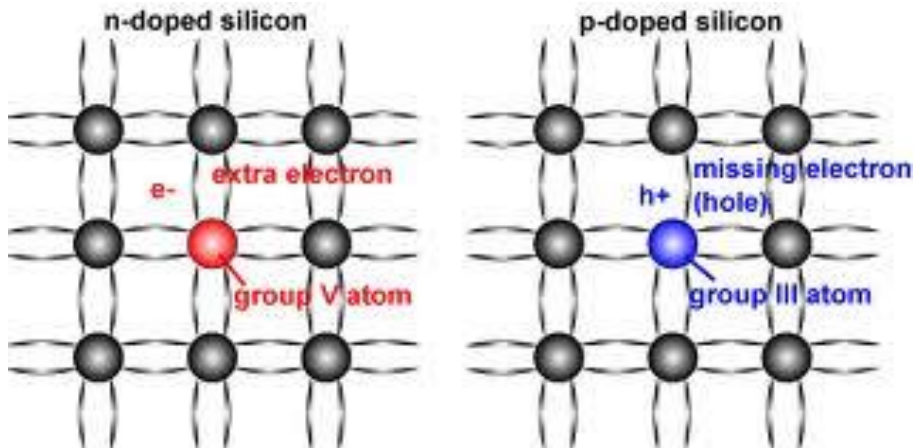
Radiation Induced Mixing



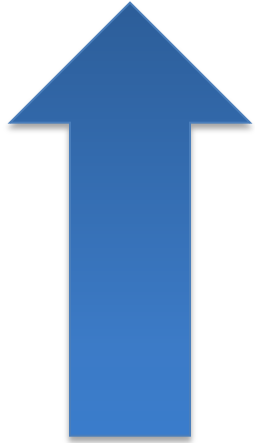
- Precipitation
- Dissolution
- Chemical Disordering
- Crystal Structure Transformation
- Amorphization

Other Radiation Effects

- Compositional Changes
- Sputtering
- Grain Growth
- Texture Changes
- Blistering and Exfoliation



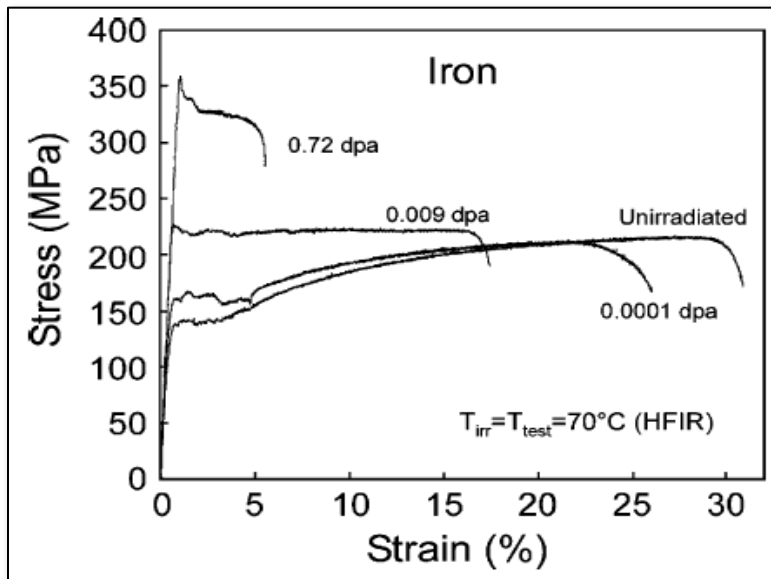
Radiation Effects on Mechanical Properties



- Yield Strength
- Ultimate Tensile Strength
- Ductile-Brittle Temperature Transition
- Young's Modulus
- Hardness
- High-Temperature Creep Rate



- Ductility
- Stress-Rupture Strength
- Density (swelling)
- Impact Strength
- Thermal Conductivity
- Electrical Conductivity

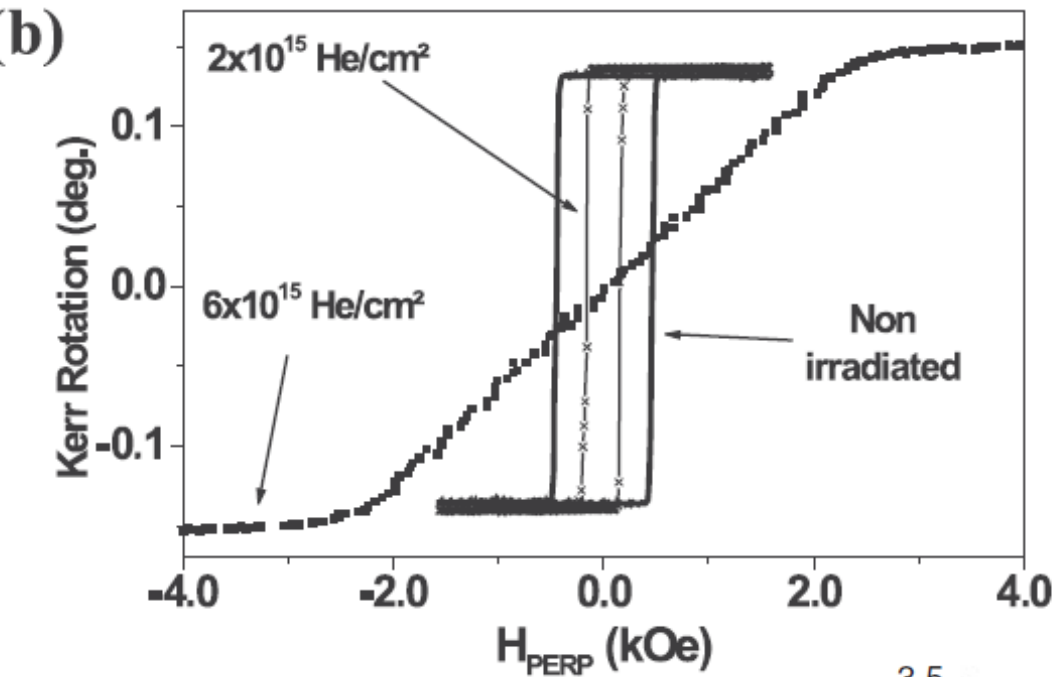


Eldrup et al. (2002)



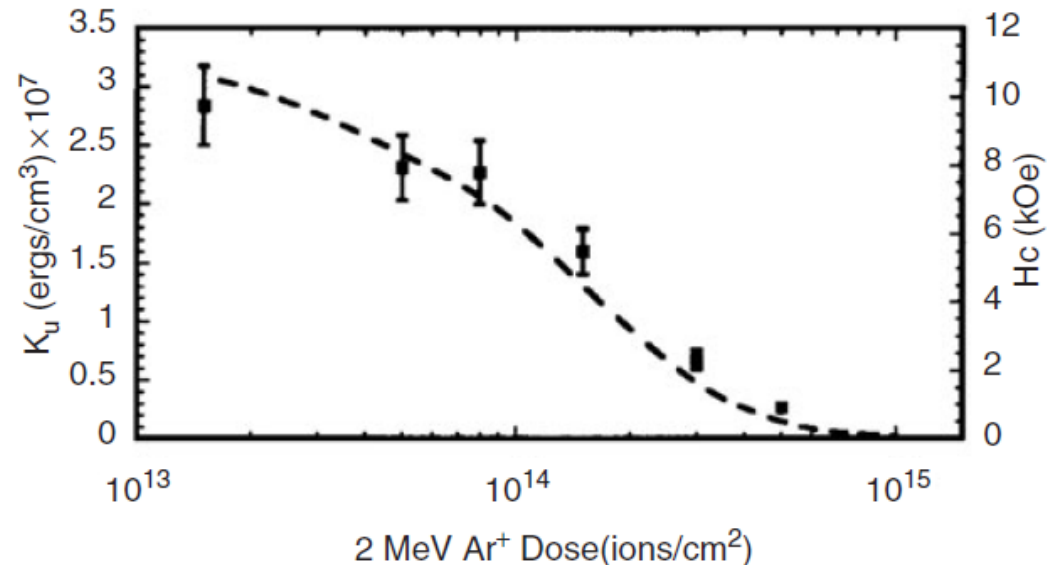
Mansur (1994)

Radiation Effects on Magnetic Properties

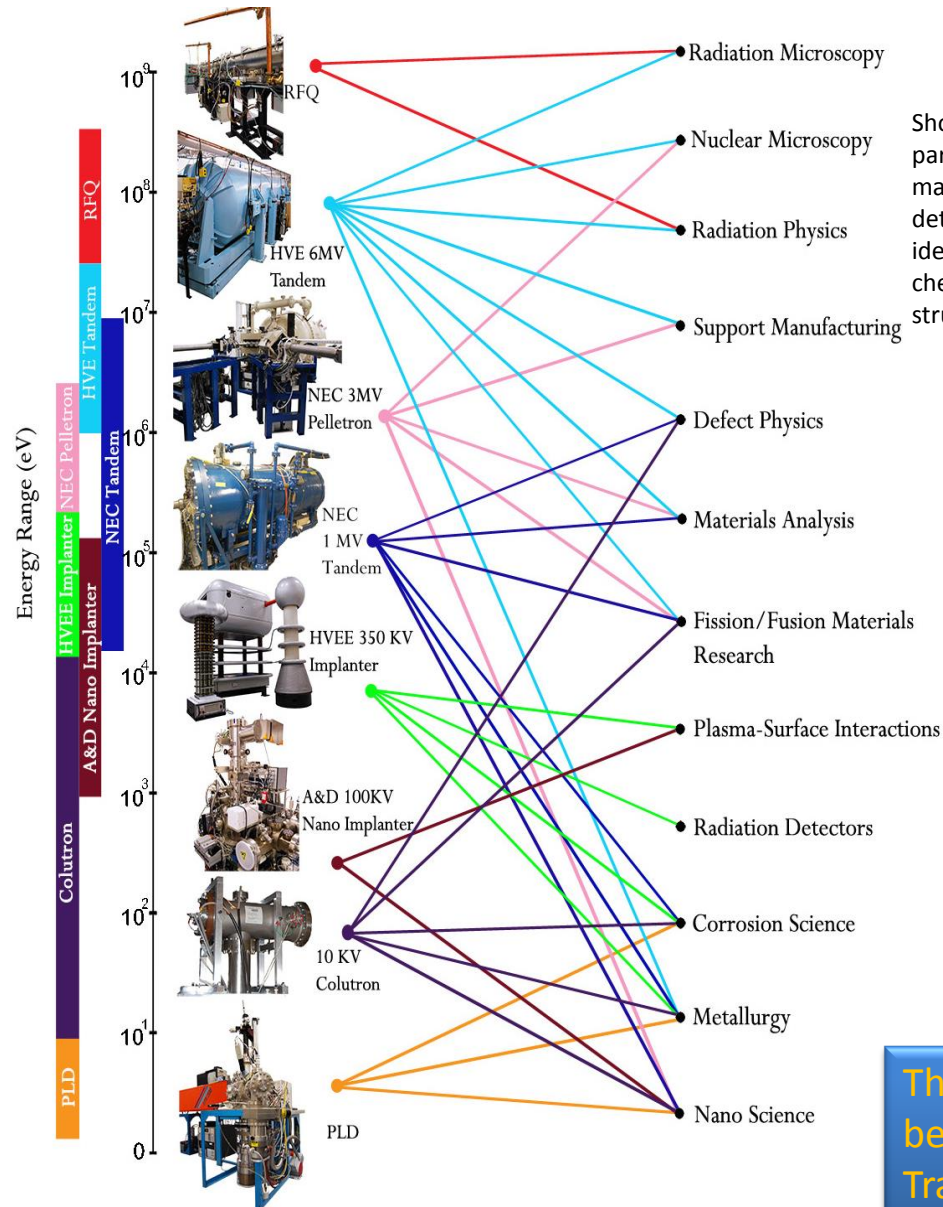


Hysteresis loops of Pt(2.8)-6x[Pt(0.6nm)-Co(0.3nm)]-Pt(6.5 nm)-SiO₂ multilayer. Irradiated with 30 keV He⁺ ions.

Anisotropy and Coercivity of Co-Pt multilayer samples as a function of 2 MeV Ar⁺ Fluence



Sandia's Ion Beam Laboratory

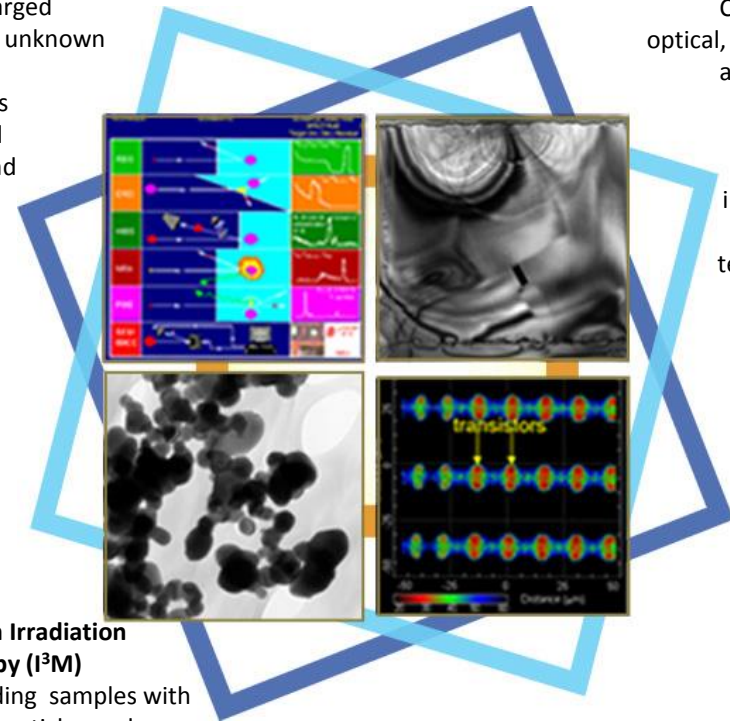


Ion Beam Analysis (IBA)

Shooting charged particle at an unknown material to determine it's identity, local chemistry, and structure.

Ion Beam Modification (IBM)

Changing the optical, mechanical, and chemical properties of materials via ion implantation to meet technological needs



In Situ Ion Irradiation Microscopy (I³M)

Bombarding samples with various particles and observing the changes in real time to understand how materials will behave in extreme environments.

Radiation Effects Microscopy (REM)

Using ion emissions to determine the Radiation hardness of microelectronics, identifying potential weaknesses.

The IBL has a unique and comprehensive capability ion beam set including and *In situ* Ion Irradiation Transmission Electron Microscopy.

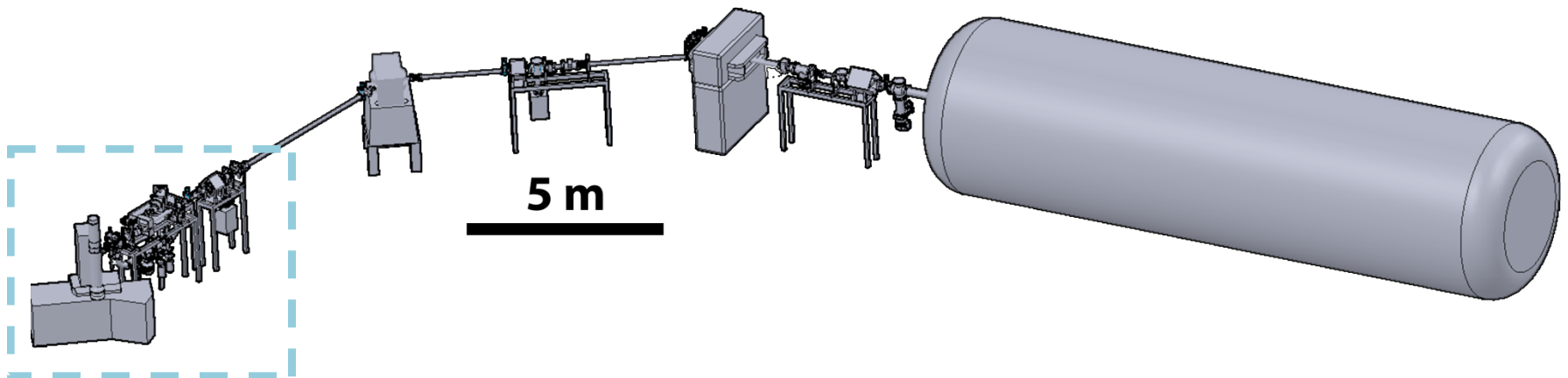
Sandia's I³TEM Facility



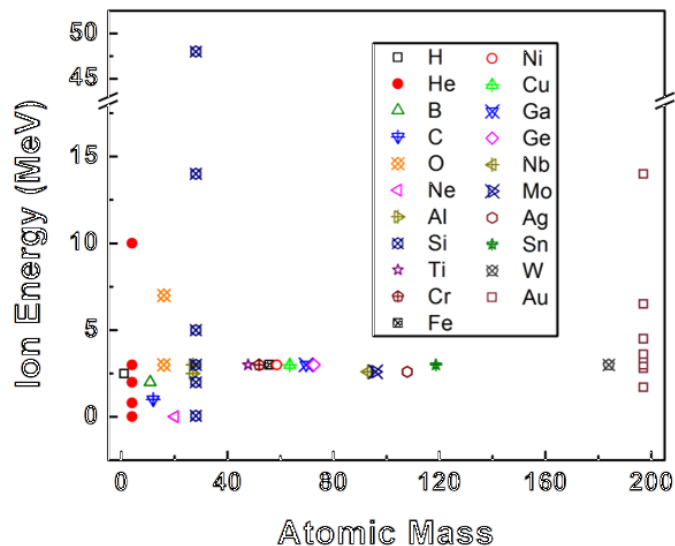
10 kV Colutron



6 MV Tandem



Sandia's I³TEM Facility



Heavy Ion Irradiation + Gaseous Implantation

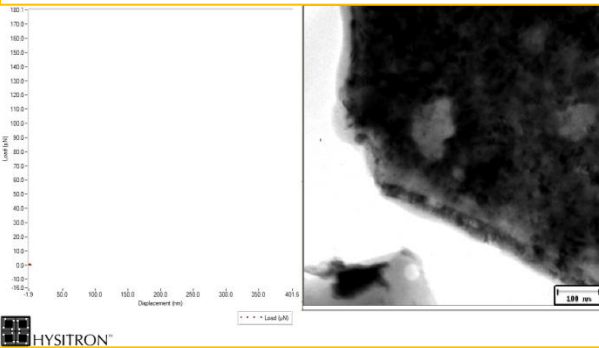
Control ratio of dpa and gas species implantation and characterize coupling effects

200 kV JEOL 2100 TEM

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

Radiation & 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

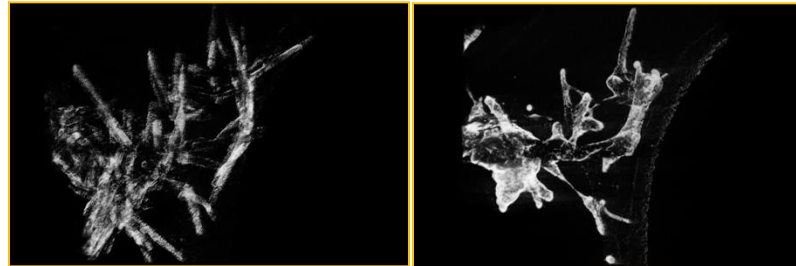
Environmental Effects

Protochips Liquid and Gas Flow

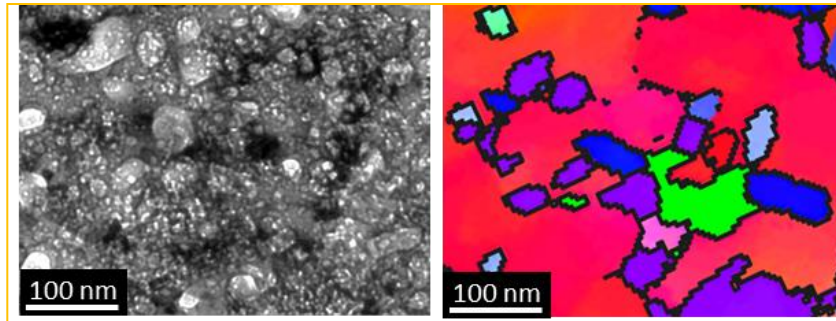
Study the material in different environments (flowing, mixing, temperature)

Structural Effects

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

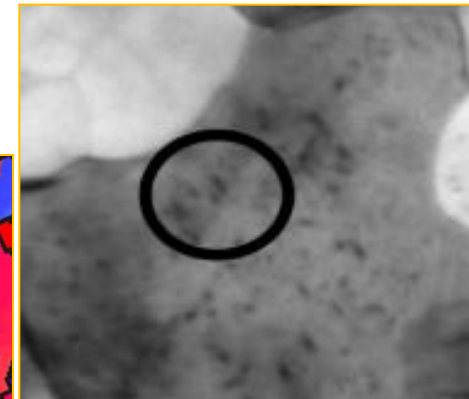


Nanomegas ASTAR
Grain structure changes as a result of radiation and implantation



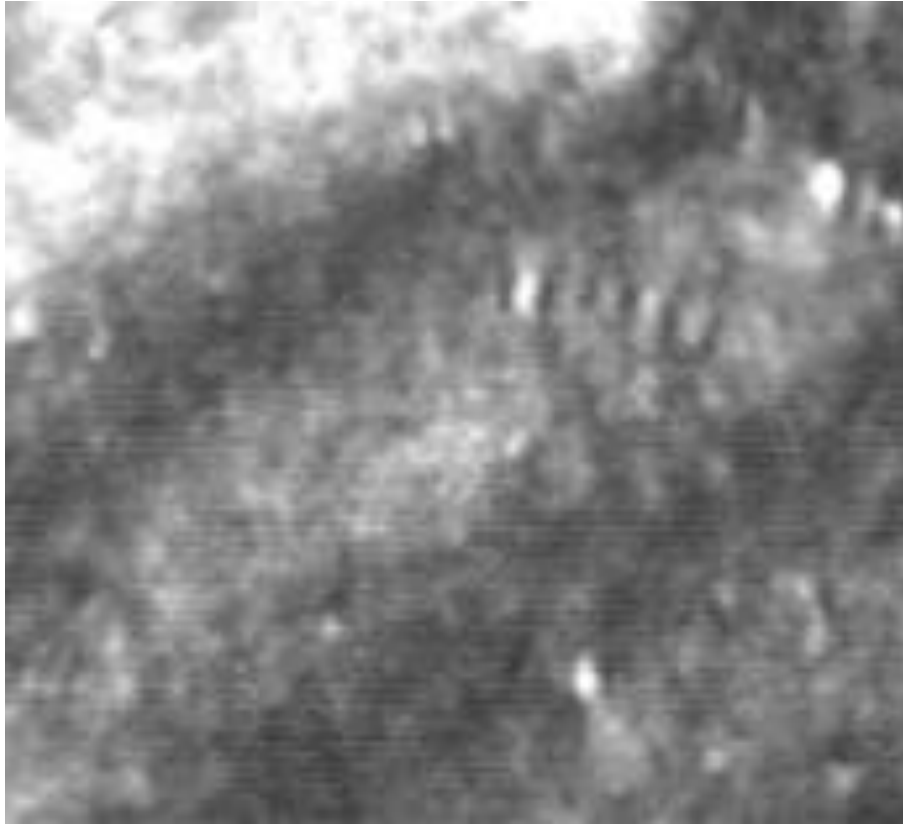
Thermal Effects

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

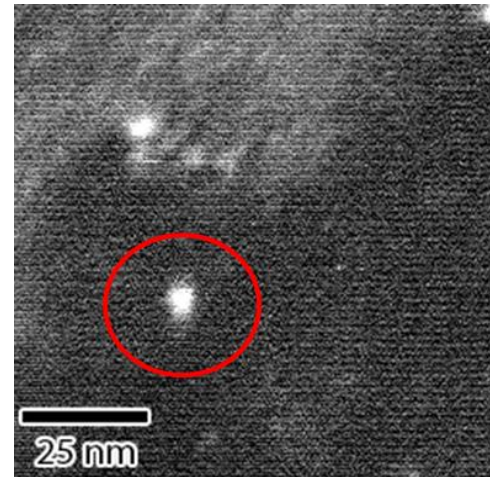
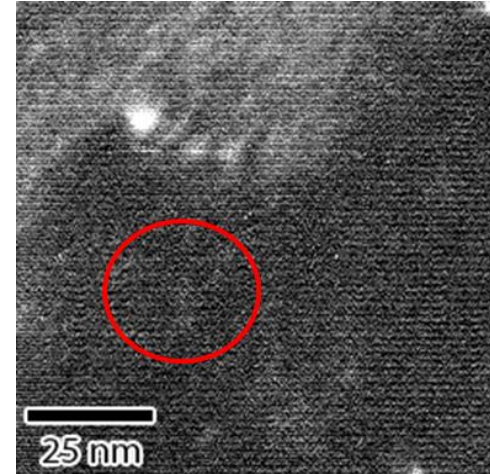


The application of advanced microscopy techniques to characterize synergistic effects in a variety of extreme environments

Ion Strikes in the TEM

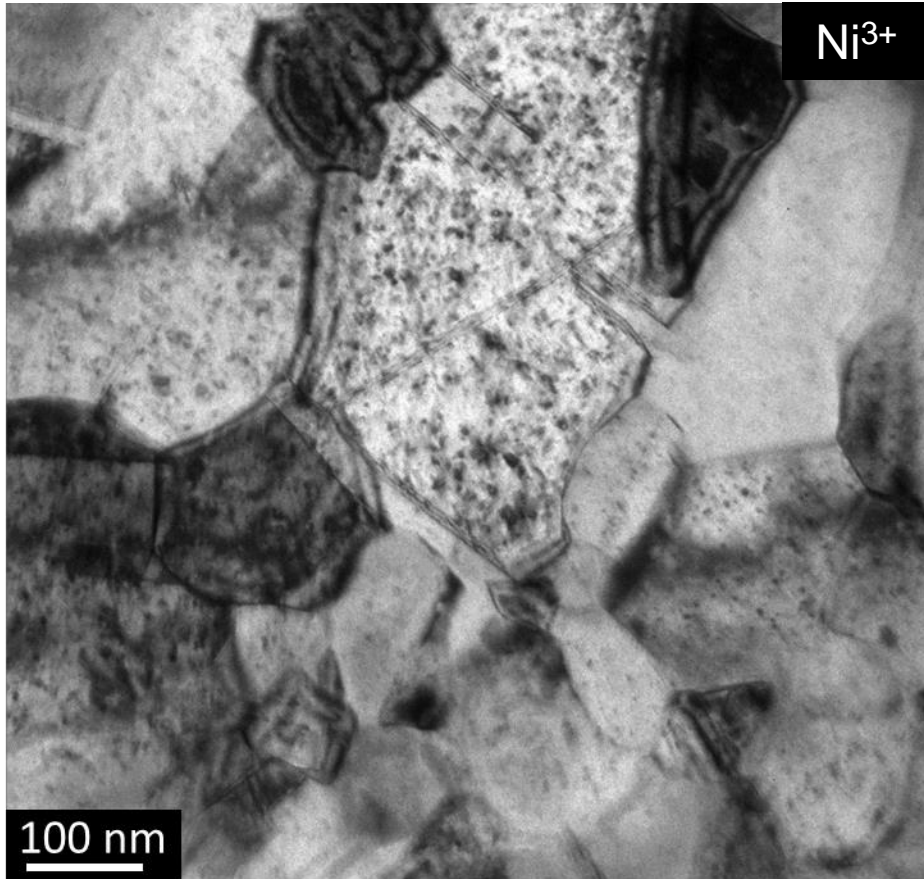


- 1.7 MeV Au³⁺ into Steel
- Single Ion Strikes Visible

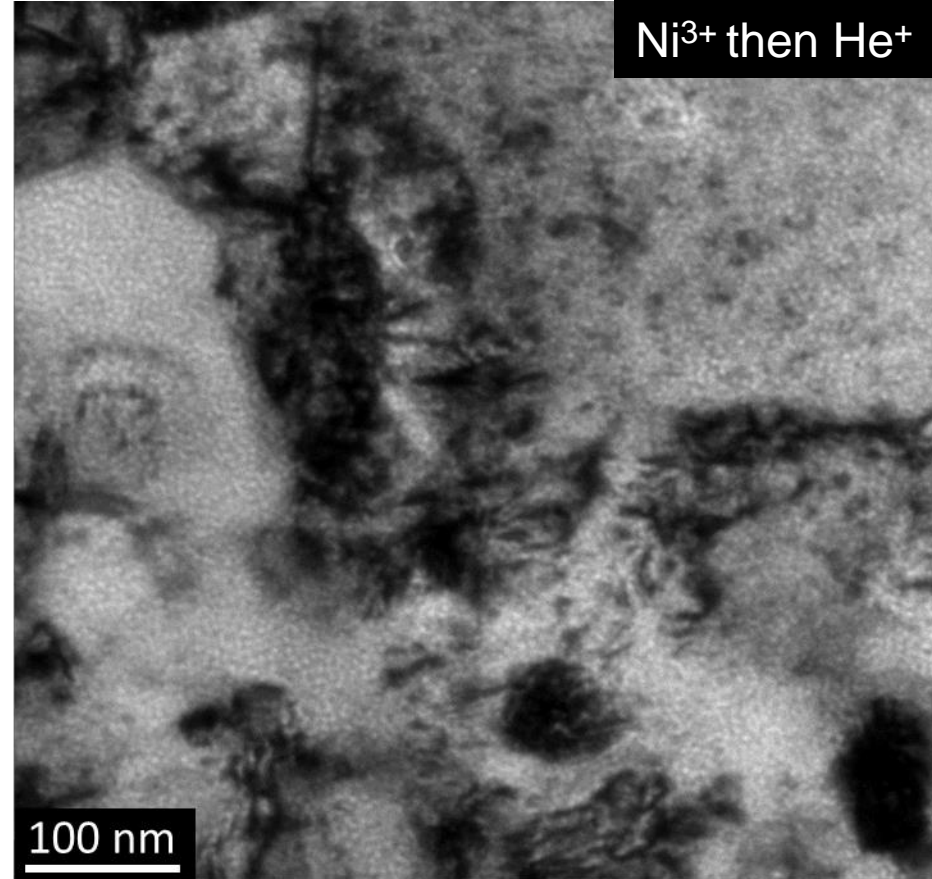


Strikes appear in 1/30 s

3 MeV Ni³⁺ Irradiation followed by 10 keV He⁺ Implantation

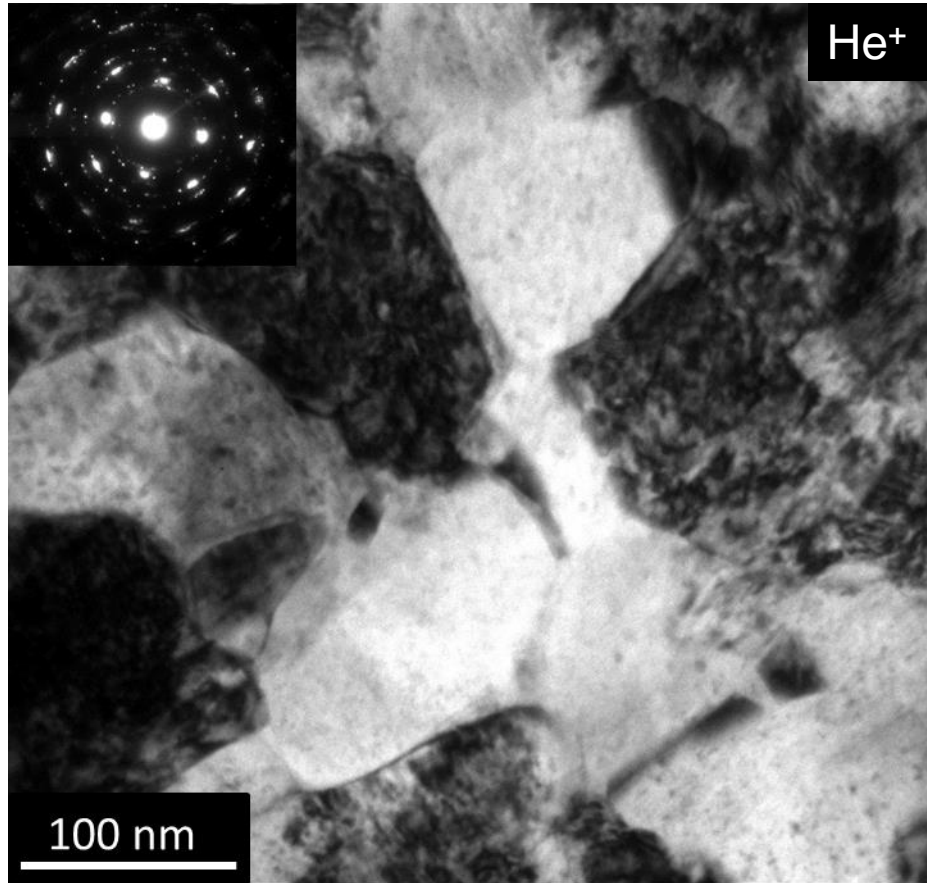


1.8 dpa Ni³⁺ irradiation
Dislocation loops and SFT are present

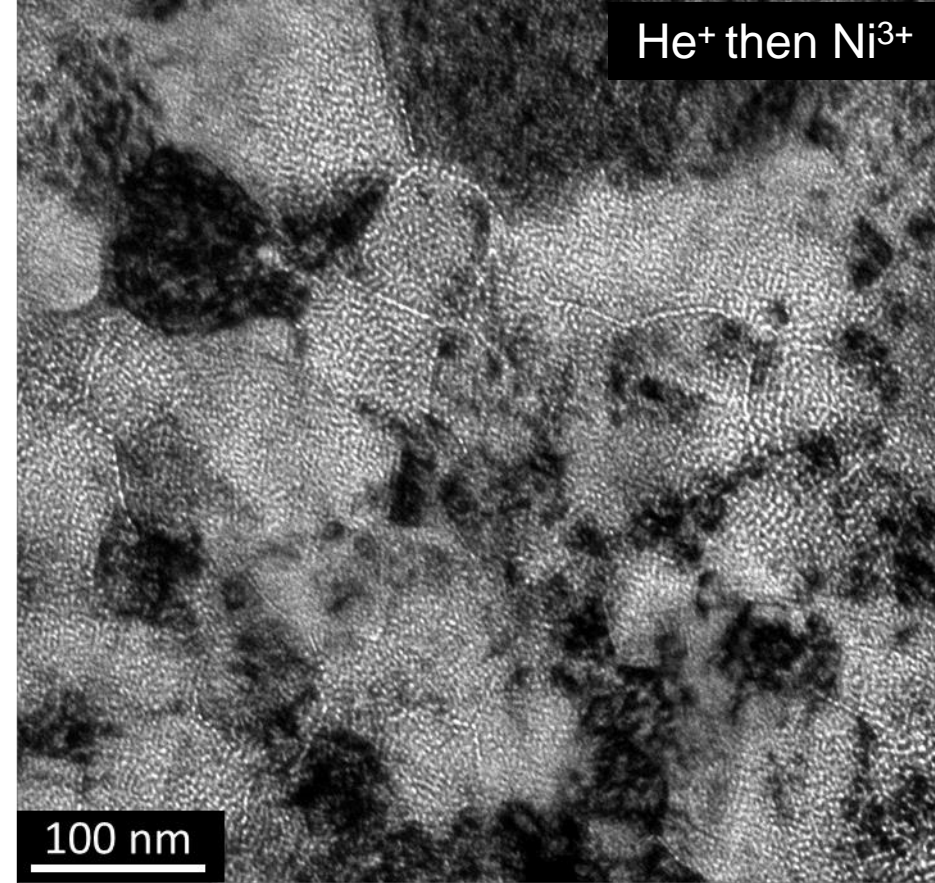


Additional 2x10¹⁶ He⁺/cm²
Evenly distributed nanometer
size cavities

10 keV He⁺ Implantation followed by 3 MeV Ni³⁺ Irradiation



10^{17} He⁺/cm²
Visible damage



0.7 dpa Ni³⁺ irradiation
High concentration of cavities along grain boundaries

Irradiation / Implantation Sequence Effect on Cavity Structure

Ni³⁺ then He⁺

He⁺ then Ni³⁺

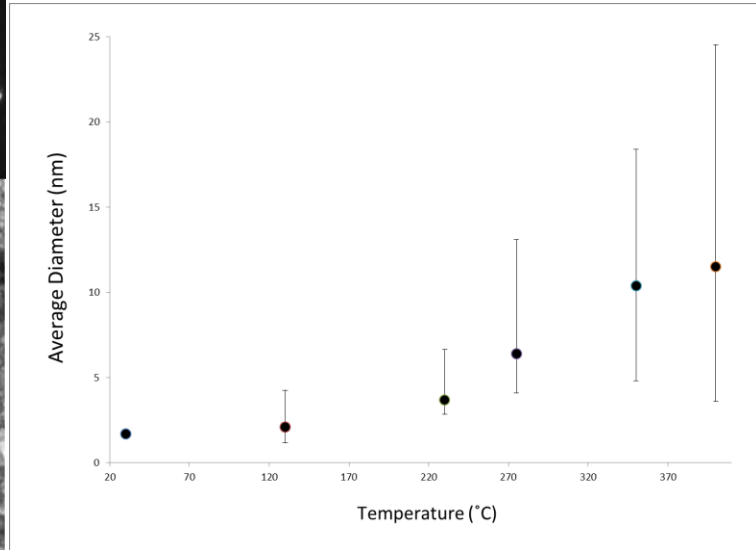
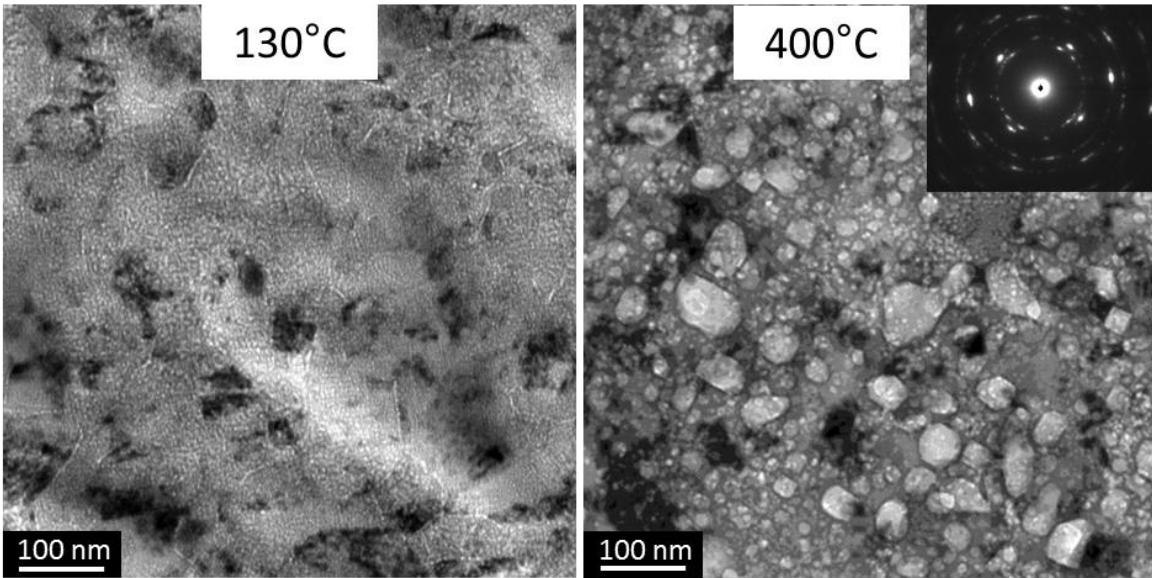
100 nm

100 nm

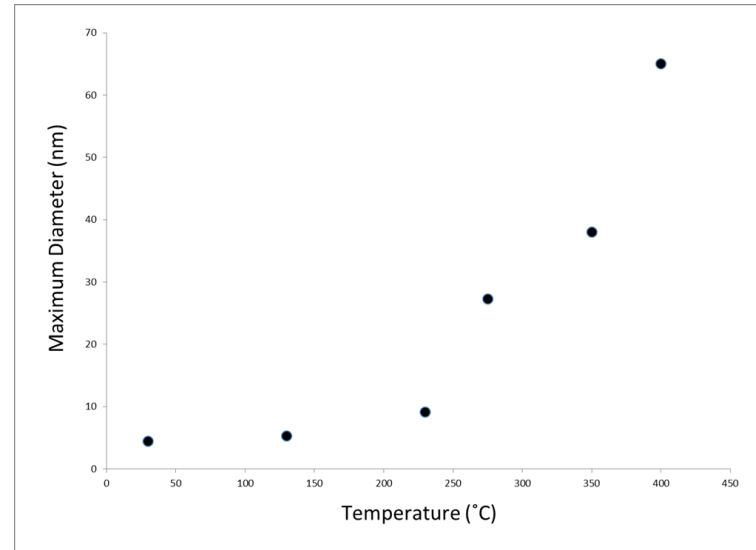
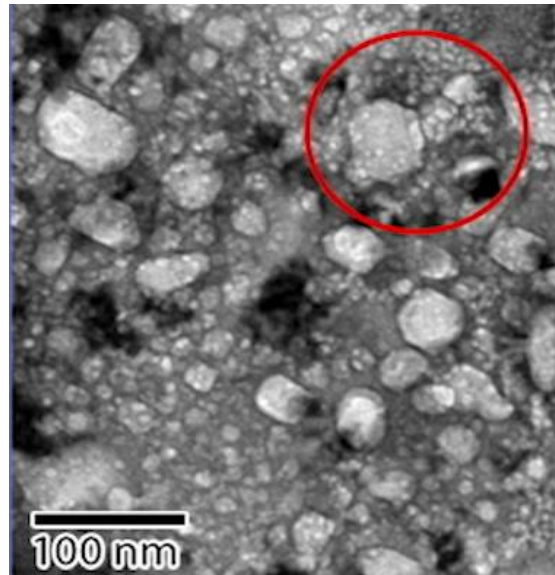
Evenly distributed cavities
over the entire grain structure

Apparent higher concentration
of cavities along grain
boundaries

Cavity Growth during In-situ Annealing of 10 keV He⁺ Implanted and then 3 MeV Irradiated Ni³⁺

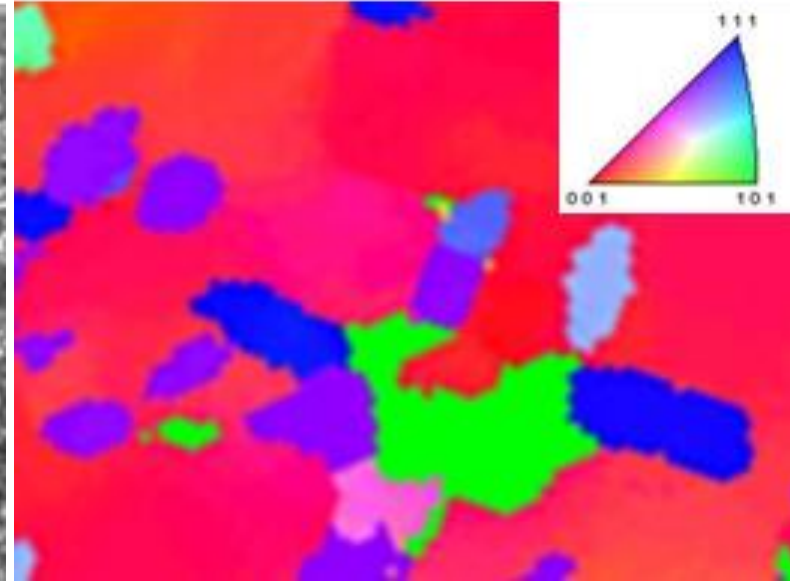
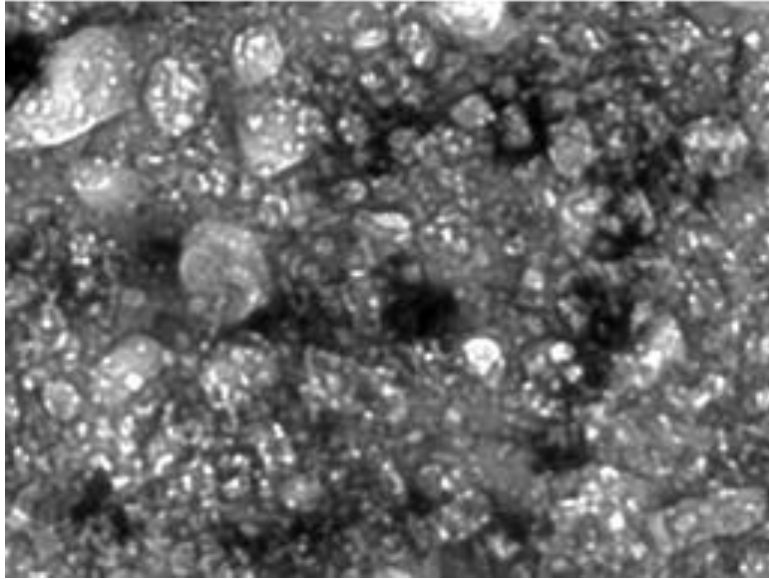


Bubble to cavity transition and cavity evolution can be directly studied

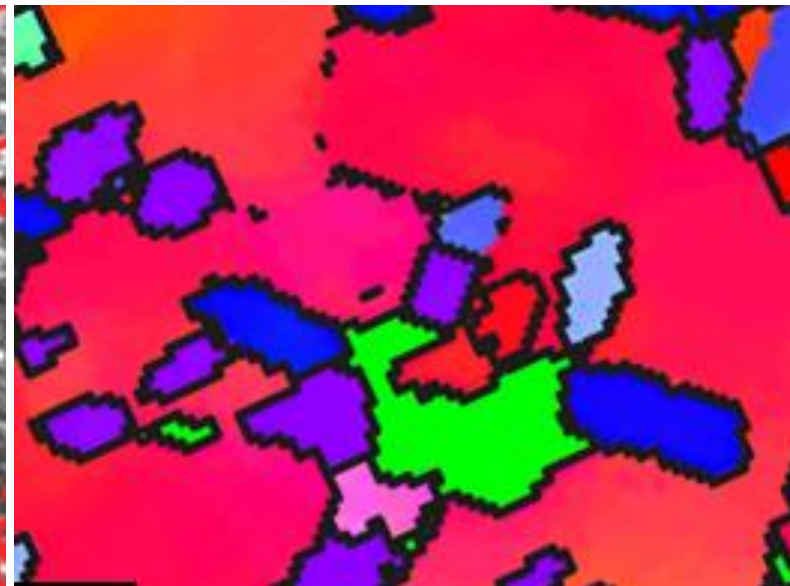
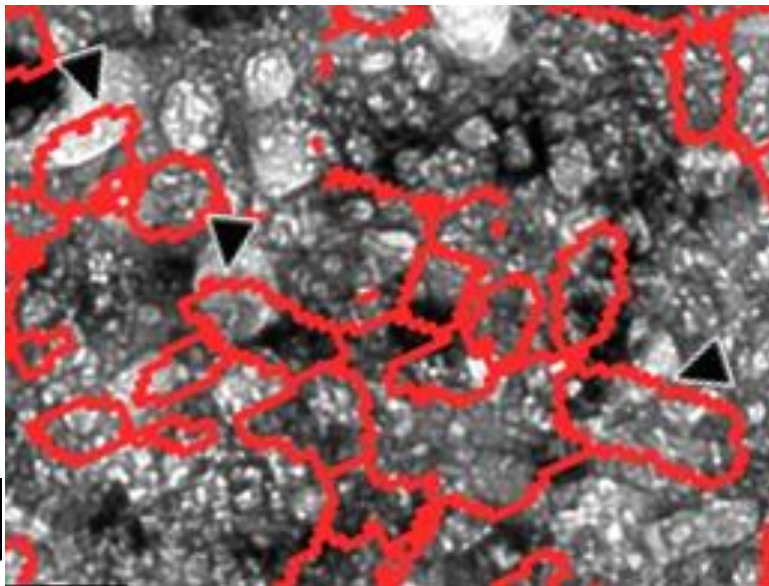


Precession Electron Diffraction Reveals Hidden Grain Structure

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



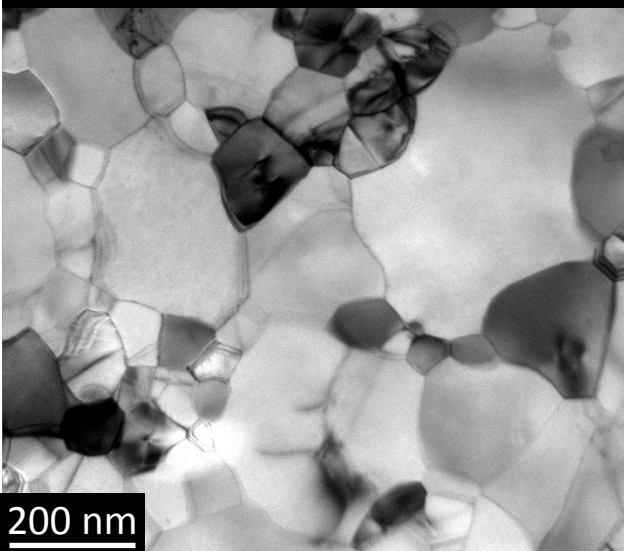
Cavities span
multiple
grains at
identified
grain
boundaries



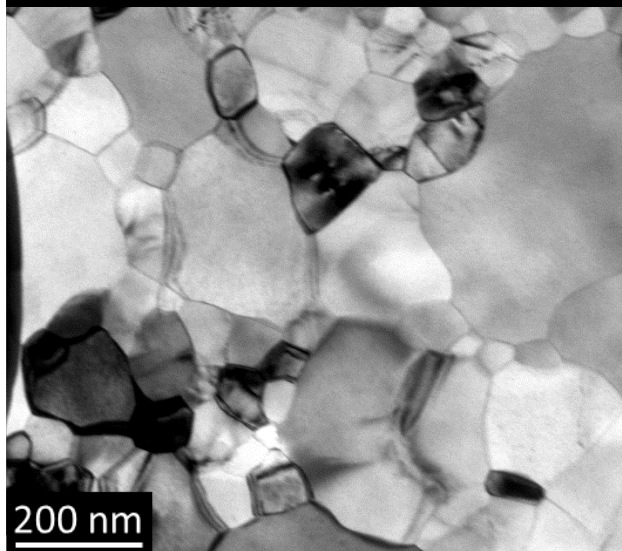
100 nm

Helium Implantation at Room Temperature

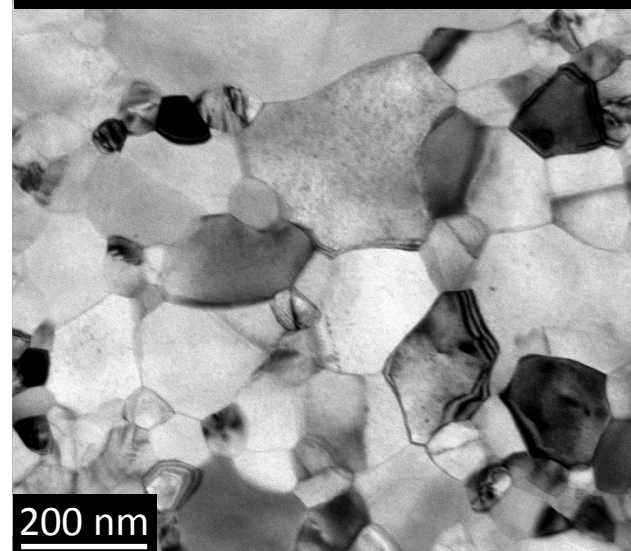
Pre-Implantation



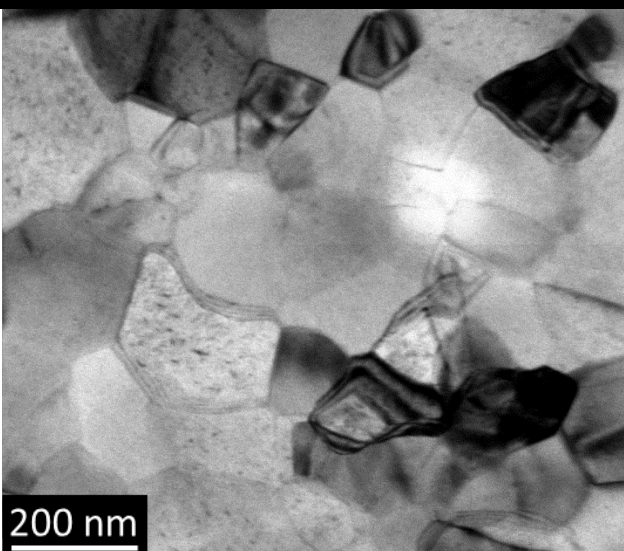
Post-Implantation, 25 °C



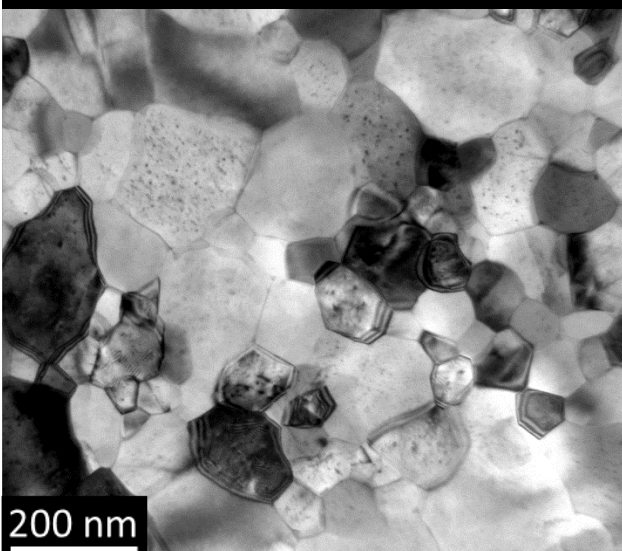
200 °C



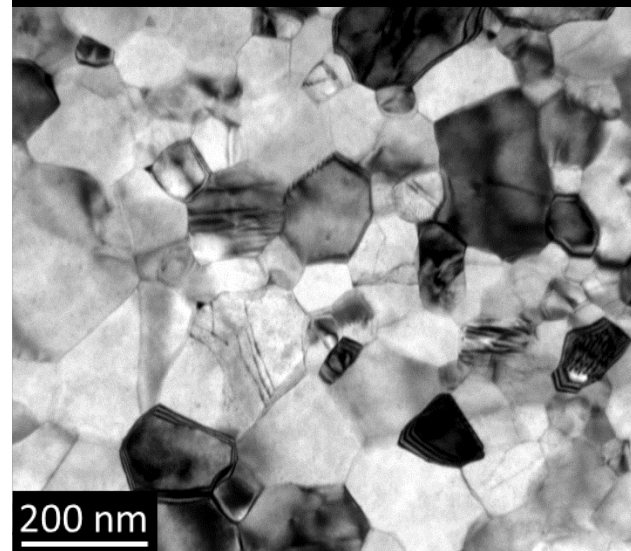
400 °C



500 °C

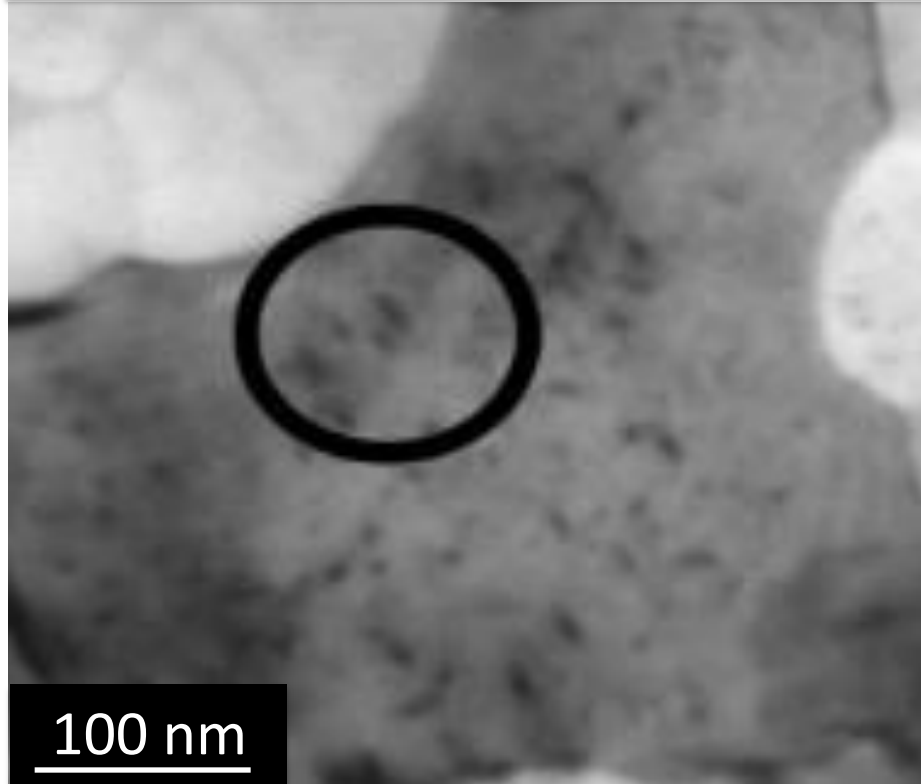


600 °C

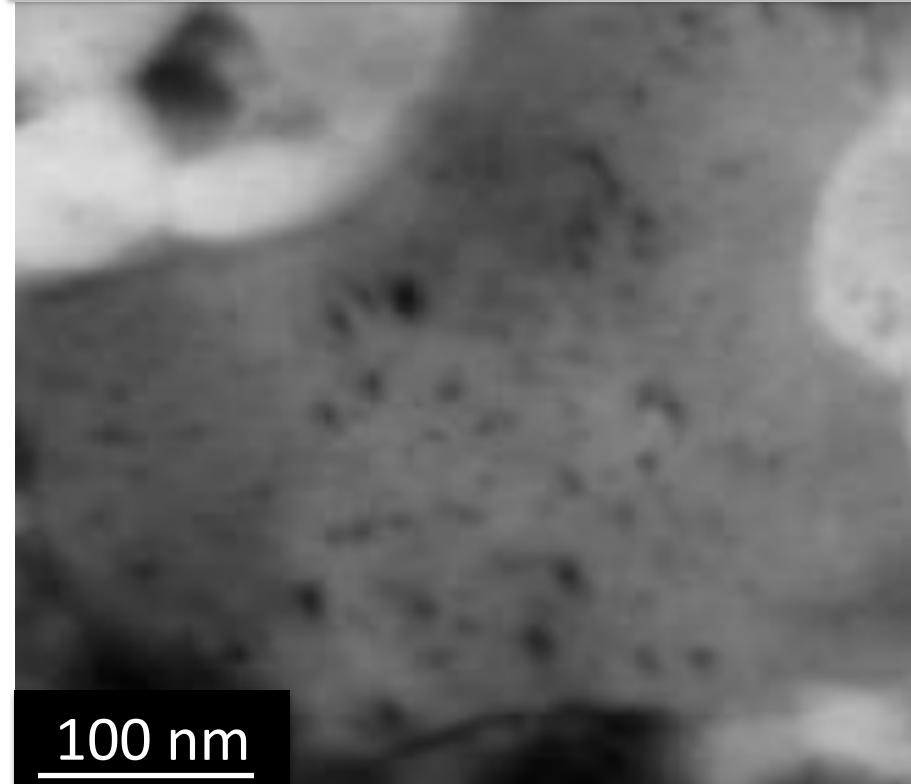


Dislocation Loop Mobility 500 °C to 600°C

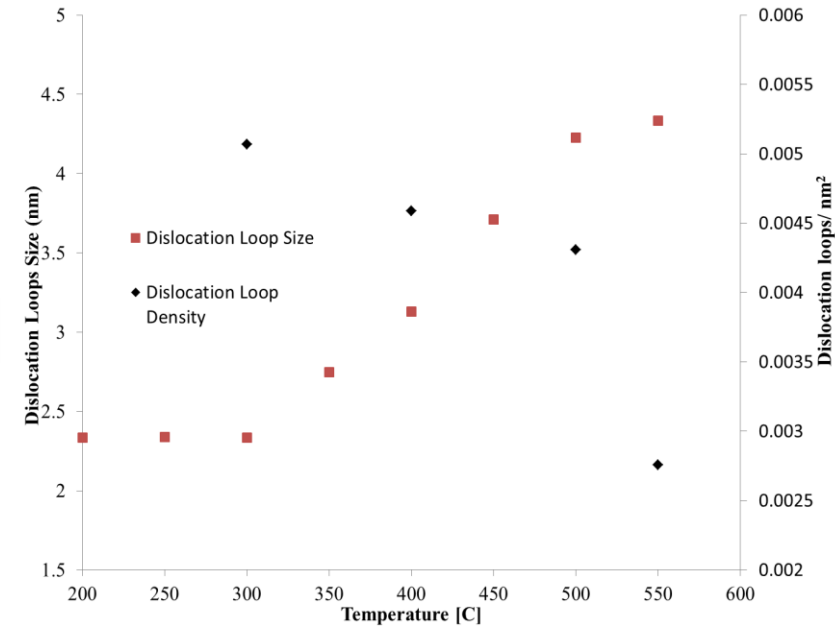
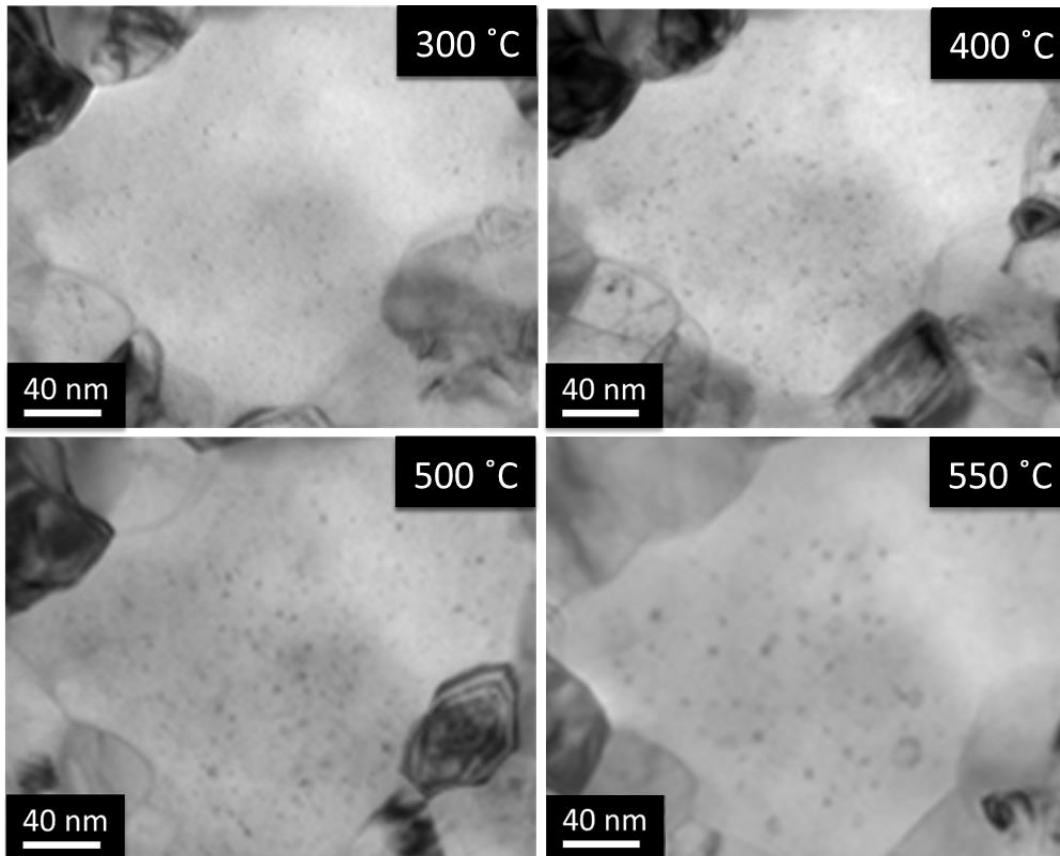
Dislocation Loop Coalescence



Dislocation Loop Migration to Surface

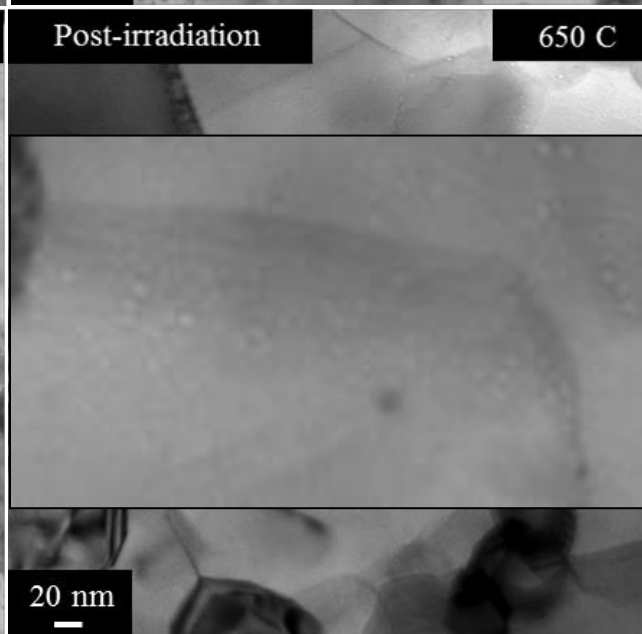
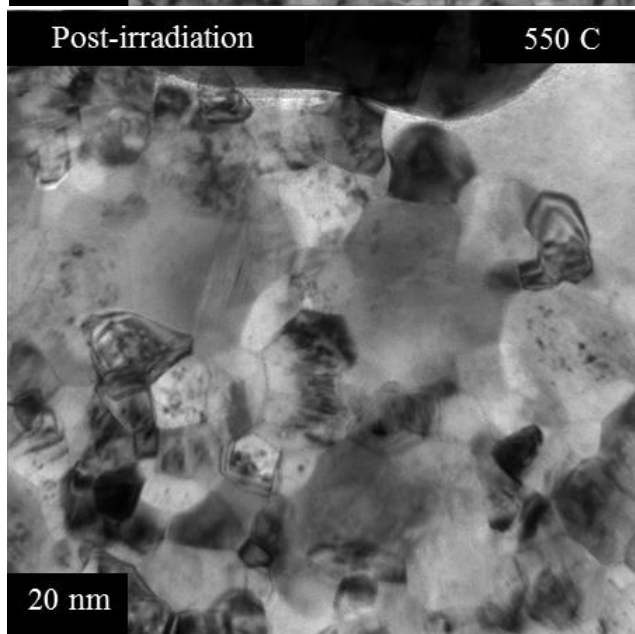
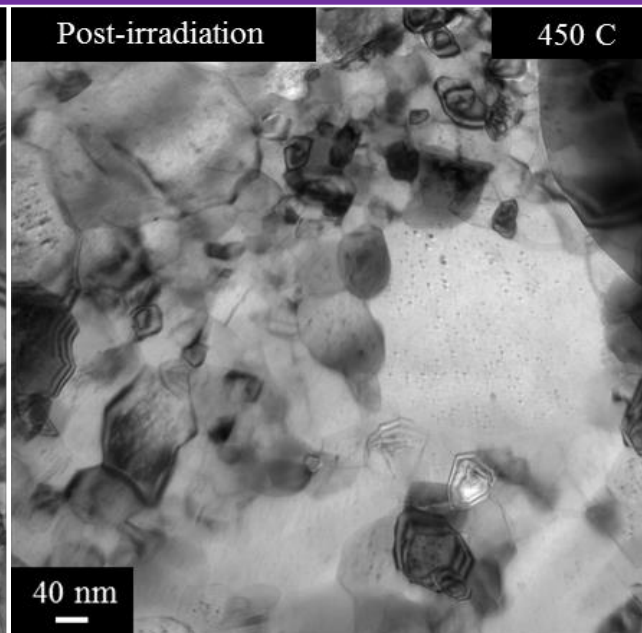
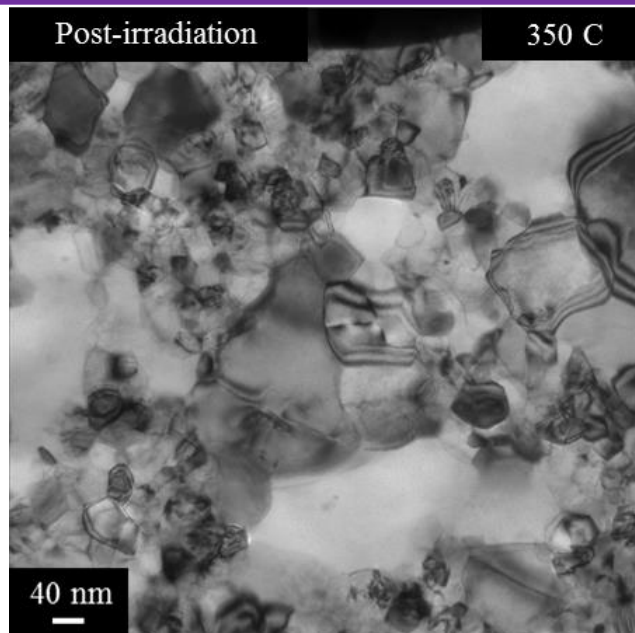


Defect Size & Density Evolution with Annealing



Defect Size Increases, Density Decreases
No visible Cavities

Elevated Temperature Helium Implantation



CINT Capabilities

Core Facility (SNL)



- TEM, SEM, FIB, XRD
- Low Temp Transport
- Scanning Probe Microscopy
- Ultra-fast Spectroscopy
- Molecular Beam Epitaxy
- Chem & Bio labs
- Molecular films
- E-beam lithography
- Photolithography
- Deposition & Etch
- Ion Irradiation

Gateway Facility (LANL)



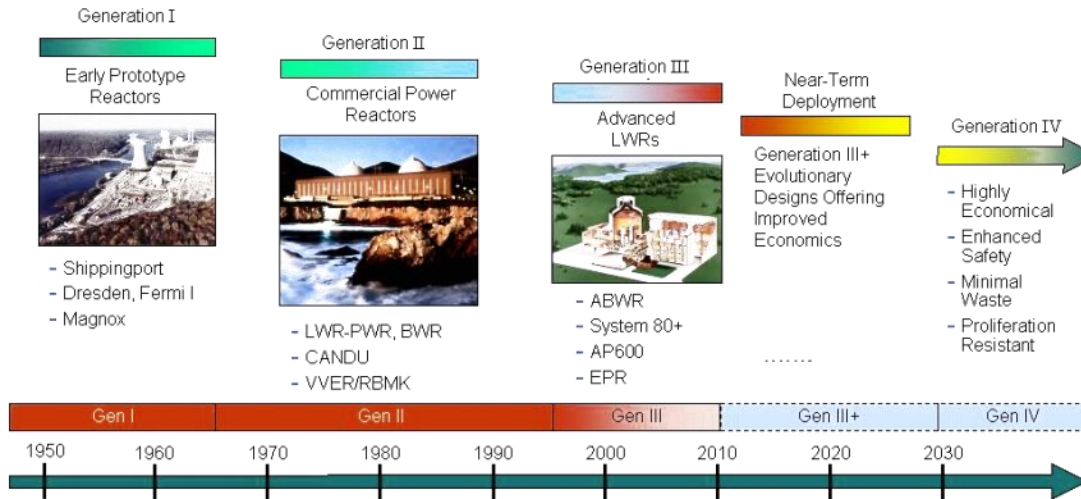
- Biomaterials & Chem synthesis
- XRD, SEM
- UV-vis, ellipsometry
- Nano-indentation
- Nanoscale optical probes
- Microscopies
- Physical Synthesis
- Pulsed Laser Deposition
- Ultra-fast Spectroscopy
- Computer Cluster
- Visualization Lab

Many instruments/techniques in this talk available for use through CINT's competitive proposal-based system.

Summary

Radiation Effects

Generation IV: Nuclear Energy Systems Deployable no later than 2030 and offering significant advances in sustainability, safety and reliability, and economics



EPR reactor design (Gen III+)

- Generation II reactor lifetime extensions: material aging
- Generation IV structural materials: safer, more radiation resistant
- Fusion materials: very high dose, helium bombardment



ITER (fusion)

CINT: a DOE Office of Science National User Facility

“A DOE/SC user facility has **unique world-class research capabilities and technologies** which are **available broadly to science community** worldwide from universities, industry, private laboratories, and other Federal laboratories for work that will be **published in the open literature.**”



The DOE/SC nanoscience centers are different from traditional user facilities

- Defined by a scientific field, not specific instrumentation.
- NSRC staff support user projects and conduct original research.
- Capabilities involve hardware plus research expertise.

