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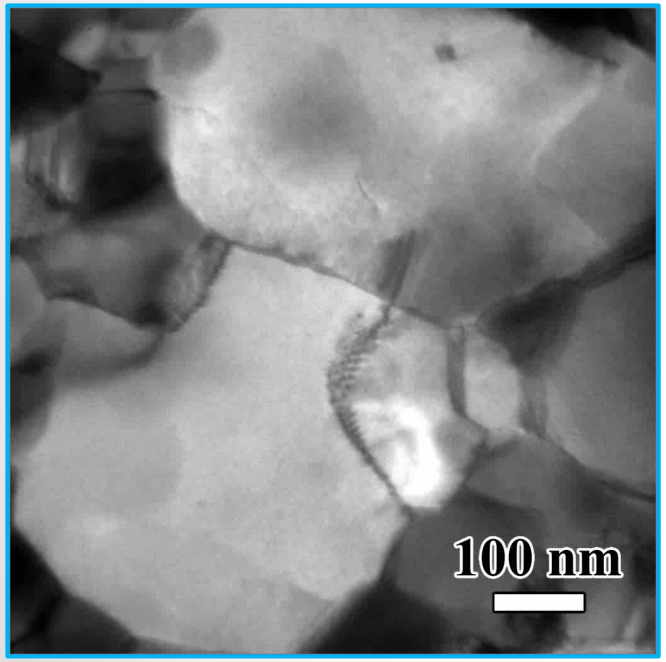
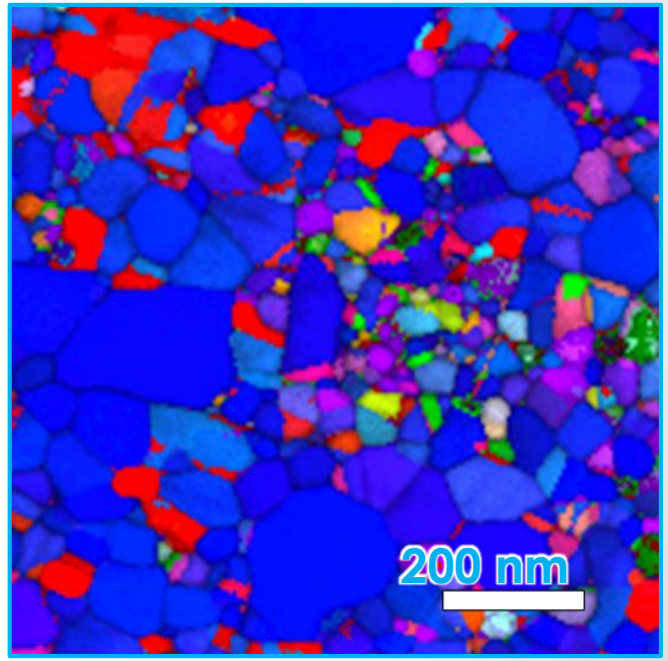
SAND2016-10013C

# Directly Observing Structural Evolution during Sequential and Concurrent Helium Implantation and Heavy Ion Irradiation




Daniel Bufford, Claire Chisholm,
Brittany Muntifering, and Khalid Hattar

Sandia National Laboratories
Albuquerque, NM, USA




U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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# Nuclear Reactors and TPBARs

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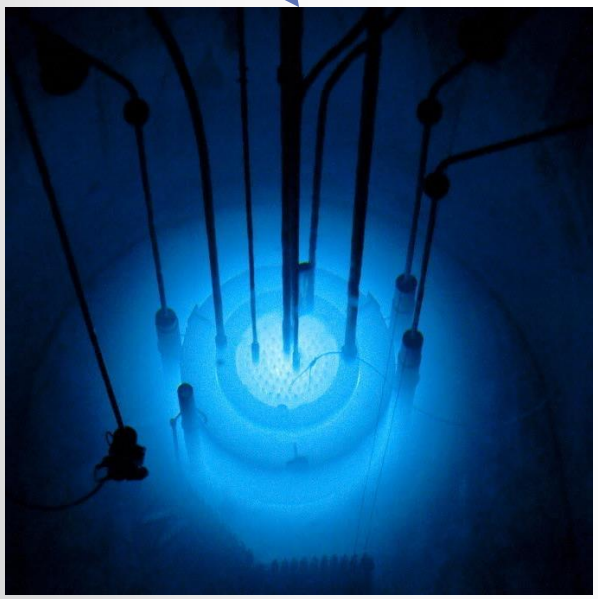
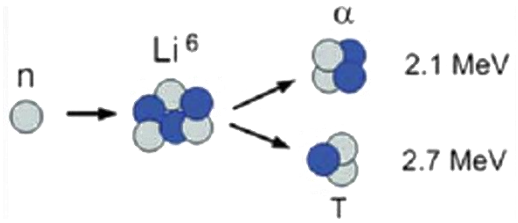
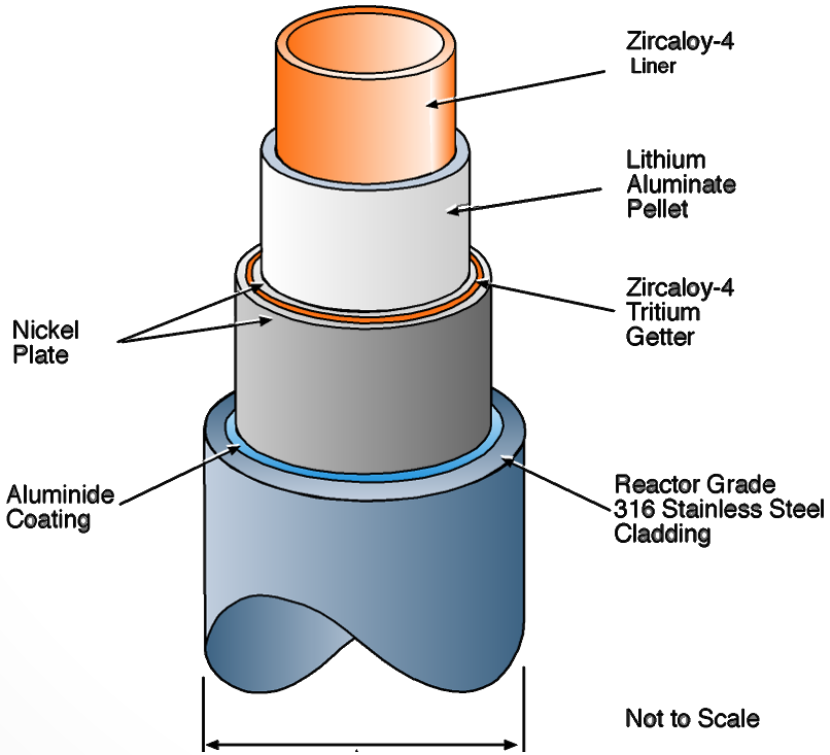
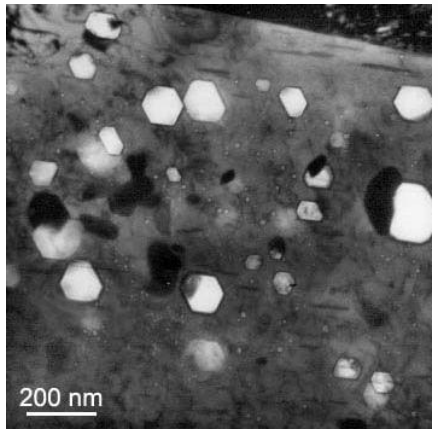
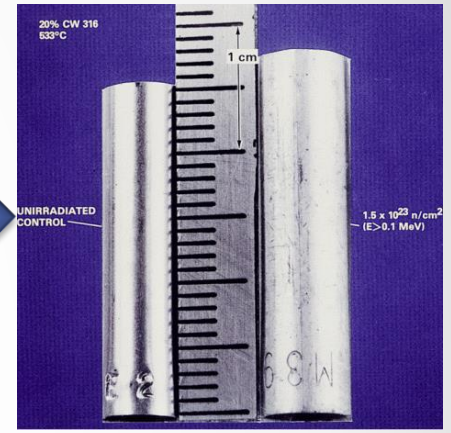
Tritium Producing Burnable Absorber Rod: device that uses neutron flux from a nuclear reactor to produce tritium.






**Tough environment for materials!**

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

**Need to understand the individual factors, and their interactions.**

# Ion beams can simulate radiation conditions to accelerate materials research and development

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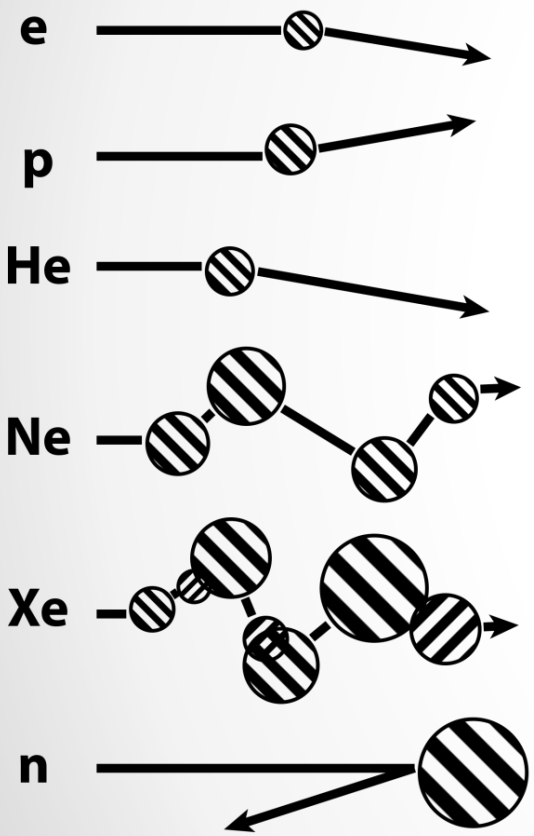
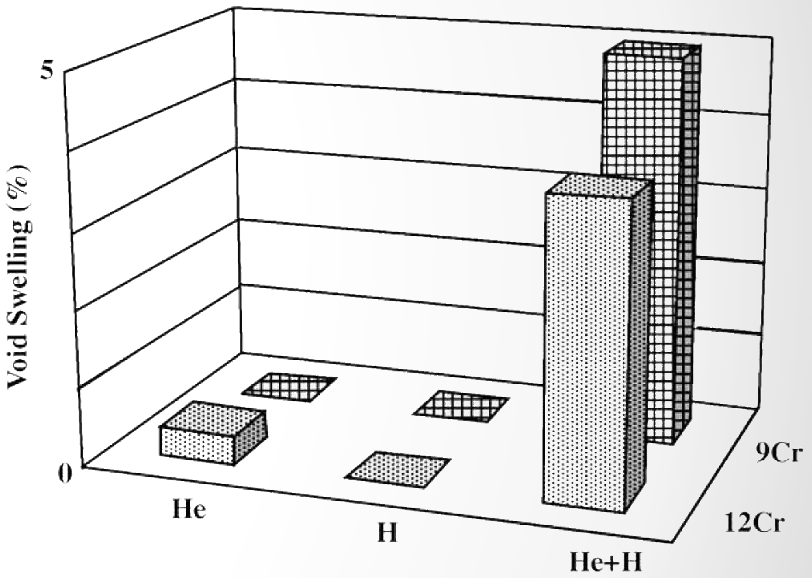



| | Neutrons | Ions |
|---|---|---|
| Radiation Conditions | Real | Simulated |
| Experiment times | Months to years | Hours to days |
| Residual radioactivity? | Yes: necessitates special material handling. | Typically none, or short term activation |
| Depths reached | Often orders of magnitude deeper than ions | Up to tens of μm |



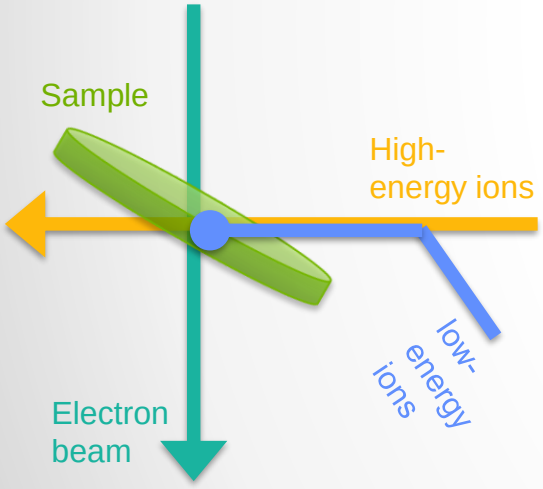

T. Tanaka, et al., J Nucl Mater 2004.

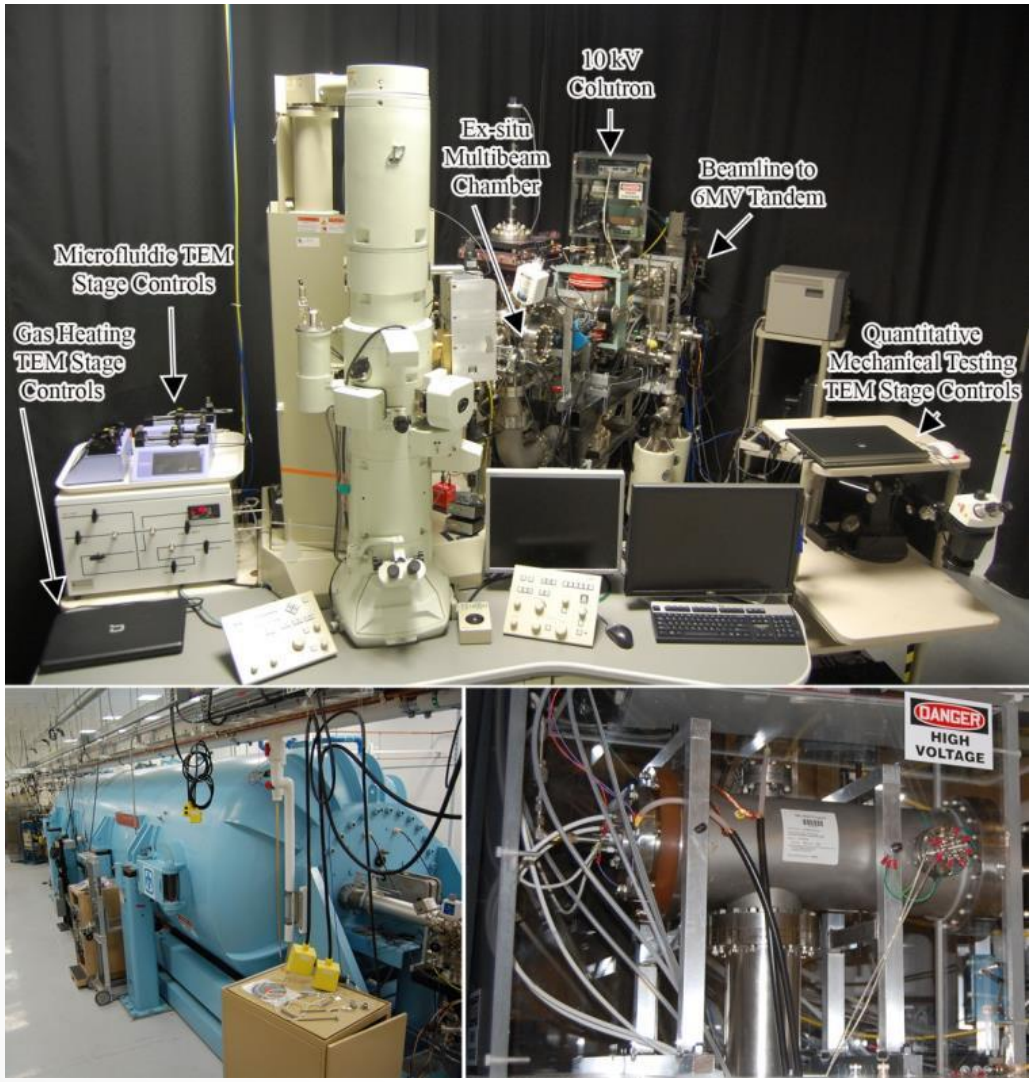
**Multiple beam concurrent irradiation produces effects not seen with sequential irradiation, however the difficulty of performing triple-beam irradiation has resulted in a limited number of facilities world wide.**

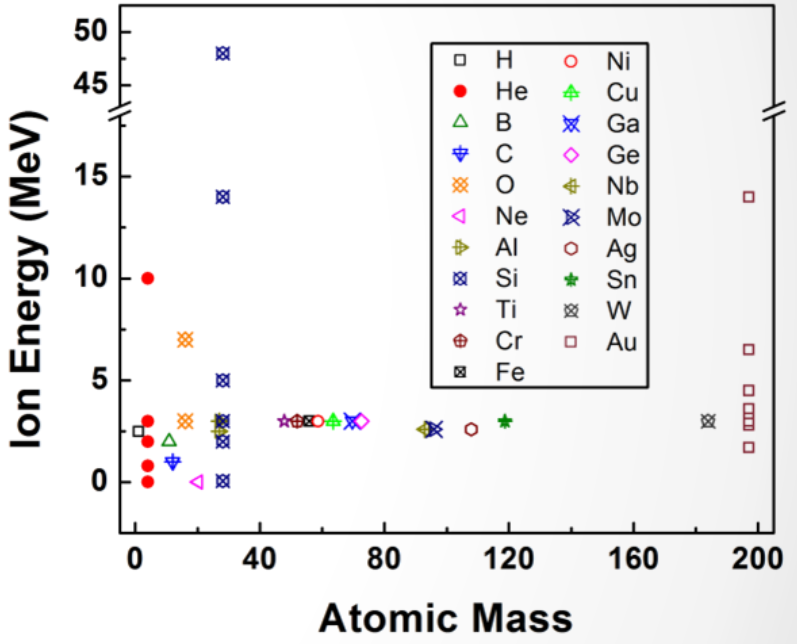
# Sandia's Concurrent *In situ* Ion Irradiation TEM ($I^3$TEM) Facility

Collaborators: D.L. Buller, K. Hattar, and J. Scott

- 10 kV Colutron
- 200 kV TEM
- 6 MV Tandem




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

# Displacement Damage *In Situ*

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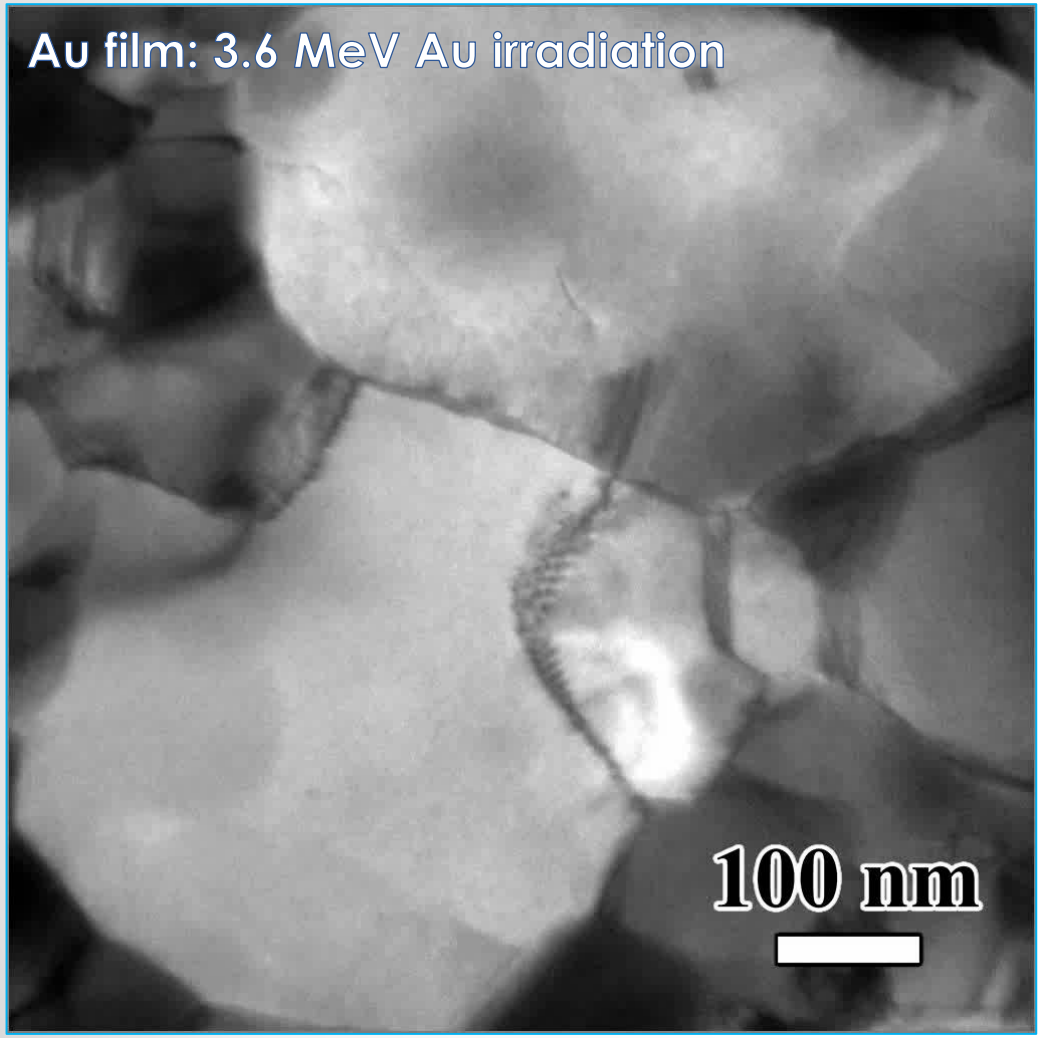
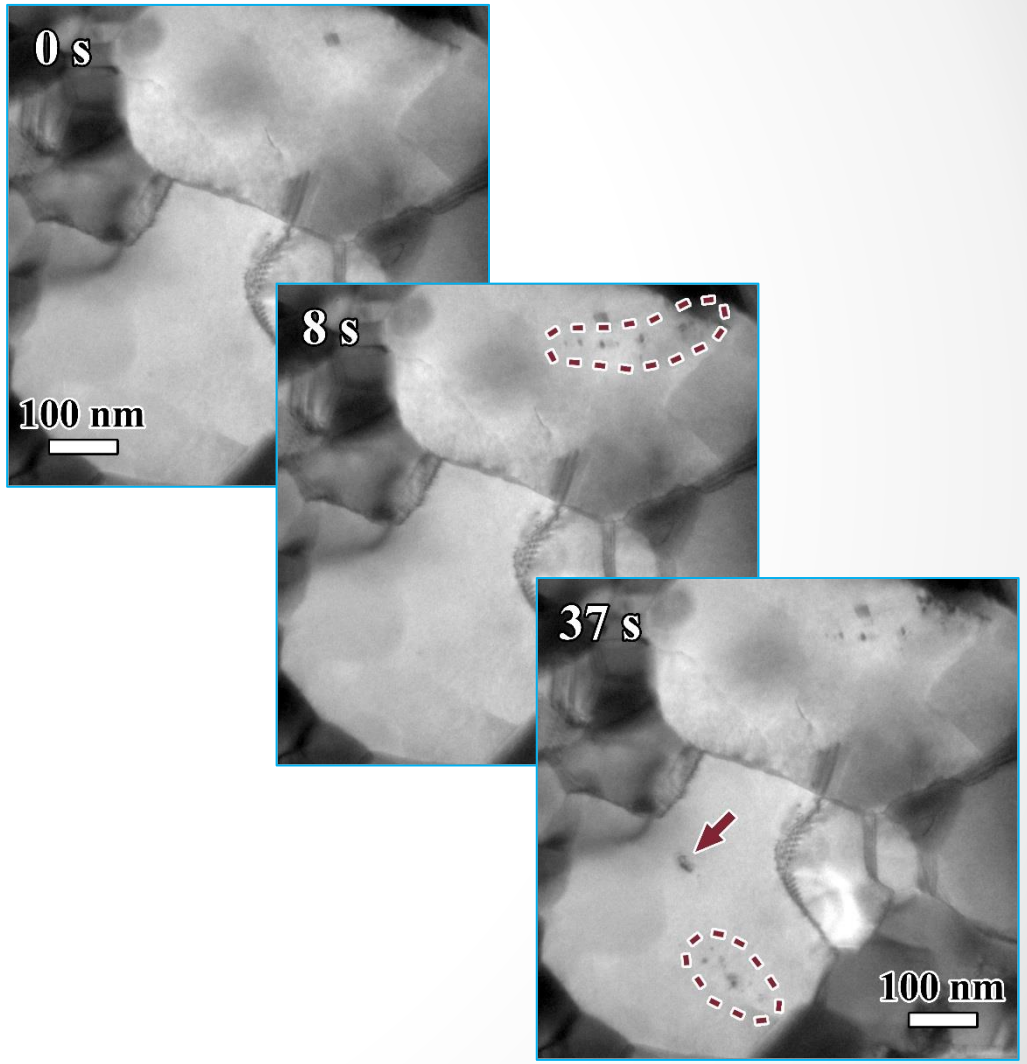
Video playback speed x5.



# Helium Implantation *In situ*

Collaborators: C. Chisholm and A. Minor

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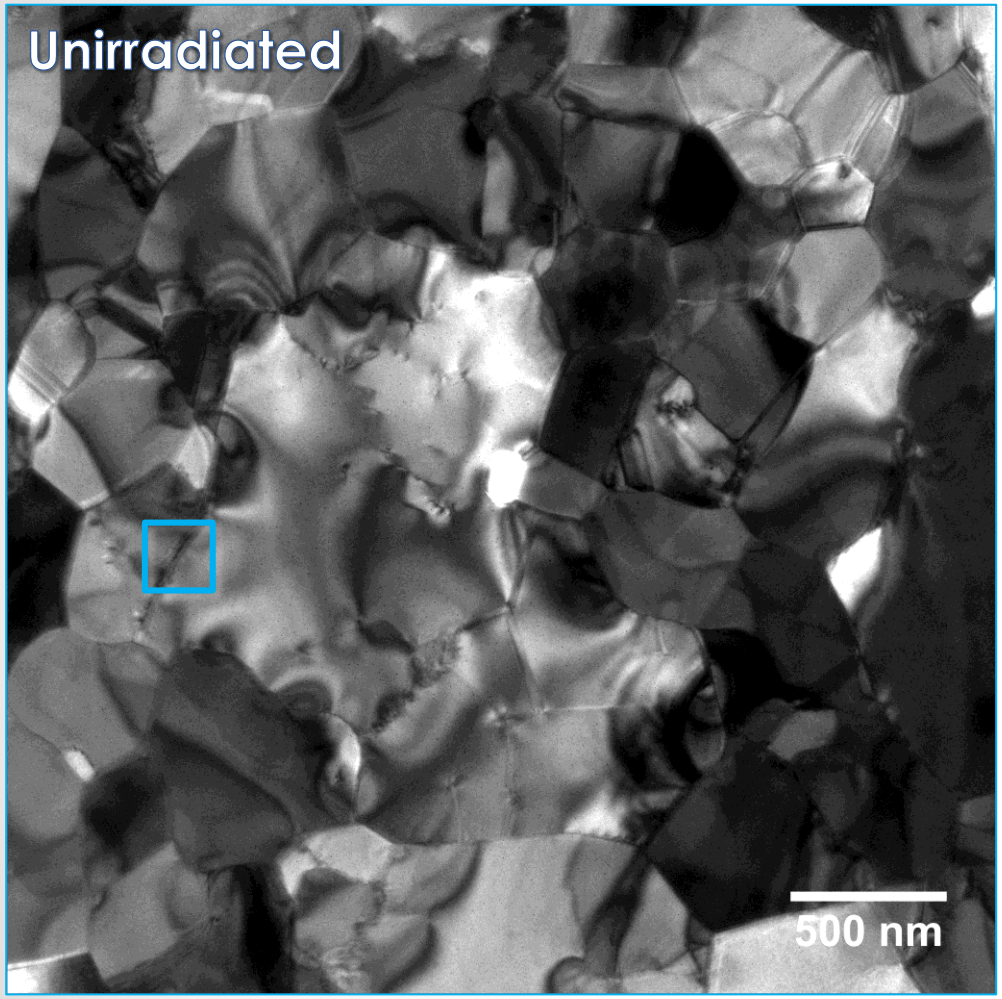
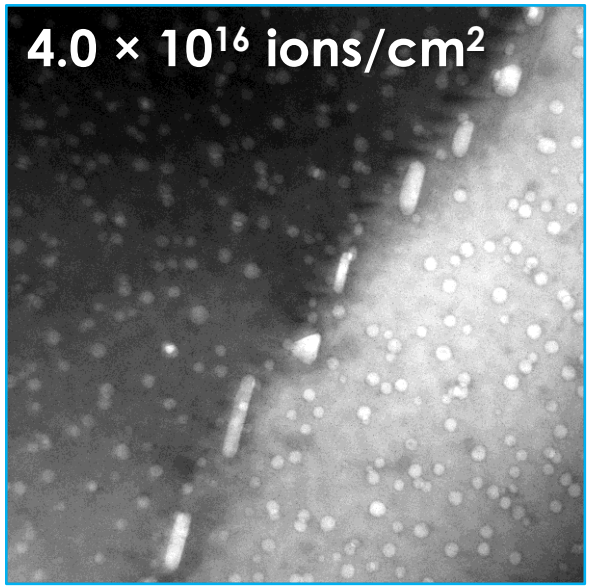
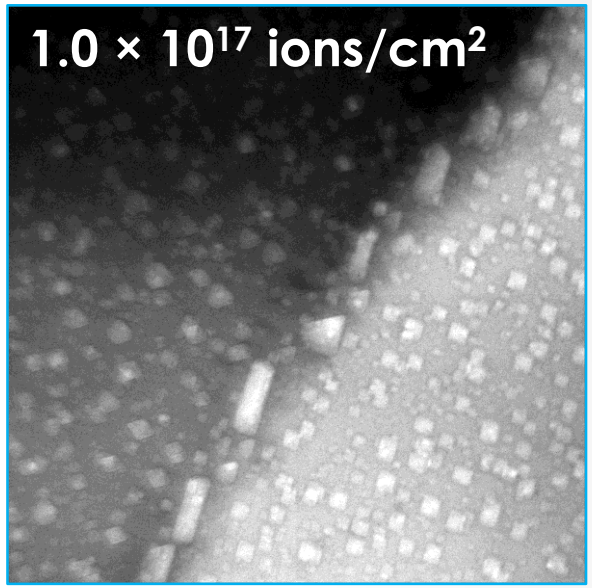
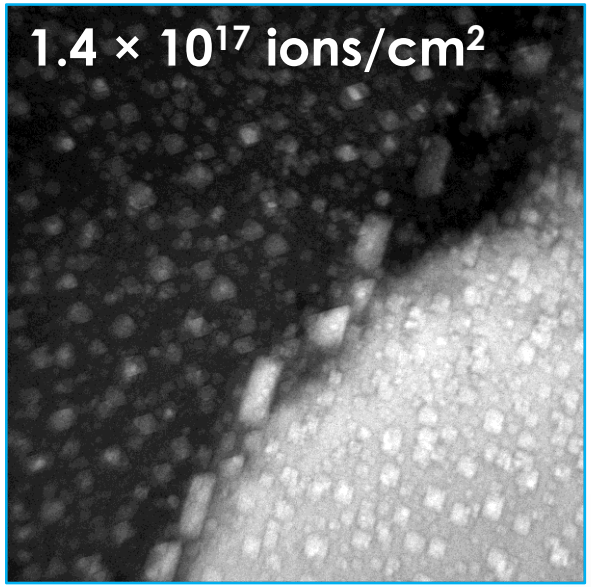
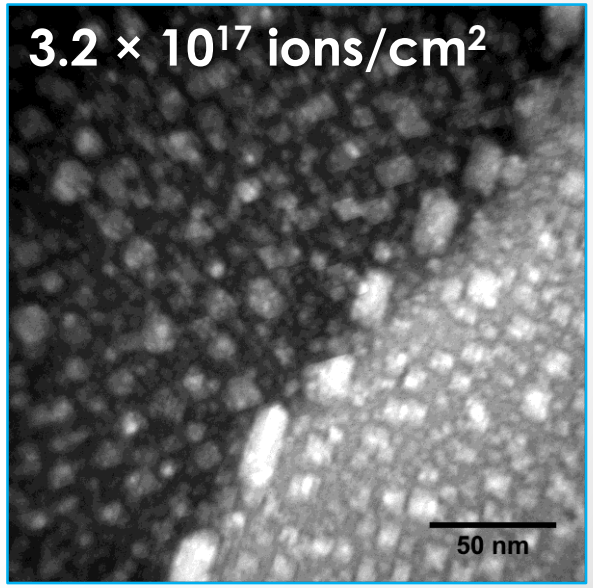


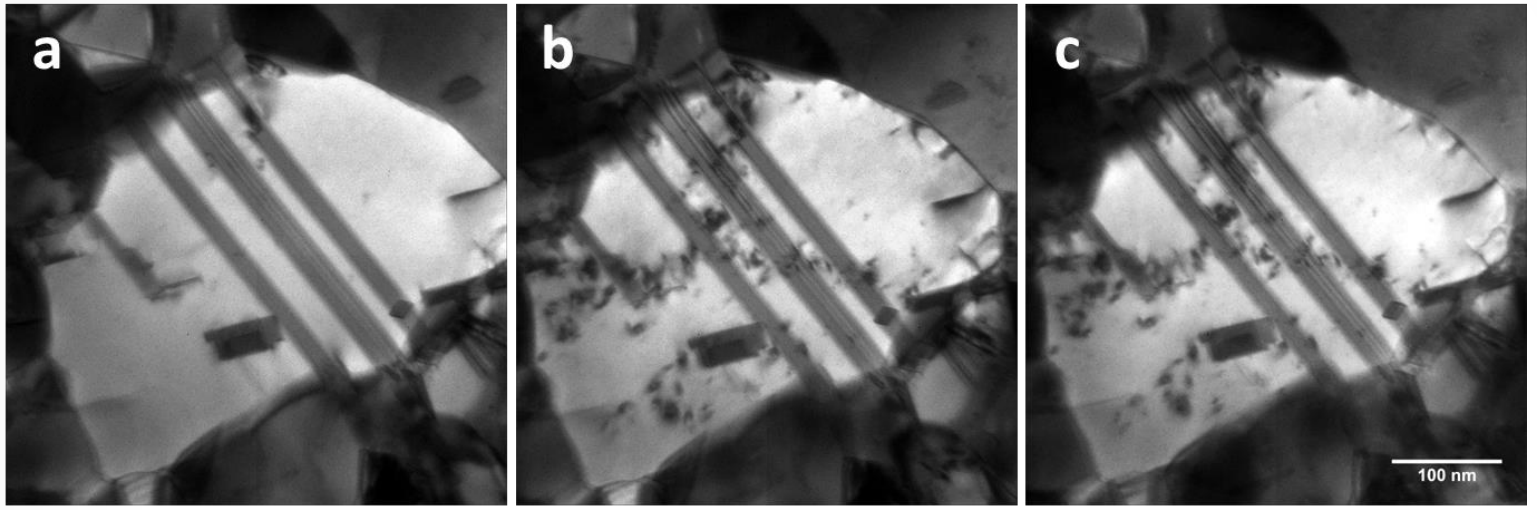




**What happens when the radiation conditions are combined?**

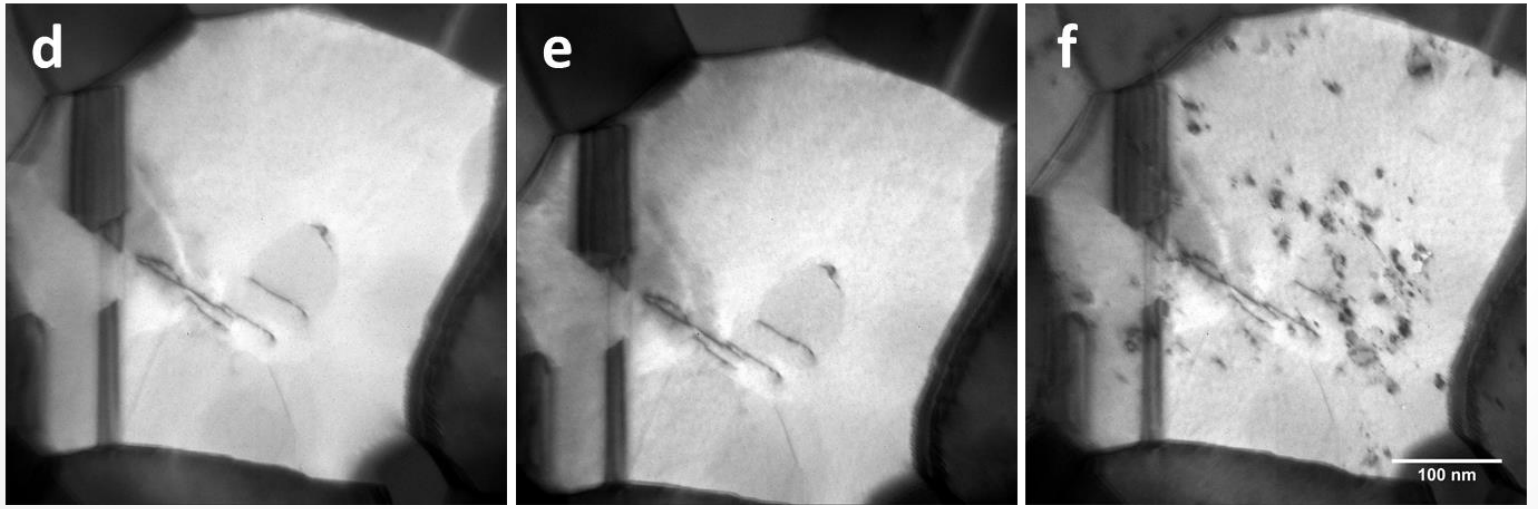
# Sequential Implantation & Irradiation

Collaborators: C. Chisholm and A. Minor

2.8 MeV Au then 10 keV He




10 keV He then 2.8 MeV Au

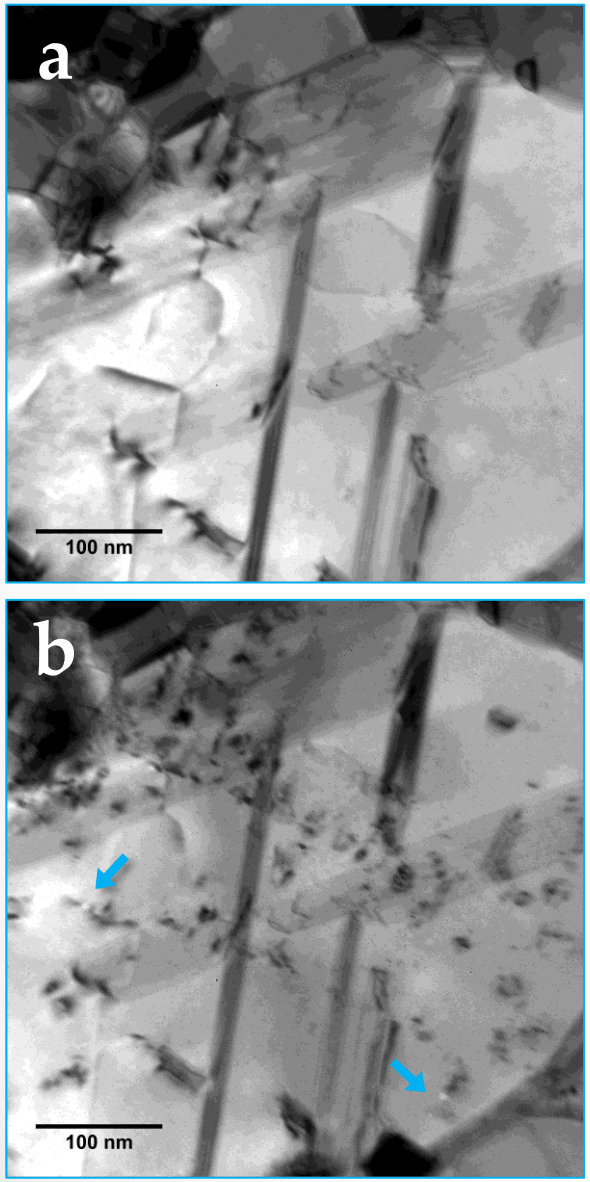


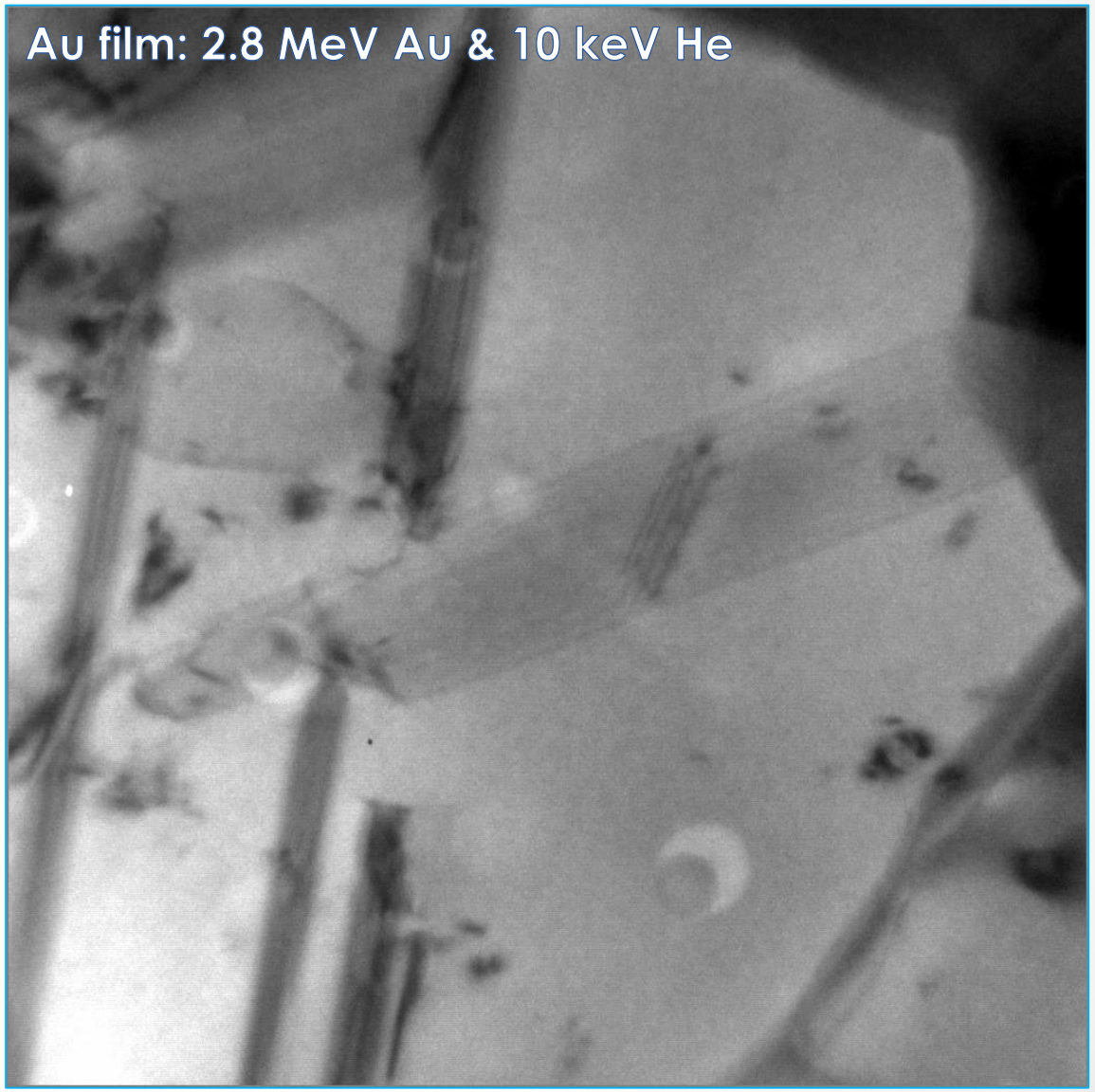

- Equal fluences:
  - Au $3 \times 10^{10}$ ions/cm$^2$
  - He $2 \times 10^{15}$ ions/cm$^2$

**No noticeable differences between these sequential irradiation conditions.**

# Concurrent Implantation & Irradiation *In Situ*

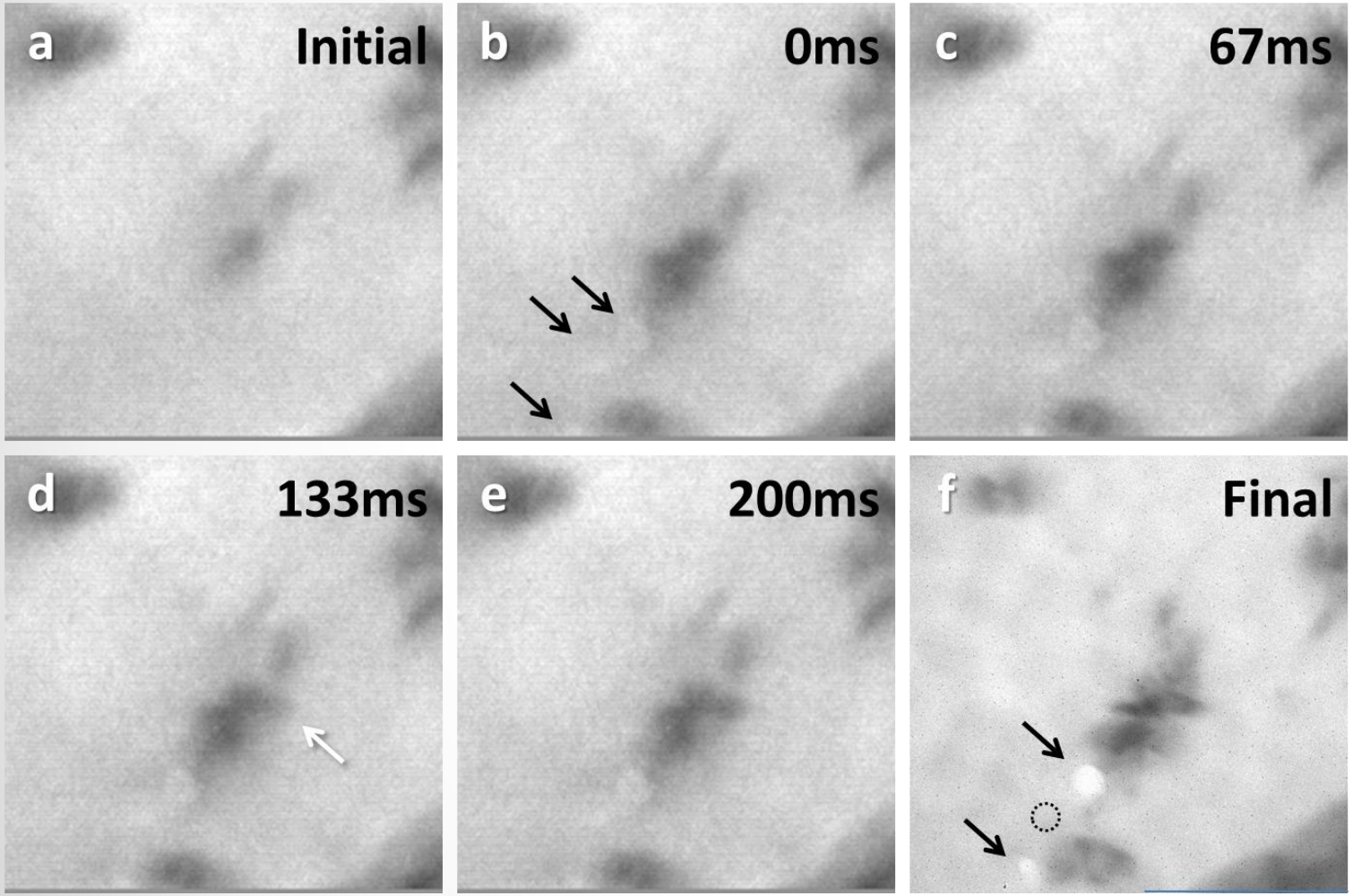
Collaborators: C. Chisholm and A. Minor



# Single Ion Strikes during Concurrent Irradiation: Direct Cavity Nucleation

Collaborators: C. Chisholm and A. Minor




a) Initial microstructure

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

d) Cascade damage still evolving

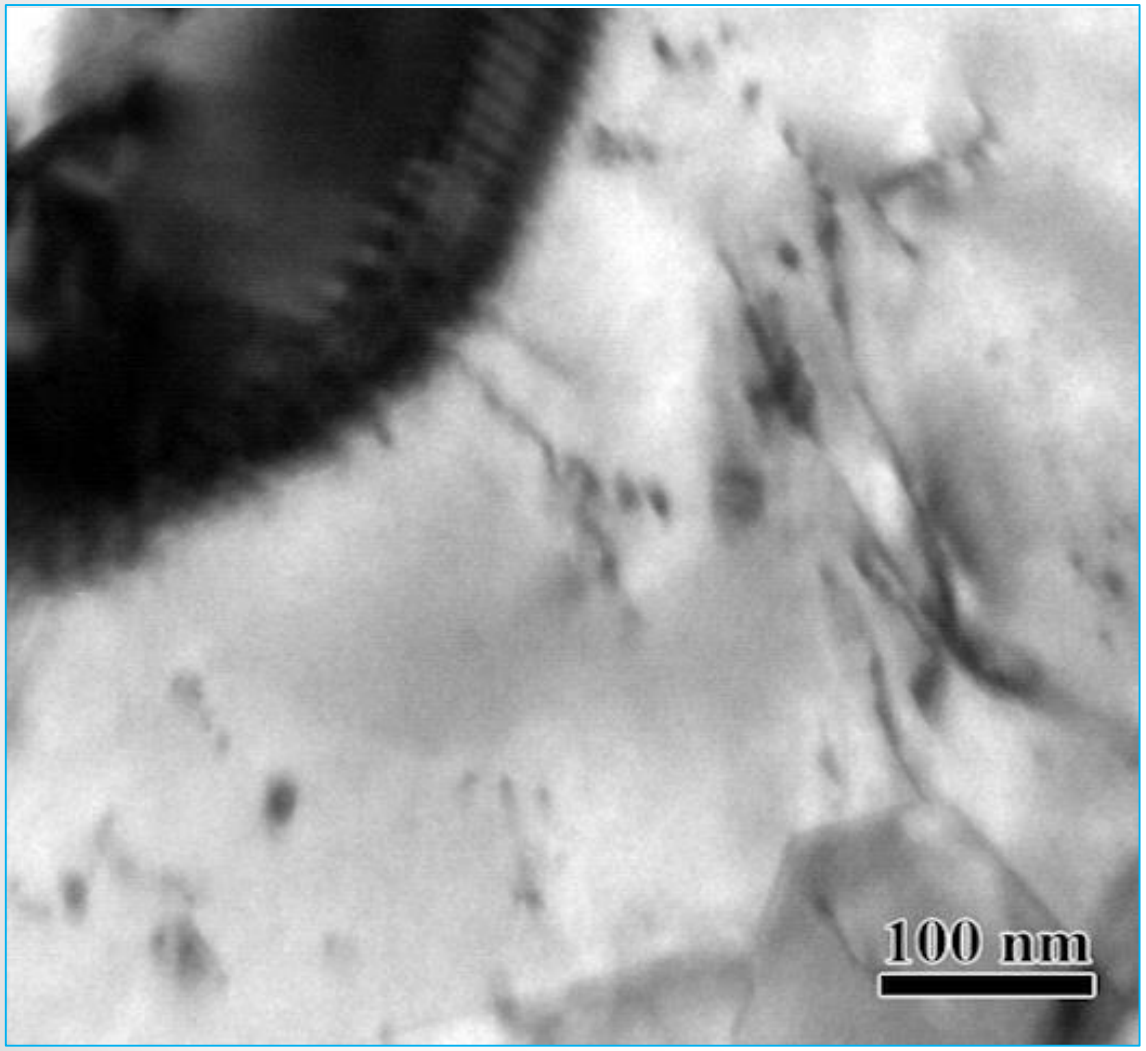
e) Apparent stability

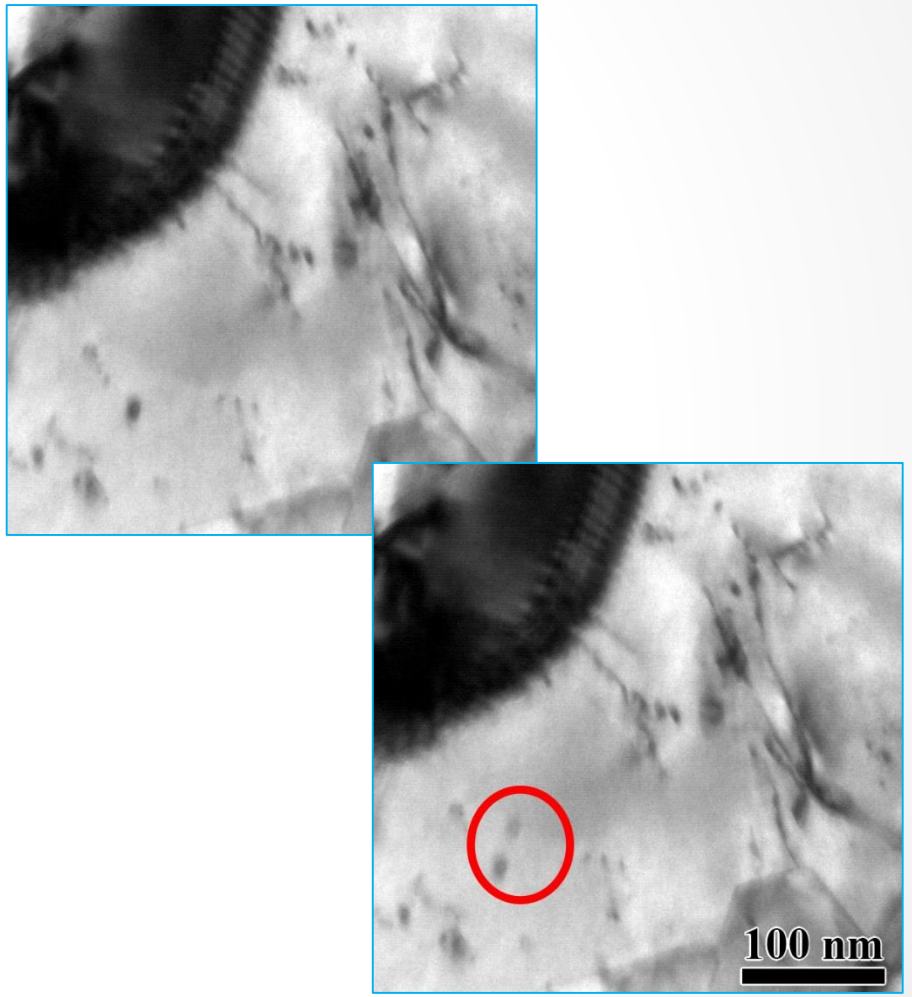
f) Final microstructure: Only two remaining cavities

**Direct cavity nucleation process seen with concurrent irradiation that was not seen with sequential beams!**

# Triple beam irradiation: Au and $He^+/D_2^+$

Video playback speed in real time.



# Triple beam irradiation: Au and $He^+/D_2^+$

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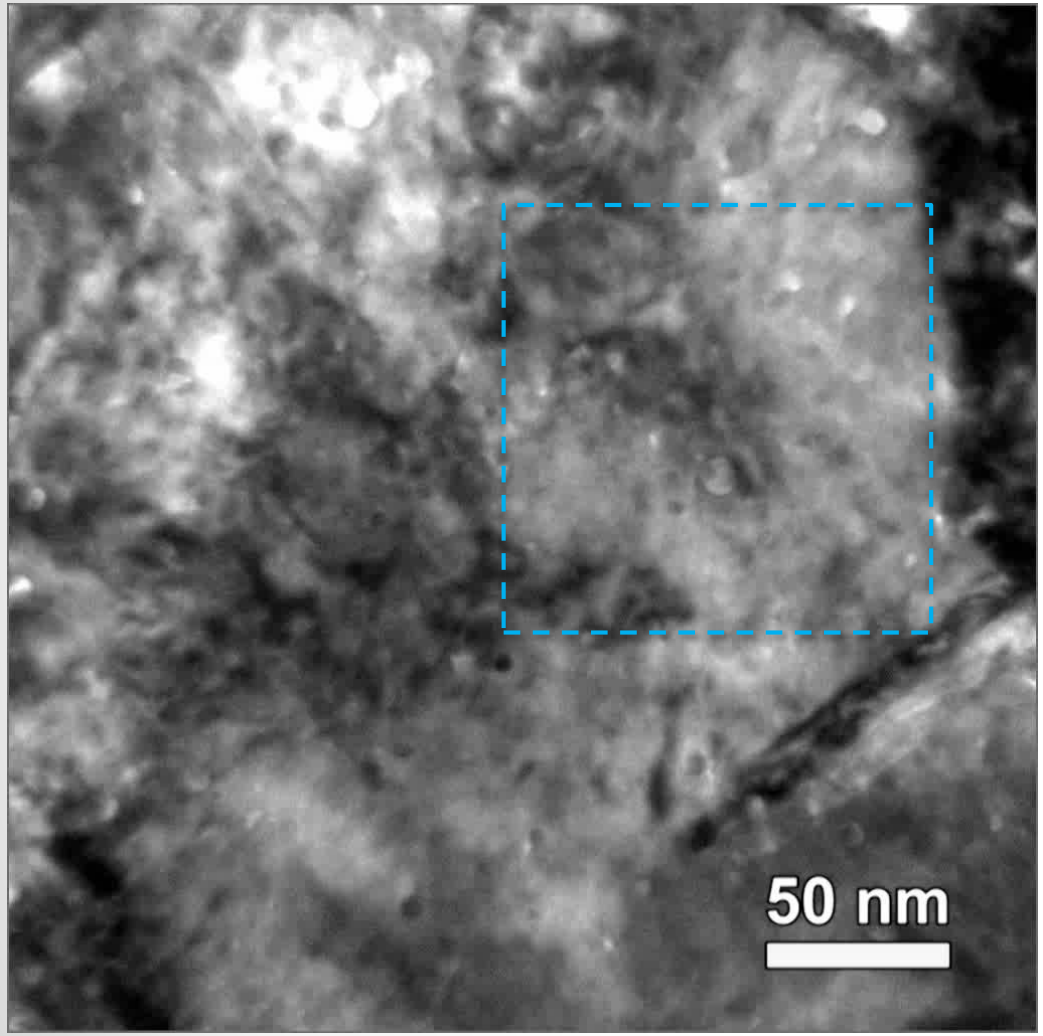
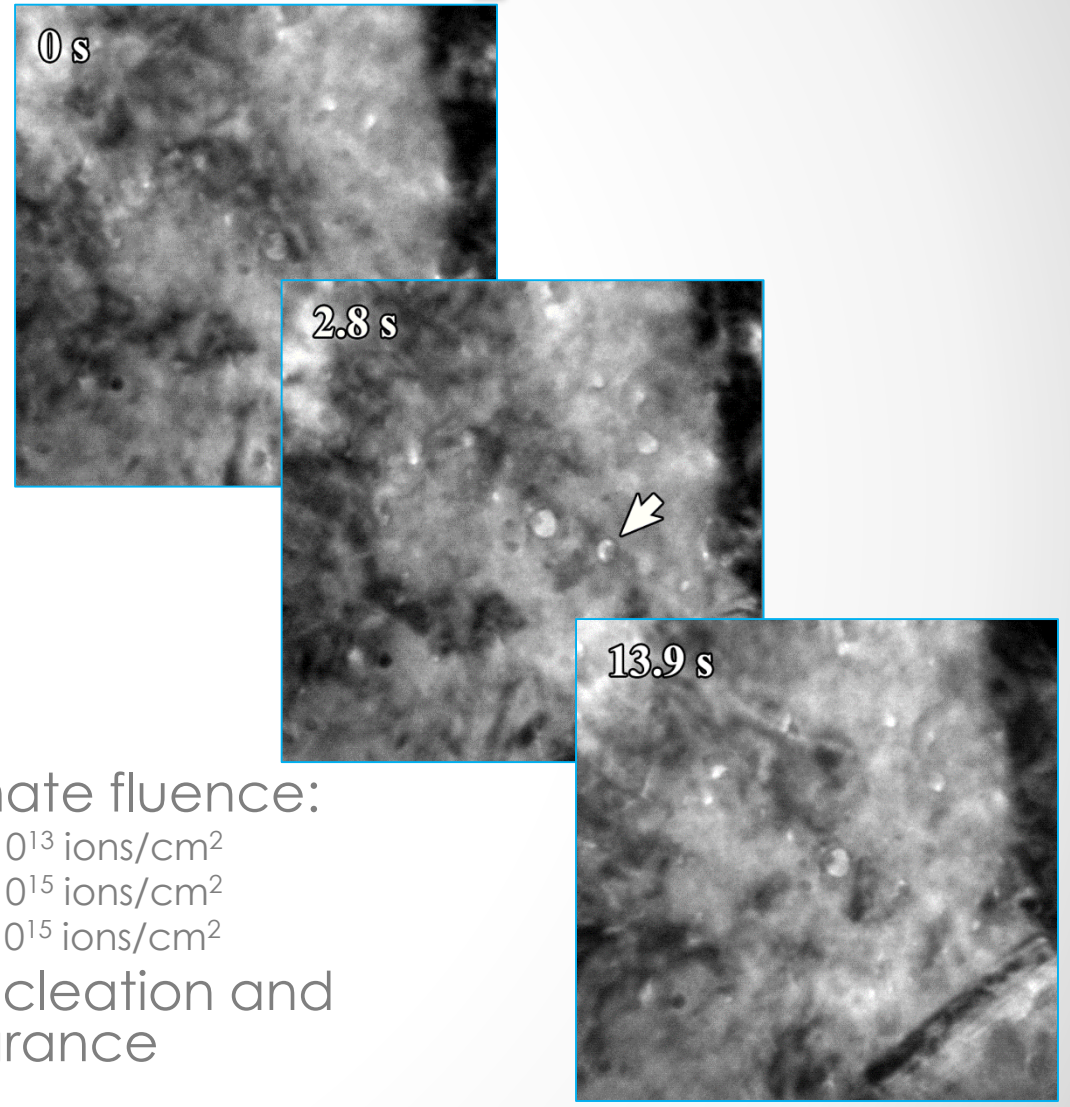
Video playback speed x1.5.




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

**Lots of activity: still working to understand this behavior**

# Irradiation / Implantation Sequence Effect on Cavity Structure

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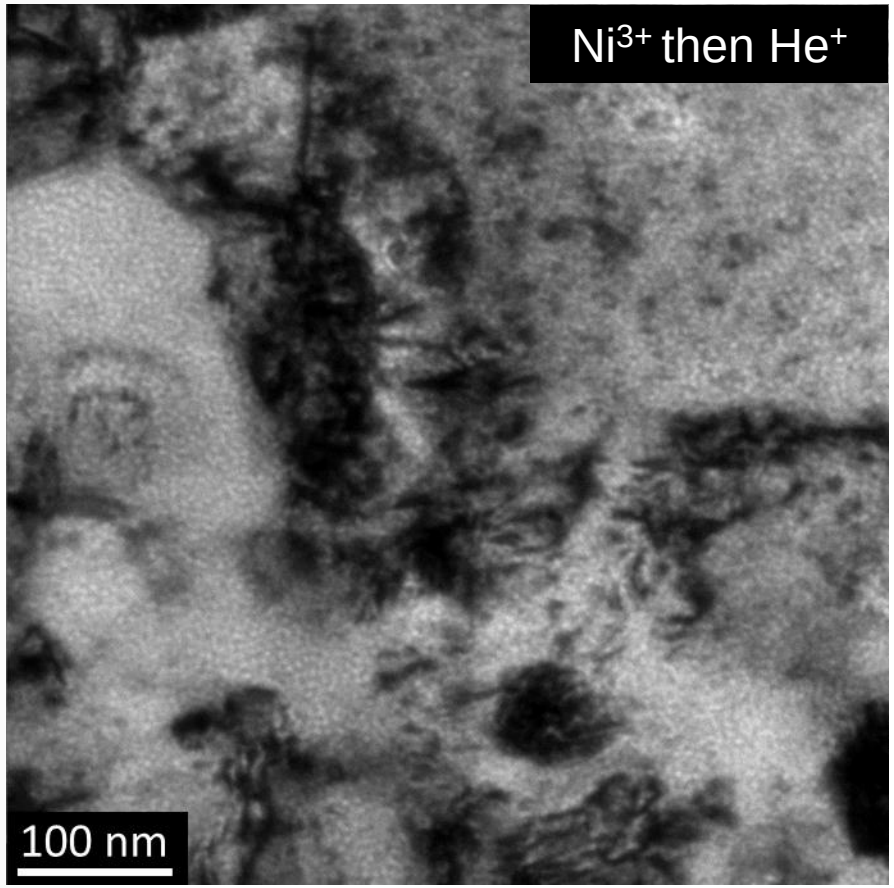
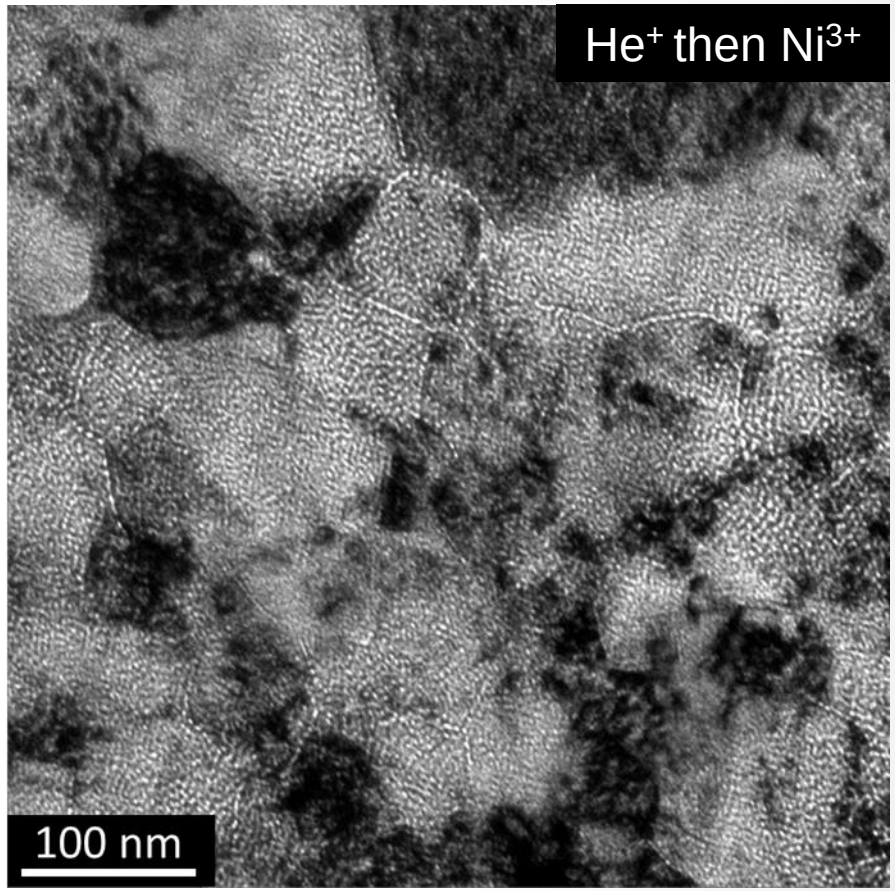
- 10 keV He
- 3 MeV Ni

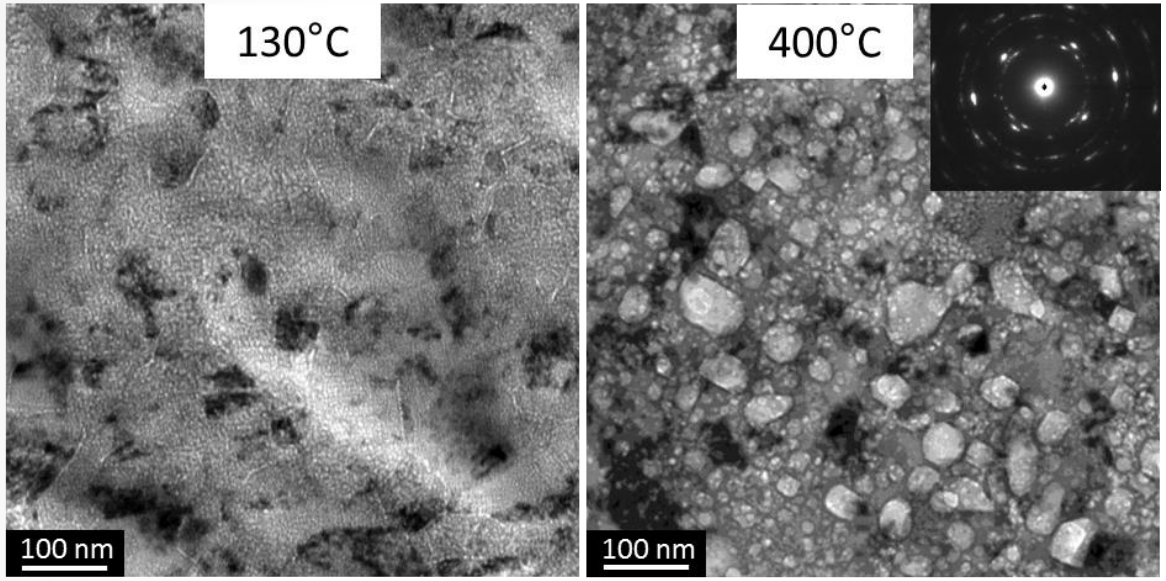


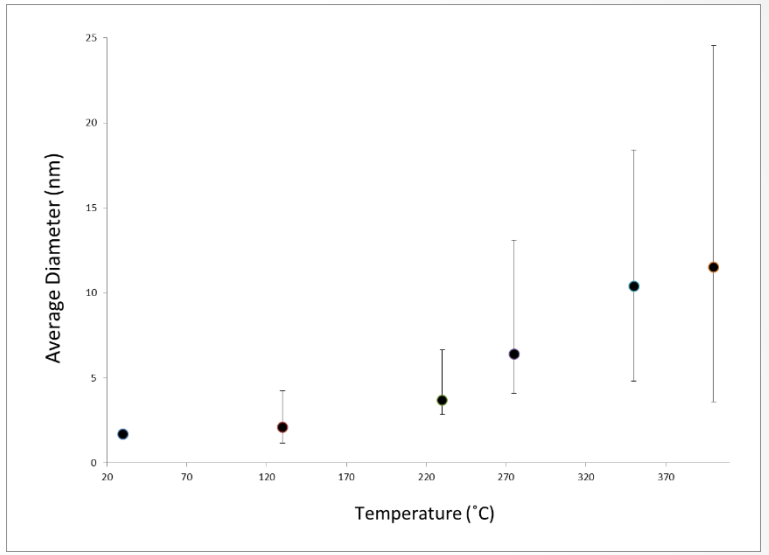

Evenly distributed cavities over the entire grain structure




Apparent higher concentration of cavities along grain boundaries

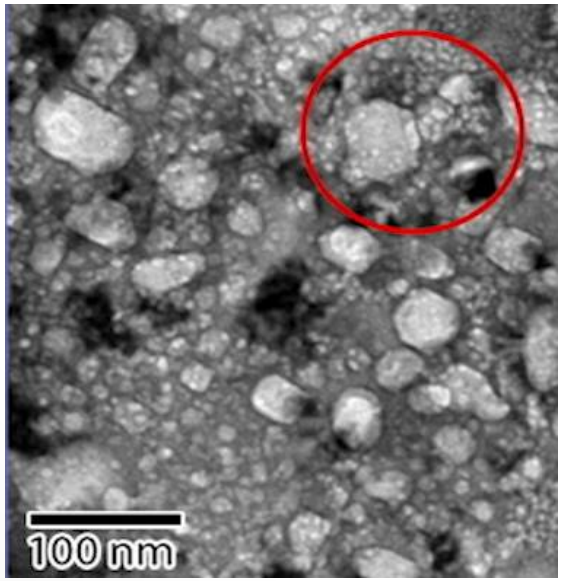
# *In Situ* Annealing of Sequentially Irradiated Ni: Cavity Growth

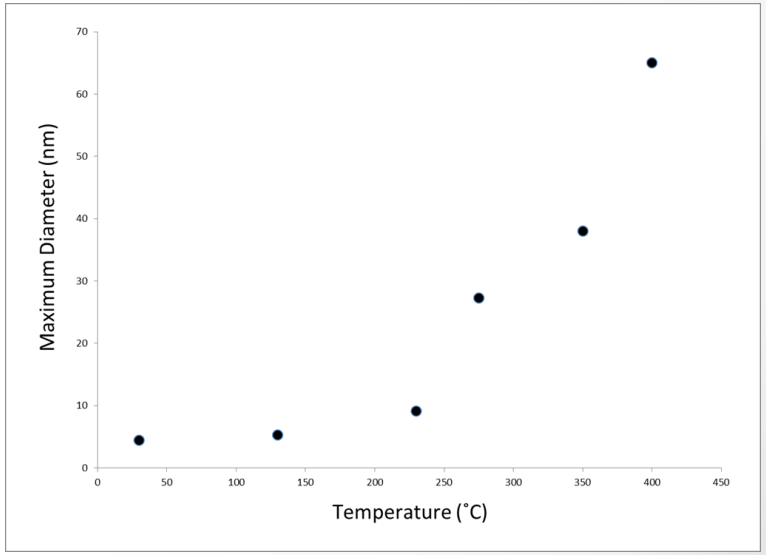



10 keV He then 3 MeV Ni

**Bubble to cavity transition and cavity evolution can be directly studied**



# Precession Electron Diffraction Reveals Hidden Grain Structure

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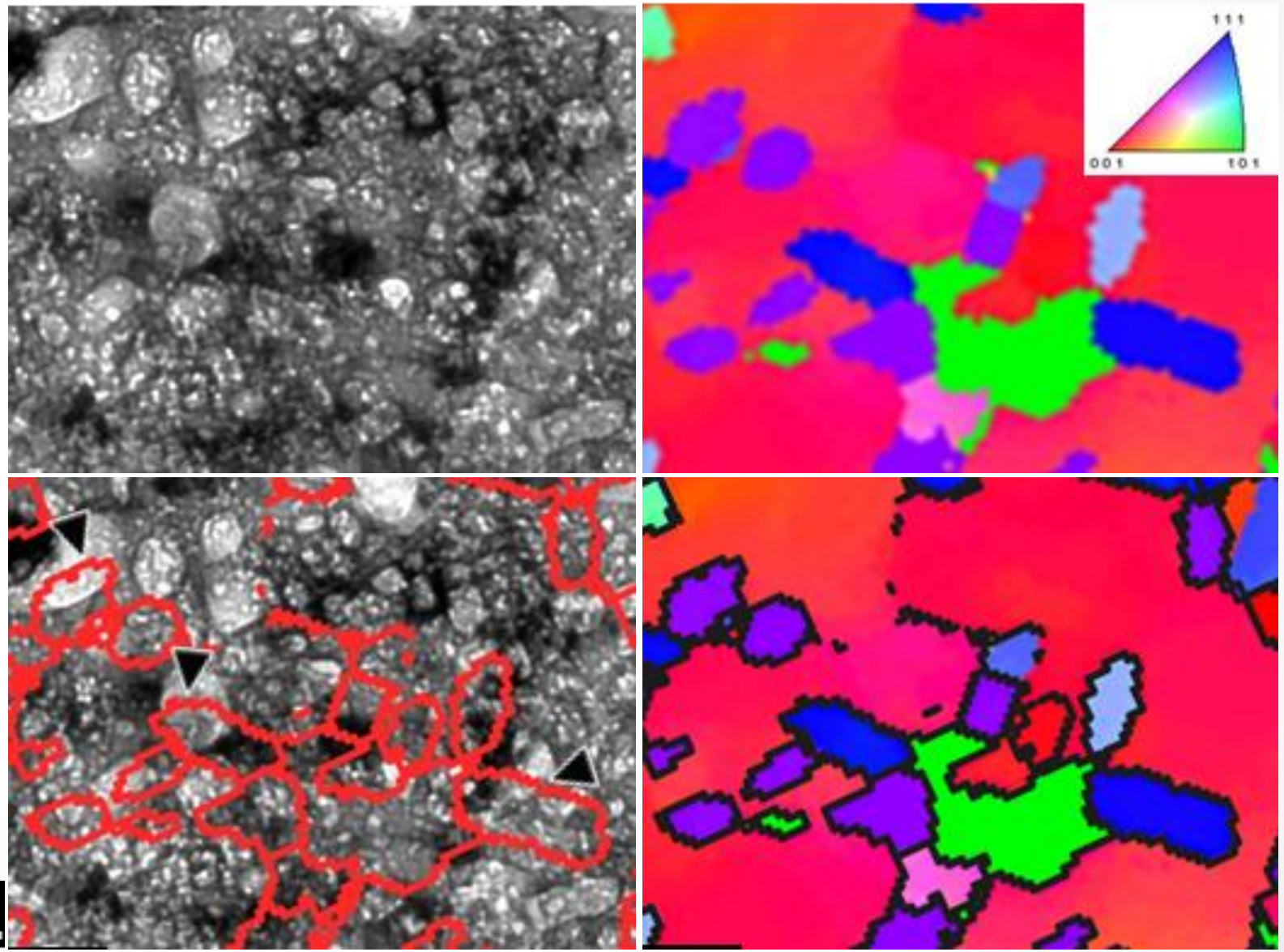
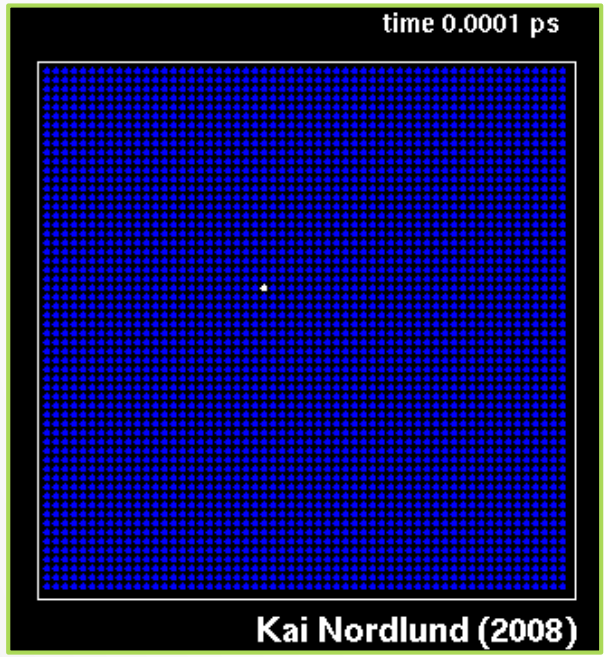
Cavities in helium implanted, self-ion irradiated, nc nickel film annealed to 400 ˚C




**Cavities span multiple grains at identified grain boundaries**

# Summary and the Future

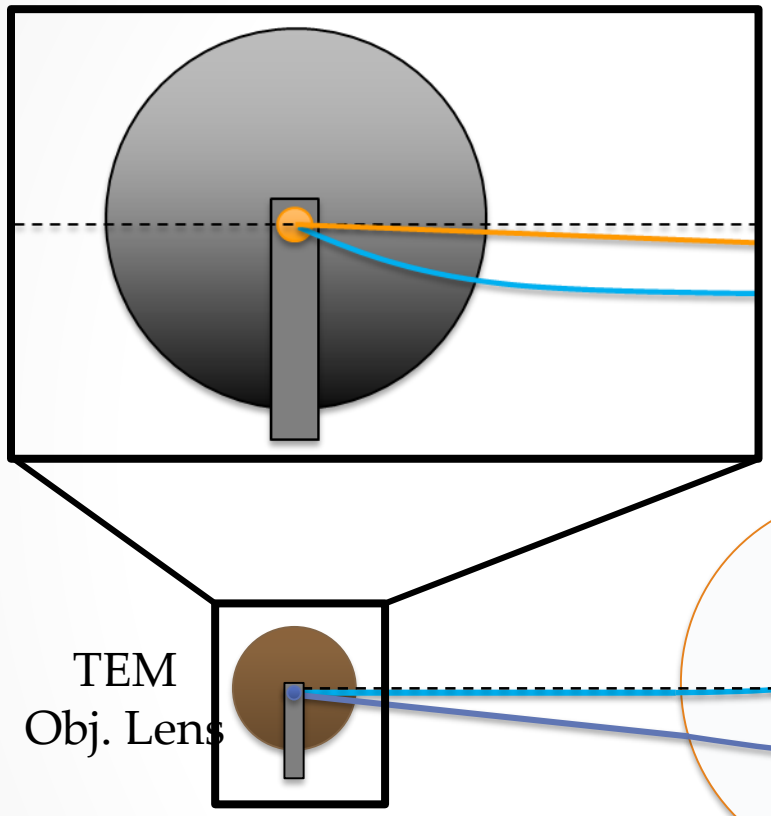
- Enhanced cavity nucleation with concurrent helium implantation and displacement damage
- Total fluence and irradiation order do affect cavity evolution, but temperature has the most dramatic effects
- Combination with orientation mapping begins to clarify the role of local microstructure

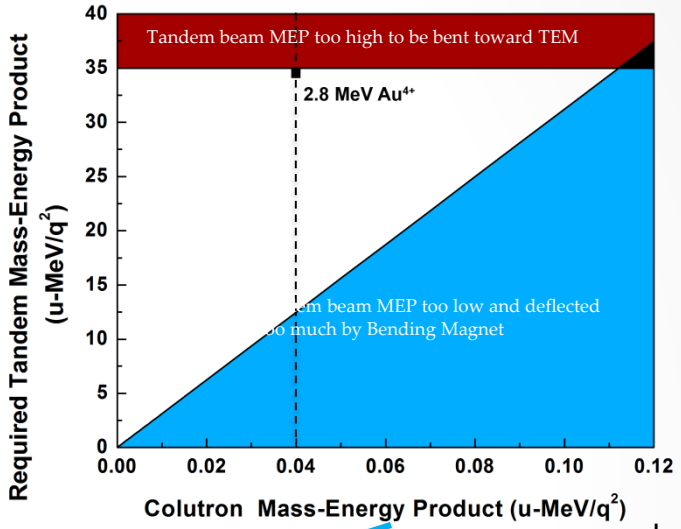


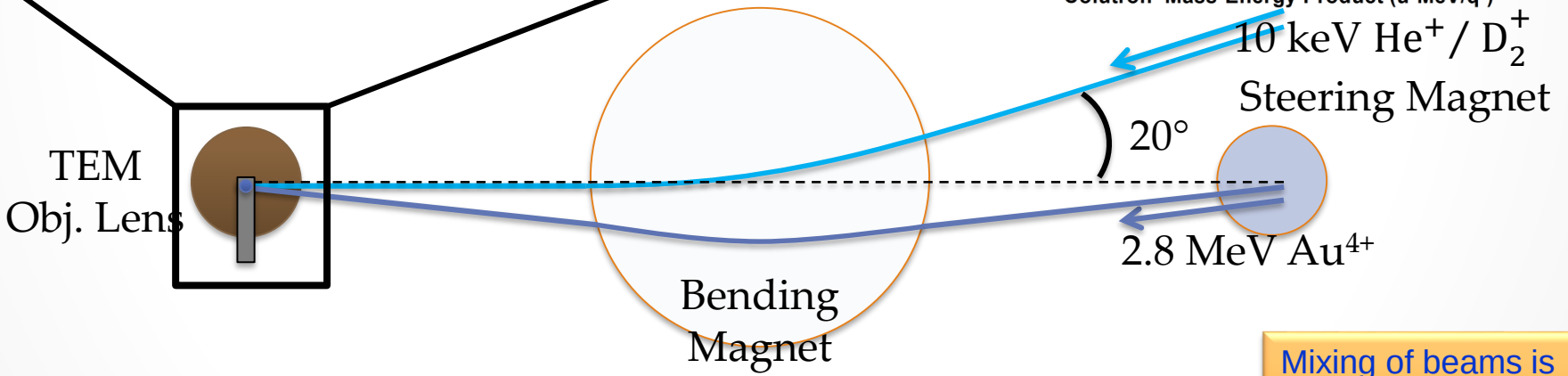

Acknowledgements: **IBL**: D.L. Buller, B.L. Doyle, C. Gong, M.T. Marshall, M. Steckbeck. **Sandia**: F.F. Abdeljawad, D.P. Adams, J.A. Scott, C. Sobczak. **External**: A. Minor, (UC Berkley), A. Darbal (AppFive), A. Leff (Drexel University). Work performed by DCB at Sandia was fully supported by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy.

# Modeling Beam Mixing and Deflection

Collaborators: M. Steckbeck & B.L. Doyle






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

Mixing of beams is possible in limited conditions!

# Ion Beam Conditions

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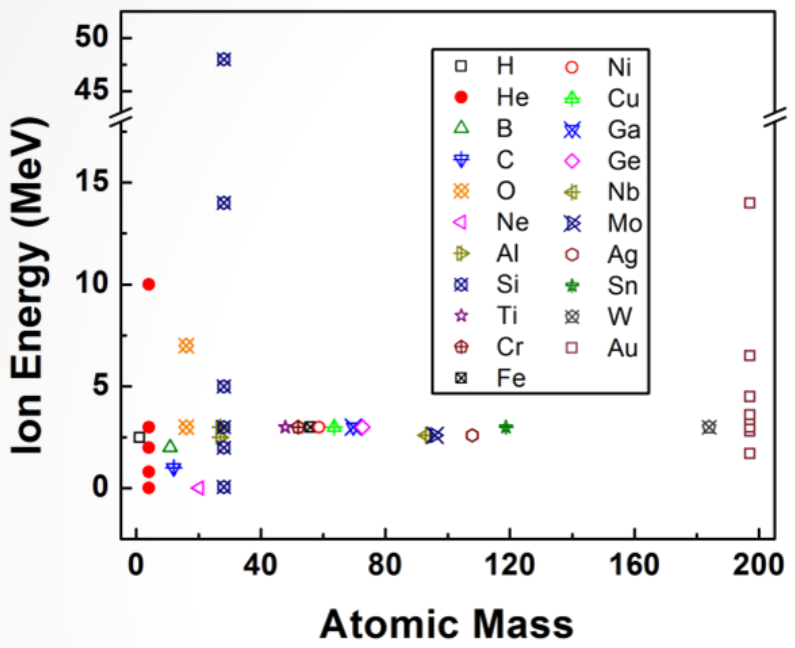


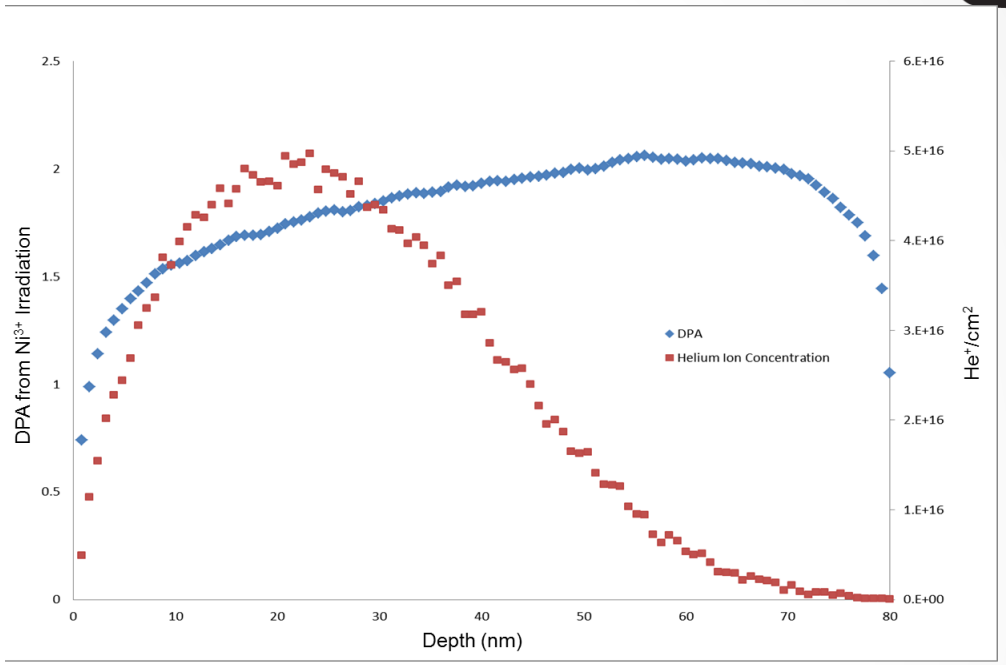


| Order | $Ni^{3+}$ Rate | $Ni^{3+}$ damage | He Rate | He concentration |
|---|---|---|---|---|
| | ions/cm$^2$s | DPA | ions/cm$^2$s | ions/cm$^2$ |
| $Ni^{3+}$, $He^{+}$ | 1.5 E11 | 1.8 | 2.6 E 13 | 3 E 16 |
| $He^{+}$, $Ni^{3+}$ | 1.5 E11 | 0.7 | 5.5 E13 | 1 E 17 |

# 3 MeV $Ni^{3+}$ Irradiation followed by 10 keV $He^{+}$ Implantation

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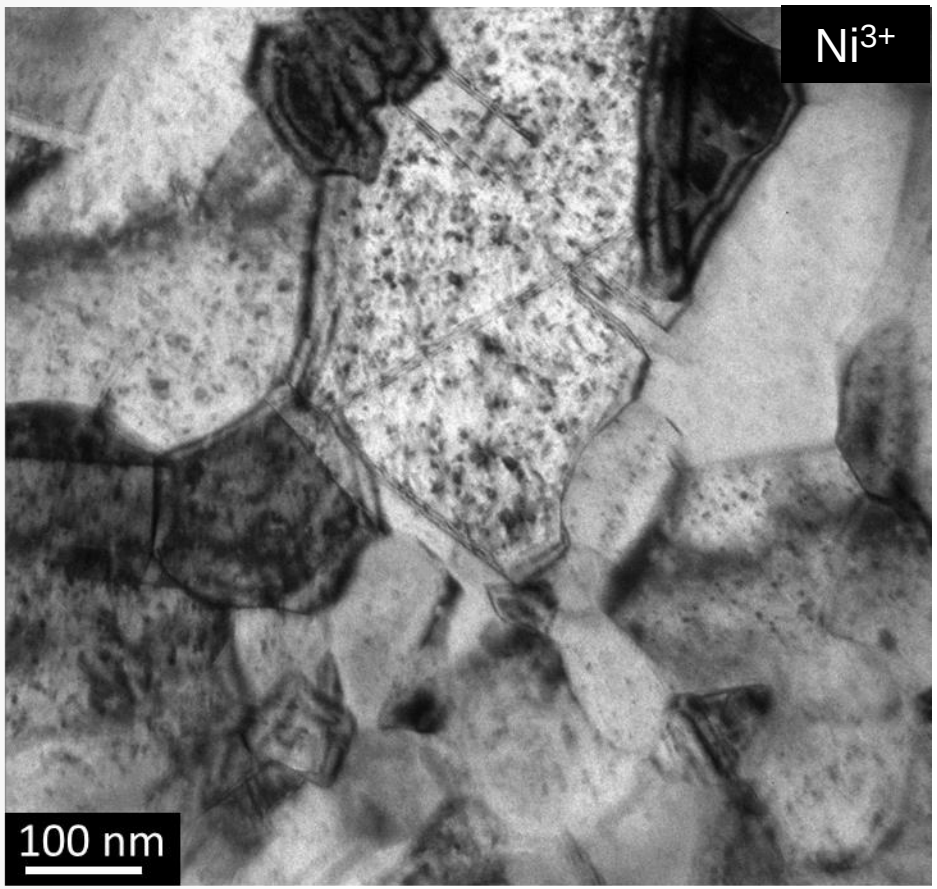
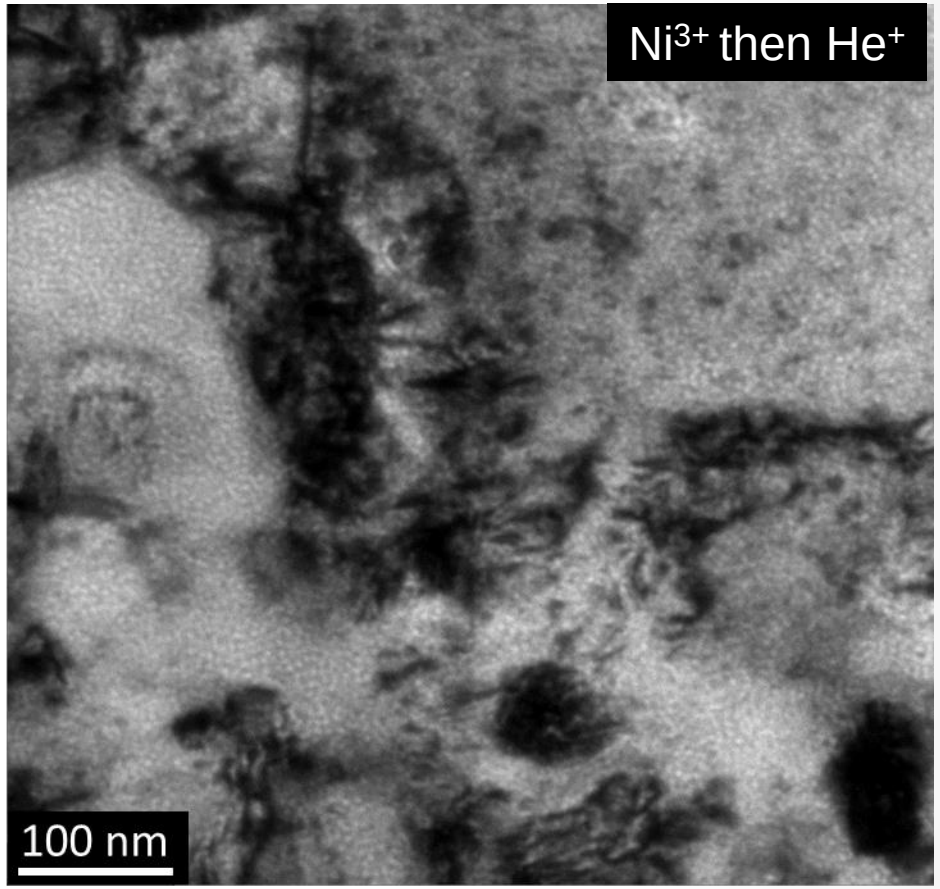
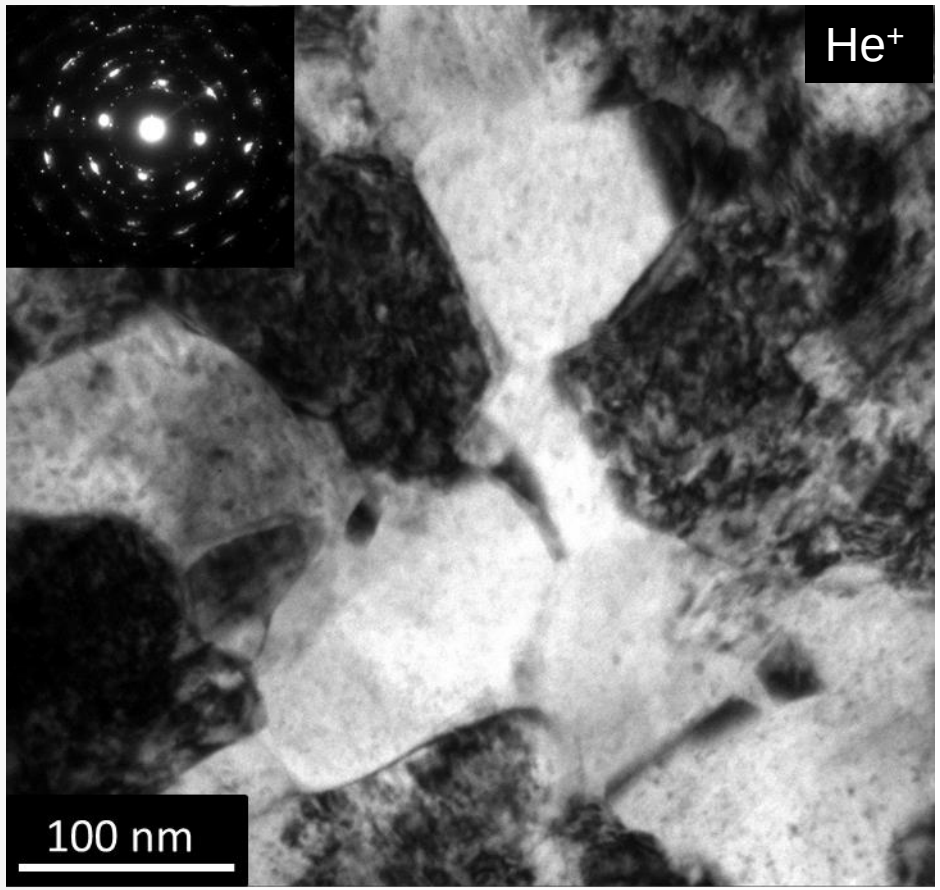


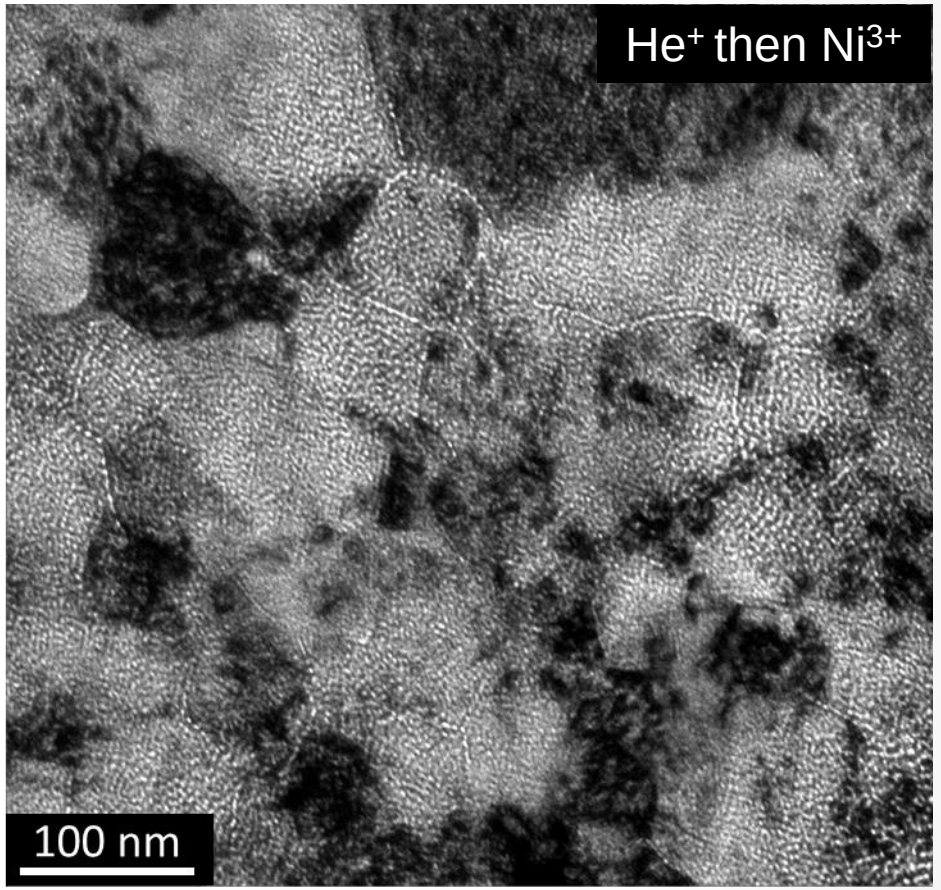

1.8 dpa $Ni^{3+}$ irradiation
Dislocation loops and SFT are present

Additional $2x10^{16}$ $He^{+}/cm^{2}$
Evenly distributed nanometer size cavities

# 10 keV $He^+$ Implantation followed by 3 MeV $Ni^{3+}$ Irradiation




$10^{17}$ $He^+/cm^2$
Visible damage




0.7 dpa $Ni^{3+}$ irradiation
High concentration of cavities along grain boundaries

# Beam Effects

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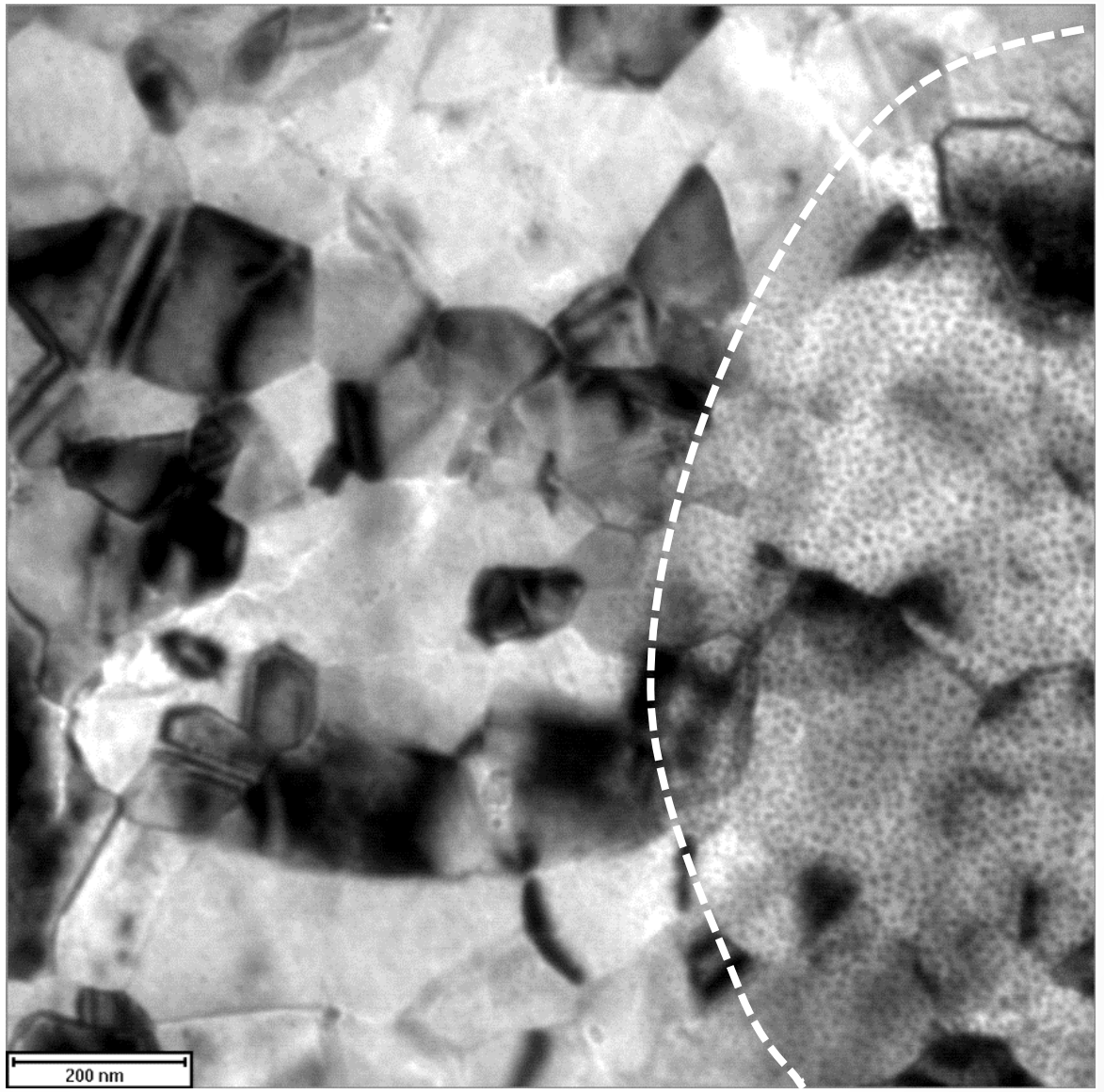
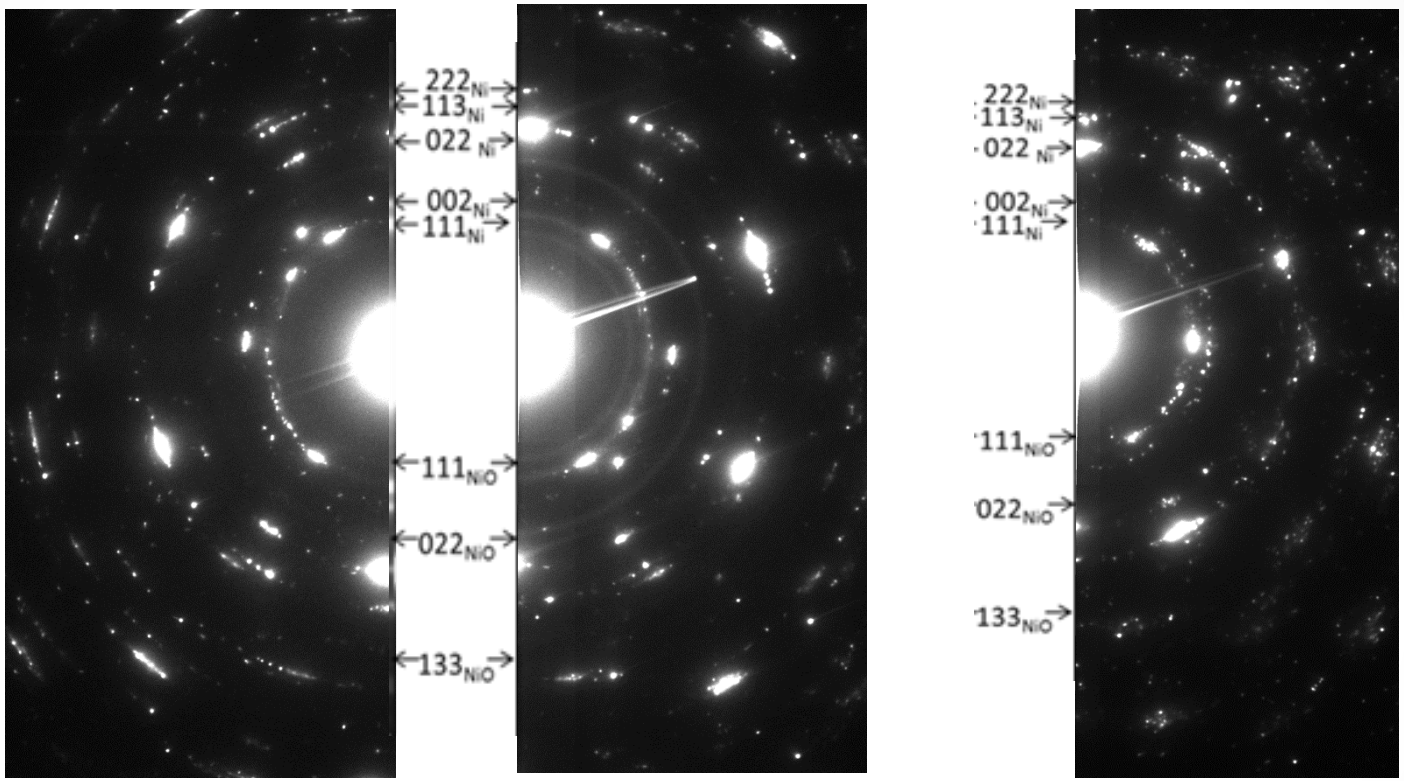
$\Delta T \approx 2$ K

Max Energy
Transferred = 14.5 eV
Threshold E = 22 eV



# Growth




Include the diffusion model…