

Insight from in-situ analysis of grain boundary character, radiation sequence, and thermal conditions on defect structure evolution in nickel

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Ion Beam Lab at Sandia National Laboratories

March, 2016



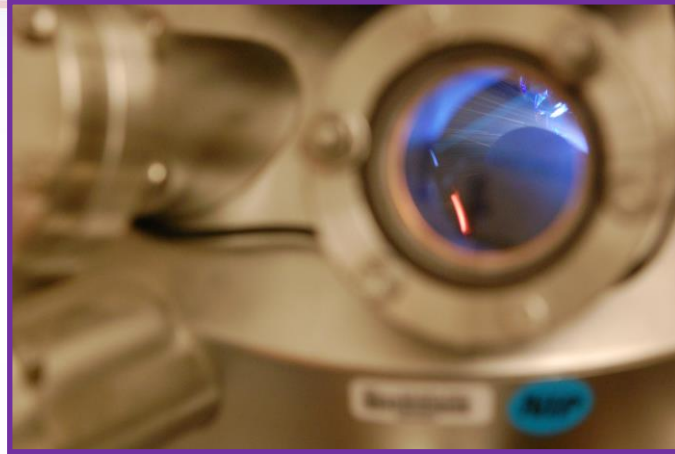
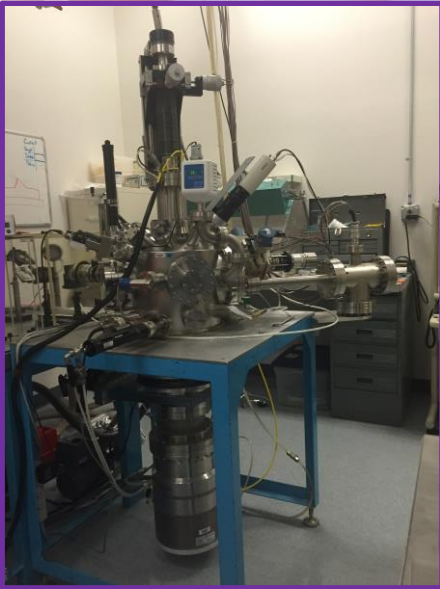
This work was supported by the US Department of Energy, Office of Basic Energy Sciences.

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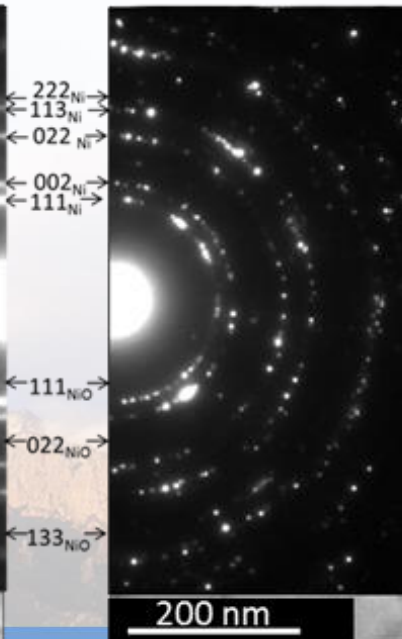
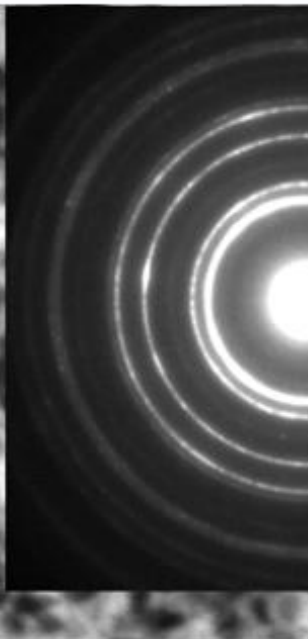
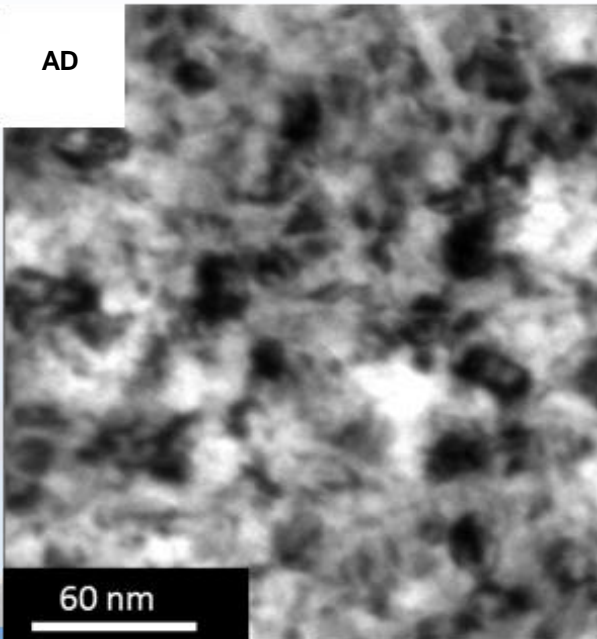
Nuclear Security Administration under contract DE-AC04-94AL85000.

Pulse Laser Deposition of Ni Films

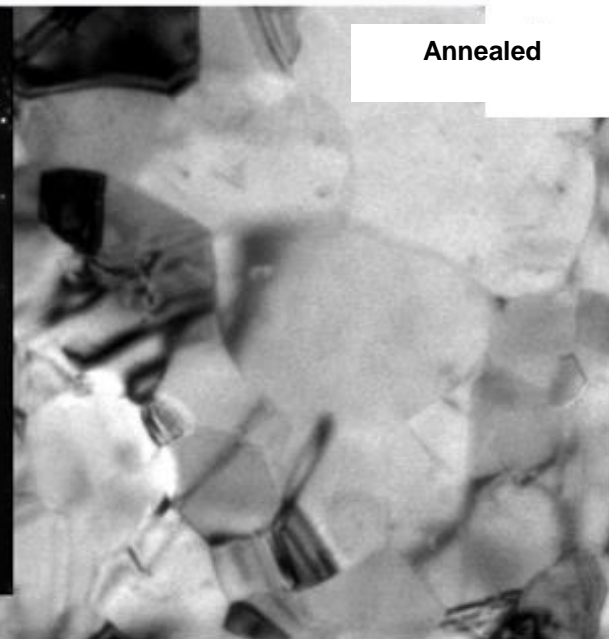


- KrF Excimer laser
 - 35 Hz
 - 4.5×10^{-5} Pa

AD



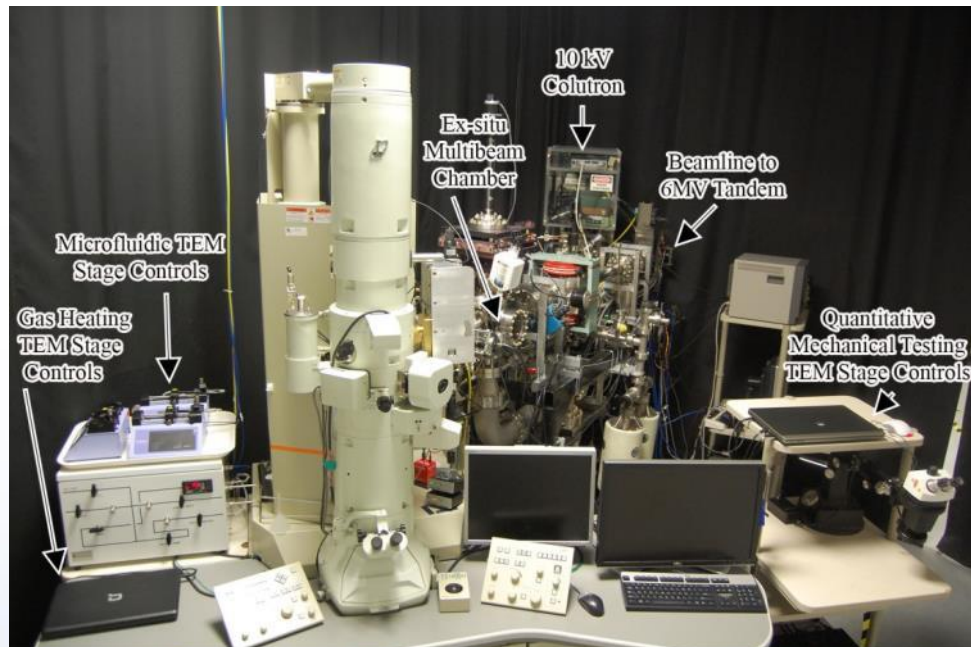
Annealed



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

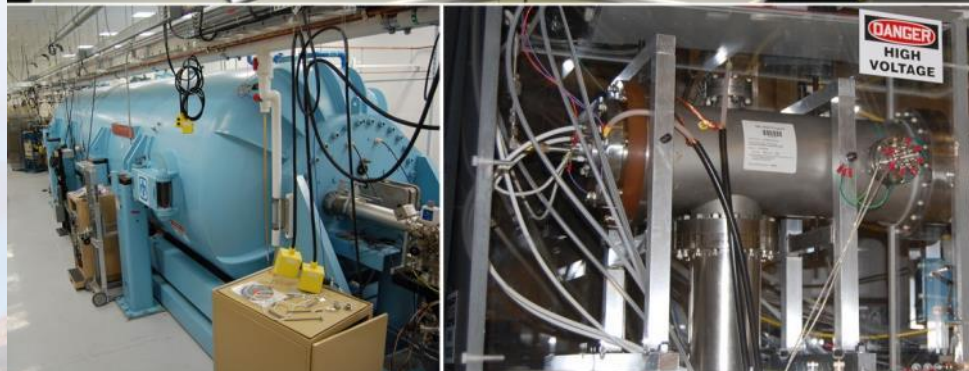
Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

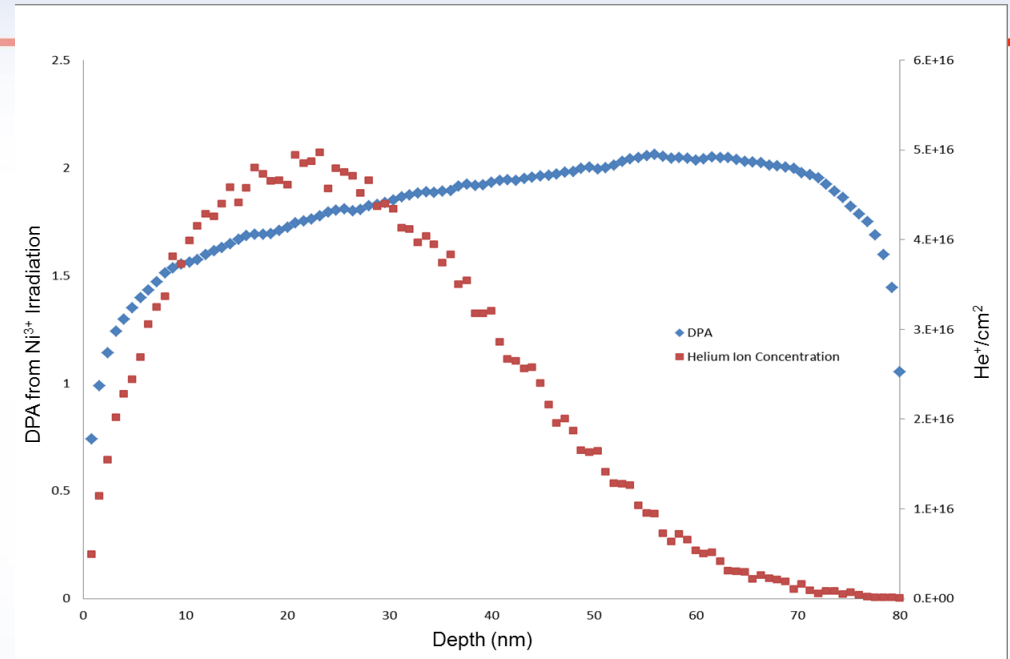
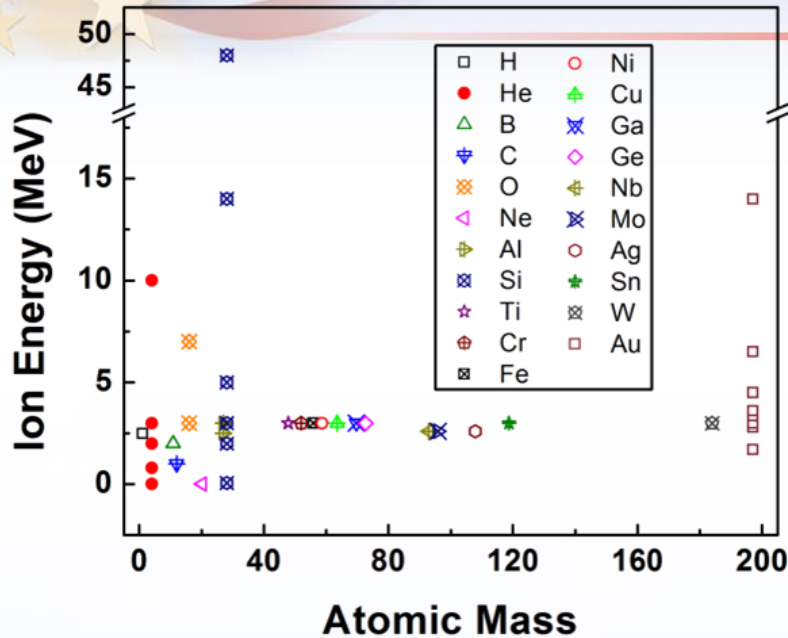


Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

For more details:
Khalid Hattar's Talk
Name
Time
Date



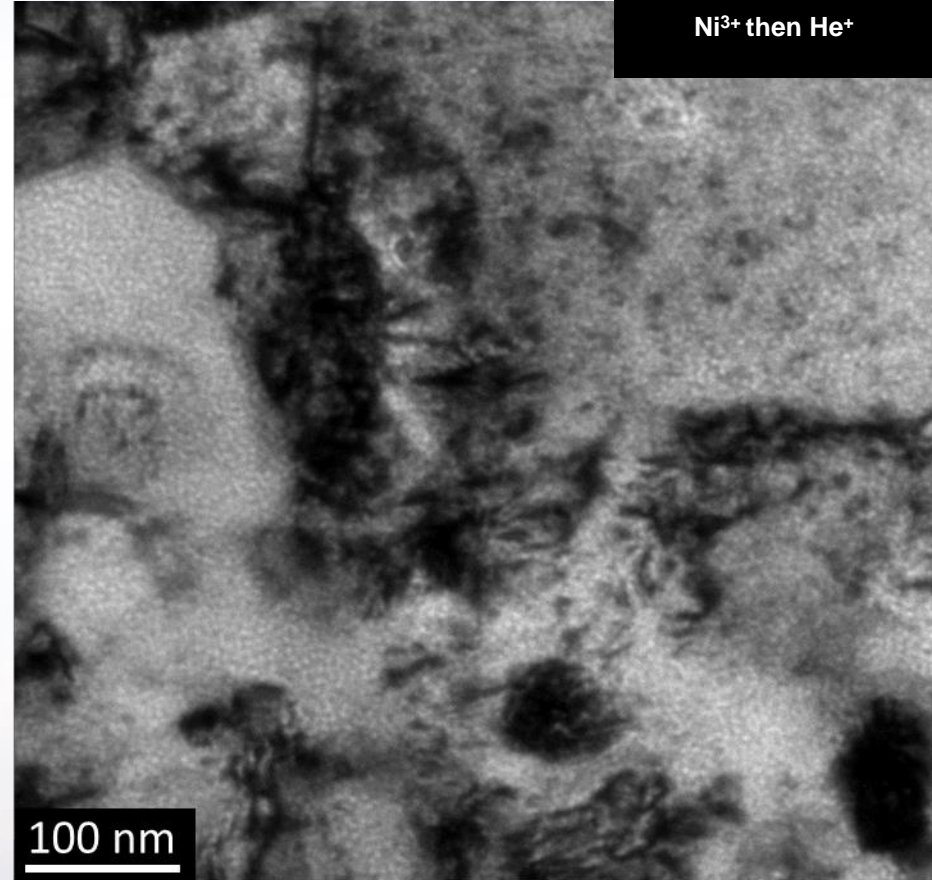
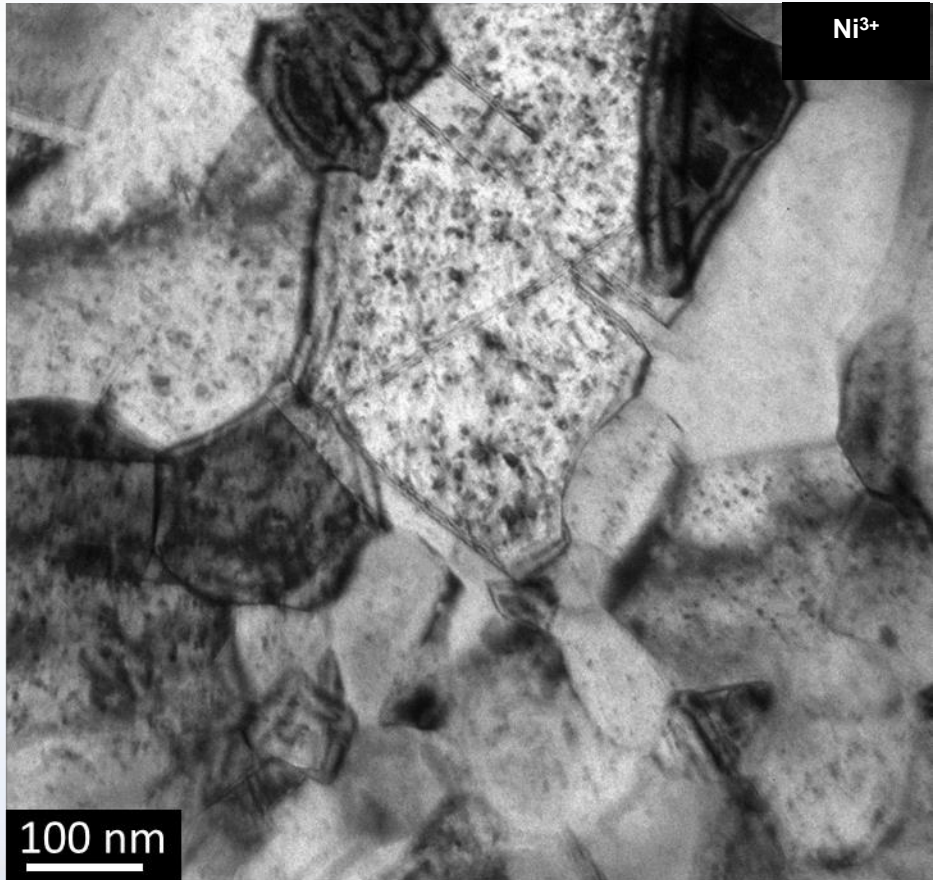
Ion Beam Conditions



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 Ni³⁺ Irradiation followed by 10 keV He⁺ Implantation

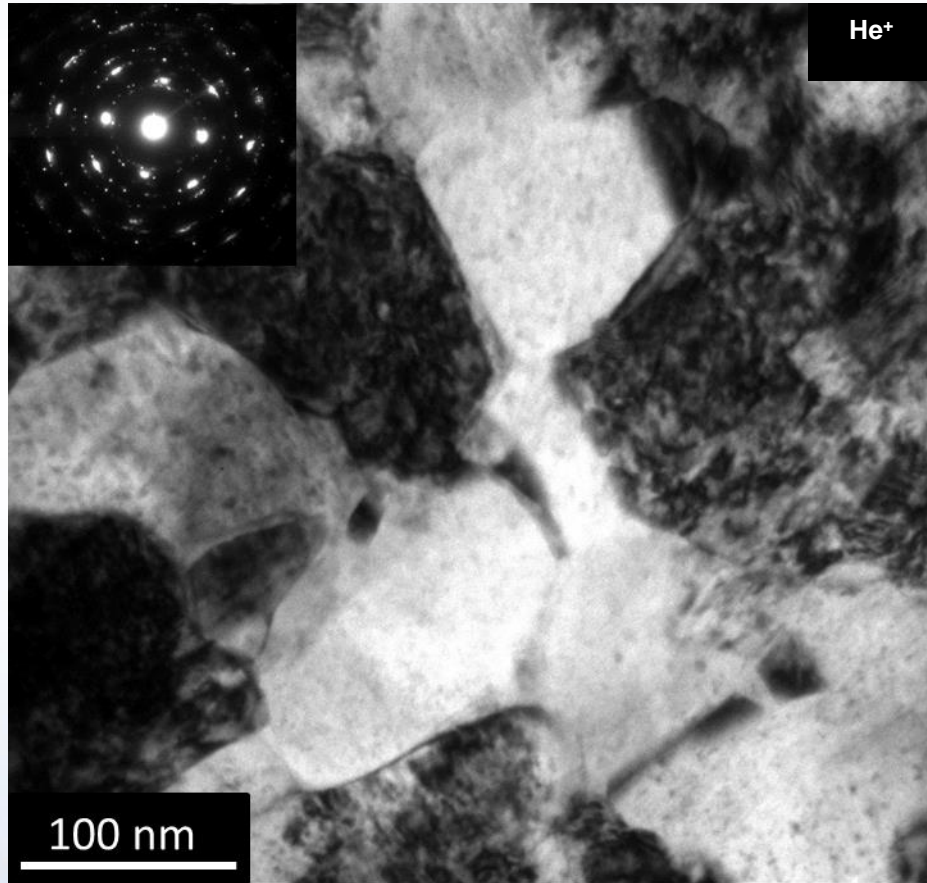


1.8 dpa Ni³⁺ irradiation
Dislocation loops and SFT are present

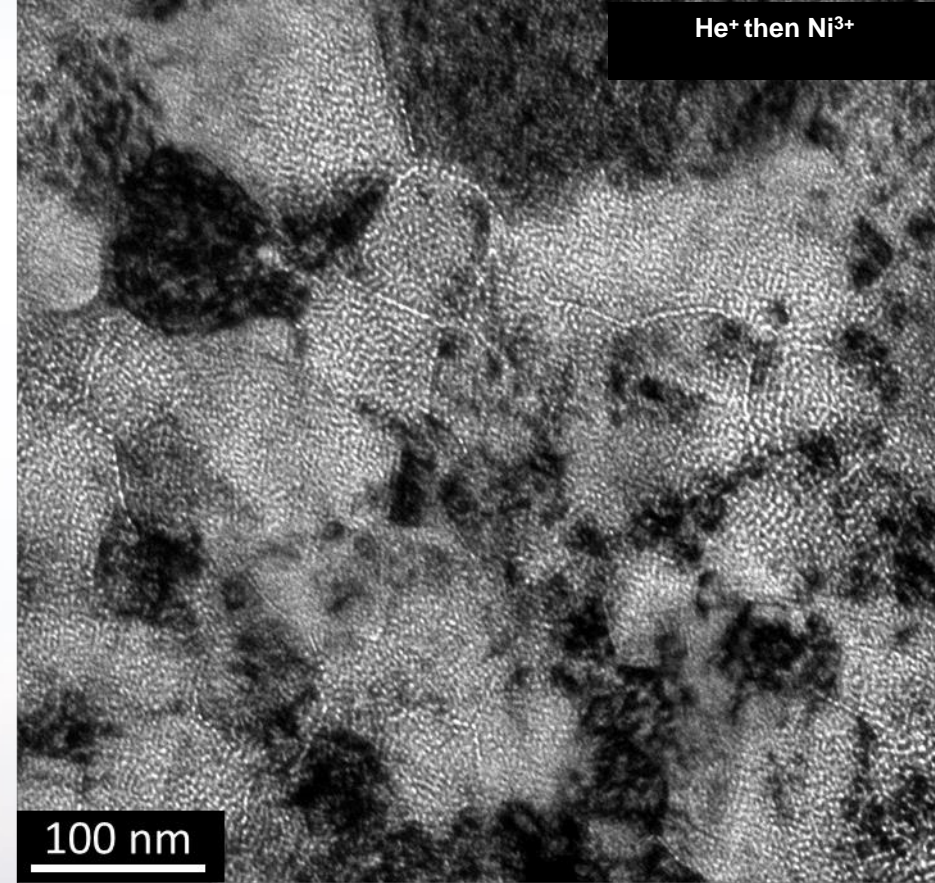
Additional 2x10¹⁶ He⁺/cm²
Evenly distributed
nanometer size cavities



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



10¹⁷ He⁺/cm²
Visible damage

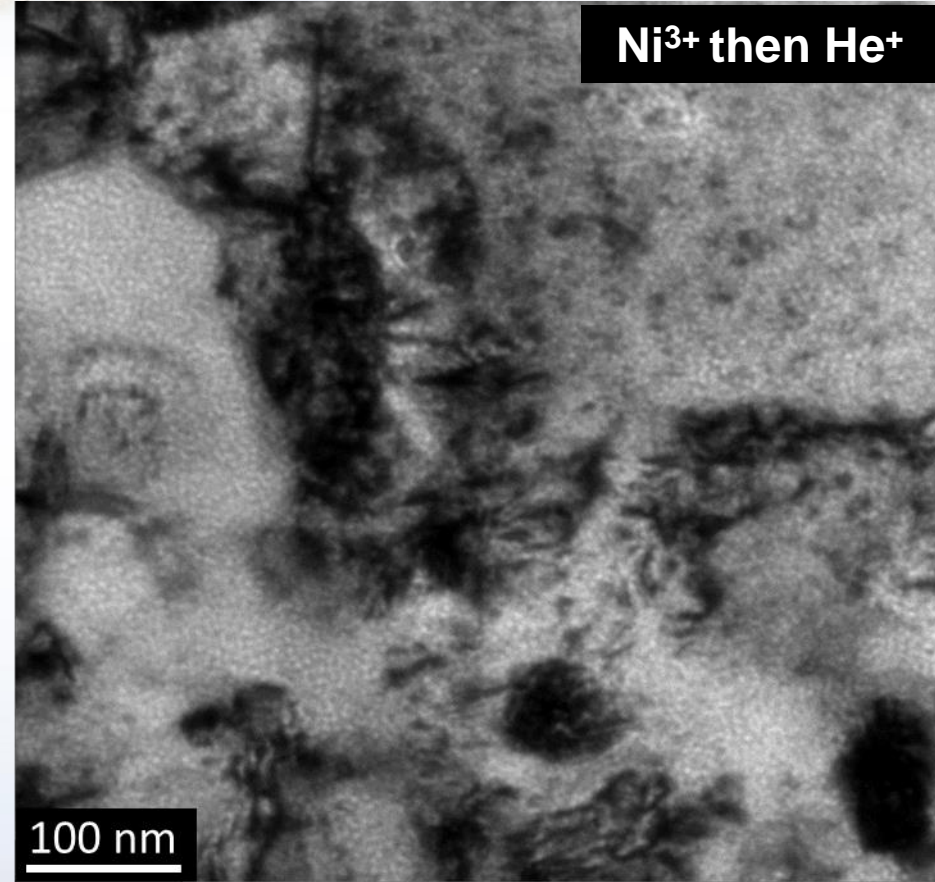


0.7 dpa Ni³⁺ irradiation
High concentration of cavities along
grain boundaries

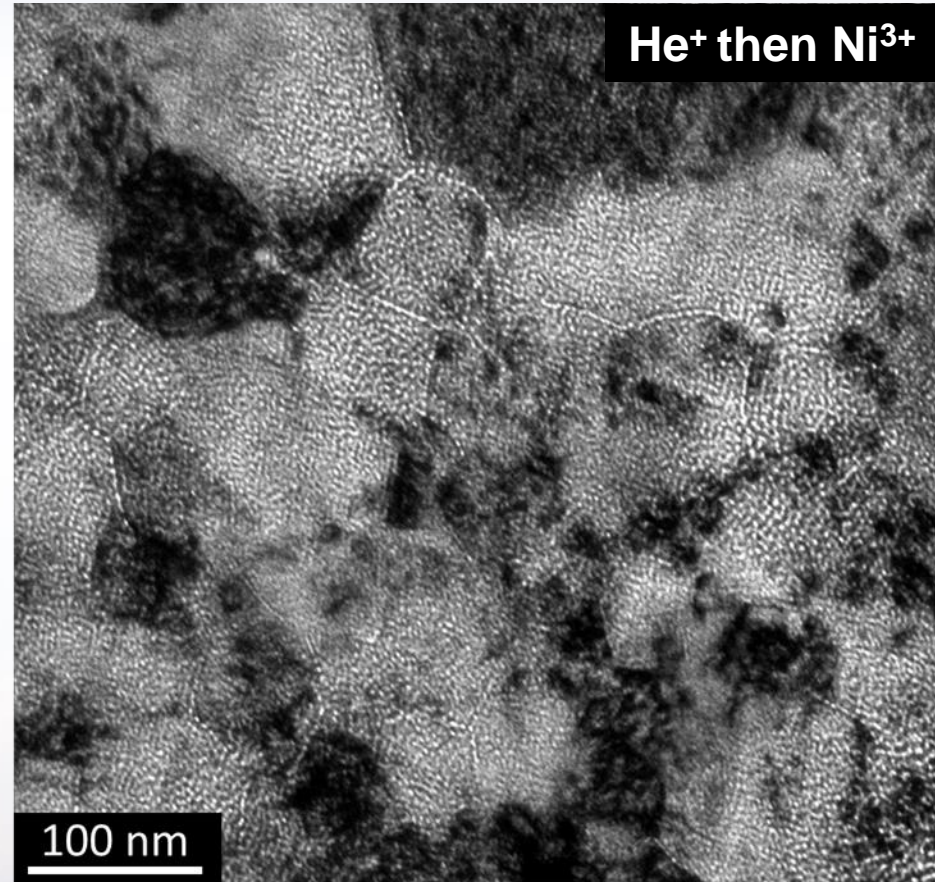


Irradiation / Implantation Sequence Effect on Cavity Structure

Ni³⁺ then He⁺



He⁺ then Ni³⁺

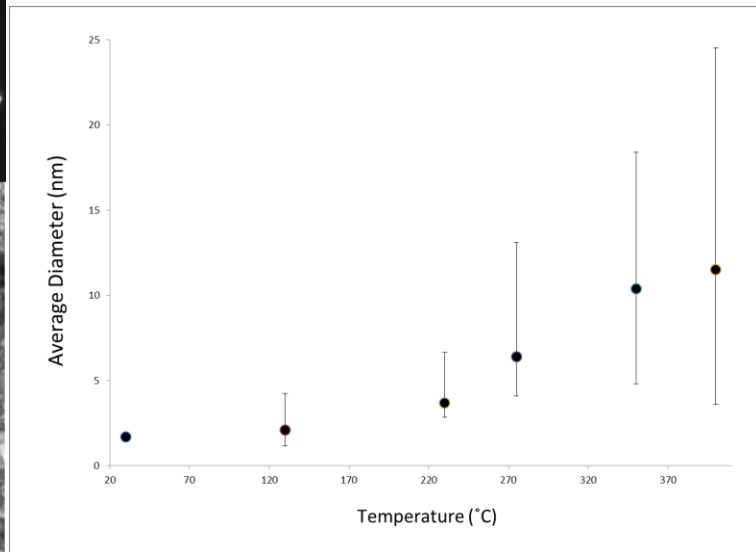
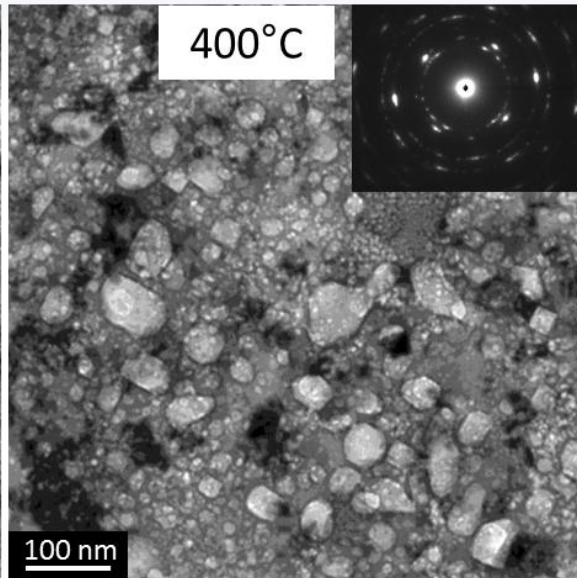
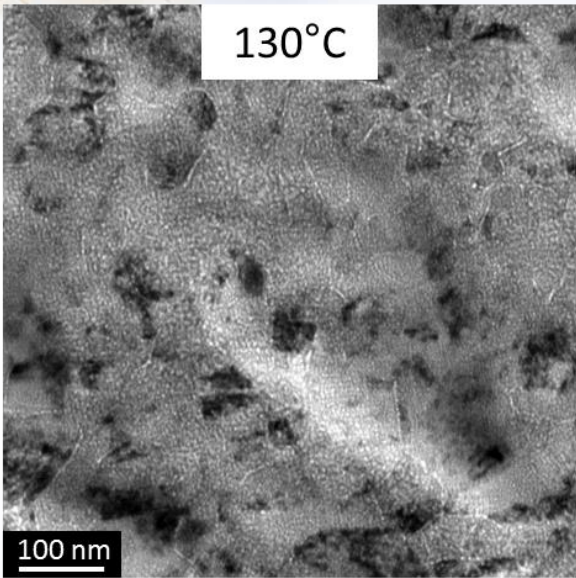


**Evenly distributed
cavities over the entire
grain structure**

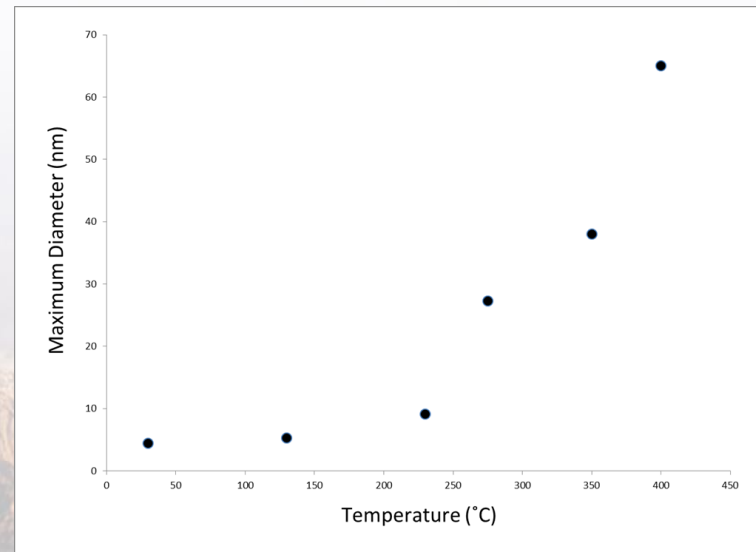
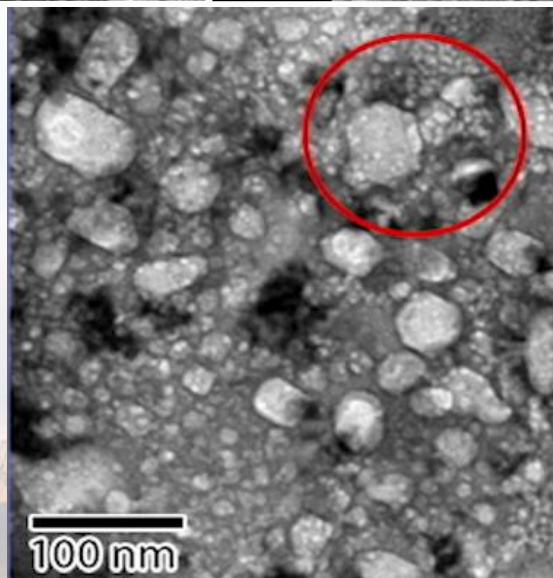
**Apparent higher
concentration of cavities
along grain boundaries**



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

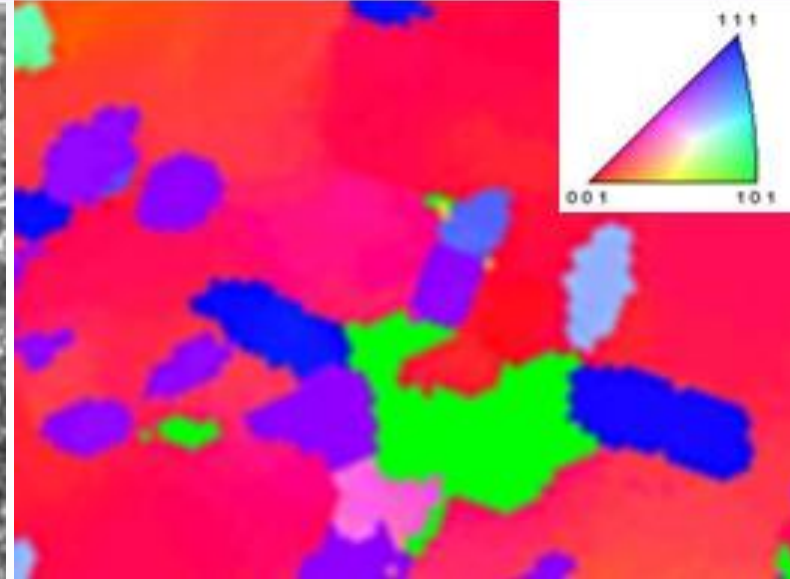
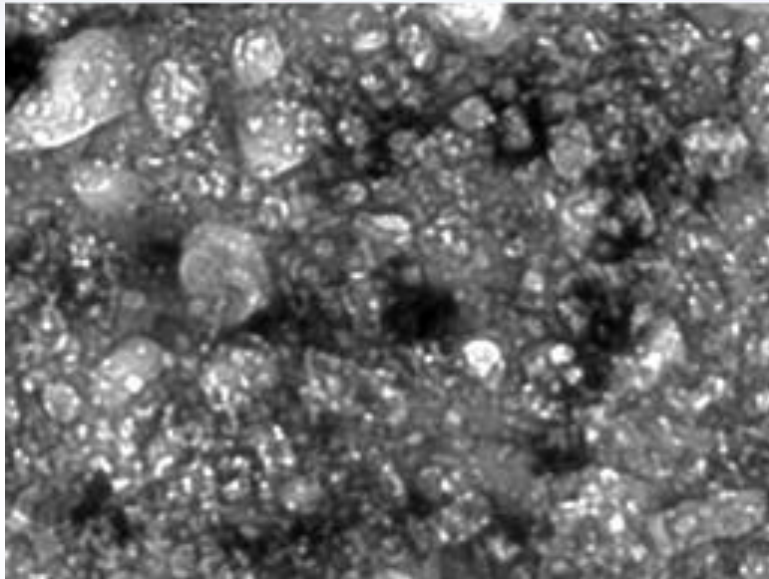


Bubble to cavity transition and cavity evolution can be directly studied

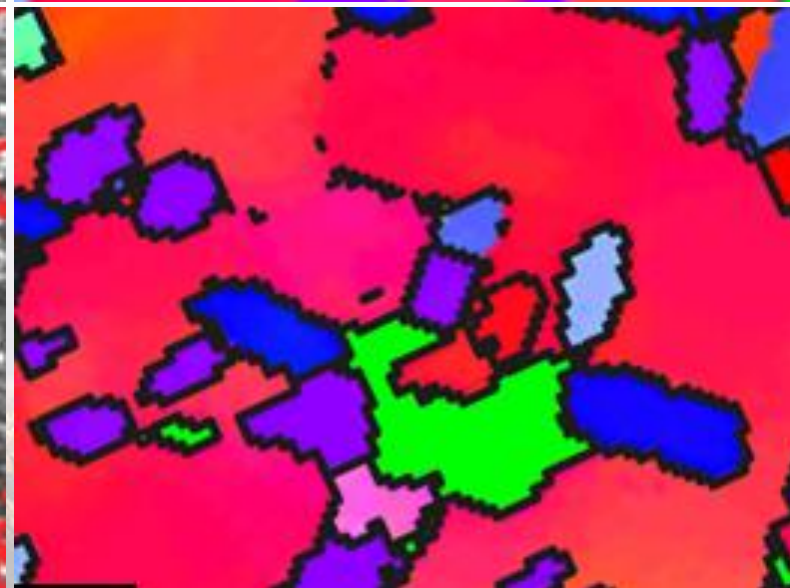
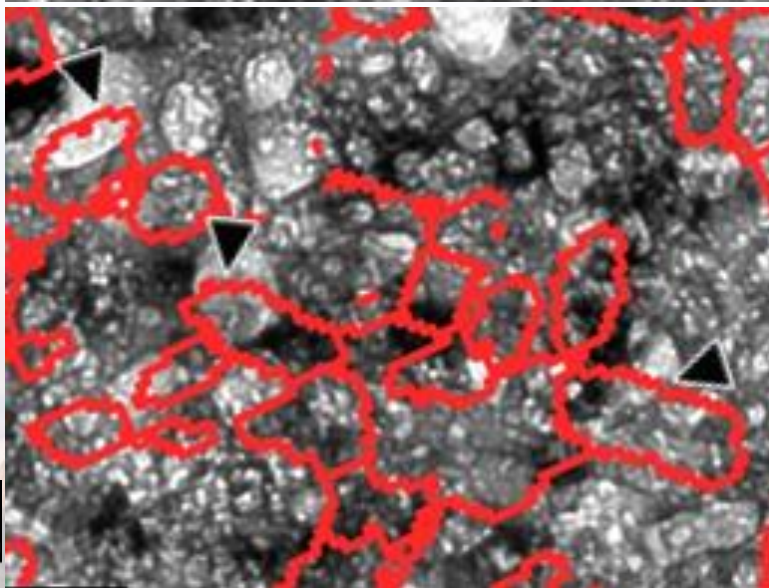


Precession Electron Diffraction Reveals Hidden Grain Structure

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

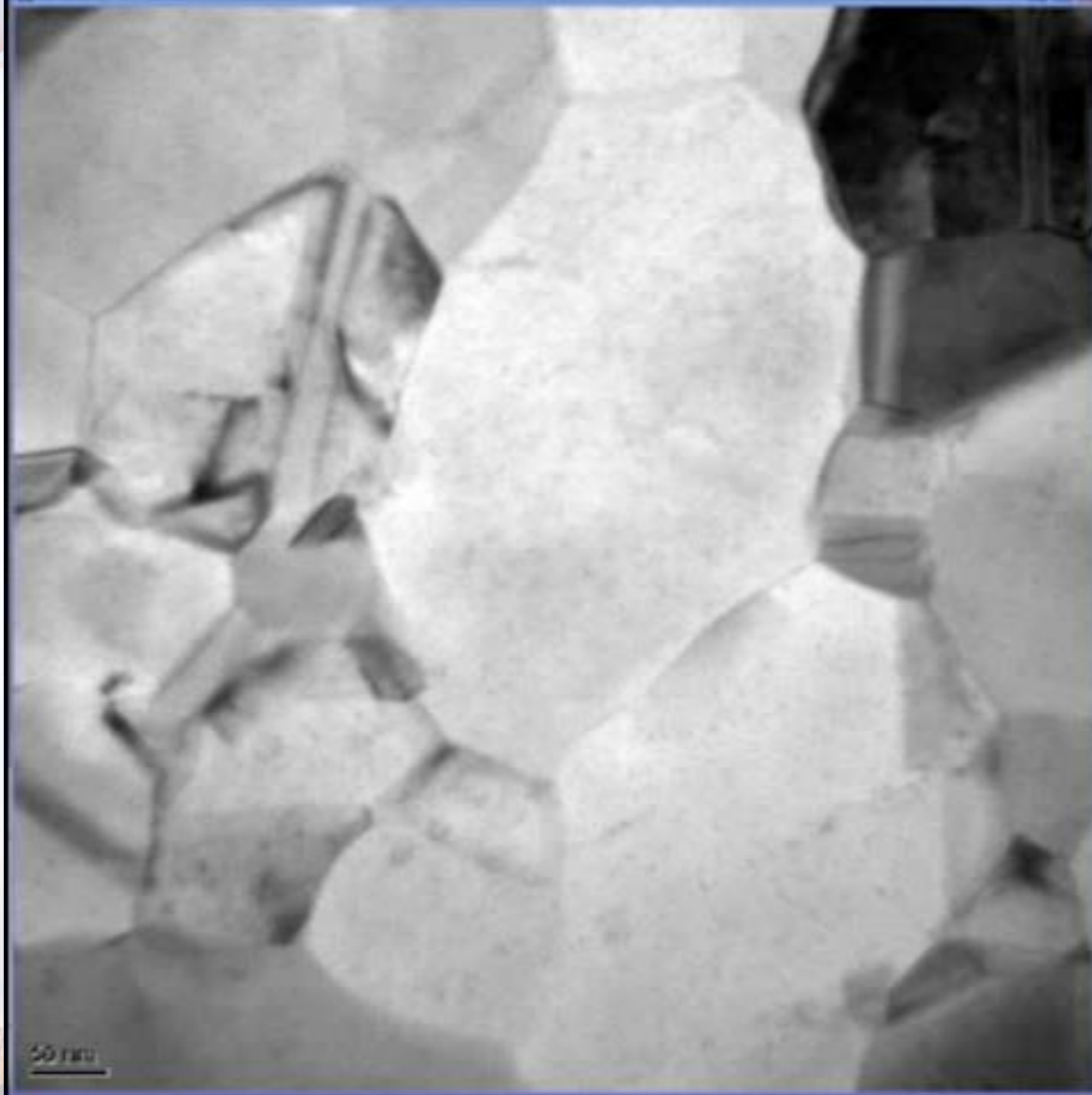


Cavities
span
multiple
grains at
identified
grain
boundaries



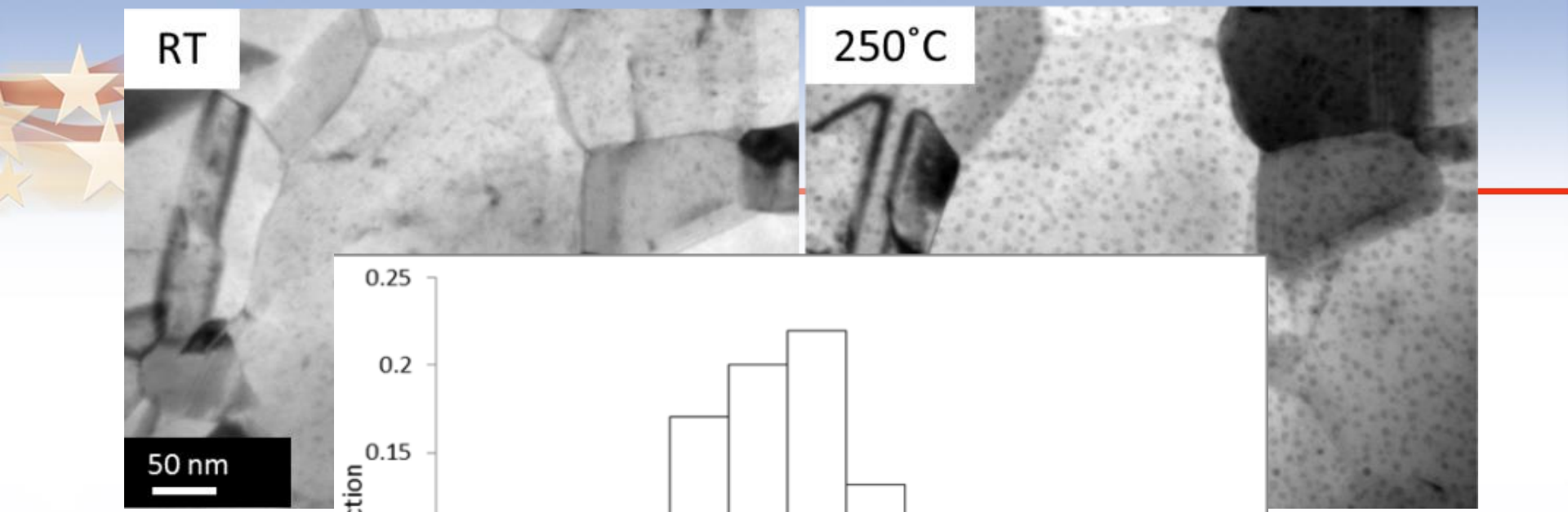
100 nm

Annealing Behavior of Self-Ion Irradiation Induced Defects



≈ 2 dpa Ni^{3+}

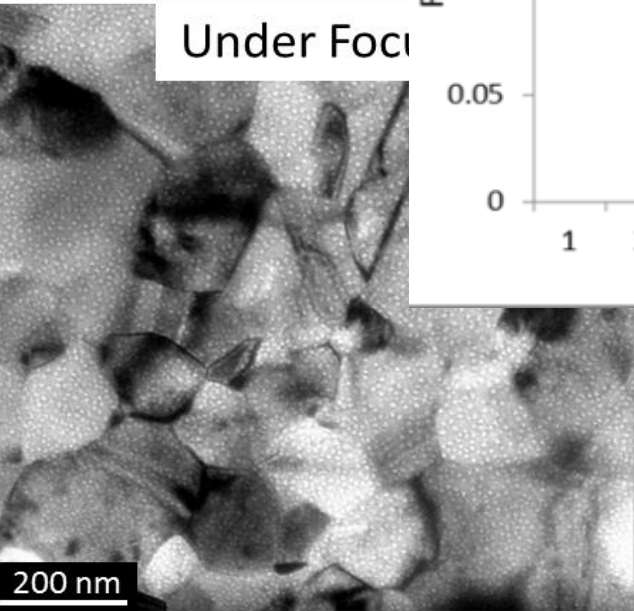
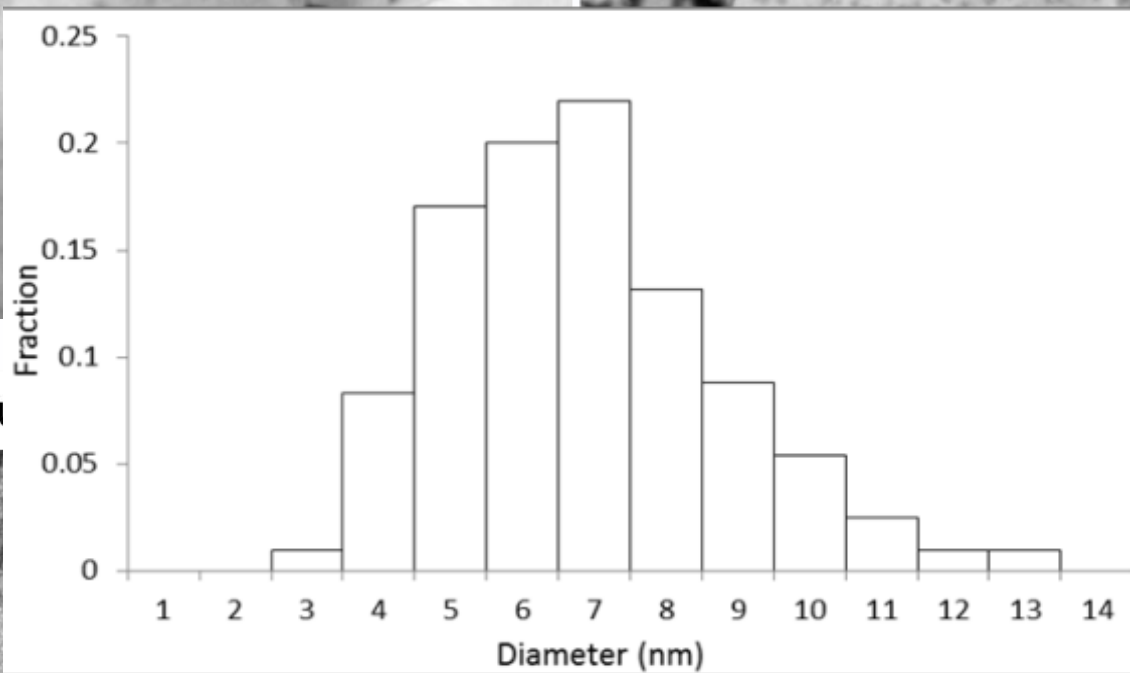




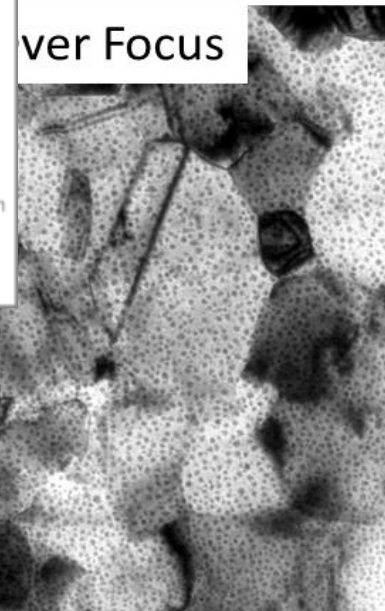
RT

250°C

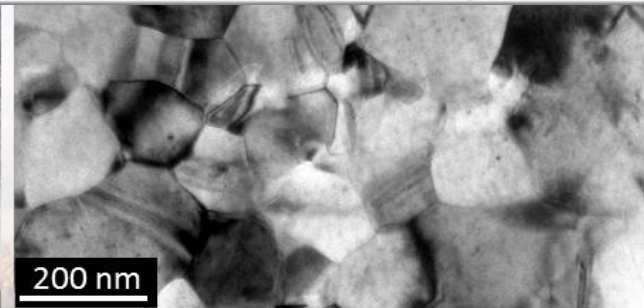
50 nm



Under Focus



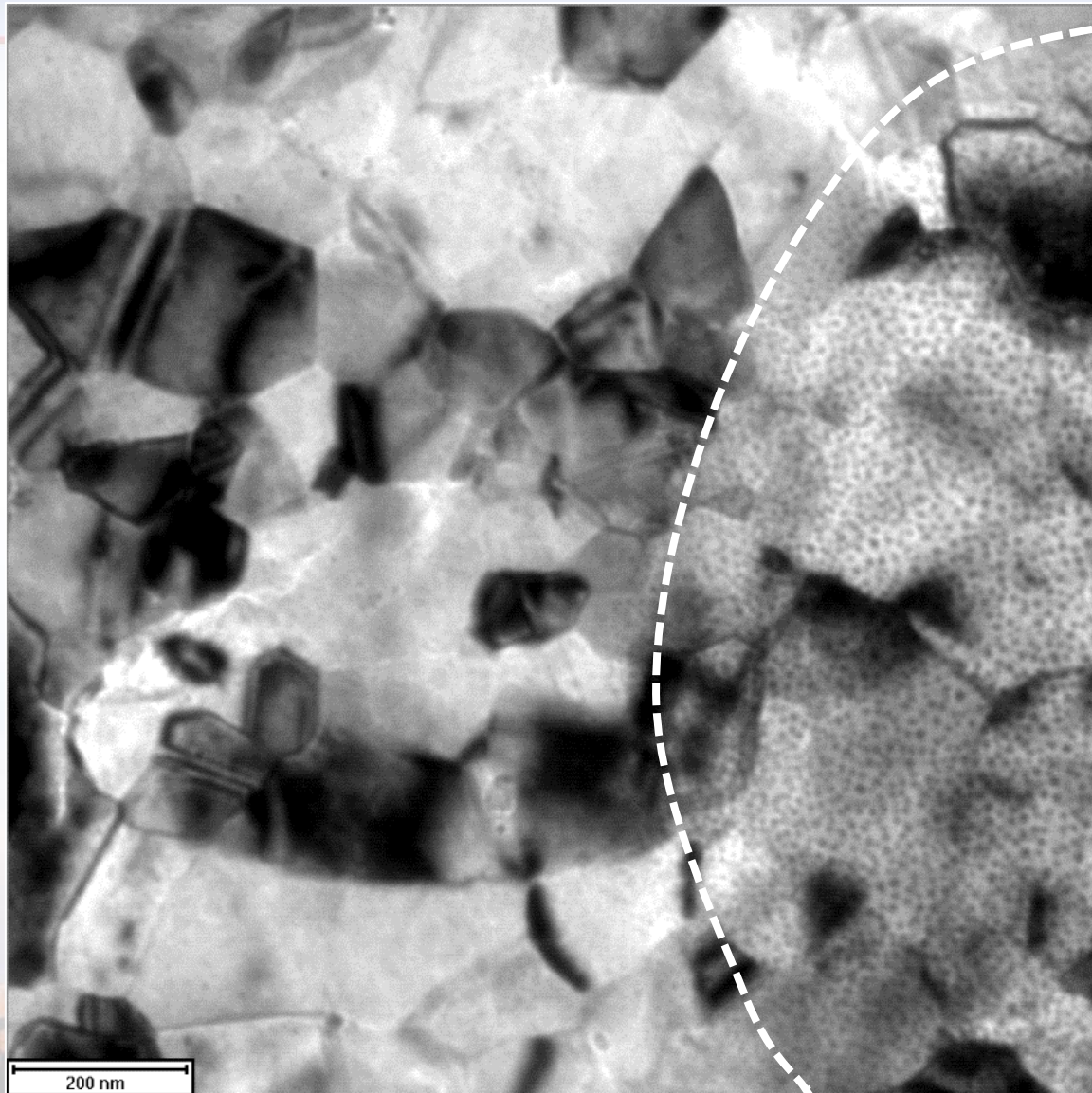
Over Focus



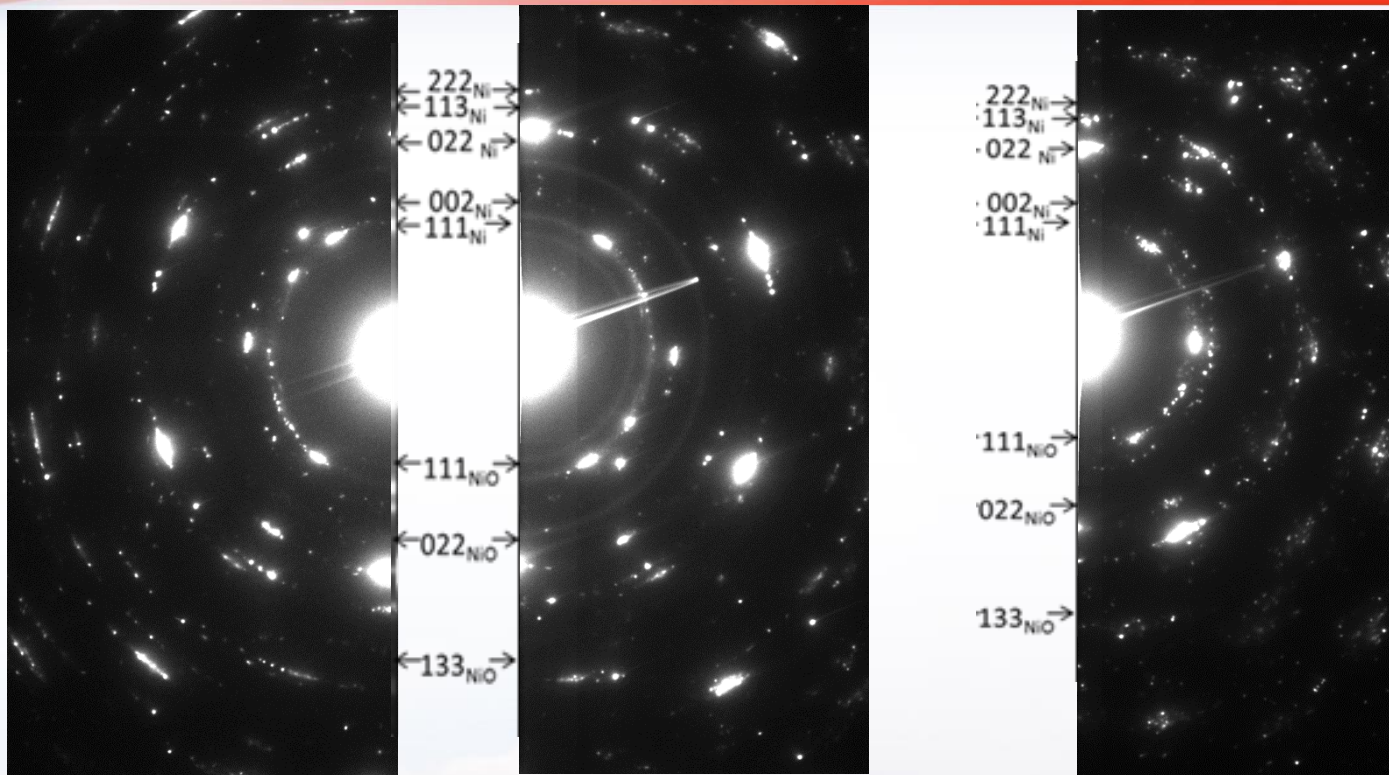
Electron Beam Effects

$\Delta T \approx 2 \text{ K}$

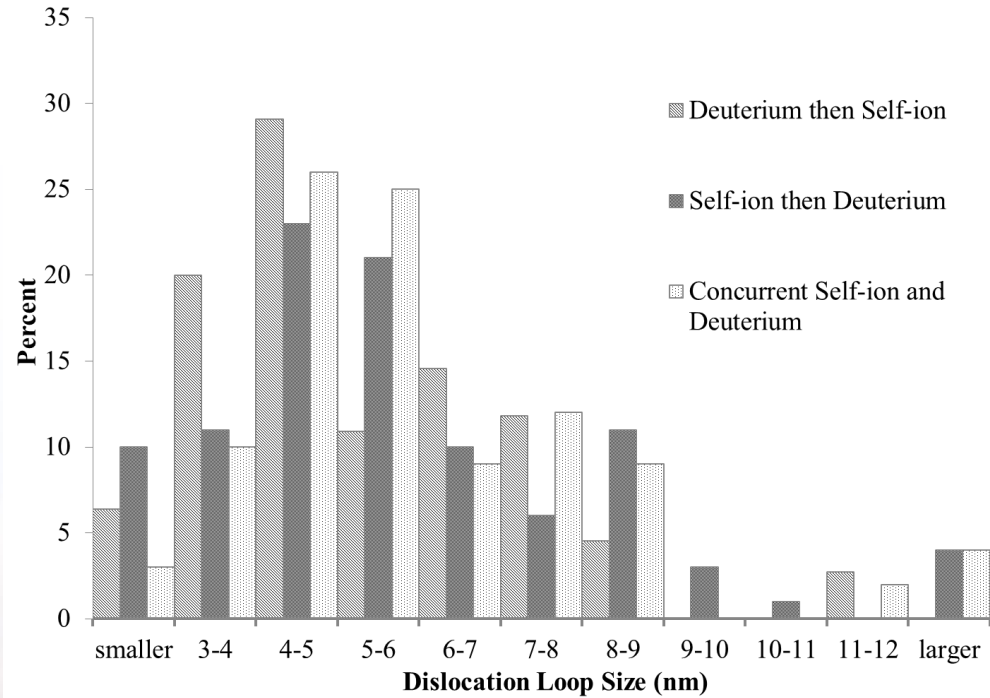
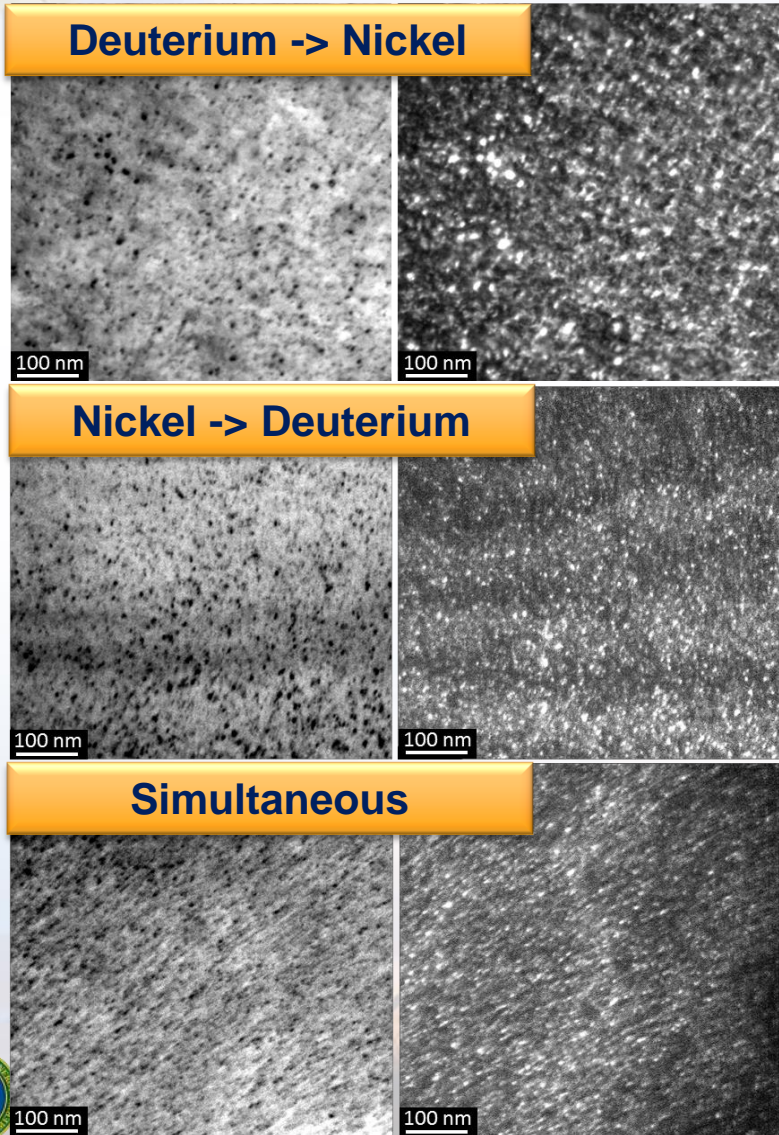
Max Energy Transferred = 14.5 eV
Threshold E = 22 eV



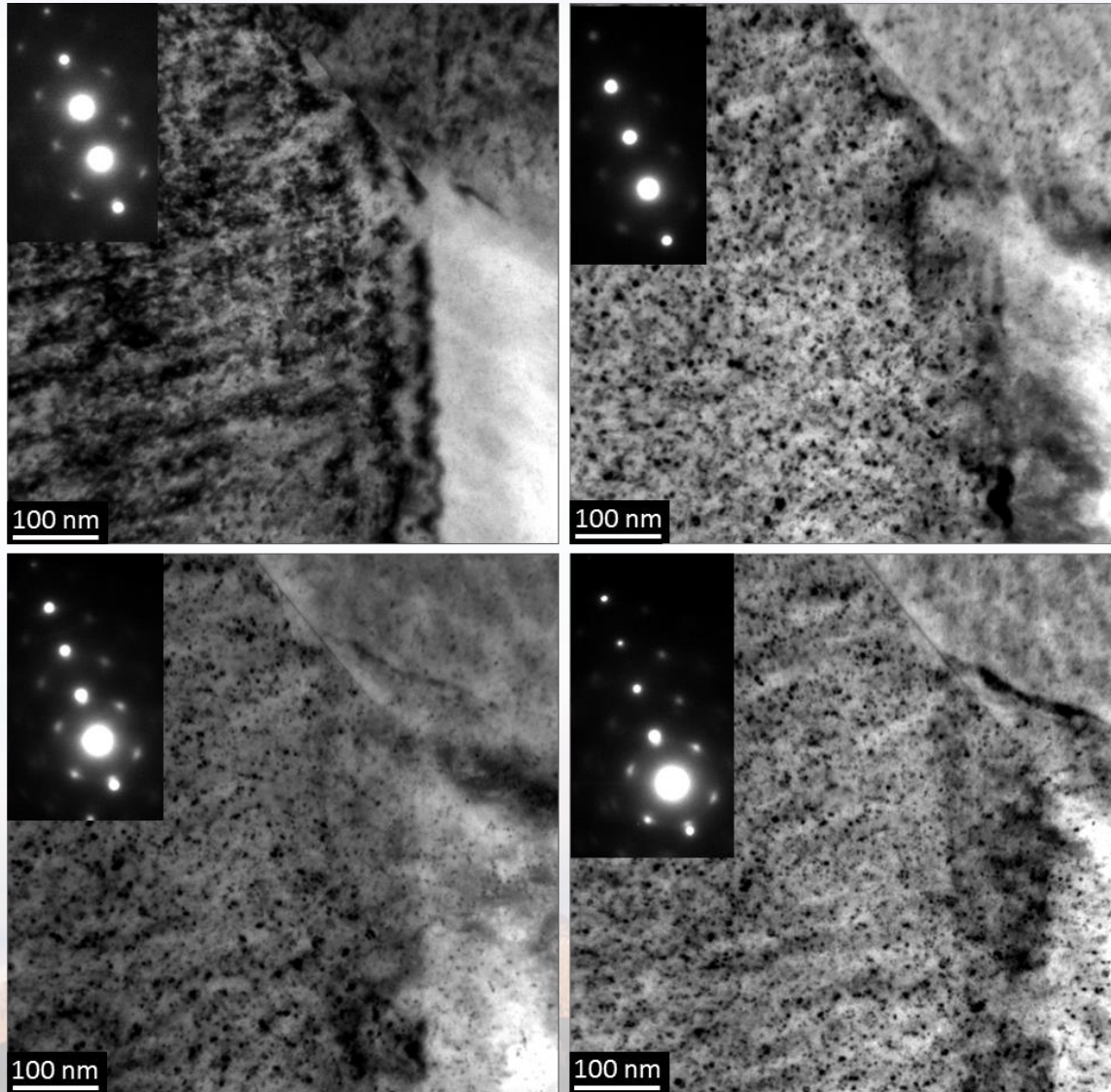
Electron Beam Enhanced Oxide Growth



Self Ion irradiation and Deuterium Implantation



Tilting Effects on Loop Appearance





Summary

