SAND2021-11606C

Comparing Neutron and Ion Irradiation by Atom Probe Tomography and In-situ Electron Microscopy

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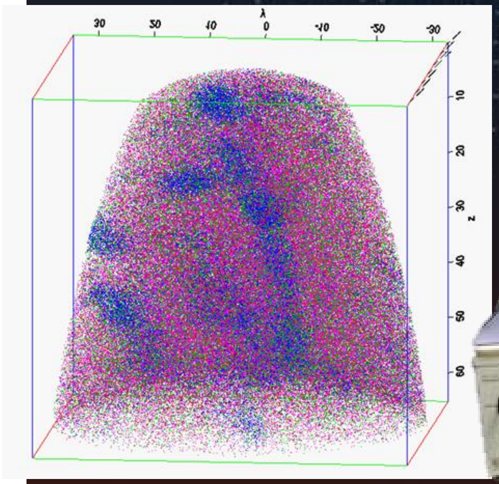

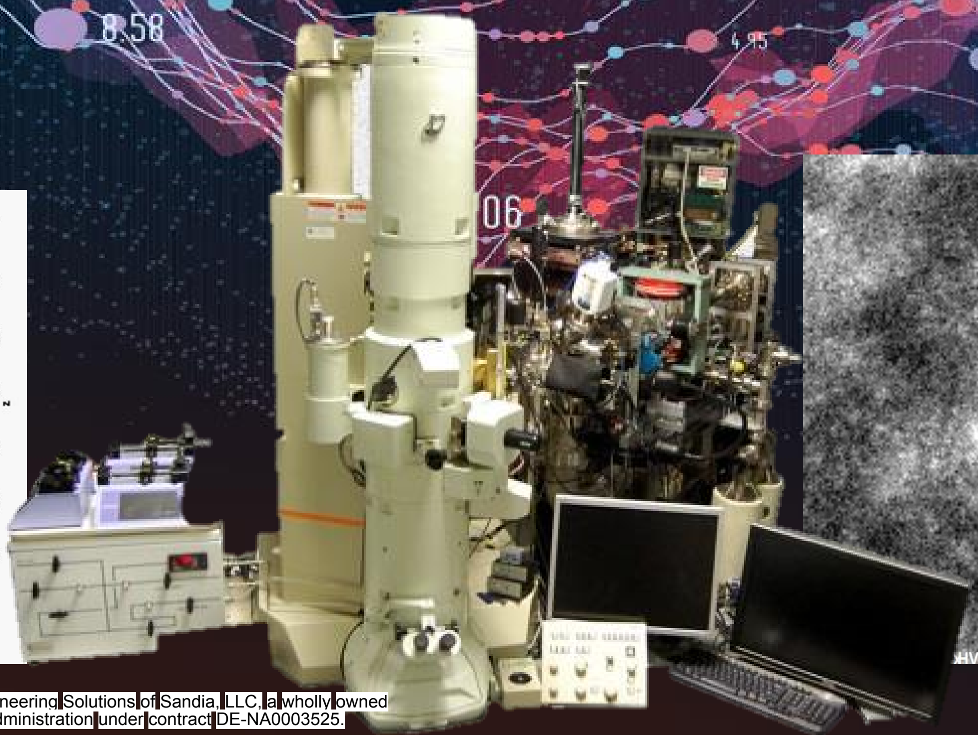

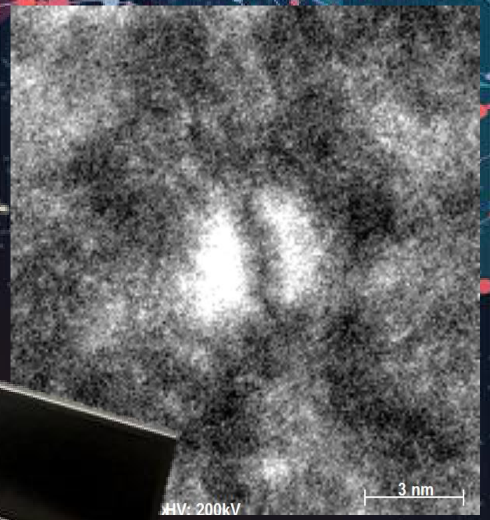

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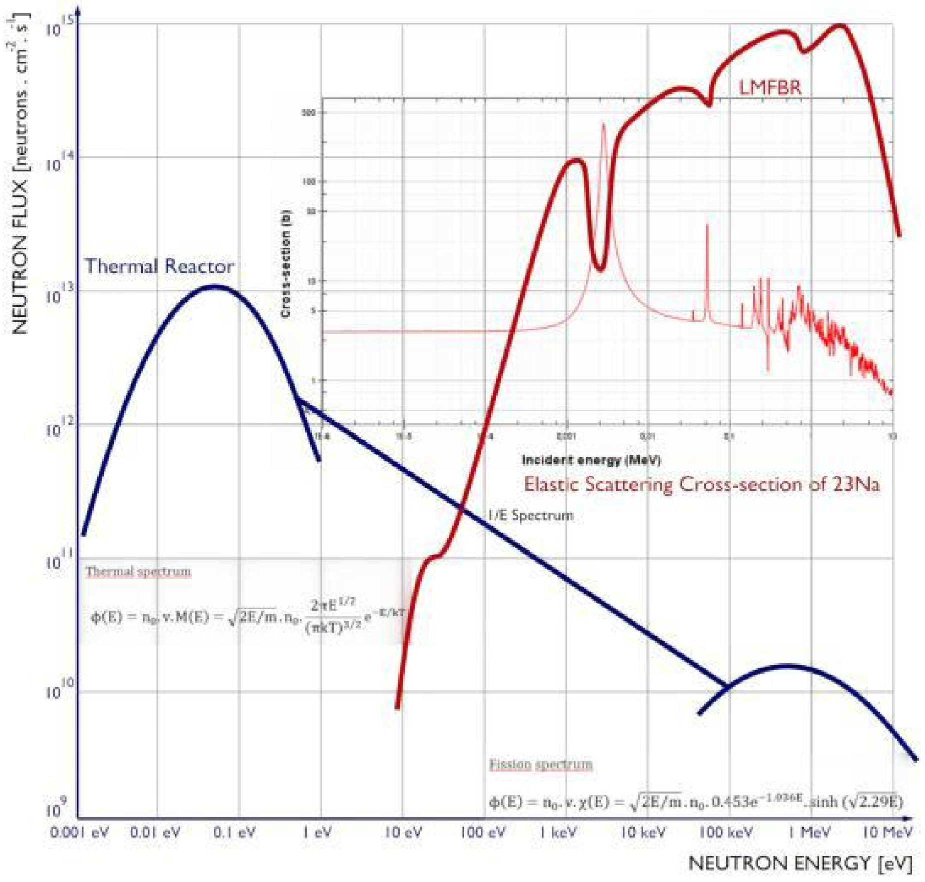
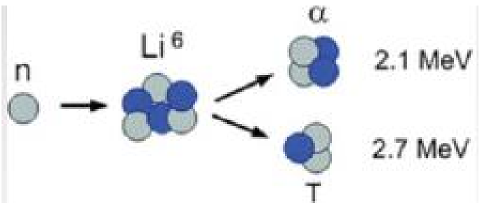
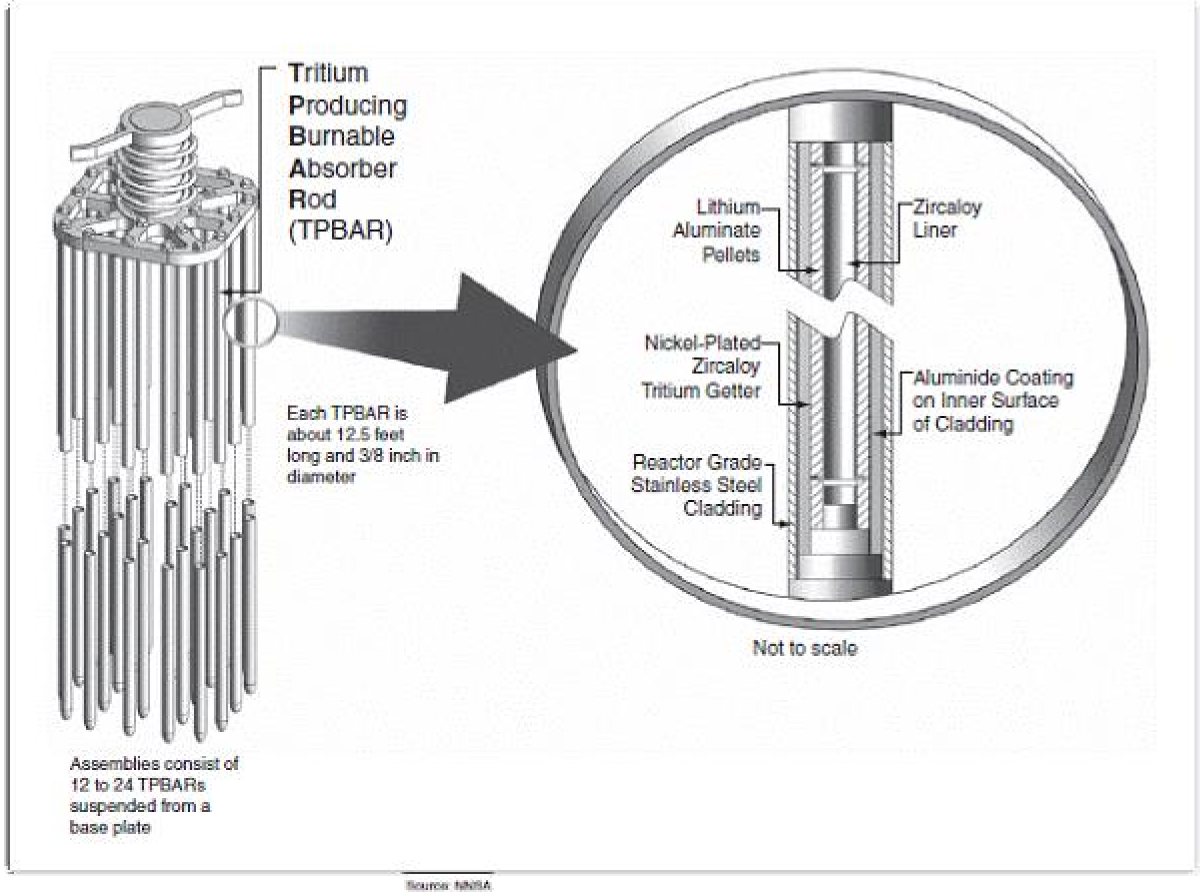
TPBAR Design and Reactor Environment

Tritium Producing Burnable Absorber Rod



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

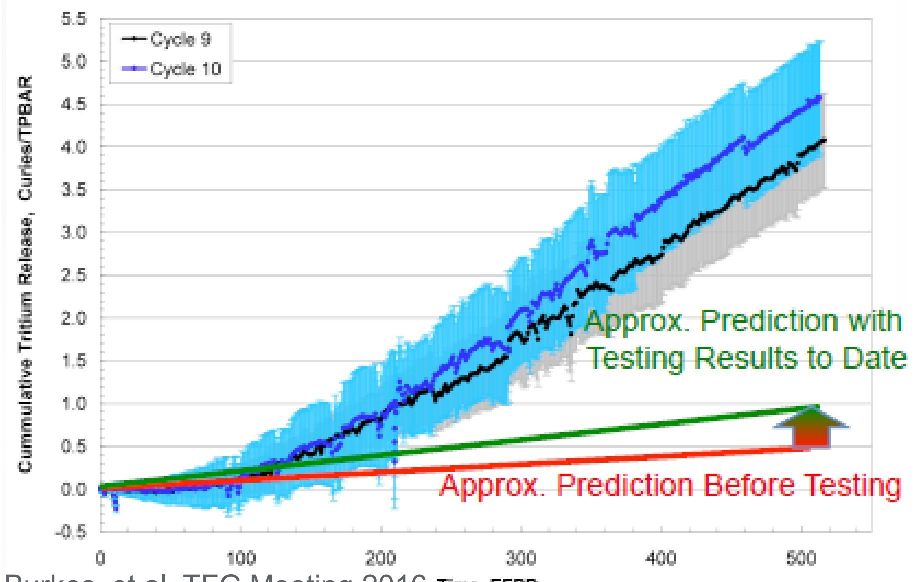




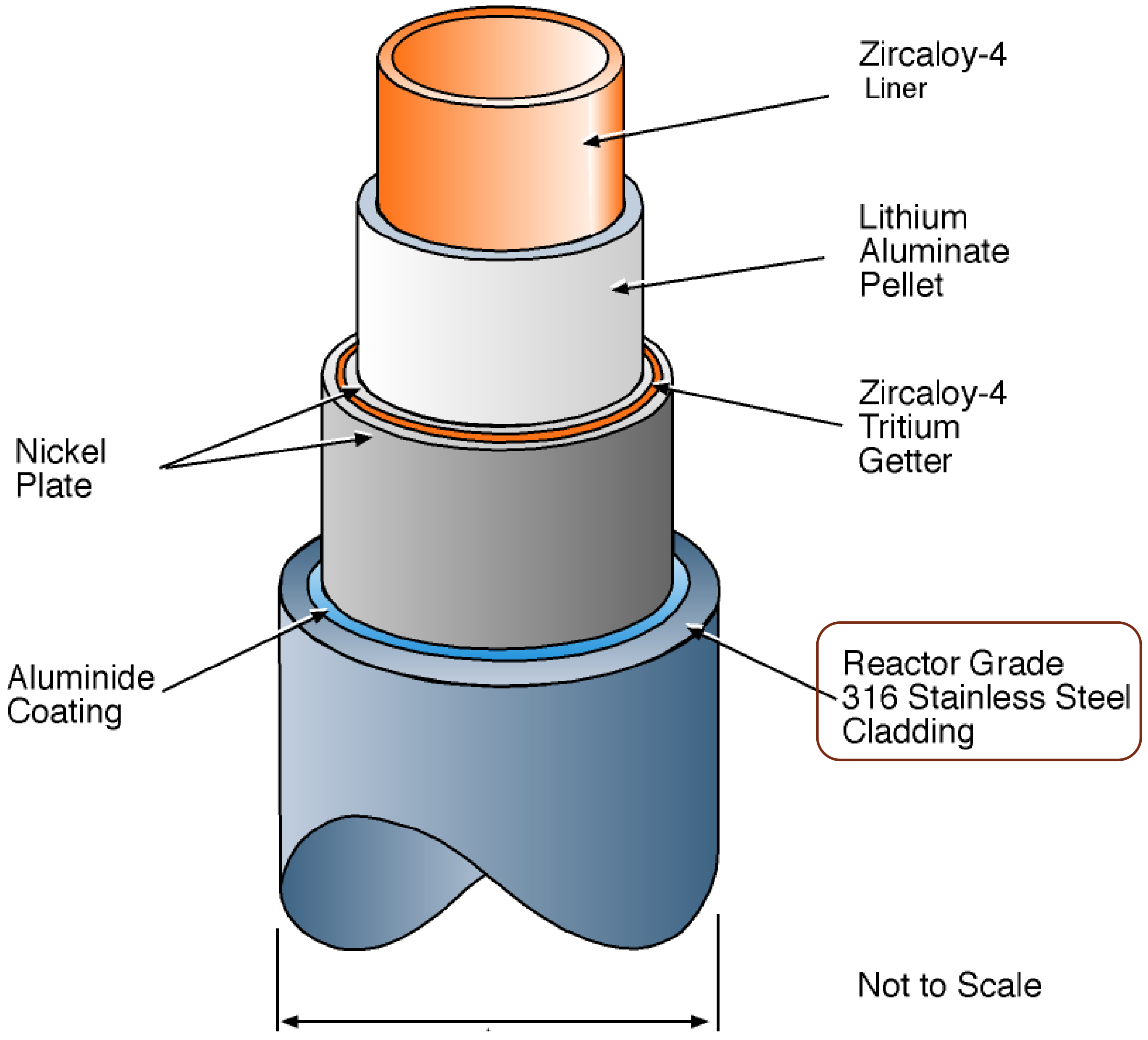
Extremely Complex Environment: Displacement Damage, Tritium Production, Helium Production, Mechanical Stress & Elevated Temperatures

Understanding ^3H evolution in TPBAR components

- Mechanisms responsible for differences between predictions and observations are not well understood
- Currently building an understanding of fundamental ^3H-He-defect interactions



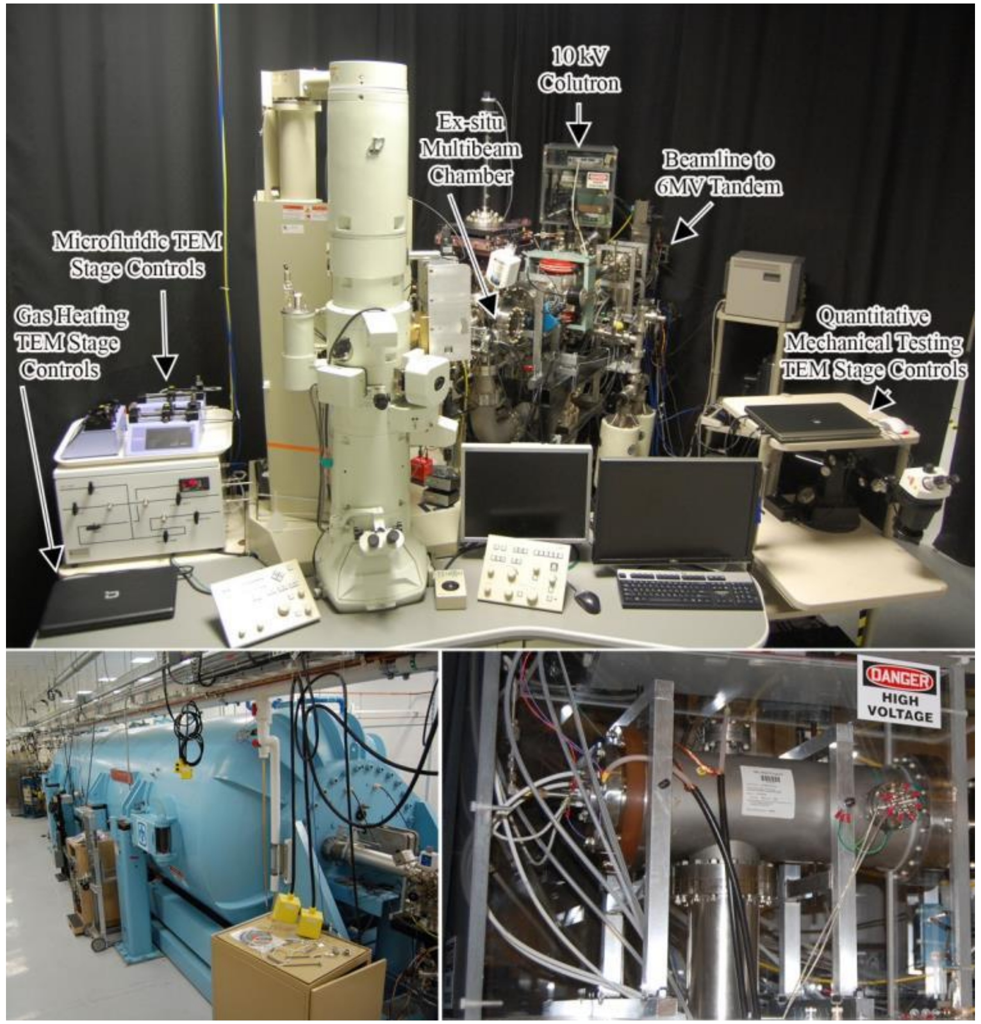
Burkes, et al. TFG Meeting 2016



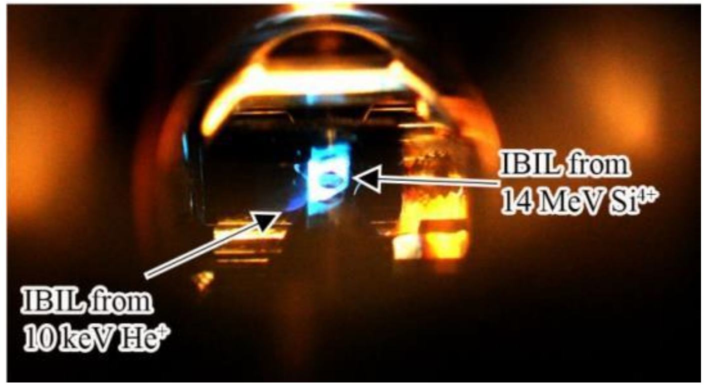
Simulating neutron irradiation in a reactor is complicated, and TPBAR adds the additional complication of ^3H production

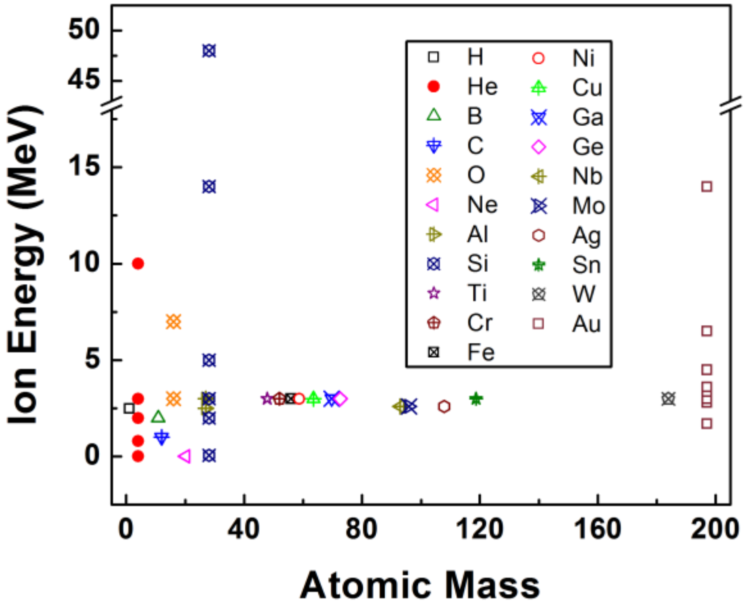
Sandia's Concurrent *In situ* Ion Irradiation TEM (I3TEM) Facility

10 kV Colutron - 200 kV TEM - 6 MV Tandem



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





Capabilities

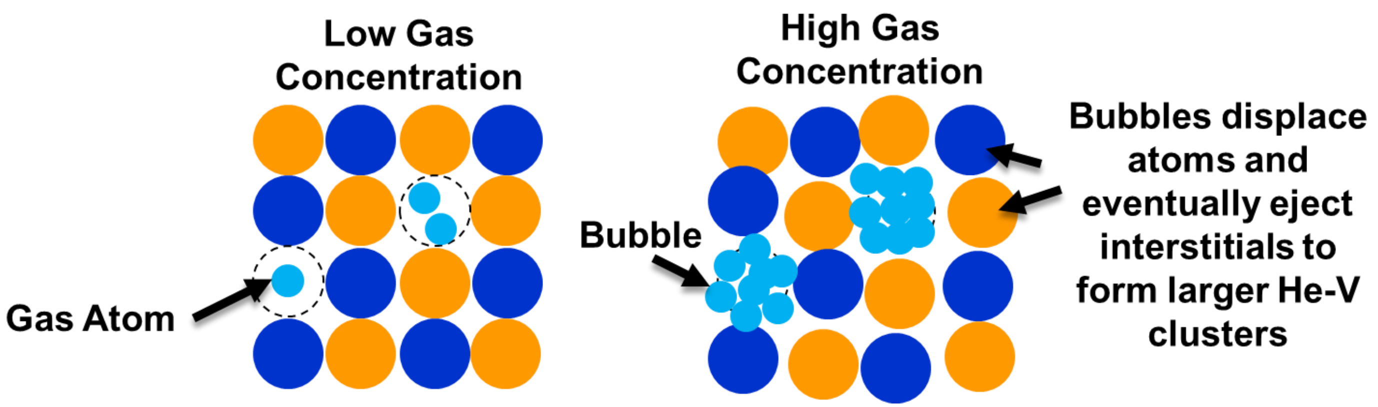
- 200 kV LaB_6 TEM
- Ion beams considered:
  - Range of Sputtered Ions
  - 10 keV D^{2+}
  - 10 keV He^{+}
- All beams hit same location

- Nanosecond time resolution (DTEM)
- Procession scanning (EBSD in TEM)
- *In situ* PL, CL, and IBIL
- *In situ* vapor phase stage
- *In situ* liquid mixing stage
- *In situ* heating
- Tomography stage (2x)
- *In situ* cooling stage
- *In situ* electrical bias stage
- *In situ* straining stage (3x)

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

Bubble formation in material may affect ^3H release

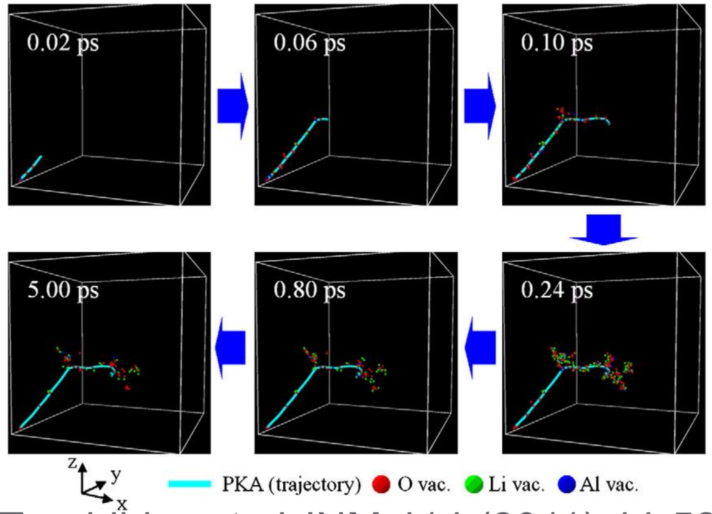
- Bubbles form due to He trapping in lattice defects



Heavy ion irradiation introduced displacement cascades from neutrons, but need to combined H and He irradiation to evaluate synergistic effects

- Neutron irradiation produces displacement cascades, providing complex defect structures for He or ^3H to trap in.

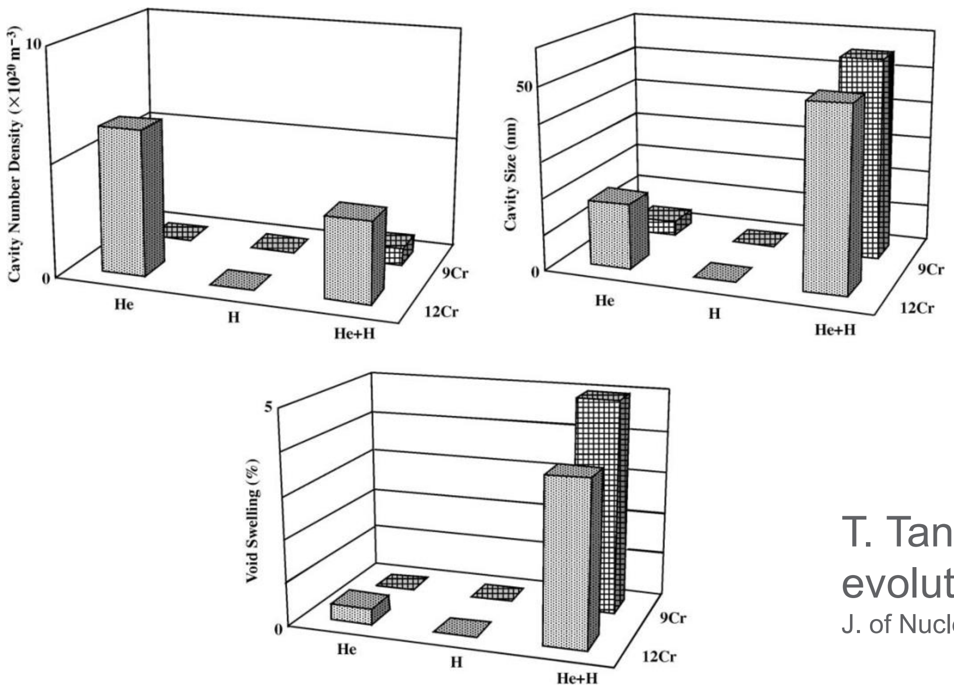
MD simulation of displacement cascade in $LiAlO_2$ (PKA = 5 keV)



Tsuchihira *et al* JNM 414 (2011) 44-52

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

Exploring triple beam synergistic effect with in-situ techniques

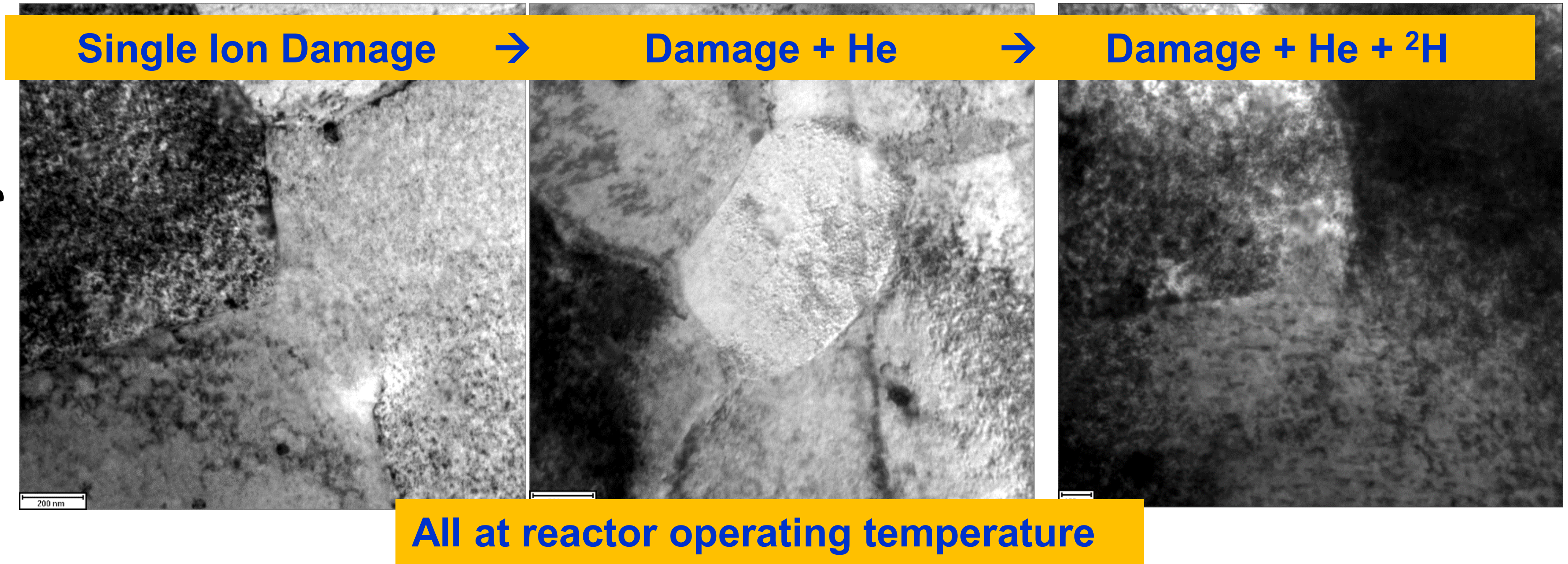


Coupling Effect

- 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

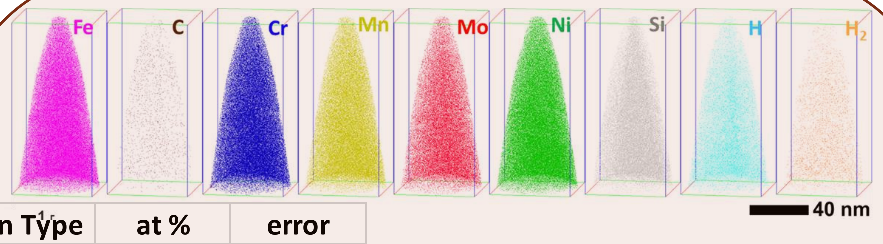
T. Tanaka et al. "Synergistic effect of helium and hydrogen for defect evolution under milt-ion irradiation of Fe-Cr ferritic alloys"
J. of Nuclear Materials 329-333 (2004) 294-298

Zr Alloys



Direct real time observation of heavy ion irradiation, and low energy D and He ion implantation with nanometer resolution

316 SS undergoes microstructural changes under neutron irradiation



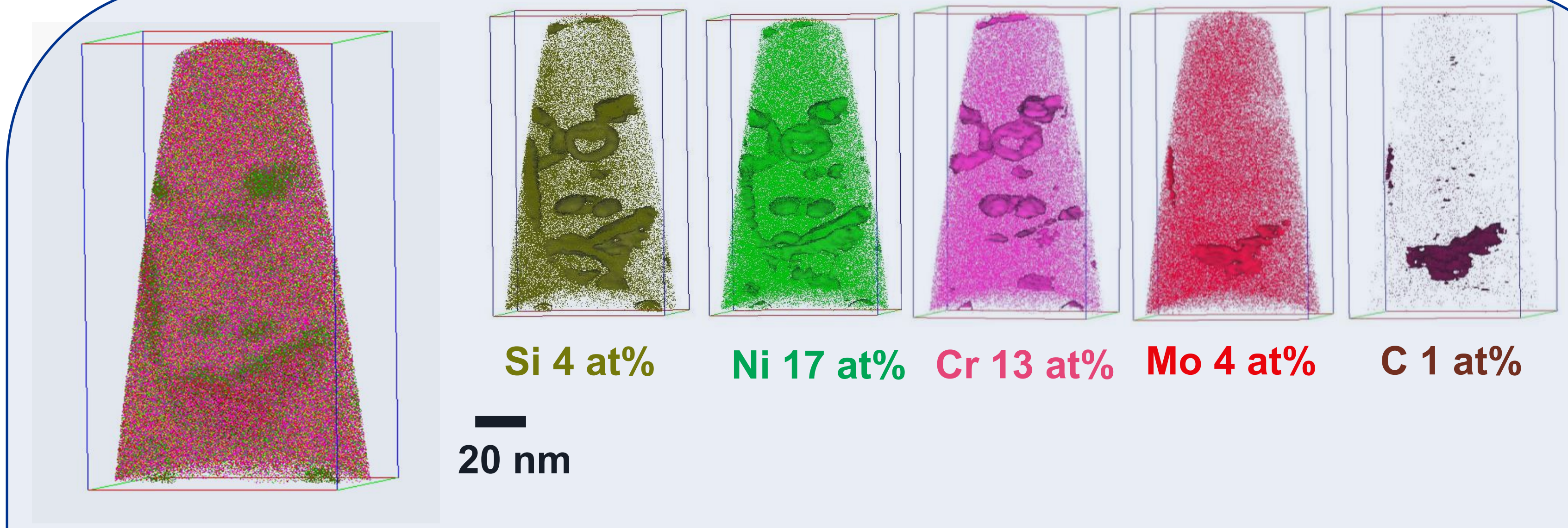
Ion Type	at %	error
Fe	65.407	0.054
Cr	18.124	0.024
Ni	11.430	0.018
Mn	1.656	0.007
Si	1.271	0.006
Mo	1.208	0.006
H	0.812	0.005
C	0.066	0.001
P	0.028	0.001

APT shows uniform distribution of all solute elements and impurity element distribution.

Pre-Irradiation

Neutron Irradiation Parameters

- **Neutron energy?**
- 5 dpa
- 300 C

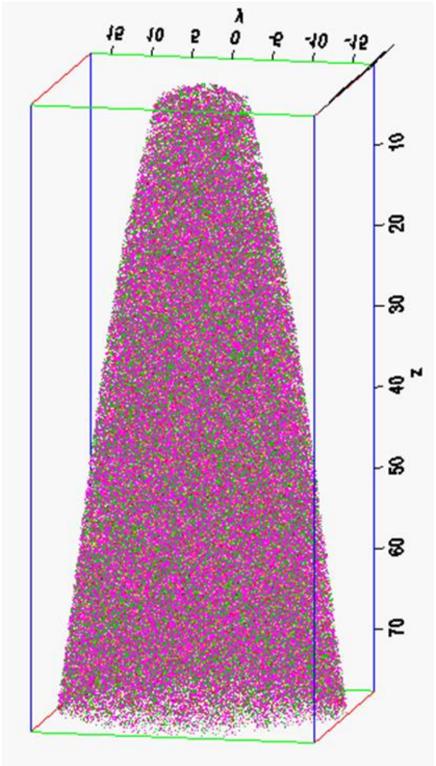


- Significant solute partitioning observed
- Ni-Si enrichment to dislocation loops corresponding Cr depletion
- Mo and C enriched regions observed

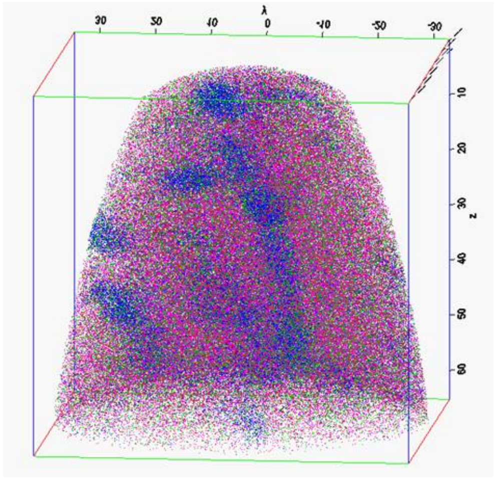
Post-Irradiation

Summary of Irradiated Stainless Steel Cladding

- Very clear segregation of elements observed
- Ni-Si enrichment to what appears are precipitates and dislocation loops with associated Cr depletion
- Mo enrichment and some isolated C enriched regions captured in the reconstruction



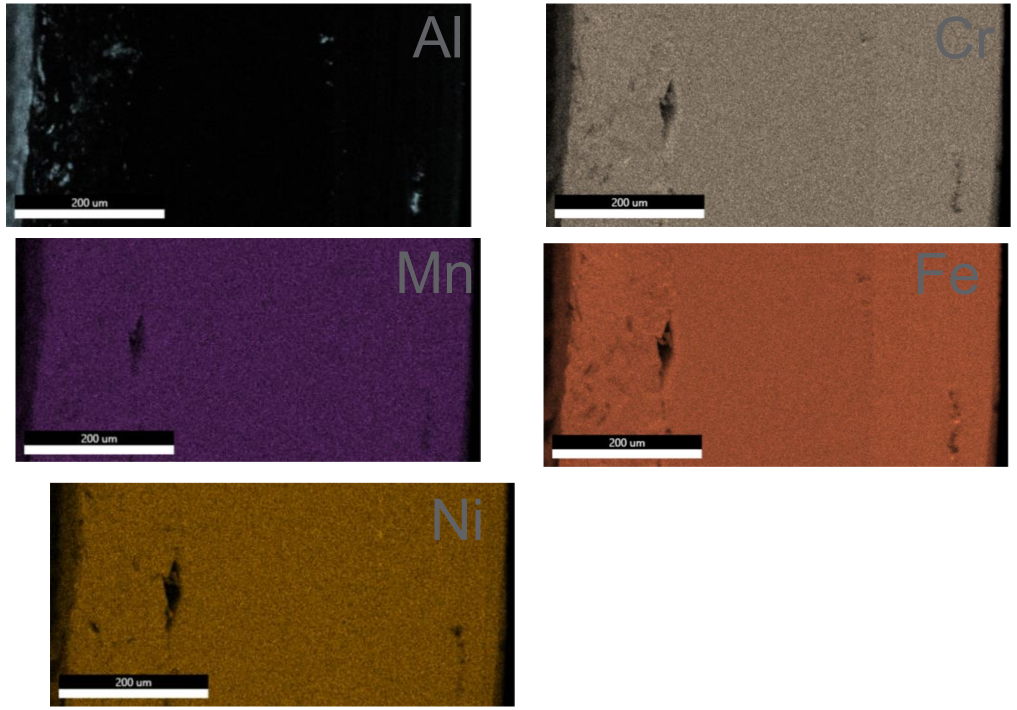
Can we mimic this damage using ion irradiation?

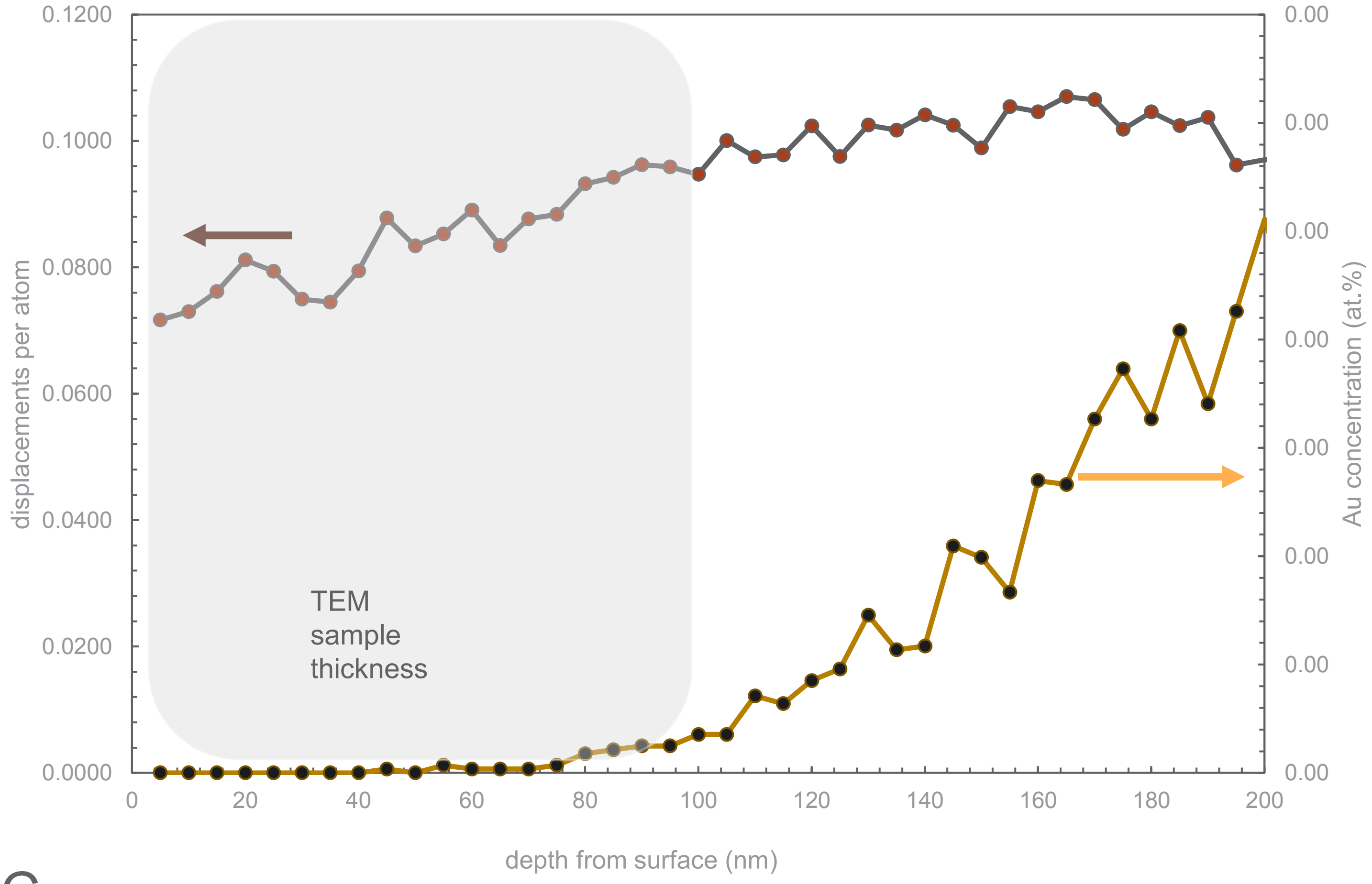


Unirradiated **irradiated**

In-situ TEM experiments to elucidate precipitation mechanisms in simulated reactor studies

Heavy ion irradiation as a surrogate for reactor irradiation allows for in-situ microstructure examination

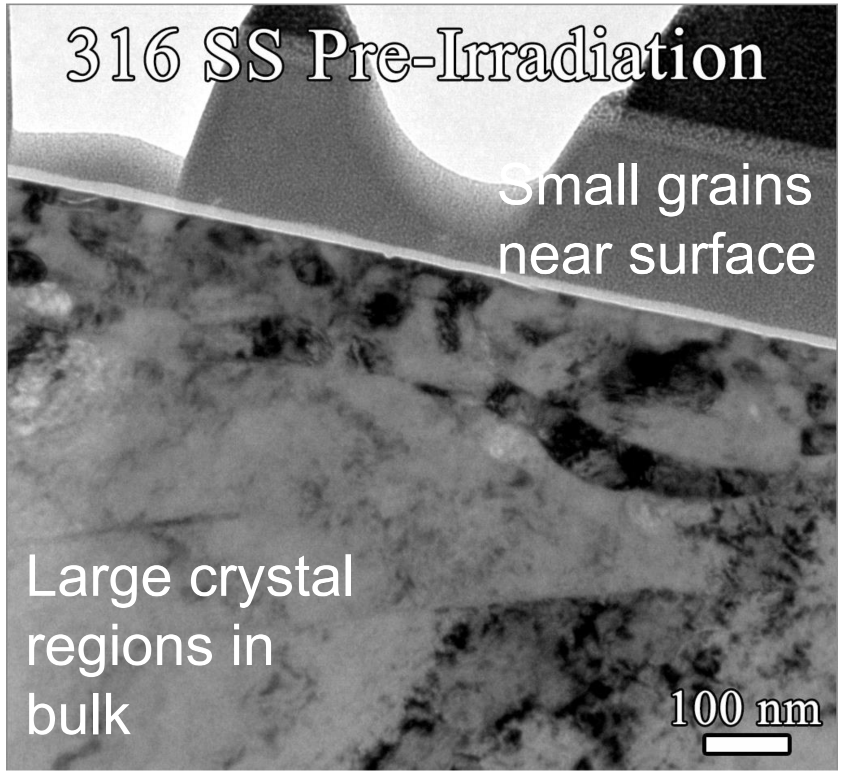




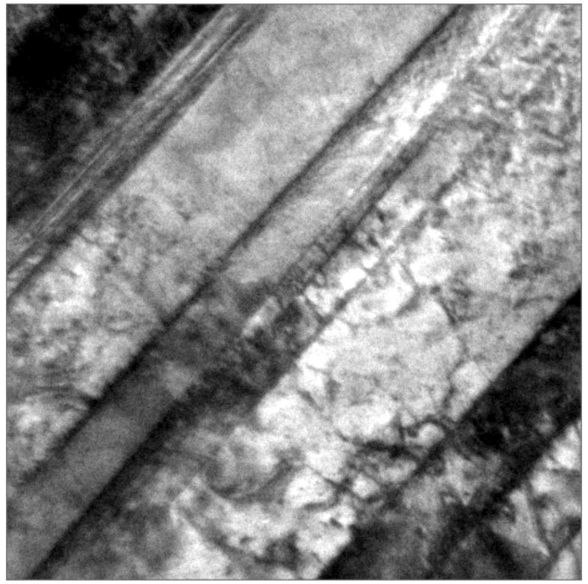
- In-situ heating for room temperature and 500°C irradiation
- Minimal Au ion implantation in the TEM sample and uniform displacement damage throughout

Left: Schematic of I3TEM at SNL for in-situ irradiation studies
Right: SRIM simulation of damage levels of 2.8 MeV Au^{+} ions into 316 SS. Shaded area represents TEM lamella thickness, indicating nearly uniform damage profile

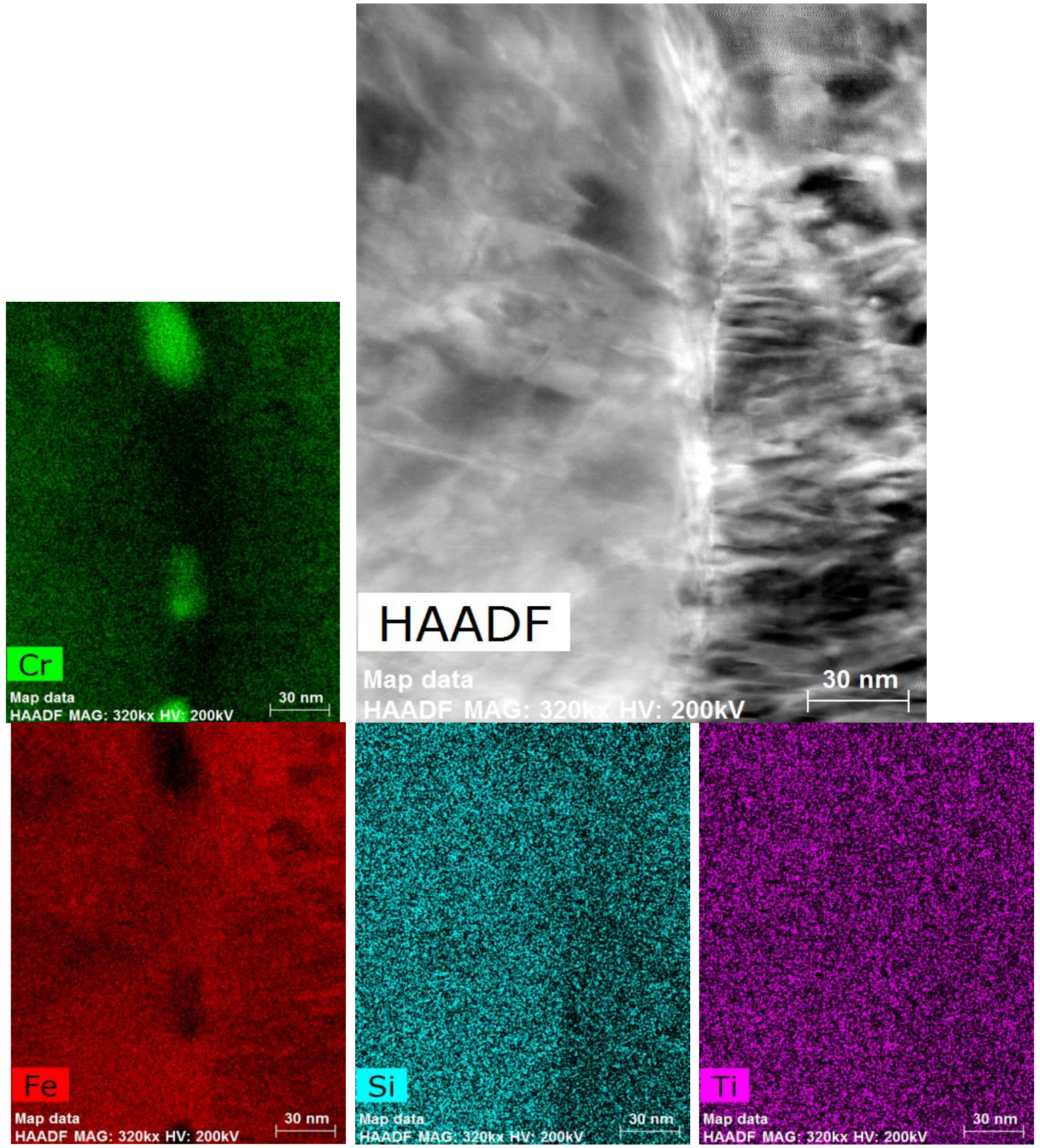
Pre-irradiation TEM shows grain boundary segregation and numerous boundaries of interest



BF-TEM showing grain structure before heavy ion irradiation

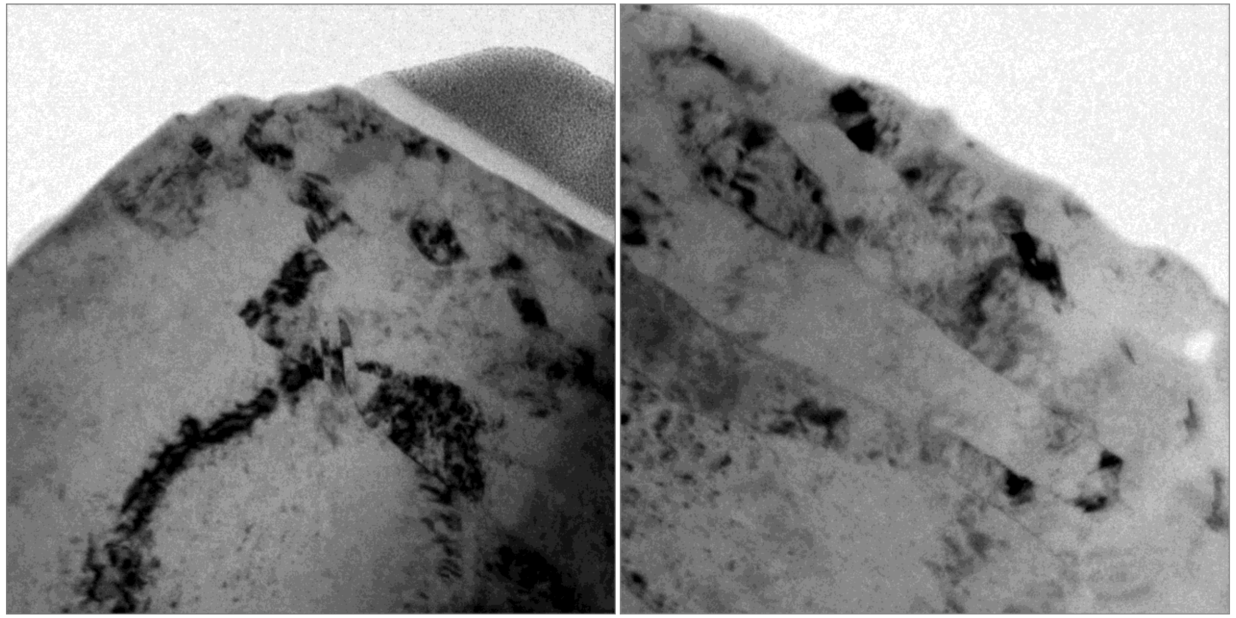


Numerous twin and grain boundaries present in the TEM samples

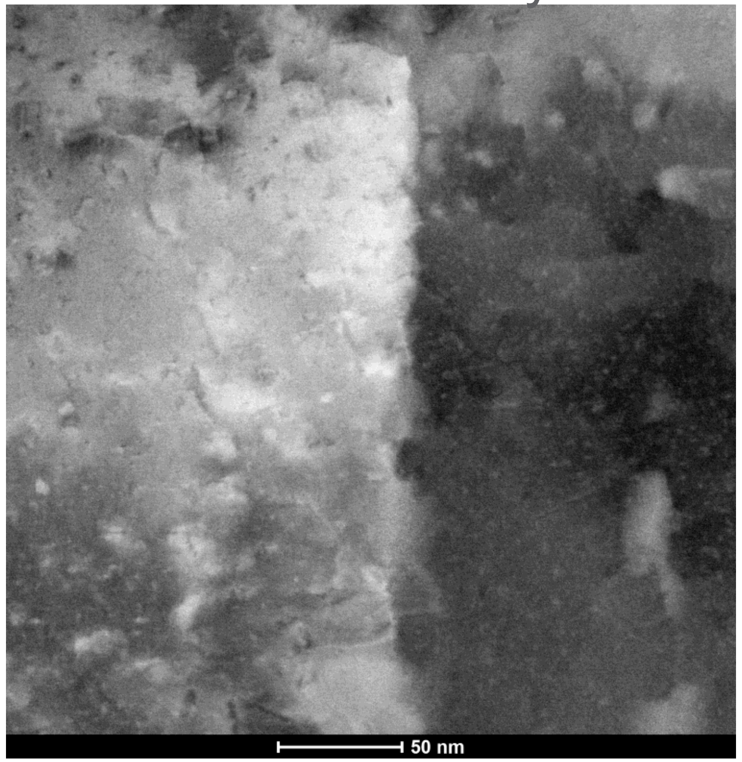


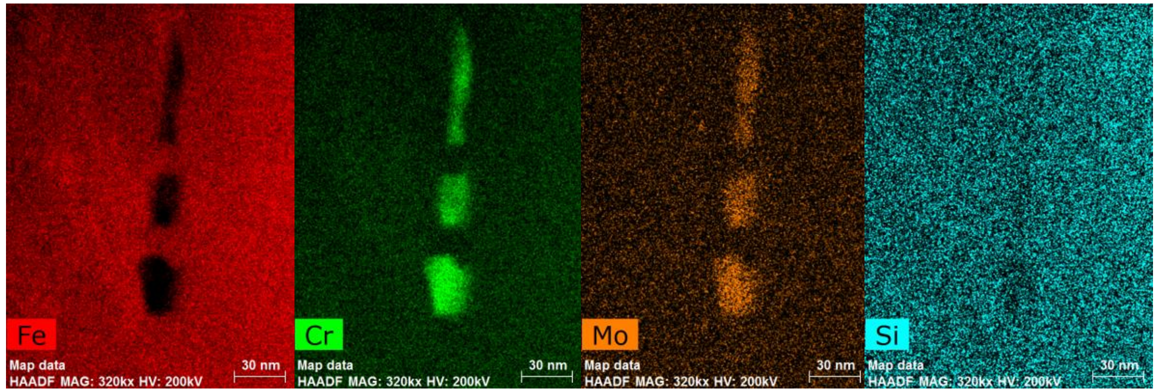
No chemical gradients observed at twin boundaries only grain boundaries

Initial in-situ experiments at ~30 C, 2.8 MeV Au show no radiation-induced segregation

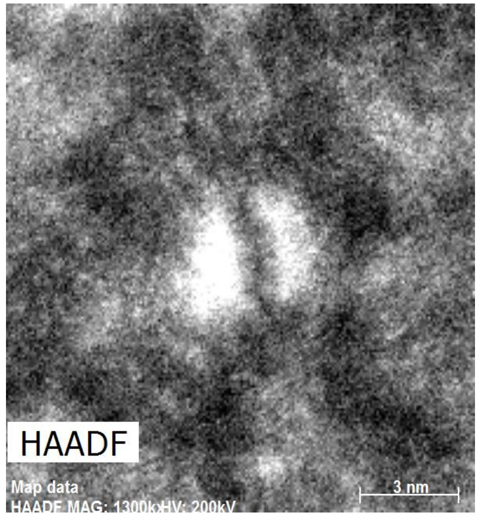


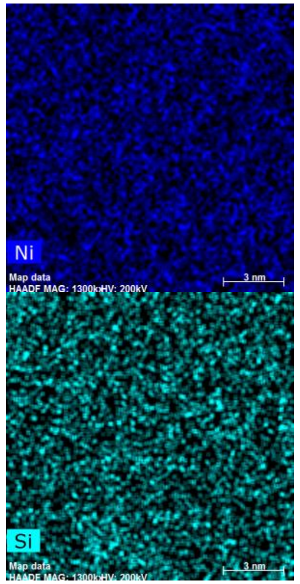
Grain Boundary





Dislocation Loop





- Dislocation loops form during irradiation
- No clear accumulation of defects at grain boundaries
- No void/bubble growth

- Still see Cr and Mo segregation at GBs as in pre-irradiation, but no Ni-Si accumulation at dislocation loops

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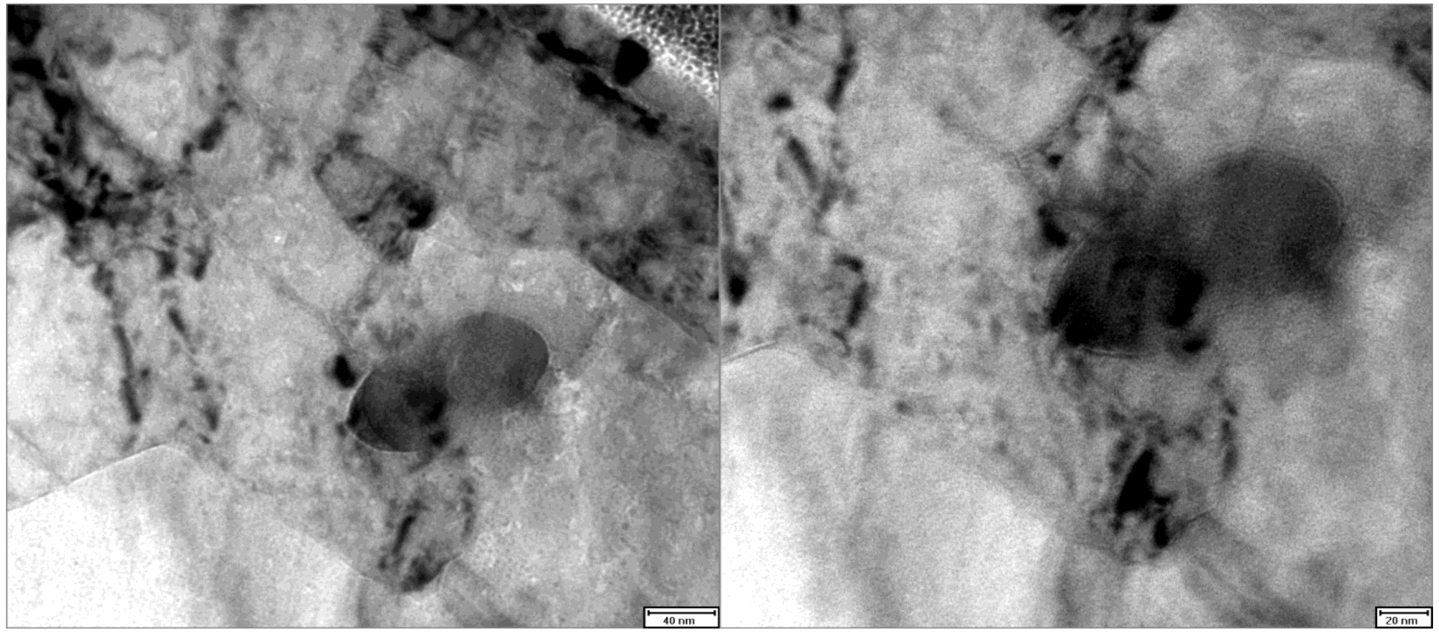
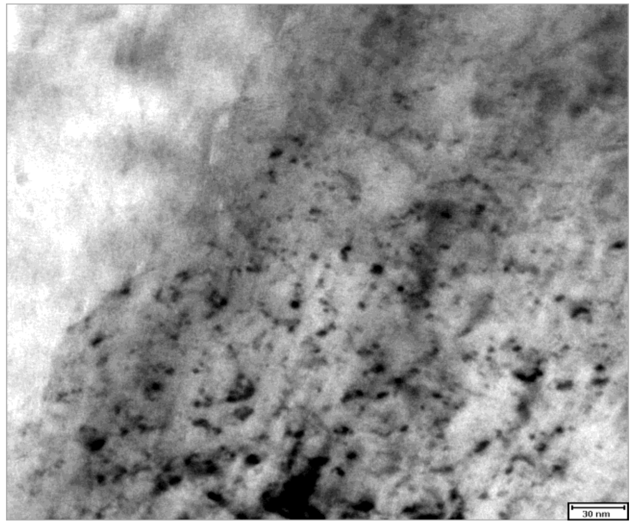
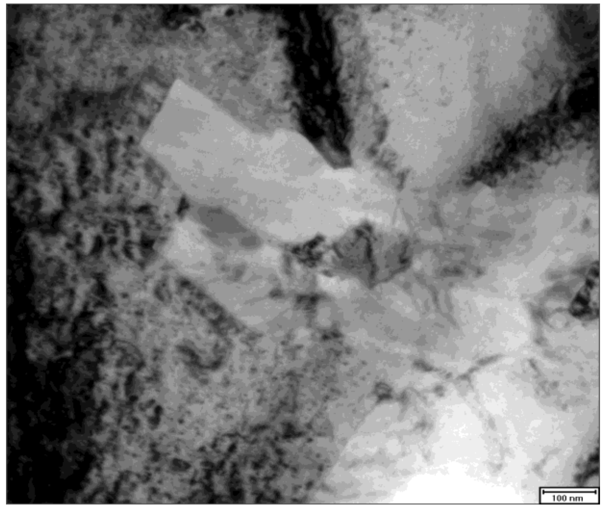
In-situ irradiation at 400 C to elucidate microstructural changes



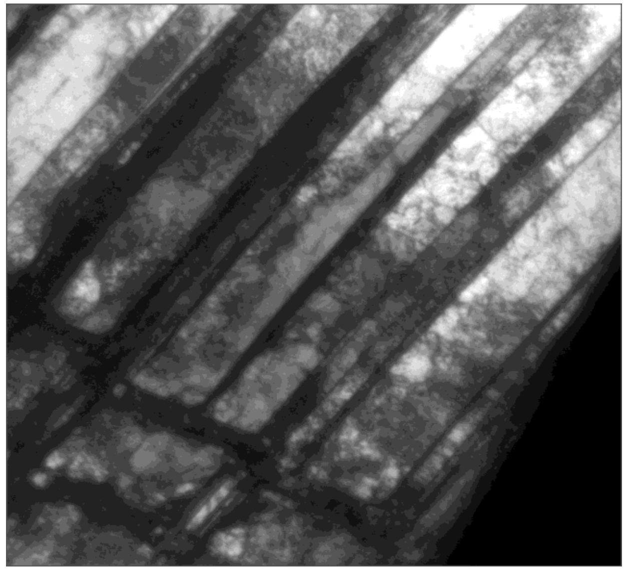
Investigating defect accumulation around nano-scale Ti precipitate within microstructure

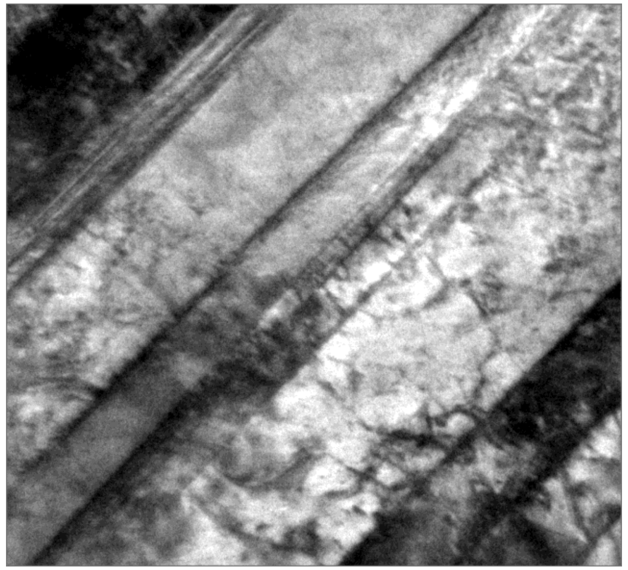


Defect loop accumulation due to Au bombardment



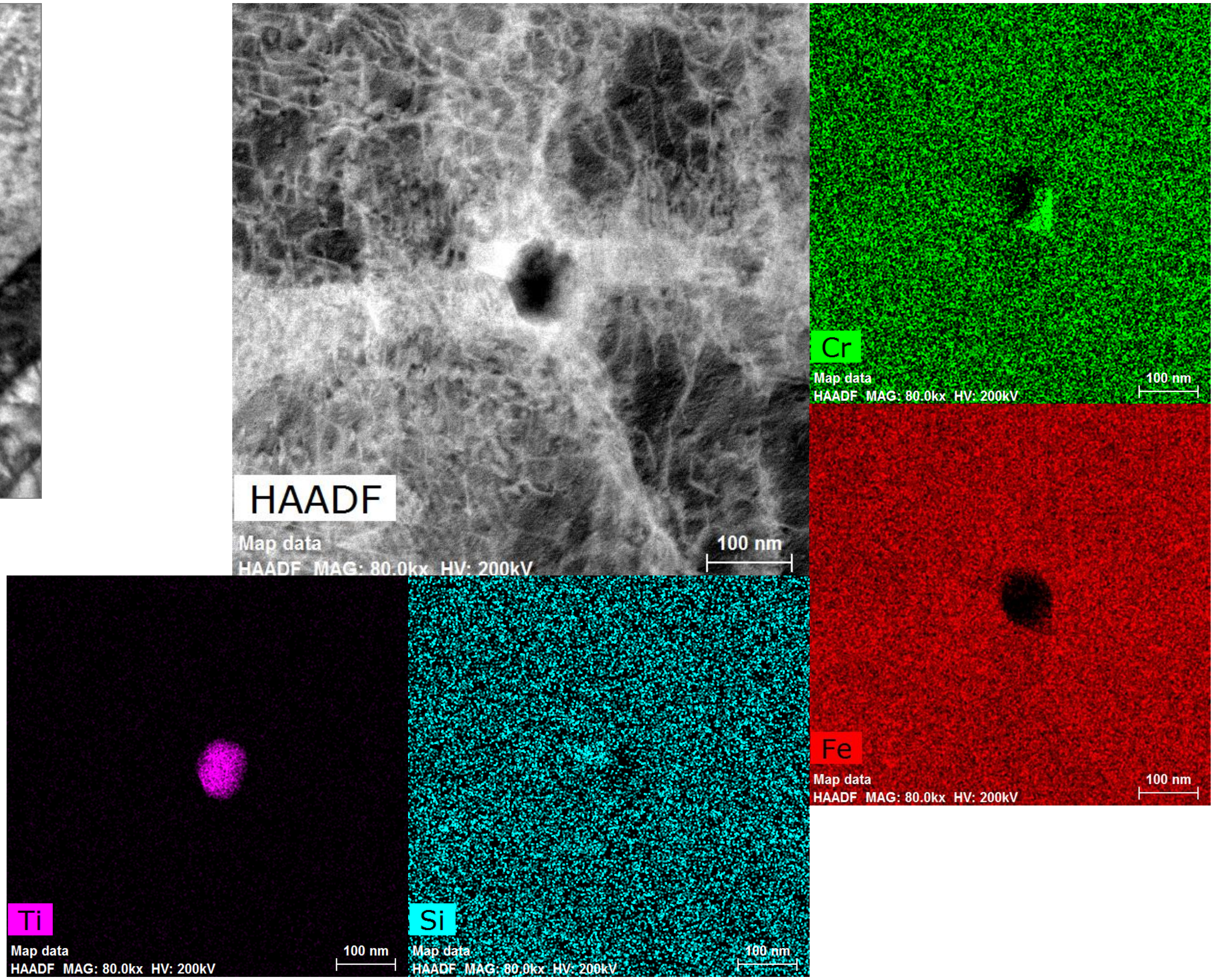
Dual-beam Au+He irradiation to better mirror reactor environment irradiation conditions





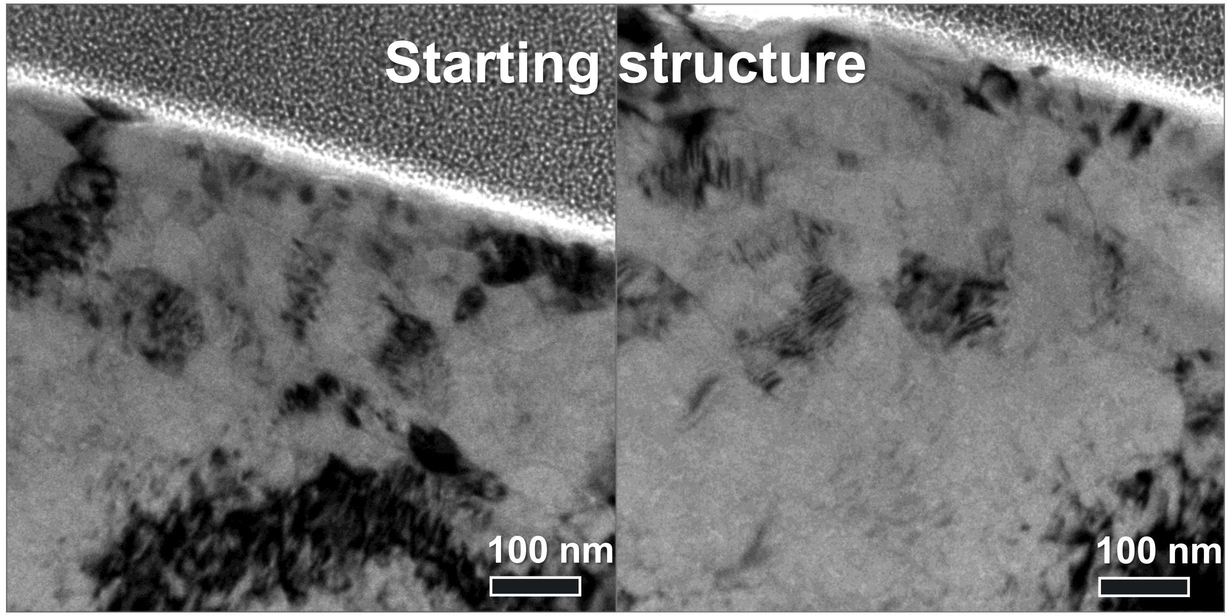
Subsequent follow up studies will investigate He bubble formation after time delay

Studying He bubble accumulation at Ti precipitates within microstructure

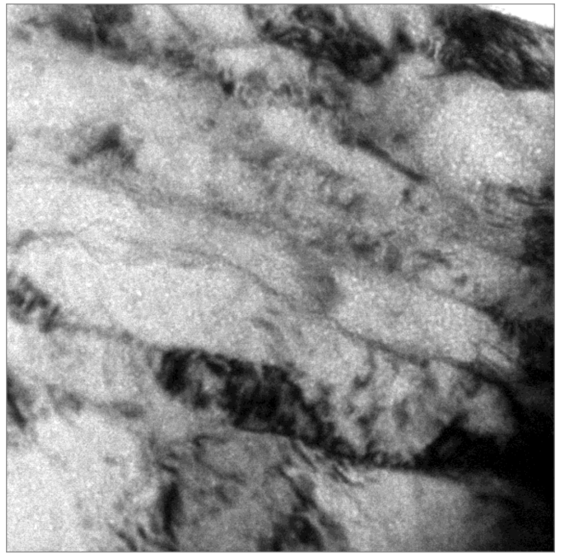


Elemental segregation at GBs comparison to unirradiated sample

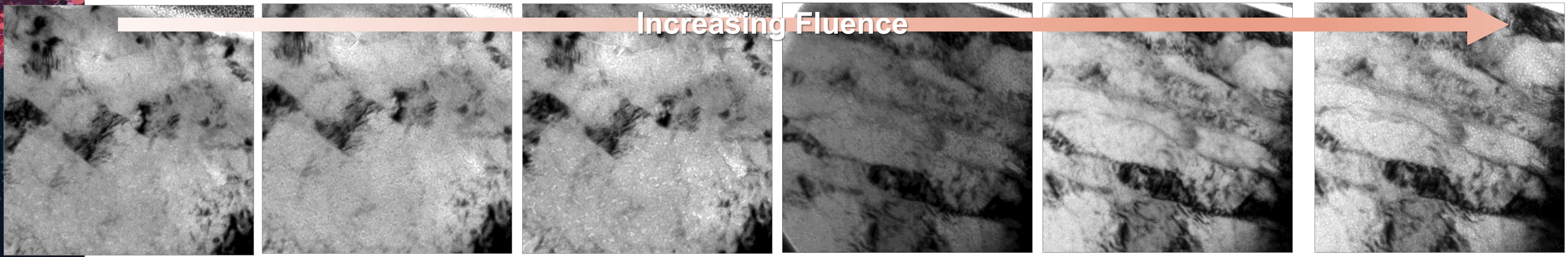
Initial in-situ experiments at 400 C, 2.8 MeV Au shows microstructural changes as fluence increases



2E14 cm^{-2} fluence



Development of features within grains as irradiation progressed



1E14 cm^{-2} fluence

2E14 cm^{-2} fluence

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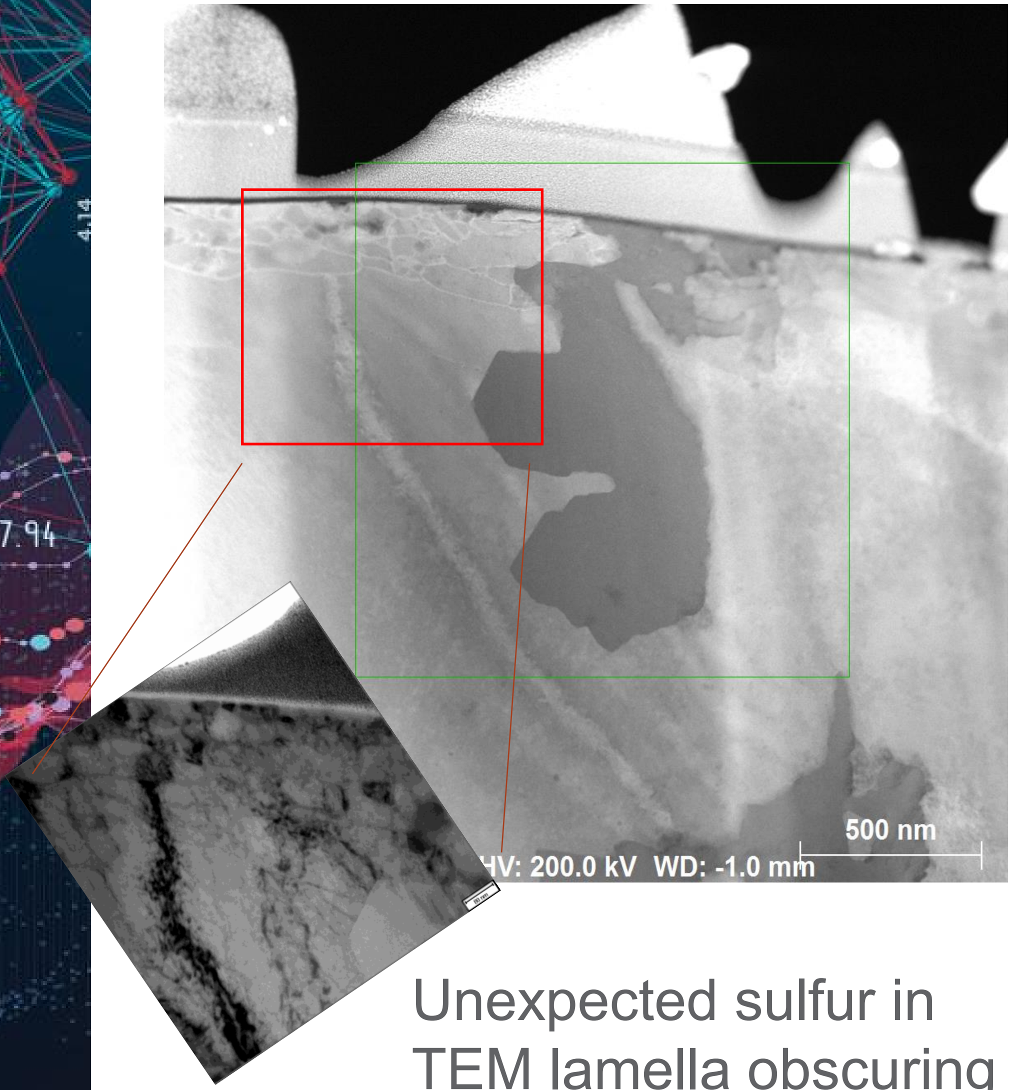
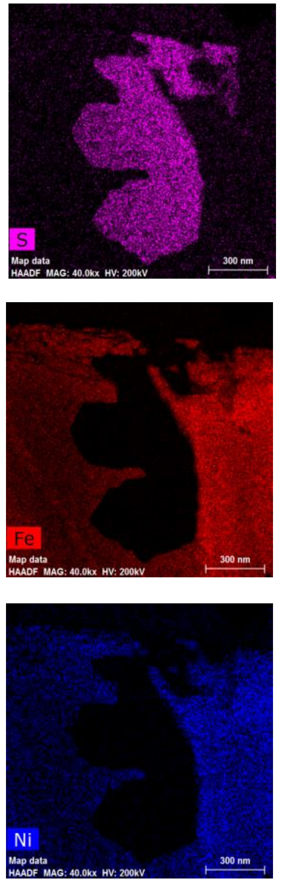
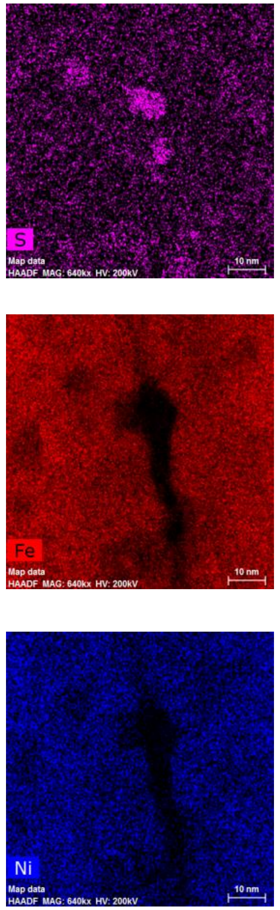
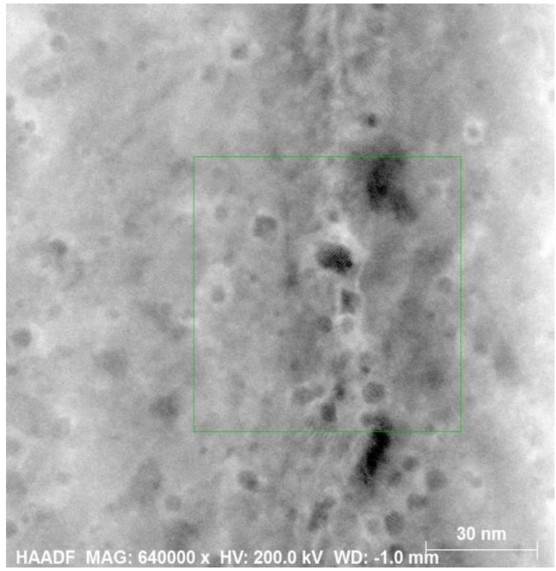
In-situ irradiation at 400 C shows sulfide formation at the micron and nano-scales

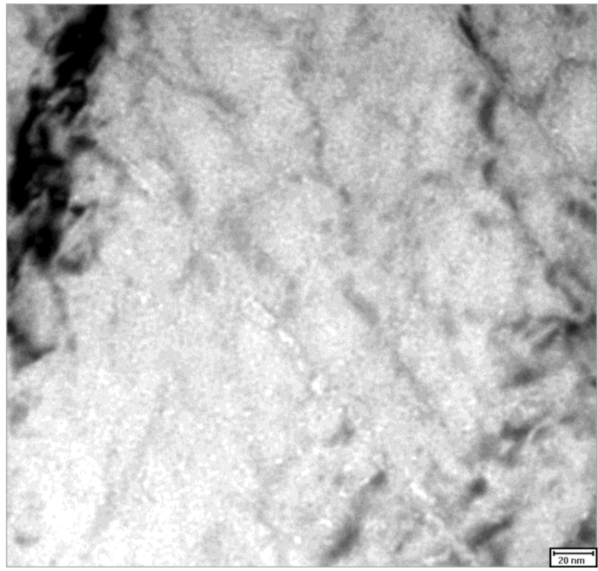




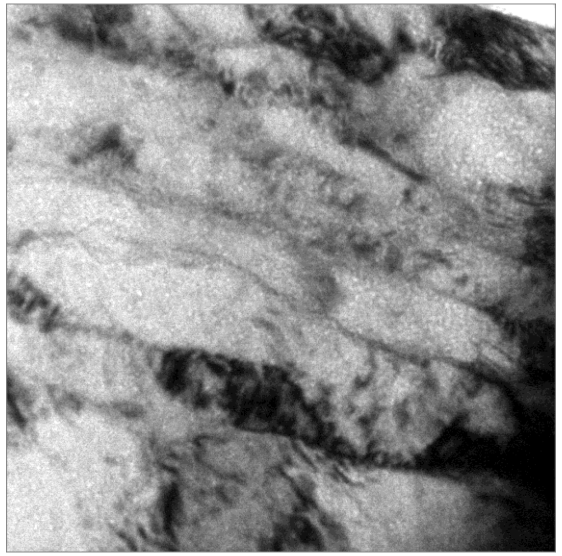
Unexpected sulfur in TEM lamella obscuring results, as unrepresentative of bulk sample



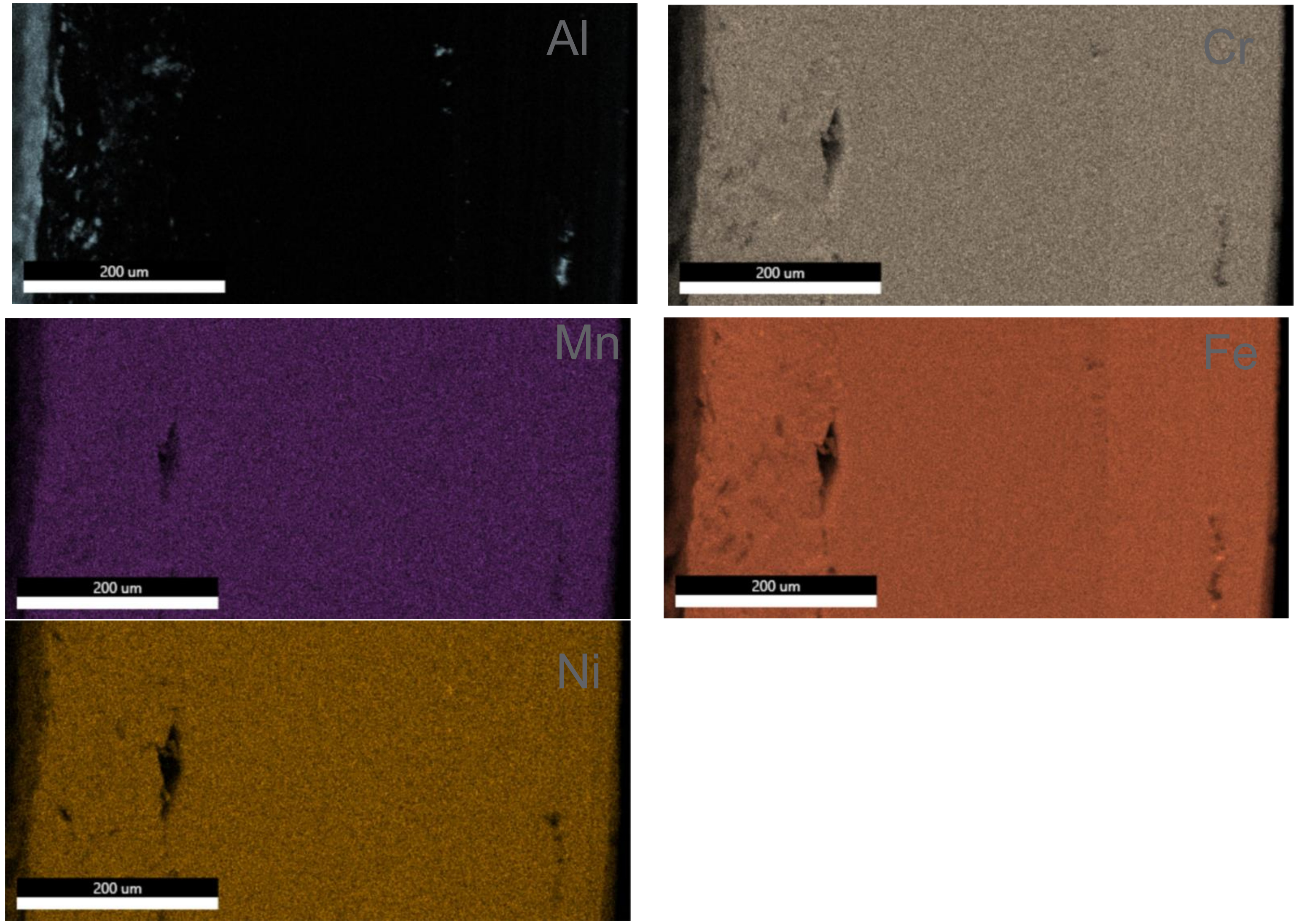




Some features observed in BF-TEM micrographs are sulfides, some are voids



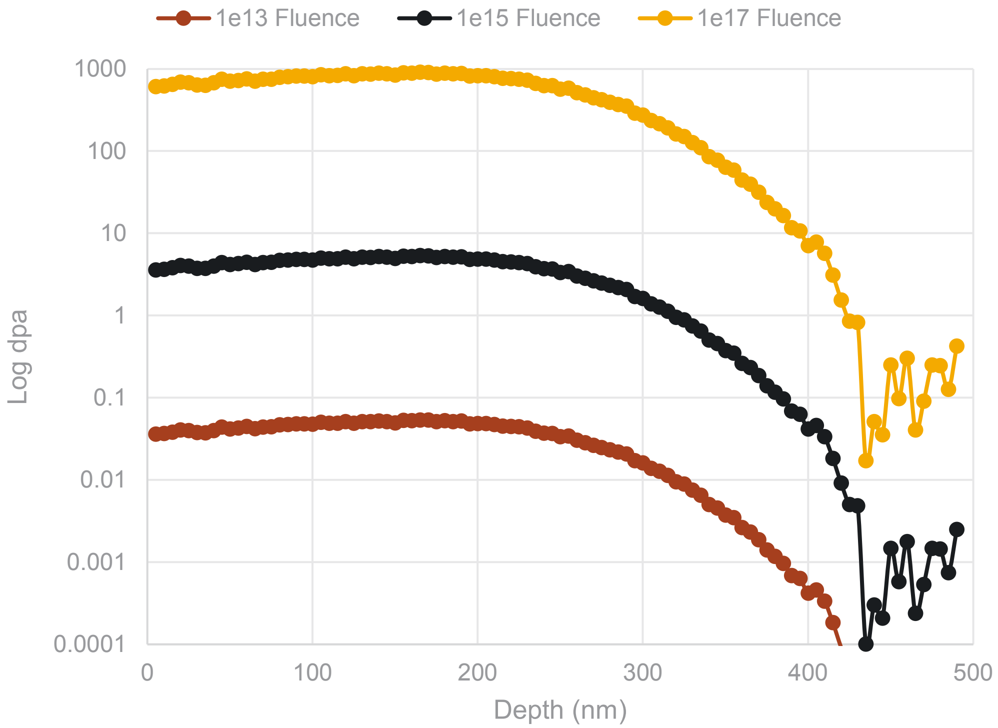
In-situ irradiation previously at 400 C showed nanosulfide development, not observed on micro-scale



No sulfur observed on SEM-EDS analysis, but sulfur concentration less than 0.03 wt.% per materials spec, so would not show up on SEM-EDS

On-going work to investigate high dose irradiation

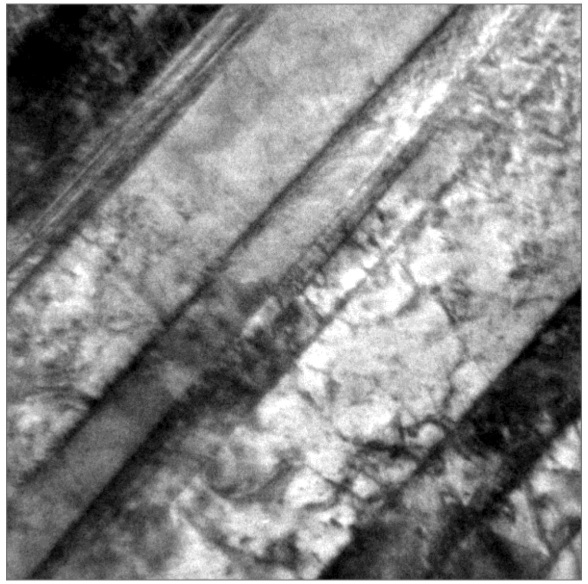
Experiments performed on bulk sample to reach higher doses, to achieve 5 dpa, albeit at room temperature

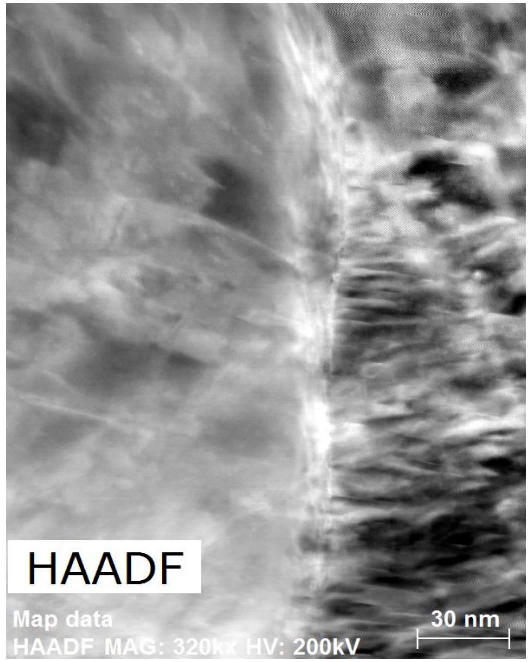


TEM and APT liftouts to be made on these samples to investigate effects of much higher ion irradiation dose

- 2.8 MeV Au, Room Temp.

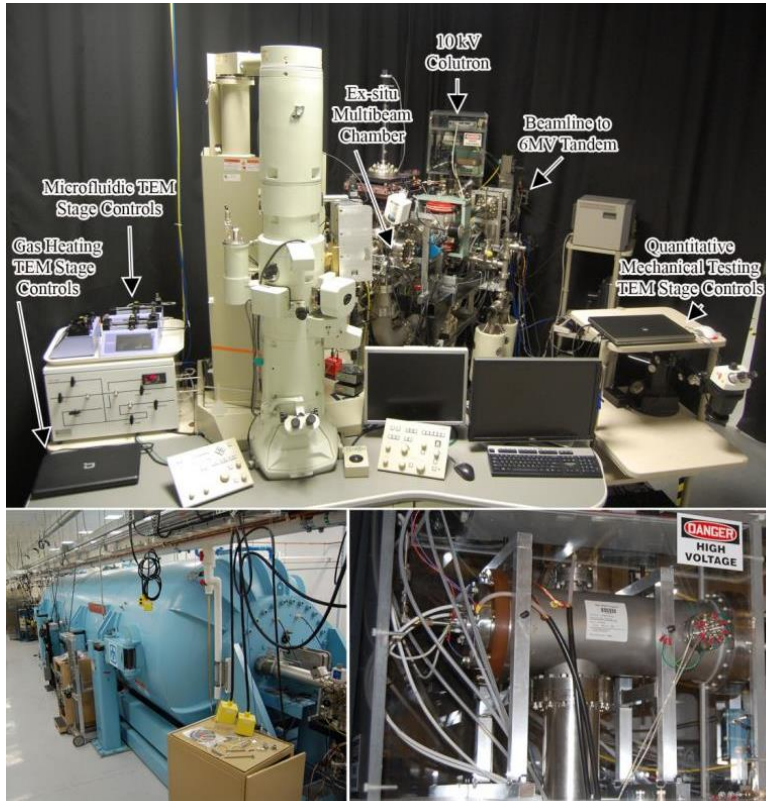
Future in-situ TEM experiments at 500 C while achieving higher fluences to better mimic 5 dpa damage level and not near a S-rich region



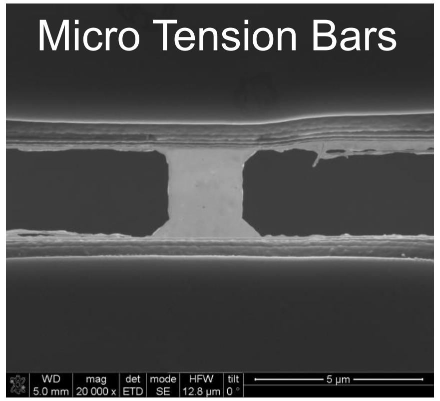


Future work to explore impacts of combined irradiation environments on mechanical properties

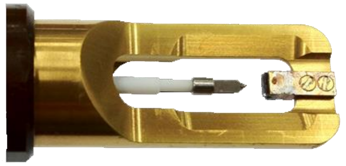
- Elucidating the mechanical properties of irradiated 316 SS through nano-scale mechanical tests
- Perform dual-beam irradiations to understand He implantation effects that better mimic neutron irradiation

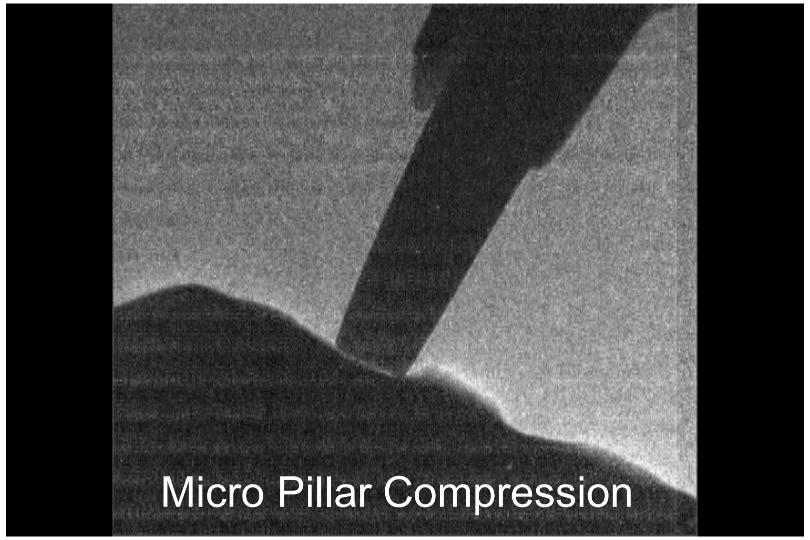


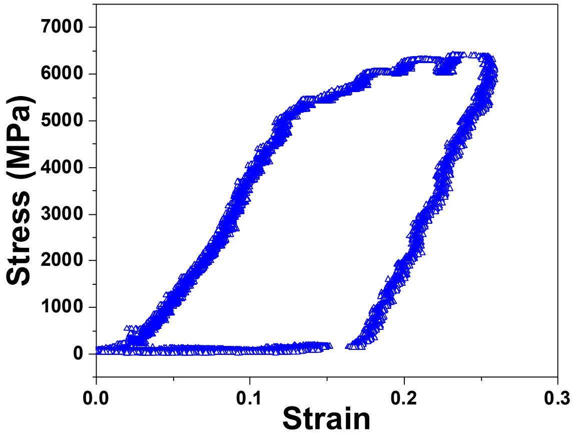
1) Indentation
2) Tension
3) Fatigue
4) Creep
5) Compression
6) Bend


Micro Tension Bars

Hysitron PI-95 Holder



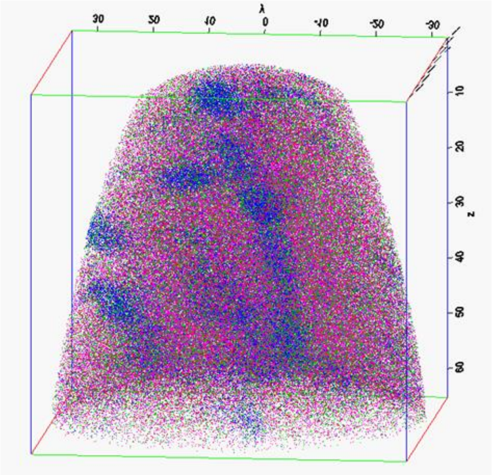

Micro Pillar Compression

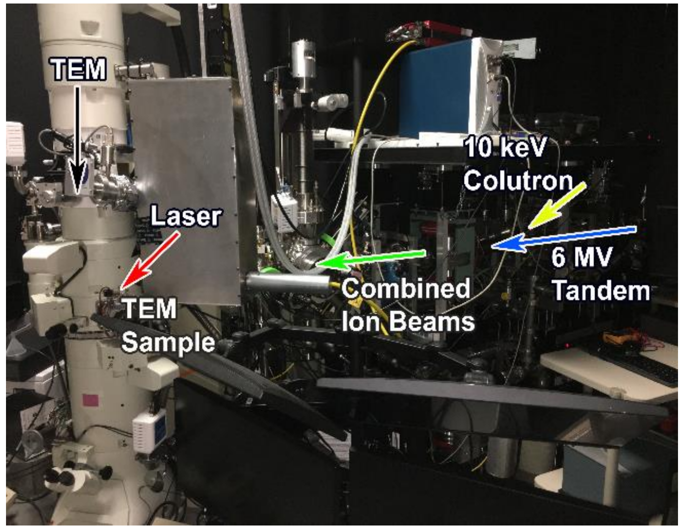


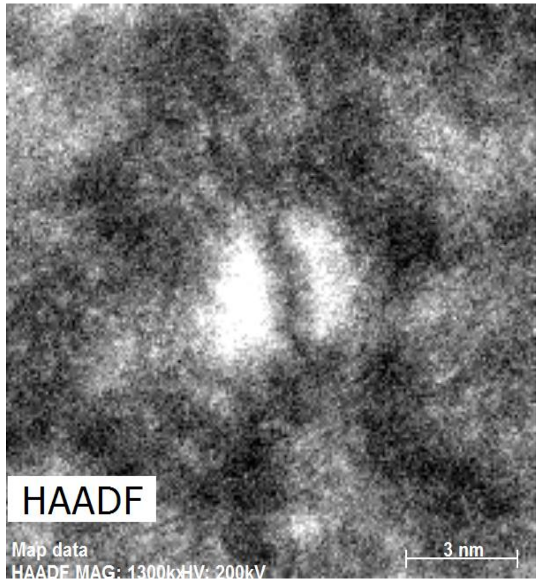
In-situ TEM nano-pillar compression to extract stress-strain data in irradiated zone

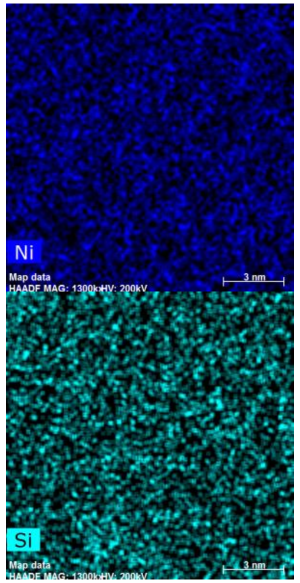
Summary

- Stainless steel TPBAR components undergo neutron radiation-induced segregation and dislocation loop formation
- Comparison experiments with ion beams accelerate the damage, and visualize the damage process with in-situ microscopy
- In-situ Au irradiation causes defect formation, but no elemental segregation









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Acknowledgements

Access to the I^3TEM and associated facilities is now available through both the Nuclear Science User Facilities (NSUF) and the Center for Integrated Nanotechnologies (CINT).

NSUF Nuclear Science User Facilities

https://nsuf.inl.gov

Center for Integrated Nanotechnologies — CINT — Sandia — Los Alamos

http://cint.lanl.gov

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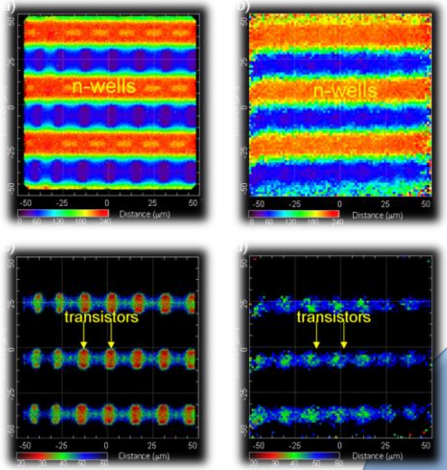
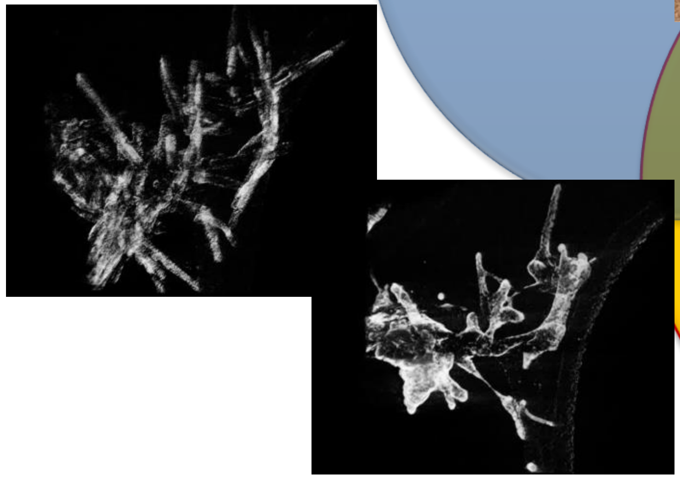
Sandia's USER Capabilities

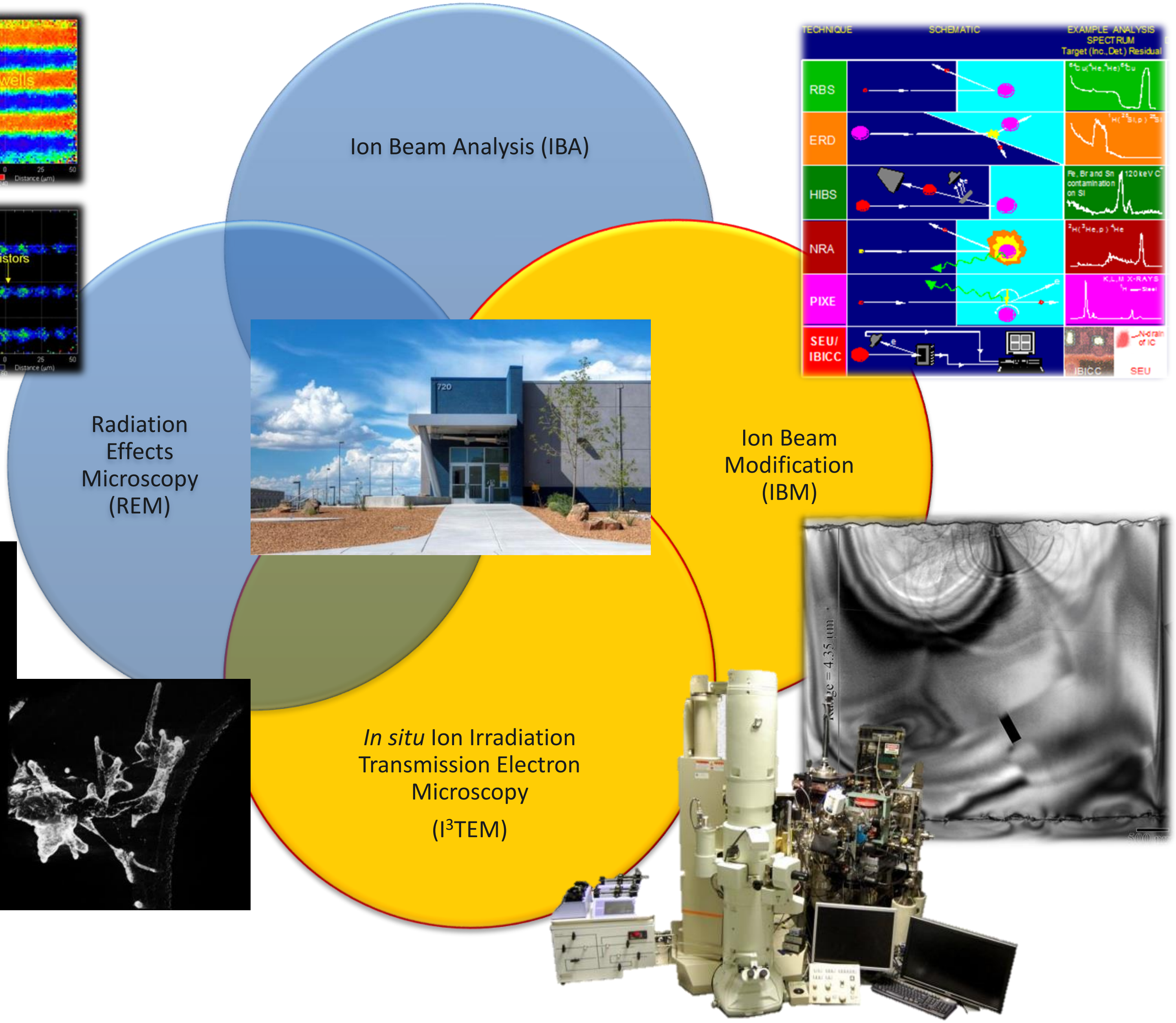
Core Facility - SNL

- Nanophotonics & Optical Nanomaterials
- Soft- Biological & Composite Nanomaterials
- Quantum Materials
- In-situ Characterization and Nanomechanics

Gateway Facility - LANL

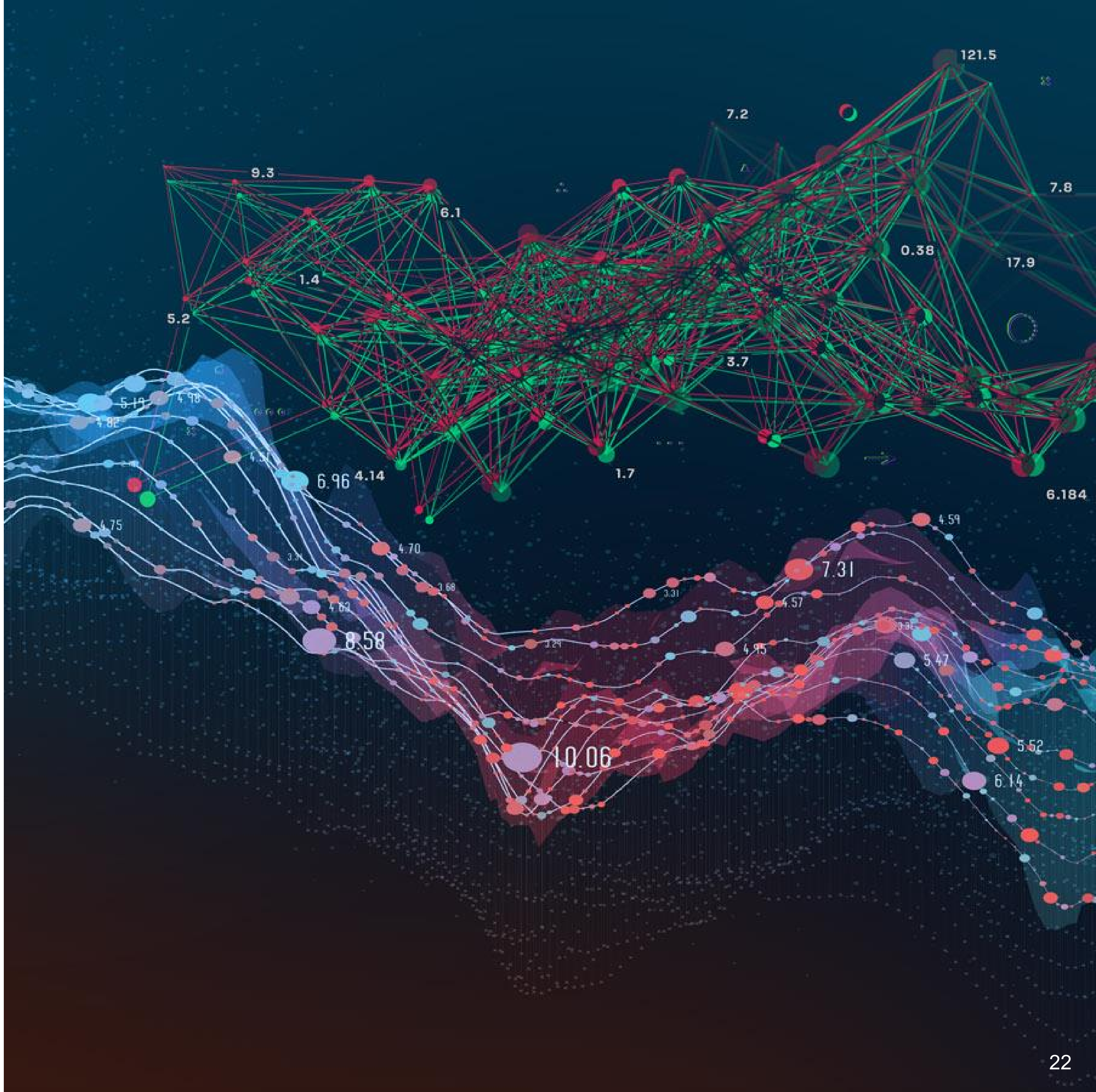




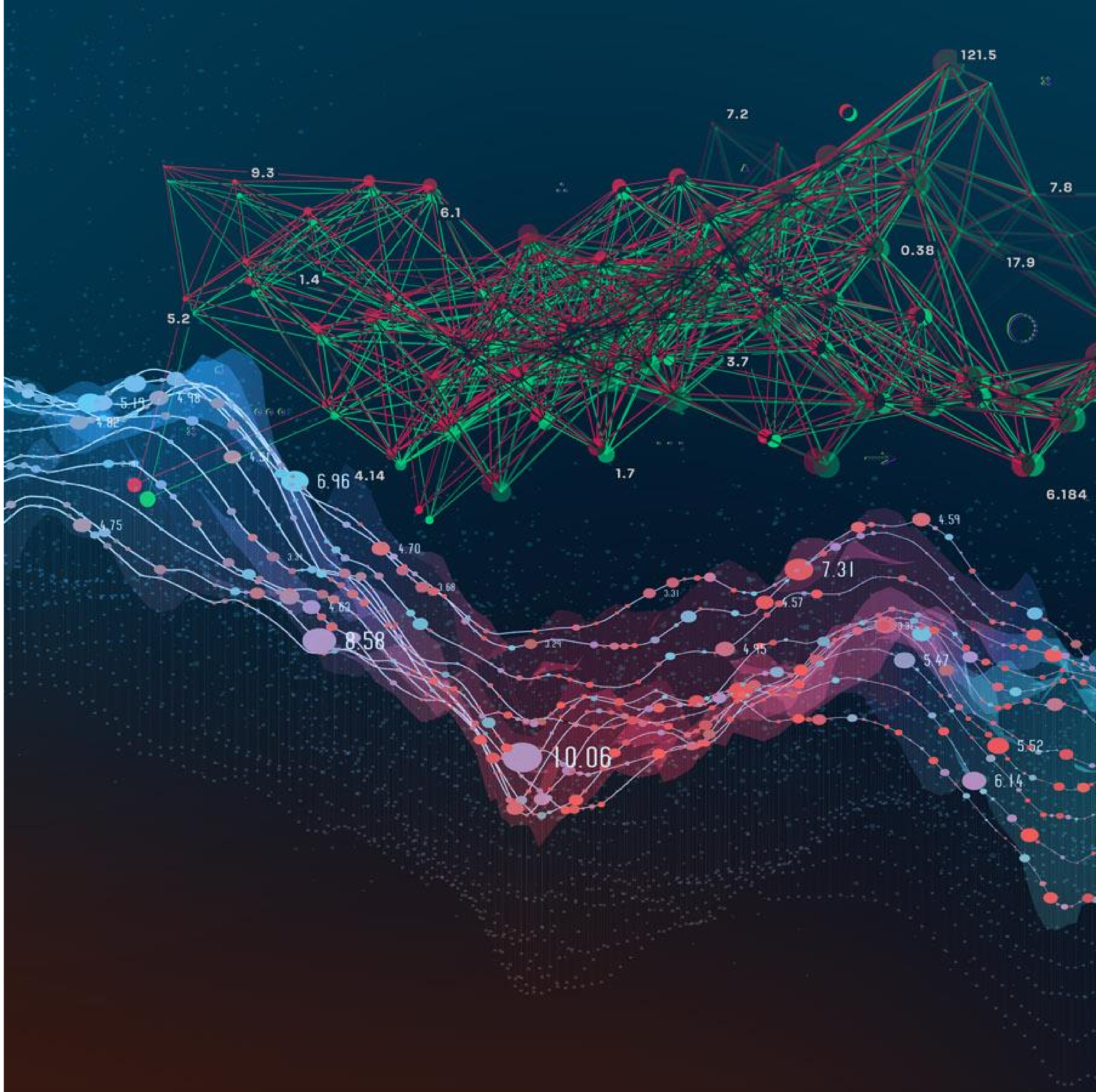


Pacific Northwest
NATIONAL LABORATORY

Thank you



Back-up Slides



U.S. DEPARTMENT OF ENERGY BATTELLE

PNNL is operated by Battelle for the U.S. Department of Energy

Ion irradiation used to mimic neutron damage

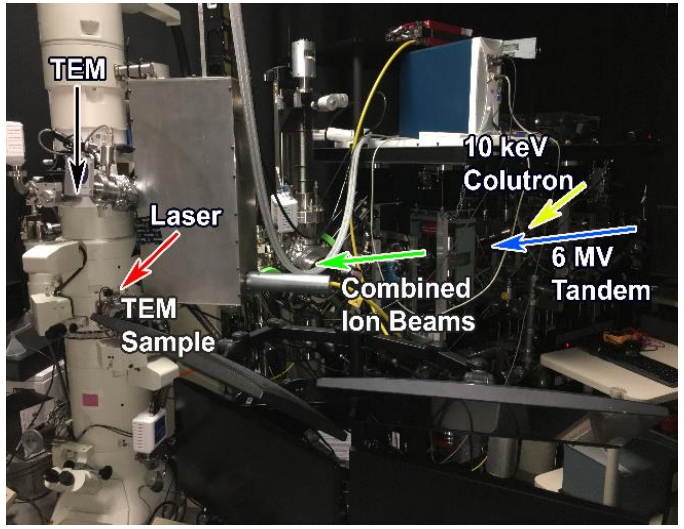
Why utilize ion beams?

- Higher damage rates
- No activation
- Faster and lower cost

But,

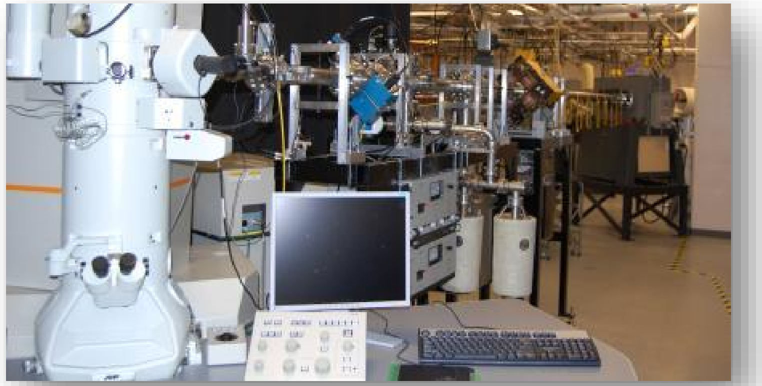
- Only study knock-on damage
- Need higher temperature

I3TEM at SNL



6 MV Tandem Accelerator

Sandia National Labs is well-suited to study ion irradiation induced effects on materials



***In situ* Ion Irradiation TEM (I^3TEM)**

Ion Beam Lab (IBL)