Revealing the Behavior of Gas Species in Materials with *In situ* TEM

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Sandia National Laboratories

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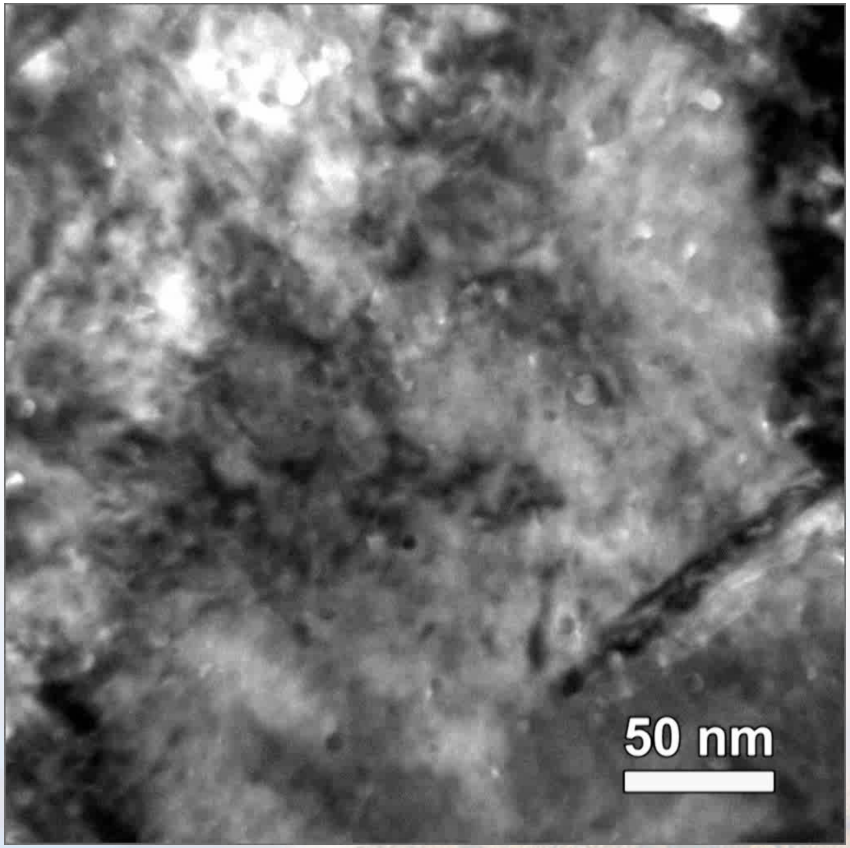

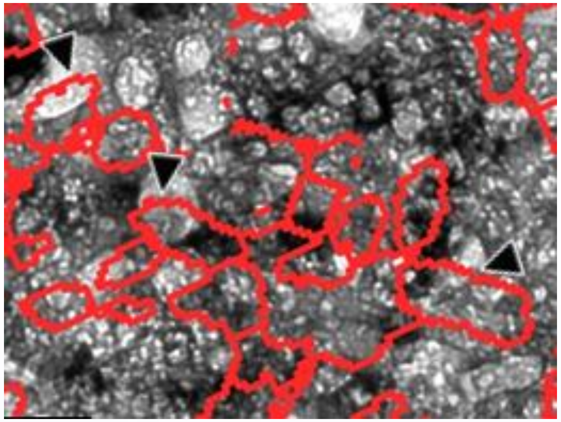

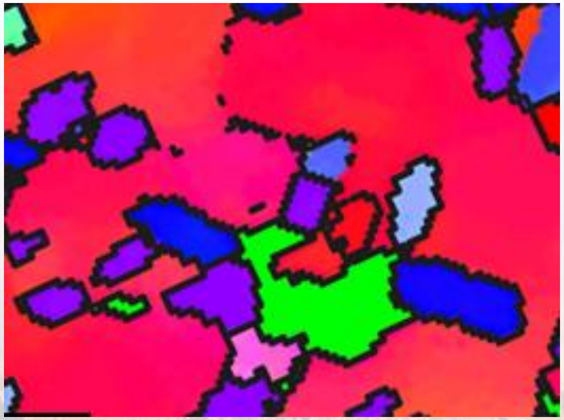

Overview

- Capability to directly observe materials response to displacement damage and/or implantation
- Direct correlation to the local microstructure with nanometer resolution
- Range of complex and overlapping environments

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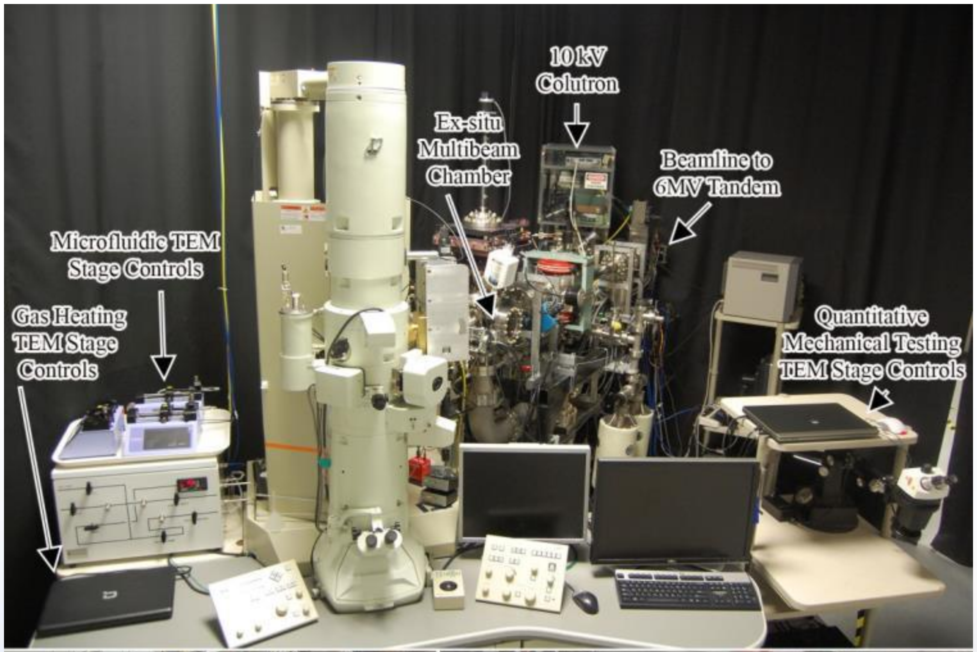
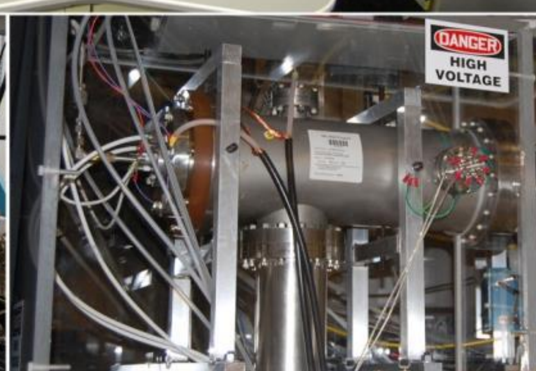
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

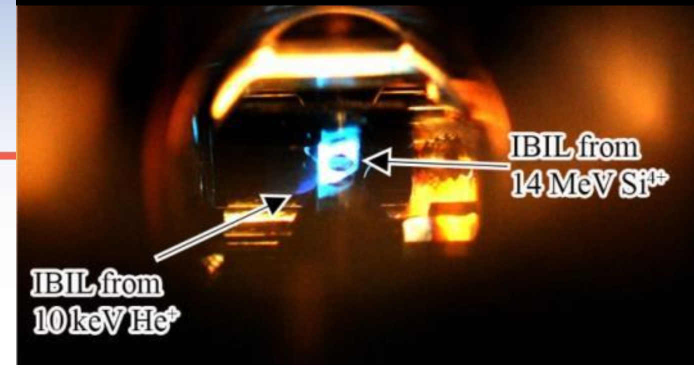
Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem



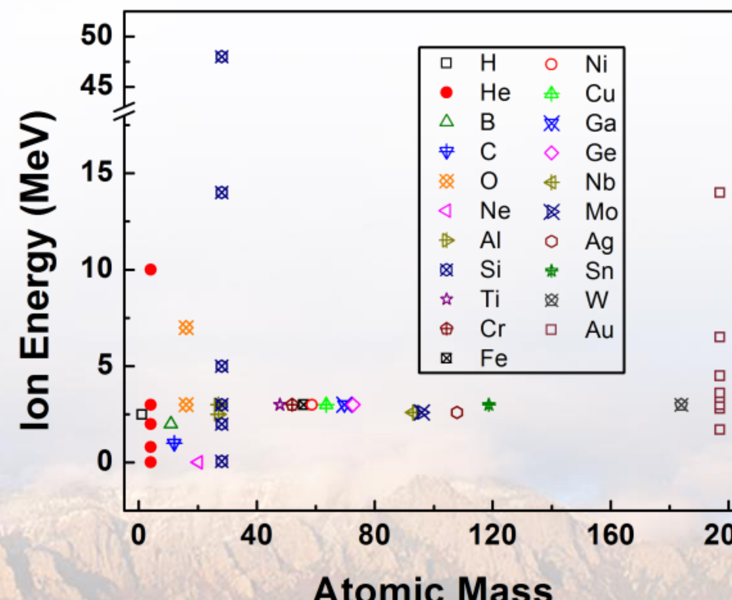


IBIL from a quartz stage inside the TEM

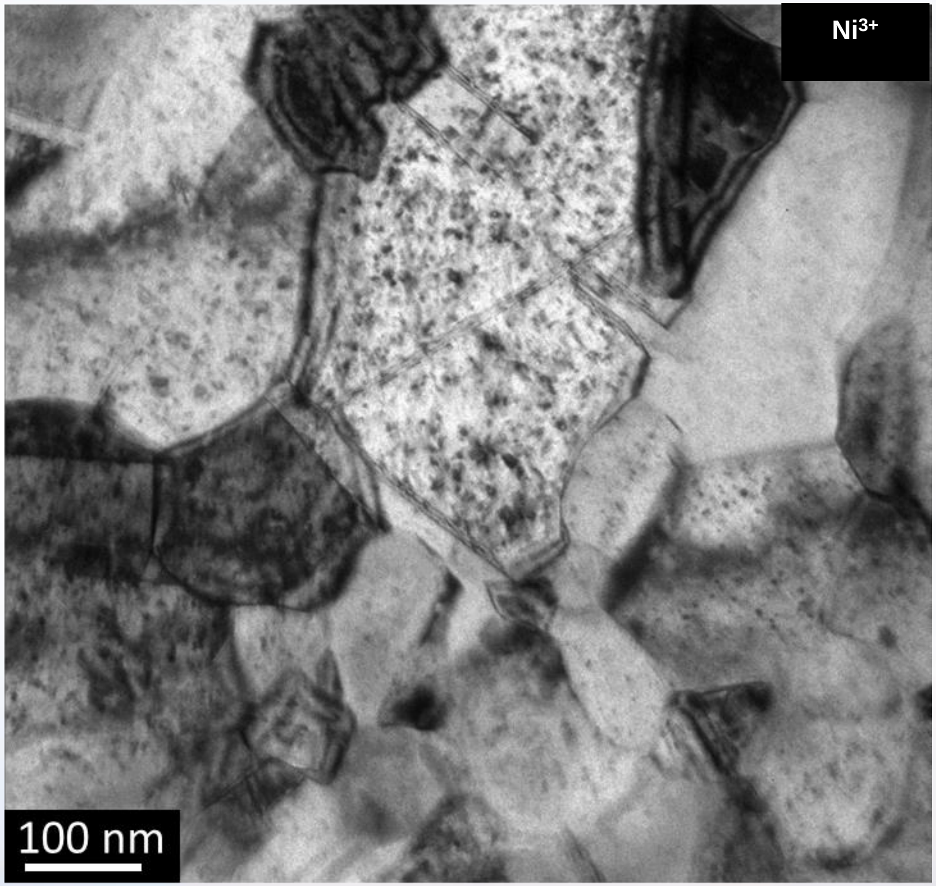


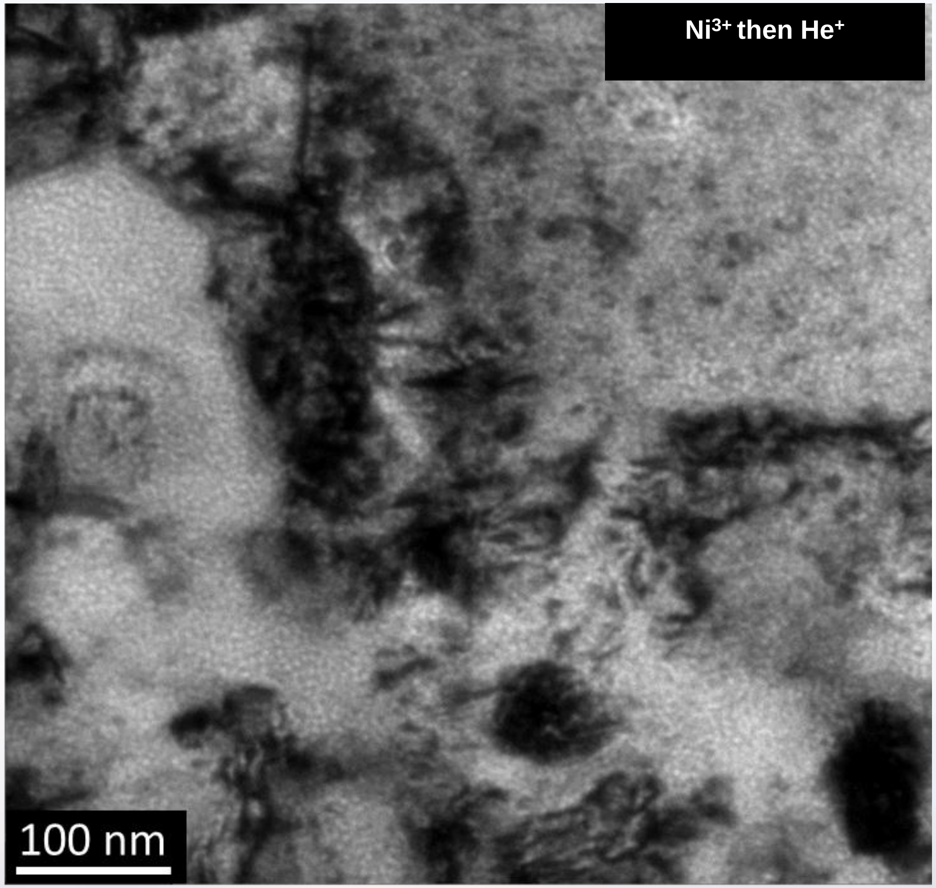
Direct real time observation of ion irradiation, ion implantation, or both with nanometer resolution

Ion species & energy introduced into the TEM



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





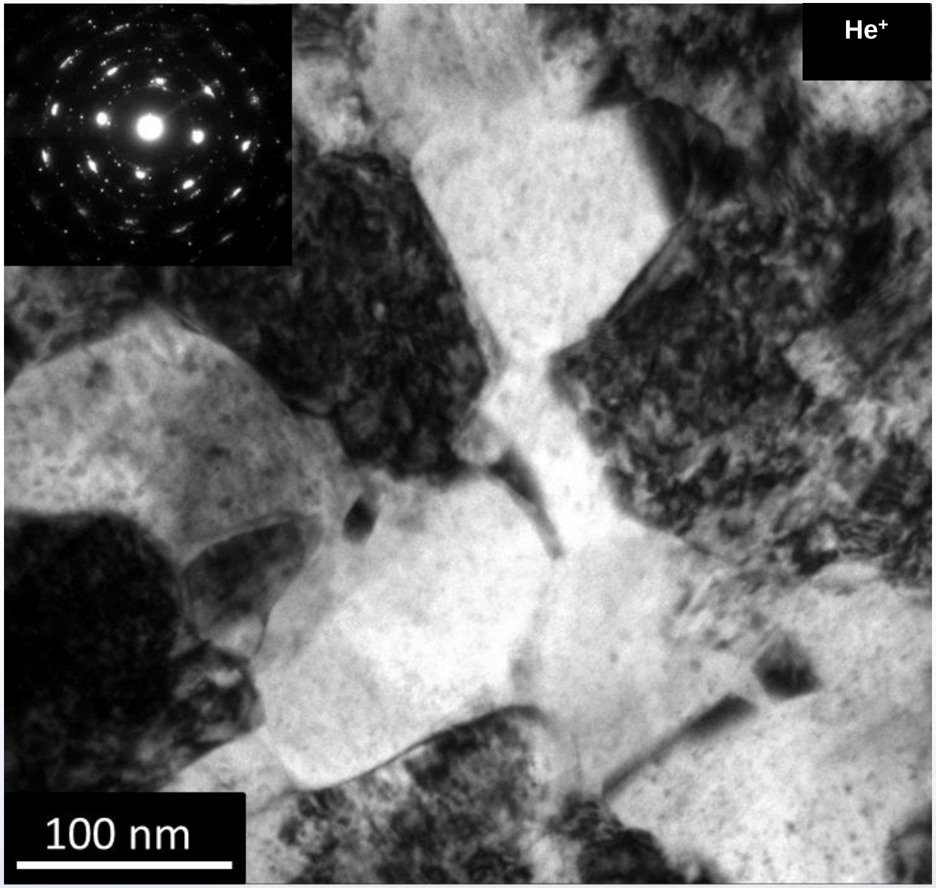
1.8 dpa Ni^{3+} irradiation

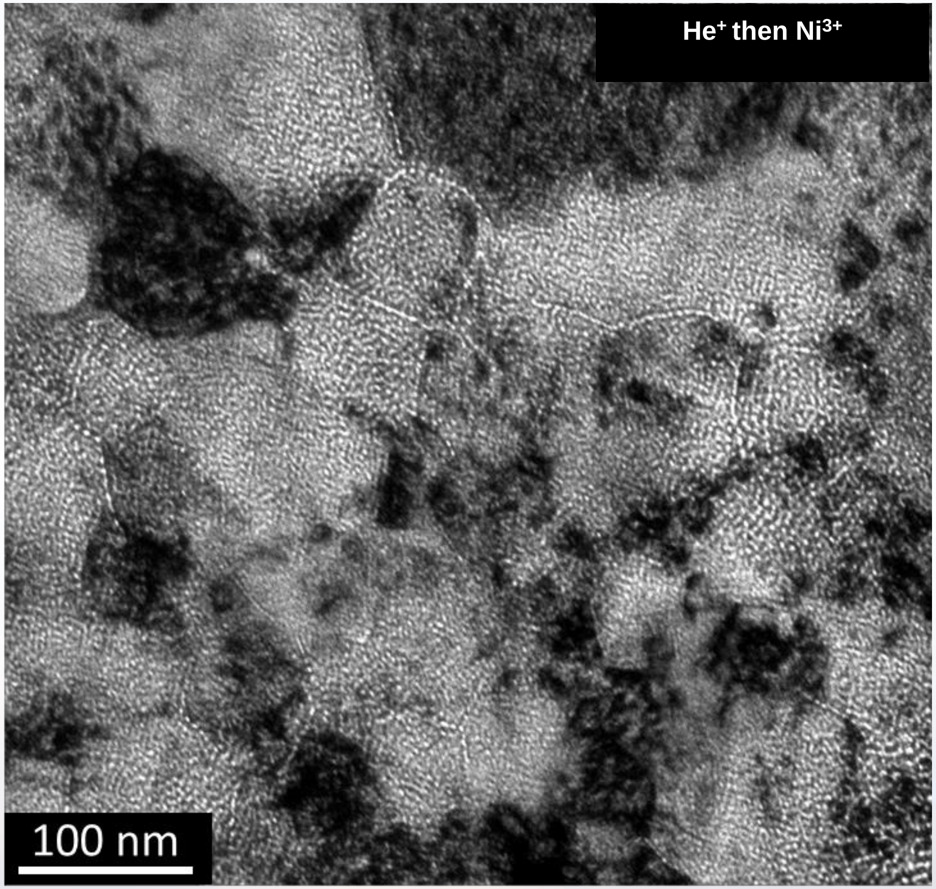
Dislocation loops and SFT are present

Additional $2x10^{16}$ He^{+}/cm^{2}

Evenly distributed nanometer size cavities

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





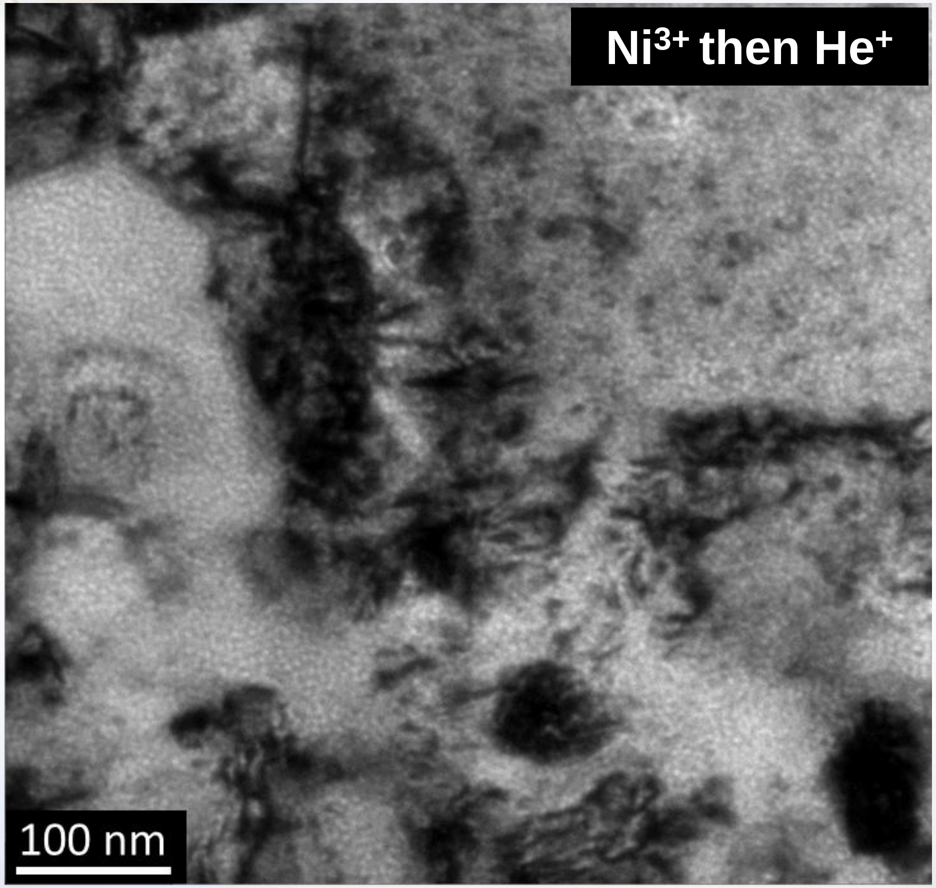
10^{17} He^+/cm^2

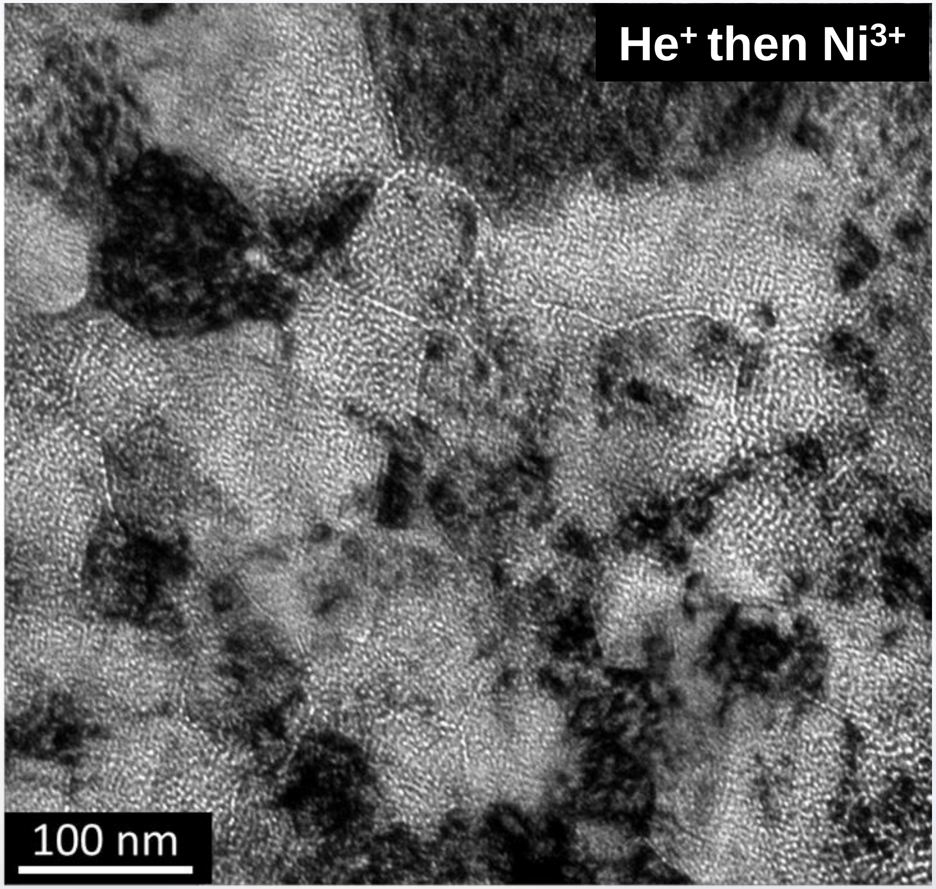
Visible damage

0.7 dpa Ni^{3+} irradiation

High concentration of cavities along grain boundaries

Irradiation / Implantation Sequence Effect on Cavity Structure

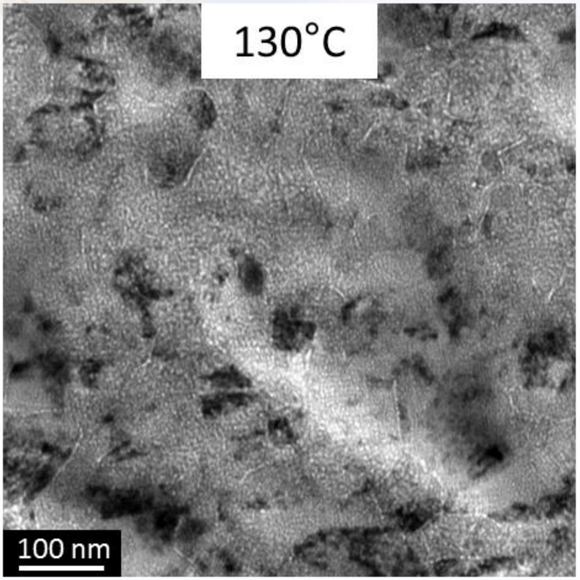


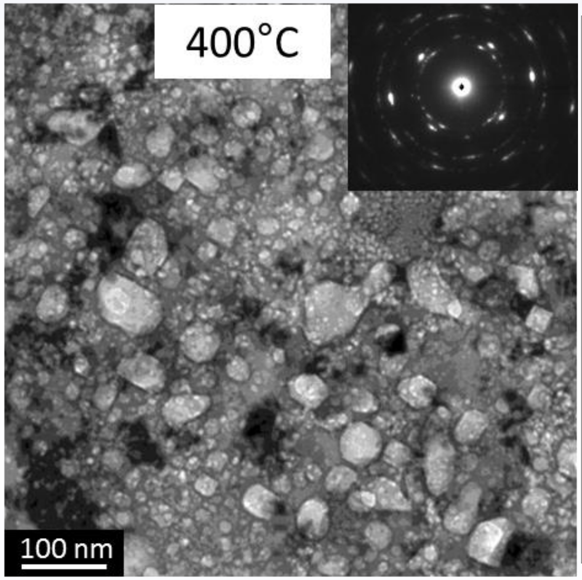


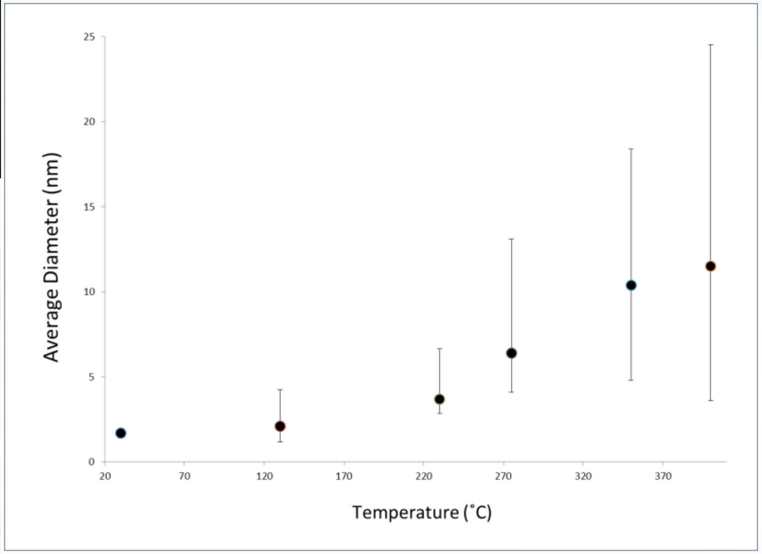
Evenly distributed cavities over the entire grain structure

Apparent higher concentration of cavities along grain boundaries

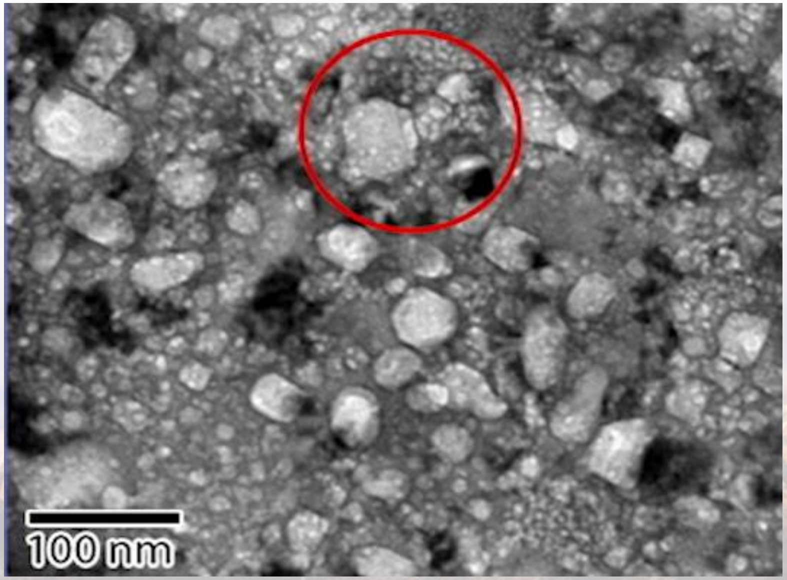
Cavity Growth during In-situ Annealing of 10 keV He^+ Implanted and then 3 MeV Irradiated Ni^{3+}

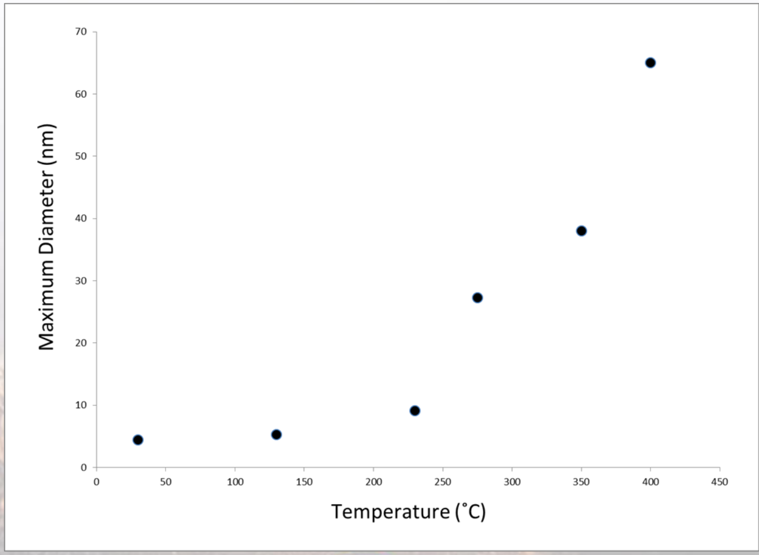






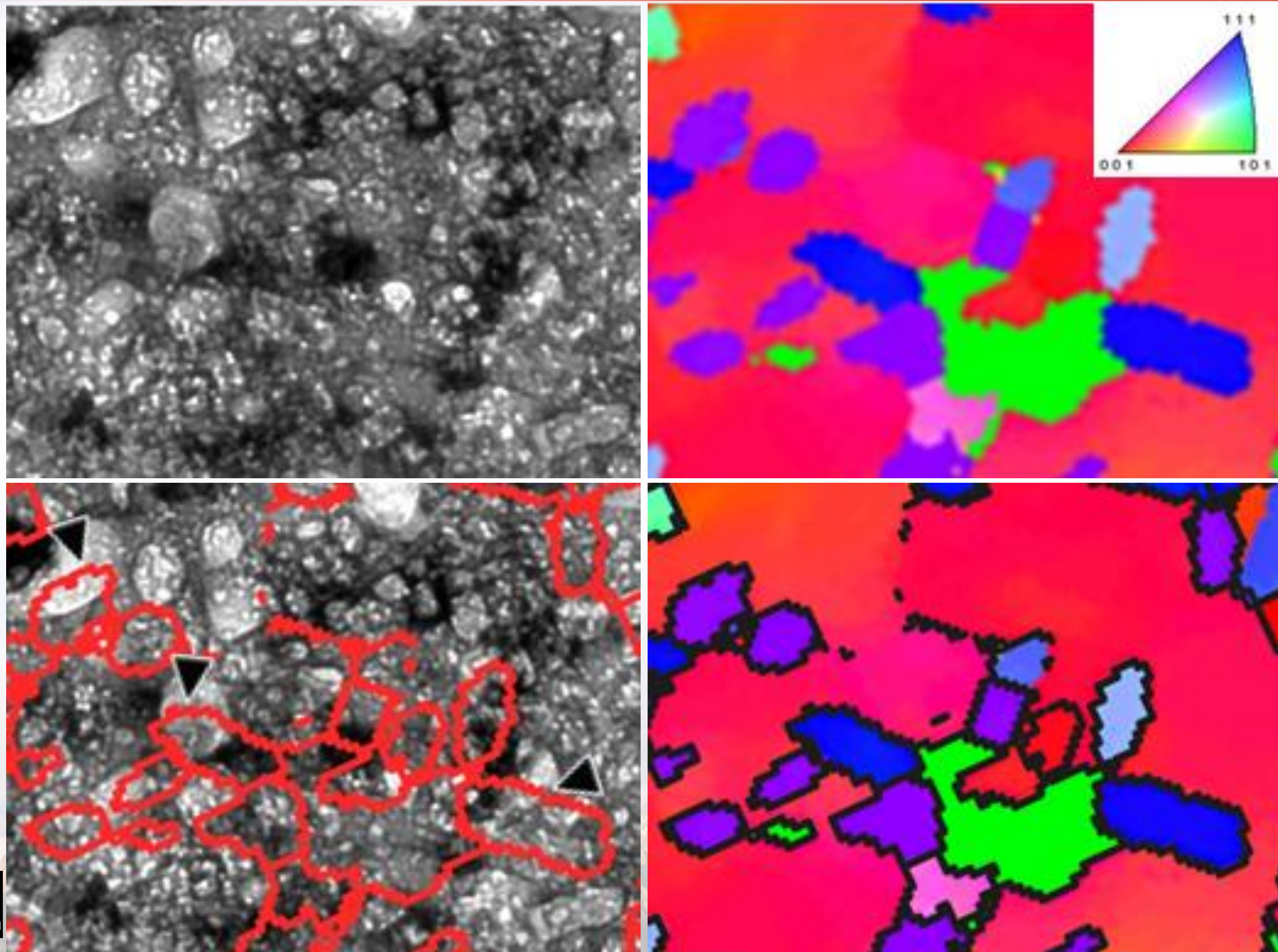
Bubble to cavity transition and cavity evolution can be directly studied





Precession Electron Diffraction Reveals Hidden Grain Structure

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

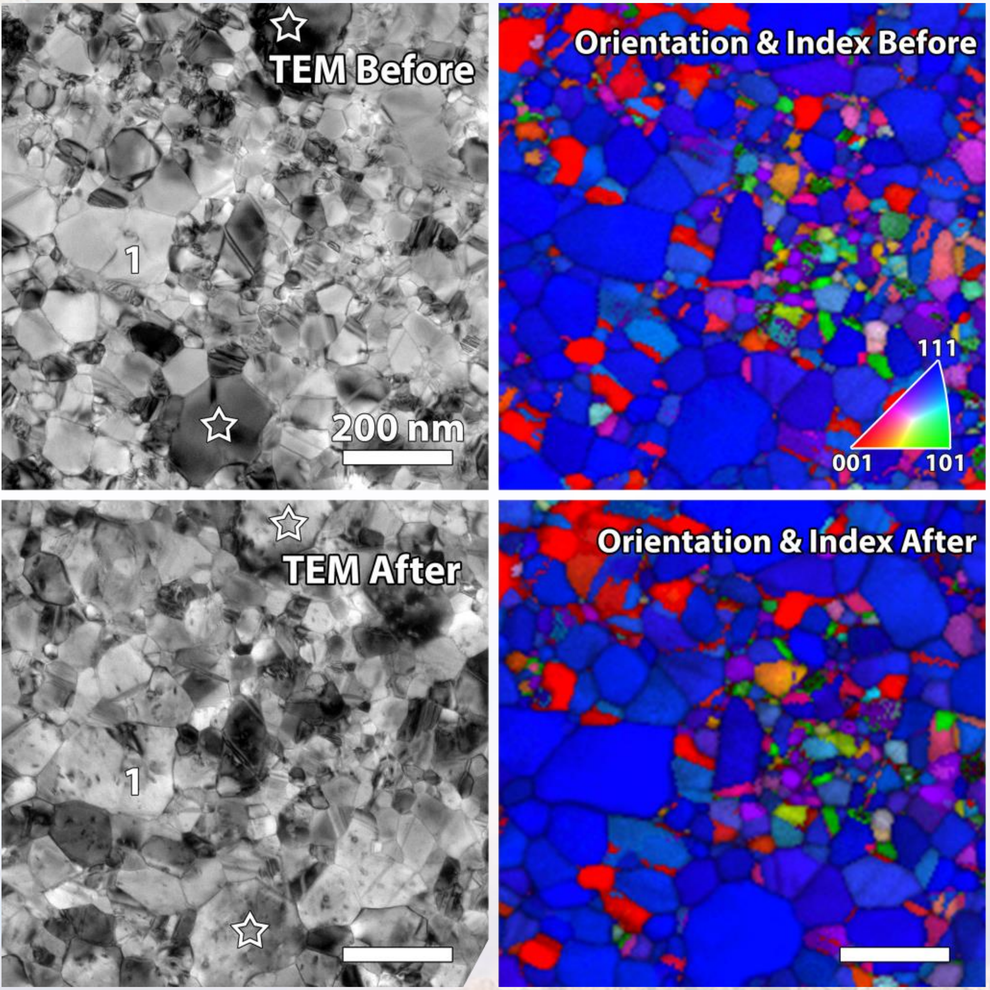


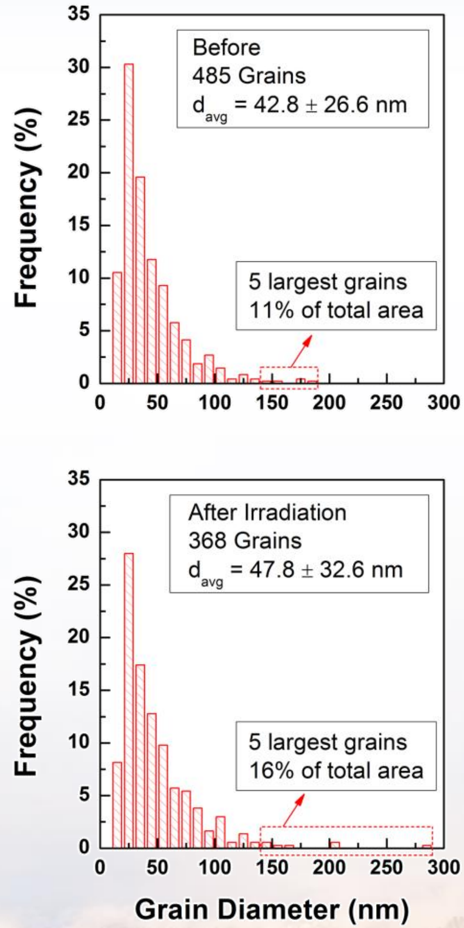
100 nm

Cavities span multiple grains at identified grain boundaries

Quantifying Stability of Nanocrystalline Au during 10 MeV Si Ion Irradiation

Collaborators: F. Abdeljawad, & S.M. Foiles

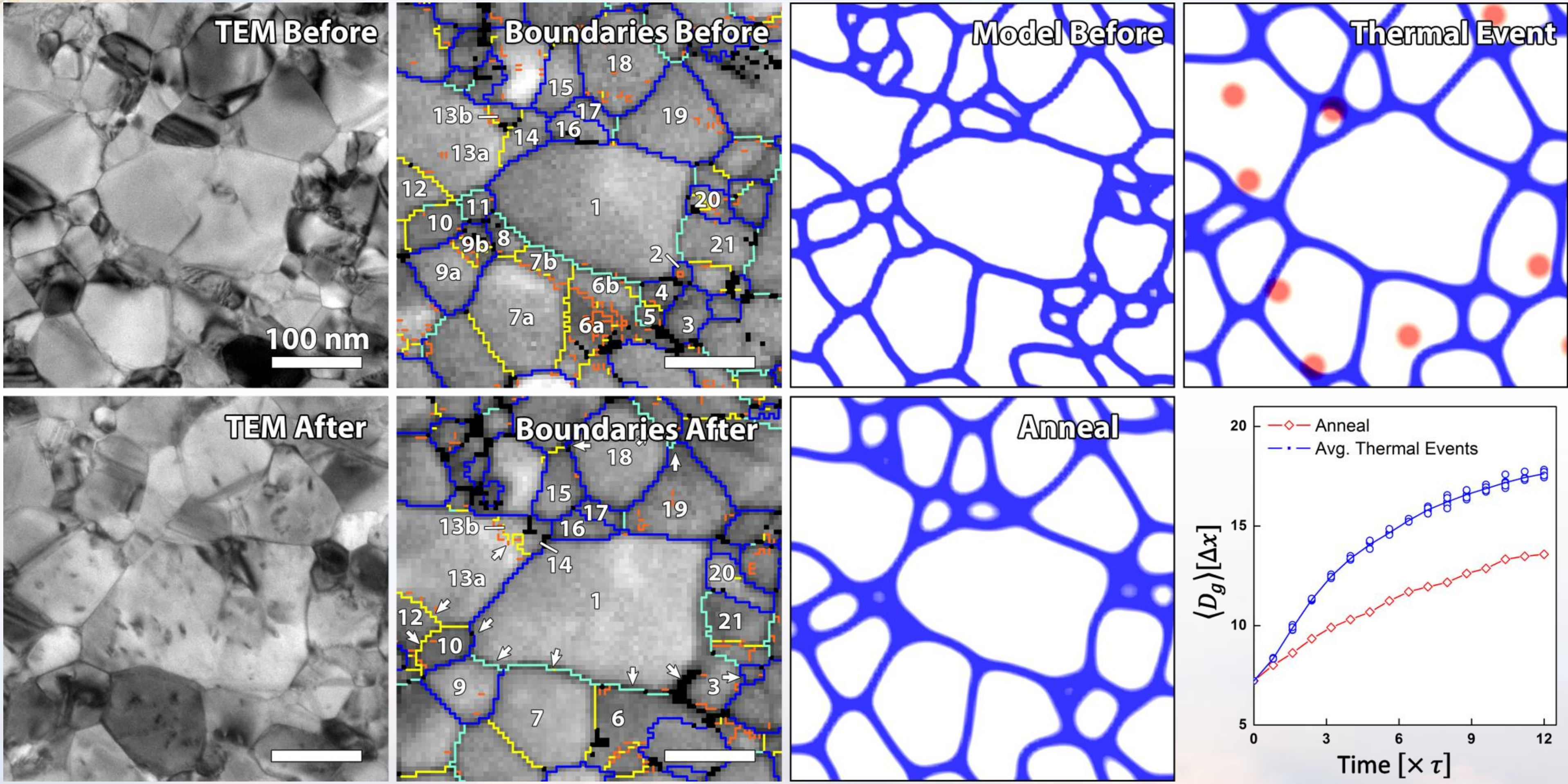




Any texture or grain boundary evolution can be directly observed and quantified

Direct Comparison to Mesoscale Modeling

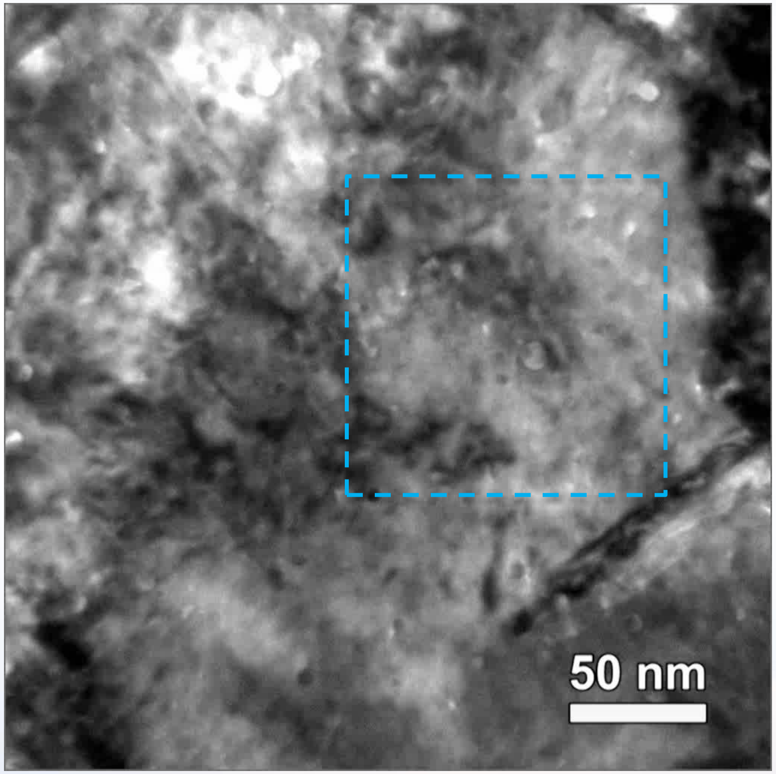
Collaborators: F. Abdeljawad, & S.M. Foiles

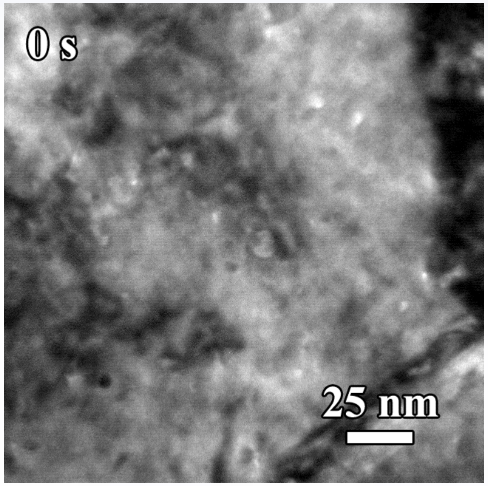


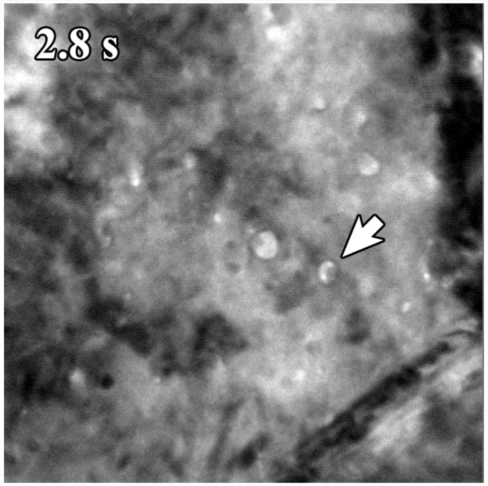
Because of the matching length scale, the initial microstructure can serve as direct input to either MD of mesoscale models & subsequent structural evolution can be directly compared.

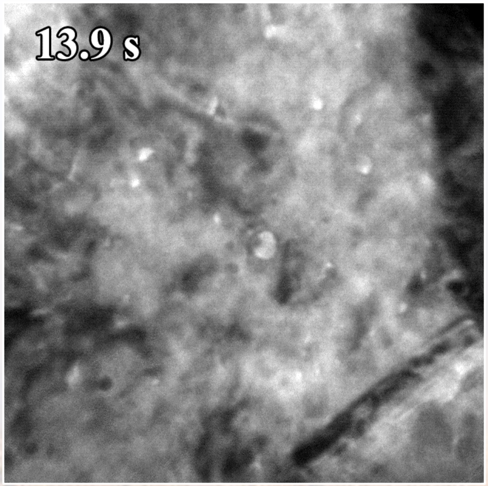
Simultaneous *In situ* TEM Triple Beam: 2.8 MeV Au^{4+} + 10 keV He^{+}/D_{2}^{+}

Video playback speed x1.5.









- **Approximate dose:**
  - 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**

In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I^3TEM!

Intensive work is still needed to understand the defect structure evolution that has been observed.

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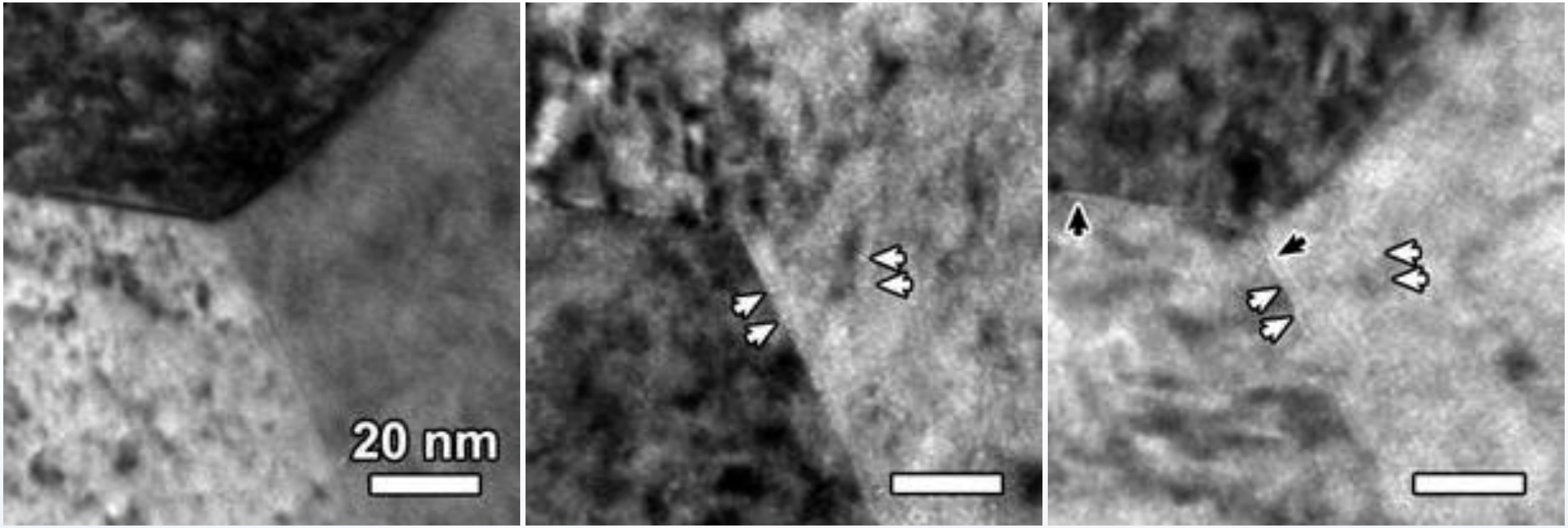
He Bubble Evolution in Deuterium Exposed Mo

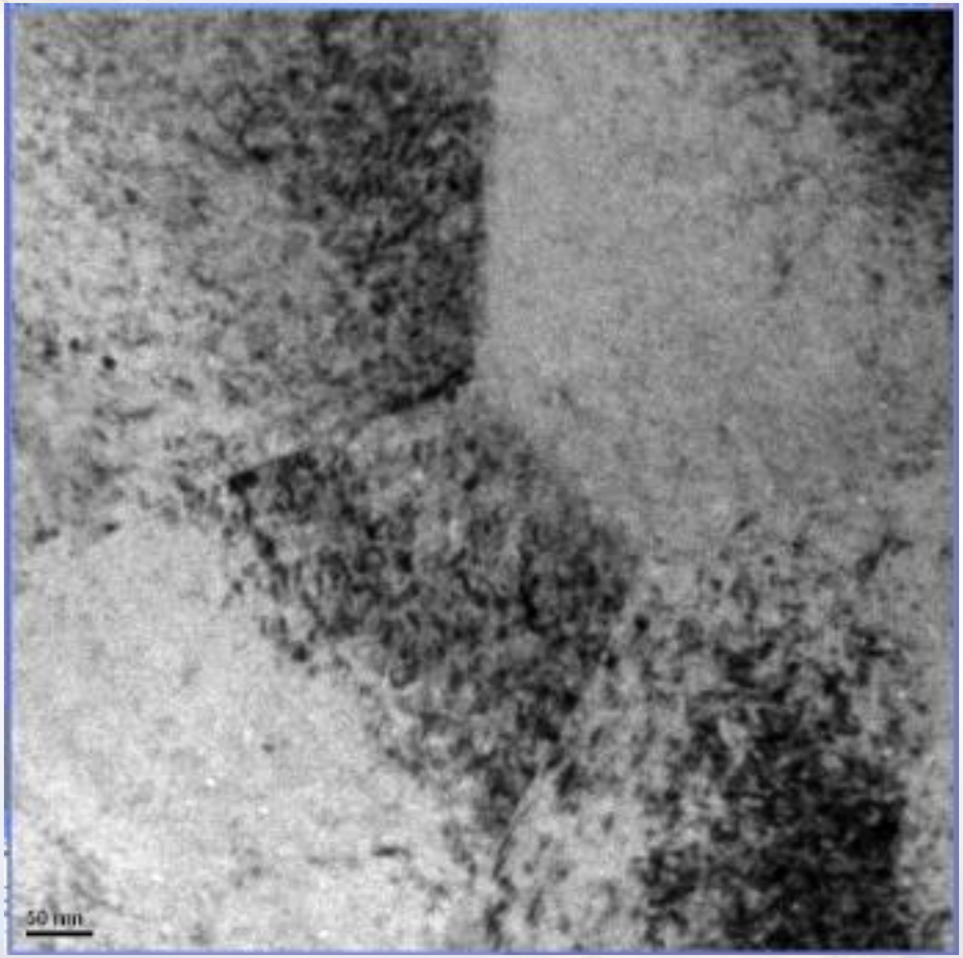
Deuterium-exposed Mo before implantation | **After 1.7 x 10^{15} He/cm^2** | **After 1.7 x 10^{17} He/cm^2**

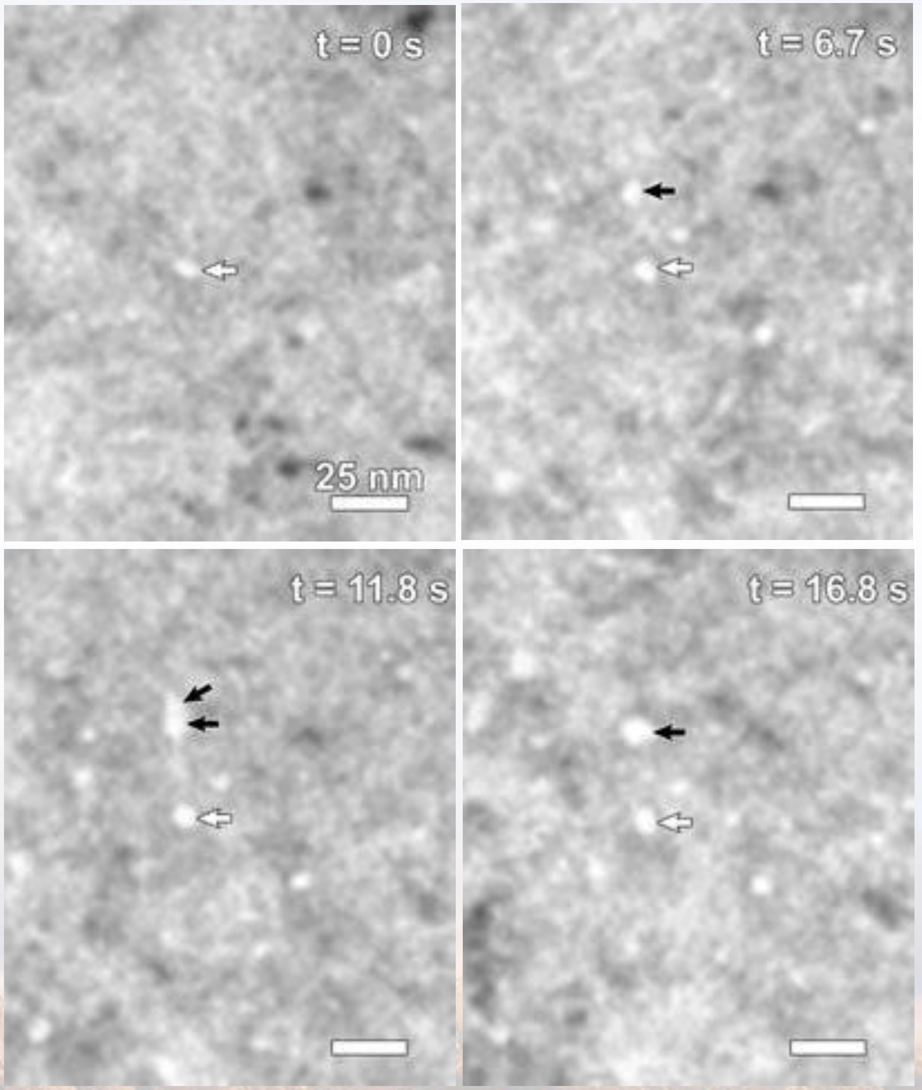


In situ Room Temperature He implantation at 10 keV shows minimal variation in bubble size or spatial distribution, but increasing density with dose.

SRIM predict 63% of He remains in the film during implantation with a peak 30 nm into TEM film

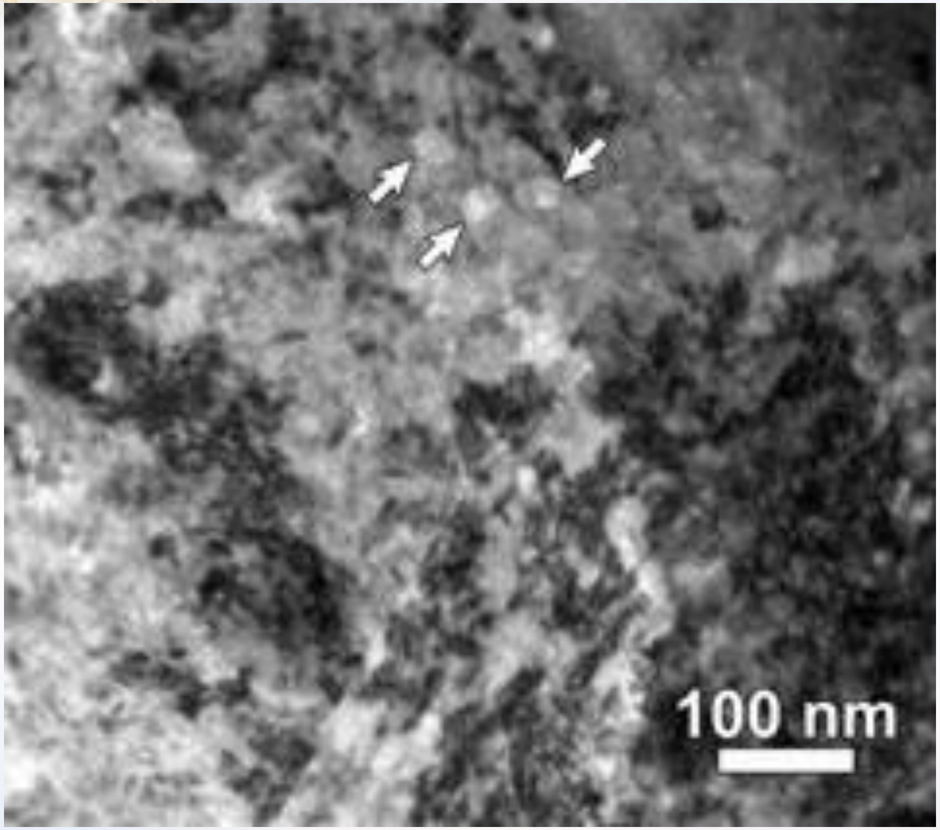
Bubble to Cavity Transition is Directly Observed during *In situ* Annealing

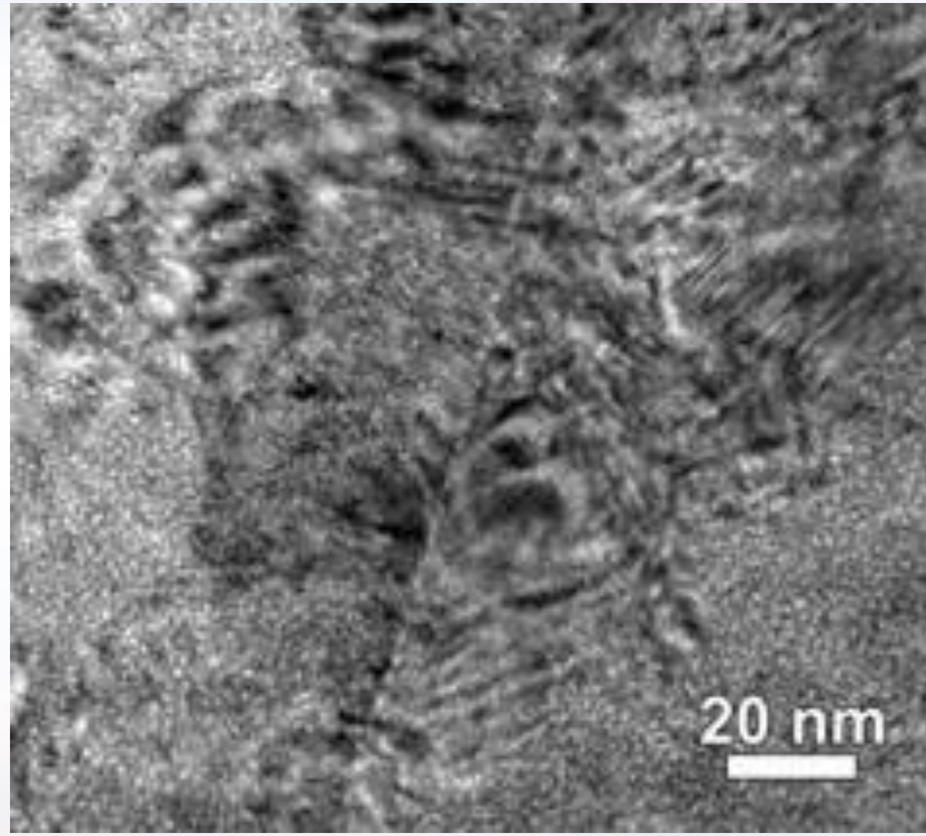


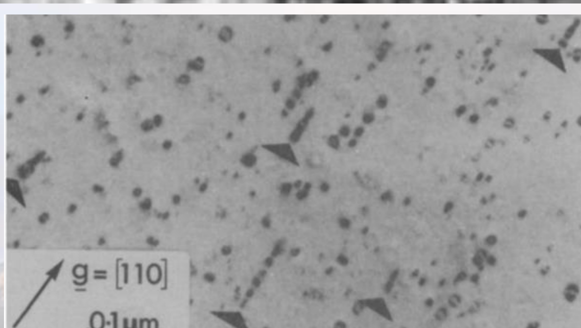


Minimal structural evolution below 750 ˚C
Video shows cavity formation at 786 ˚C

Large Cavity Formation Appears to be Significantly Effected by the Implanted Vacancy Concentration







SRIM predicts 6 vacancies/He &

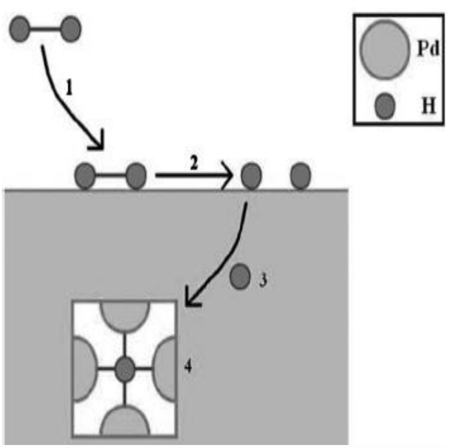
Cavity shape suggest vacancy play a major role

Future work: Explore below knock-on damage He implantation

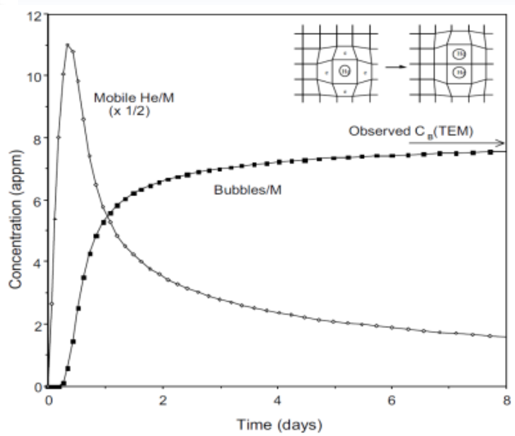
J.H. Evans, et al. Scripta Met. Vol. 15 pp. 323-326, 1981

In situ TEM Observation of Hydrogen's Effect on Pd Nanopore Stability

Contributors: B.G. Clark, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, L.R. Parent, I. Arslan. & Protochips, Inc.



R. Delmelle, J., Phys. Chem. Chem. Phys. (2011) p.11412

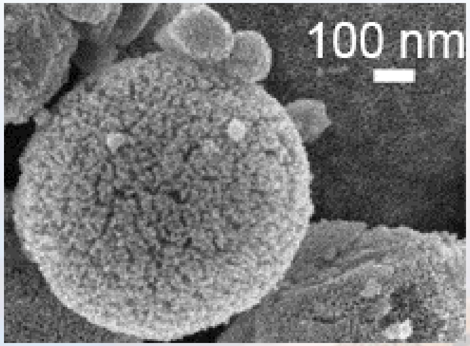


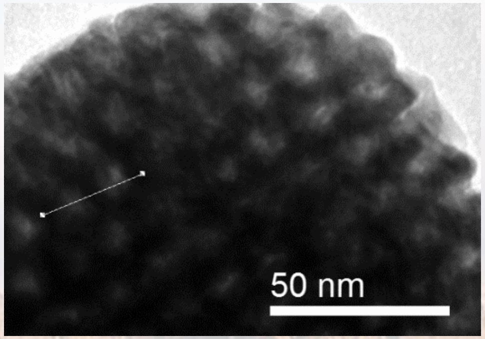
Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539
Trinkaus, H. *et al*., JNM (2003) p. 229
Thiebaut, S. *et al*. JNM (2000) p. 217

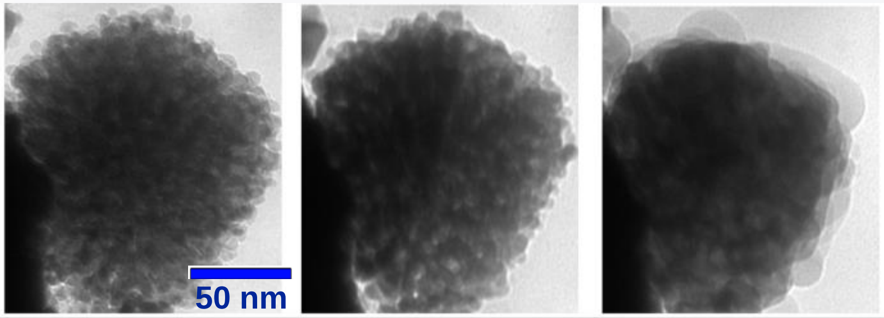
Vapor-Phase Heating TEM Stage

- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools

- 1 atm H_2 after several pulses to specified temp.







125° C 200° C 300° C

New *in situ* atmospheric heating experiments provide great insight into nanoporous Pd stability

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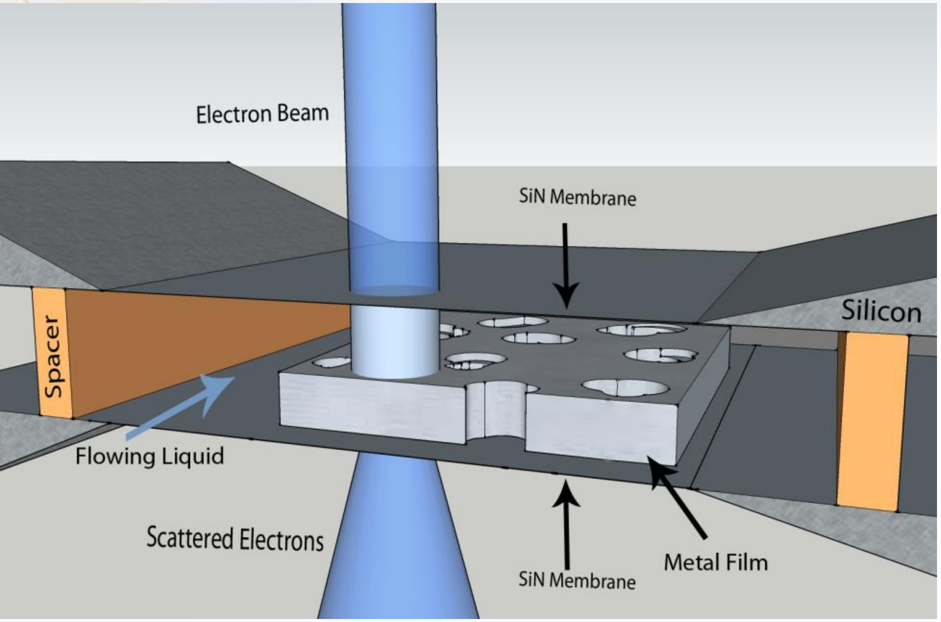
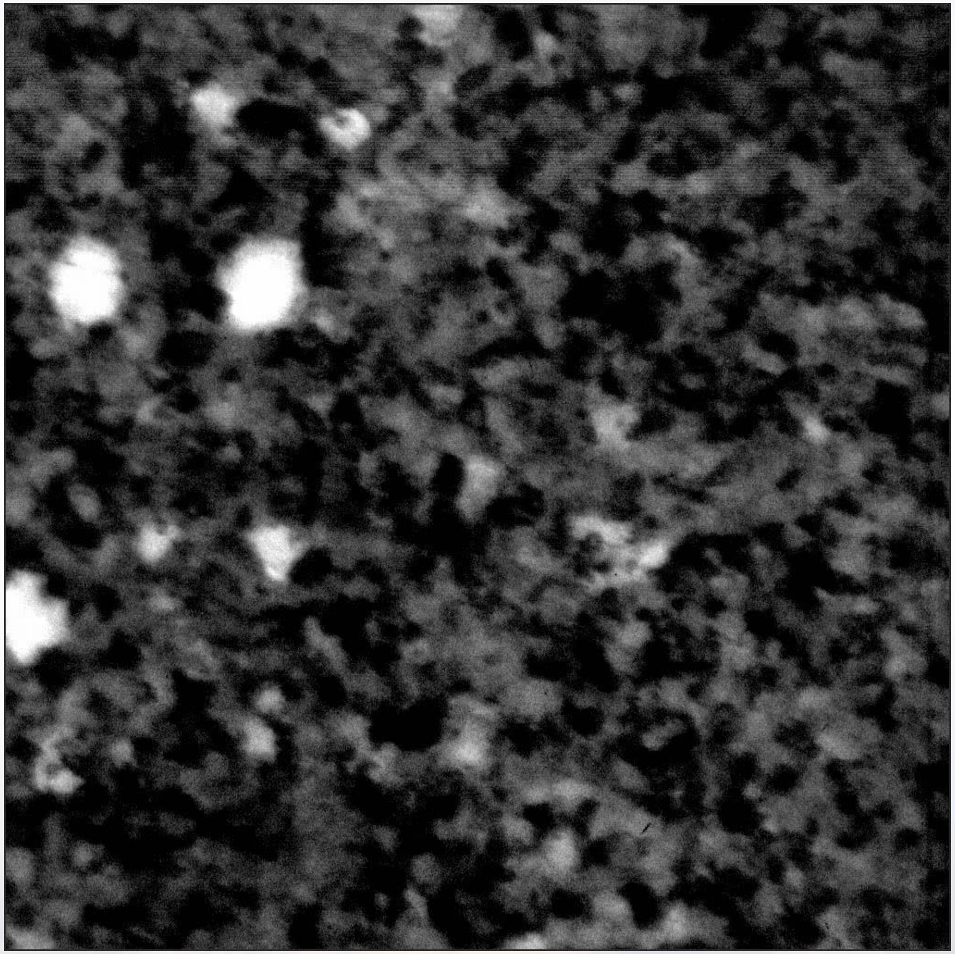
In situ TEM Corrosion Direction

Contributors: D. Gross, J. Kacher, & I.M. Robertson



Microfluidic Stage

- **Mixing of two or more channels**
- **Continuous observation of the reaction channel**
- **Chamber dimensions are controllable**
- **Films can be directly deposited on the electron transparent SiN membrane**

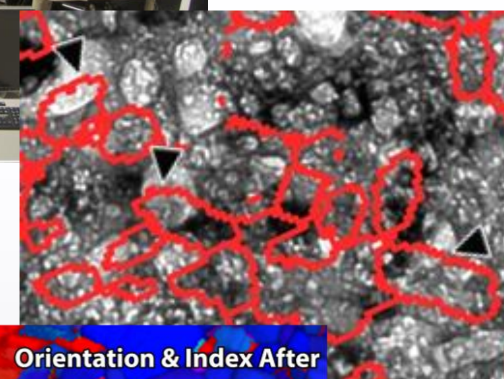


Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.

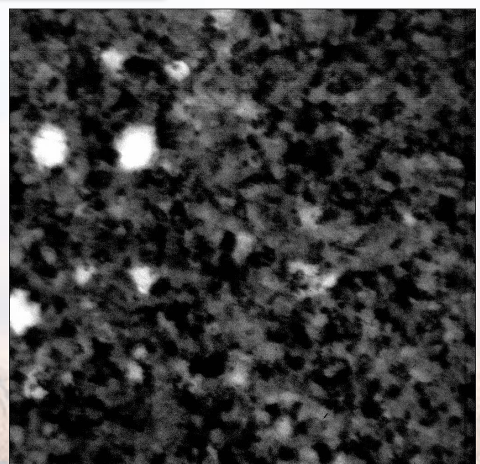
Summary

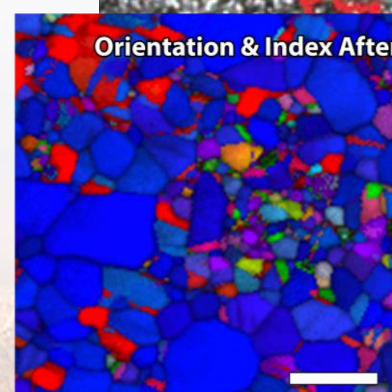
- **Sandia's I³TEM capabilities:**
  - ***In situ*** **high energy ion irradiation from H to Au**
  - ***In situ*** **gas implantation**
  - **Heating up to 1,000 ˚C**
  - **Quantitative and bulk straining**
  - **Two-port microfluidic cell**
  - **Gas flow/heating stage**
  - **Electron tomography**
  - **Precession Electron Diffraction**

Currently applying the current I³TEM capabilities to various material systems in sequential or combined harsh environmental conditions



- **Structural response of metal sample to various harsh environments:**
  - **Sequence of gas implantation and cascade formation matters for the final microstructure**
  - **Concurrent gas implantation and irradiation permits the deconvolution of environmental parameters**
  - **PED permits the correlation of bubble evolution with grain texture and boundary type**
  - **Not limited to vacuum environments**





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