Exploring Complex Evolution in a Simulated Reactor Environment via *In situ* Electron Microscopy

SAND2017-10288C

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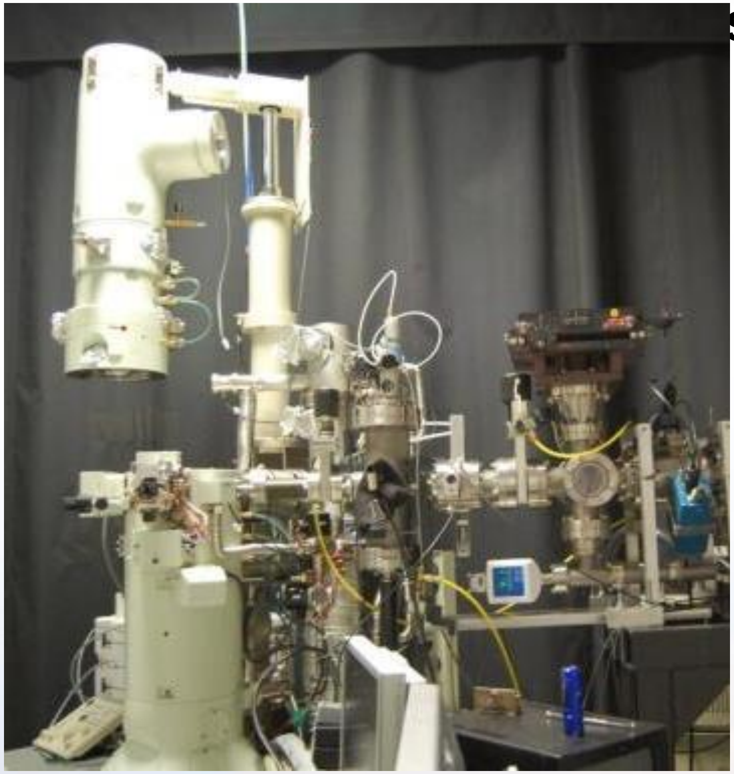
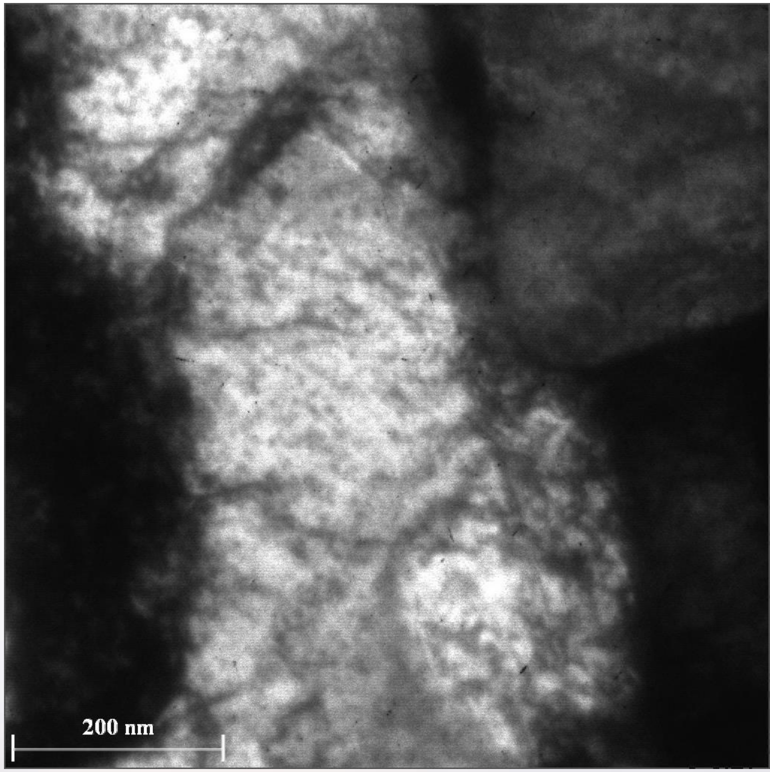
Ion Beam Lab at Sandia National Laboratories

September 6, 2017



***In situ* TEM microscopy**

has recently undergone significant growth providing capabilities to investigate the structural evolution that occurs due to various extreme environments and combinations thereof

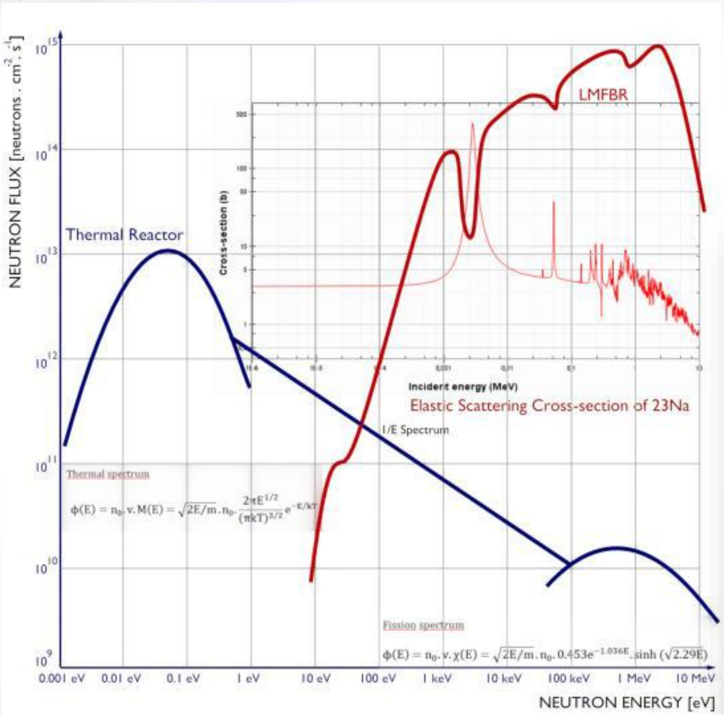


Collaborators:

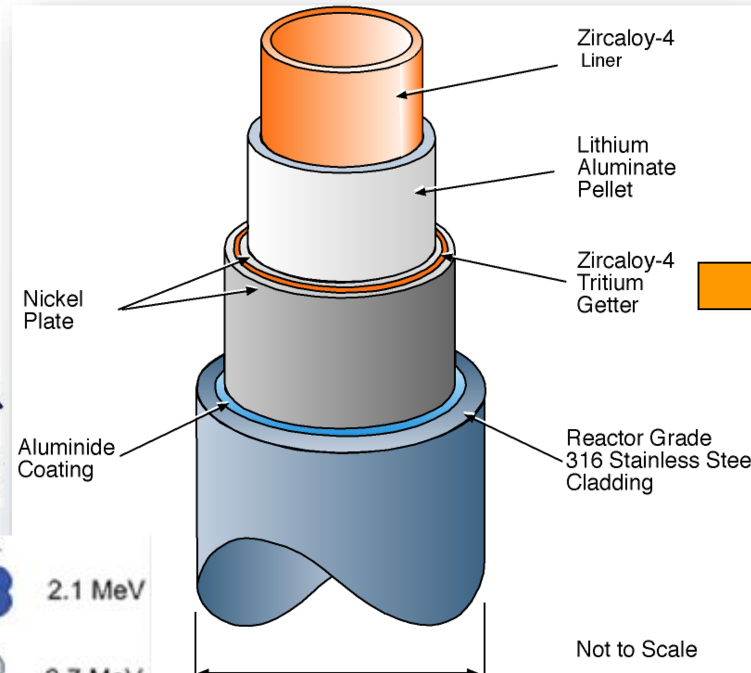
- **IBL: D.C. Bufford, D. Buller, C. Chisholm, B.G. Clark, J. Villone, B.L. Doyle, S. H. Pratt, M. Steckbeck & M.T. Marshall**
- **Sandia: B. Boyce, T.J. Boyle, P.J. Cappillino, J.A. Scott, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, J.A. Sharon, W.M. Mook, F. Abdeljawad, & S.M. Foiles**
- **External: A. Minor, L.R. Parent, I. Arslan, H. Bei, E.P. George, P. Hosemann, D. Gross, J. Kacher, & I.M. Robertson**

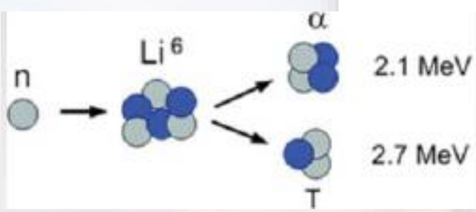
This work was partially supported by the US Department of Energy, Office of Basic Energy Sciences.

TPBAR Design & Reactor Environment



Tritium Producing Burnable Absorber Rod (TPBAR)





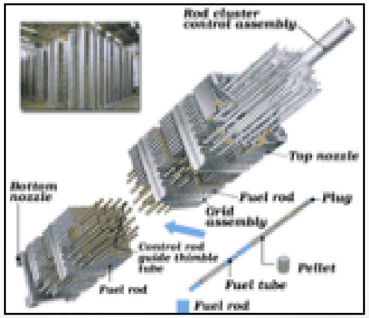
- Displacement Damage
- Helium Implantation
- Tritium Implantation
- Elevated Temperatures

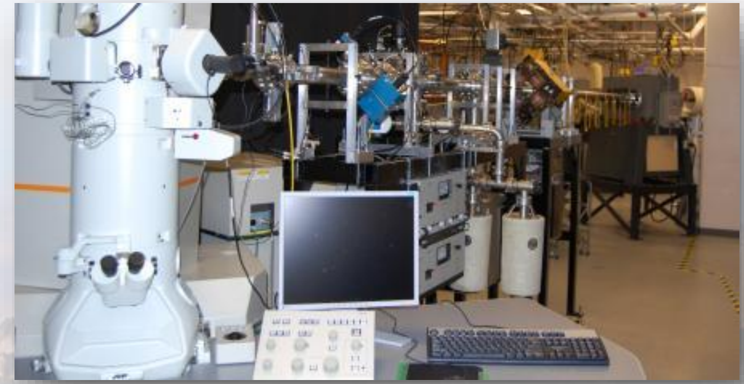
Simulating neutron irradiation in a reactor is complicated, and TPBAR adds the additional complication of ^3H production

Investigating the nm Scale to Understand the km Scale



1 nm | 1 μm | 1 mm | 1 m | 1 km | 10^7 m

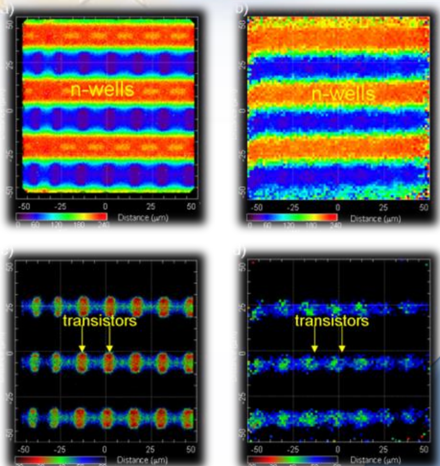





In situ Ion Irradiation TEM (I^3TEM)

Ion Beam Lab (IBL)

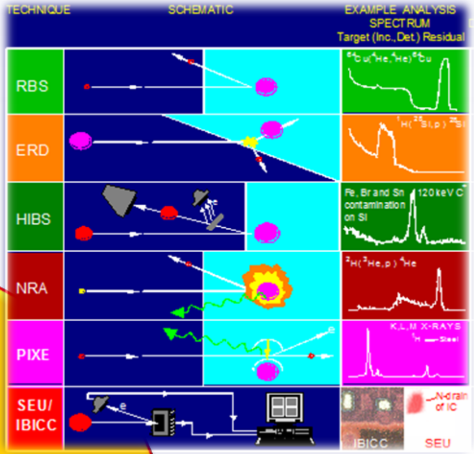
Sandia’s Ion Beam Laboratory



Ion Beam Analysis (IBA)

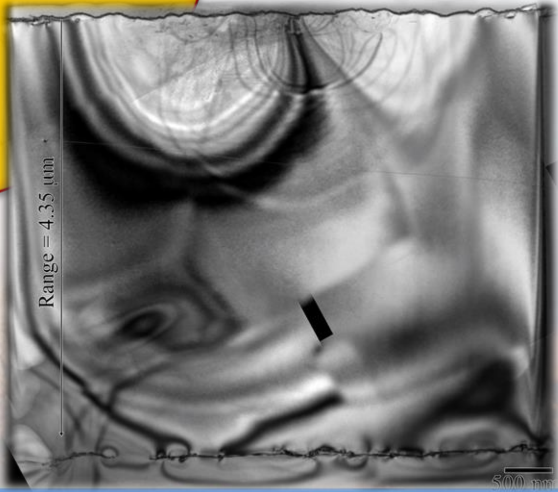
Radiation Effects Microscopy (REM)

Ion Beam Modification (IBM)



In situ Ion Irradiation Transmission Electron Microscopy

(I^3TEM)



Benefits & Limitations of *in situ* TEM

Benefits

1. **Real-time nanoscale resolution observations of microstructural dynamics**

Limitations

1. **Predominantly limited to microstructural characterization**
   - Some work in thermal, optical, and mechanical properties
2. **Limited to electron transparent films**
   - Can often prefer surface mechanisms to bulk mechanisms
   - Local stresses state in the sample is difficult to predict
3. **Electron beam effects**
   - Radiolysis and Knock-on Damage
4. **Vacuum conditions**
   - 10^{-7} Torr limits gas and liquid experiments feasibility
5. **Local probing**
   - Portions of the world study is small

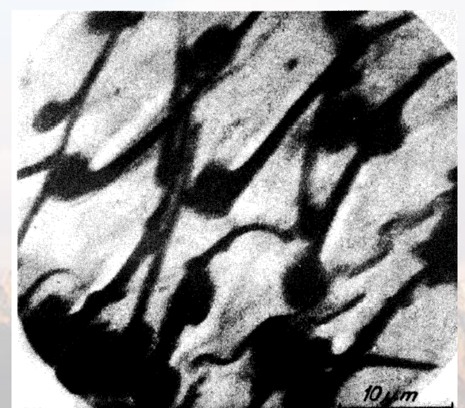


Fig. 6: Wing surface of the house fly.
(First internal photography, U = 60 kV, M_{el} = 2200)
(Driest, E., and Müller, H.O.: Z. Wiss. Mikroskopie 52, 53-57 (1935)

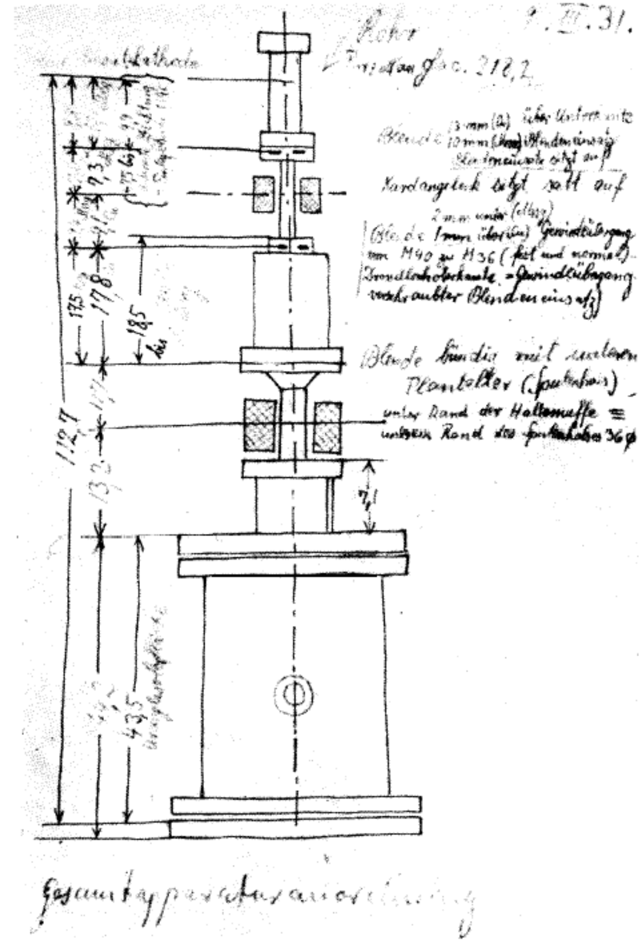


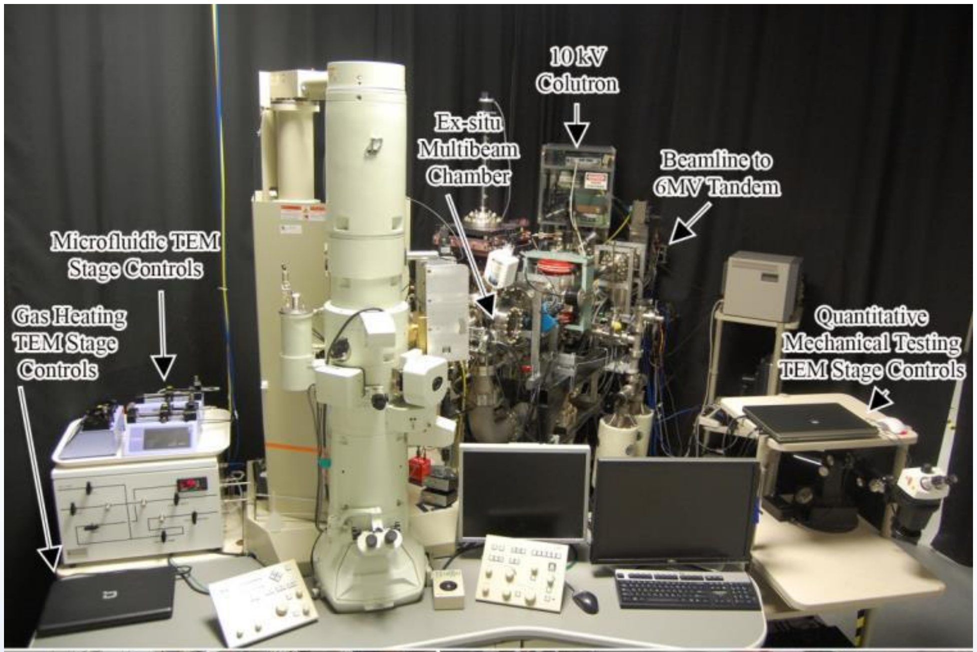
Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].

E. Ruska Nobel lecture (1986)

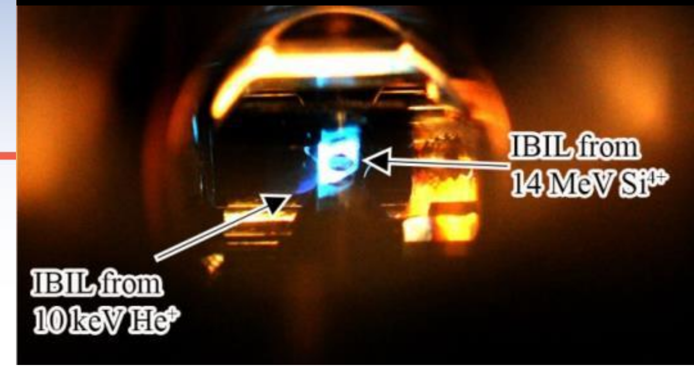
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem

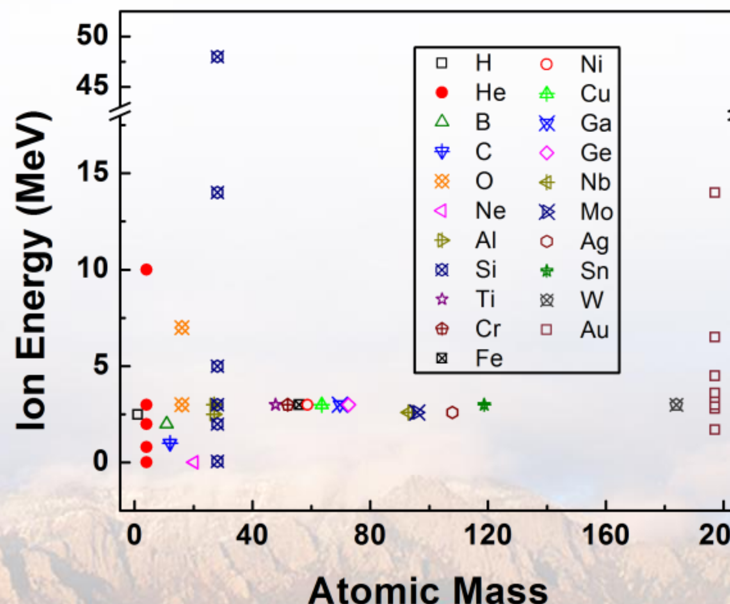


IBIL from a quartz stage inside the TEM



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

Ion species & energy introduced into the TEM

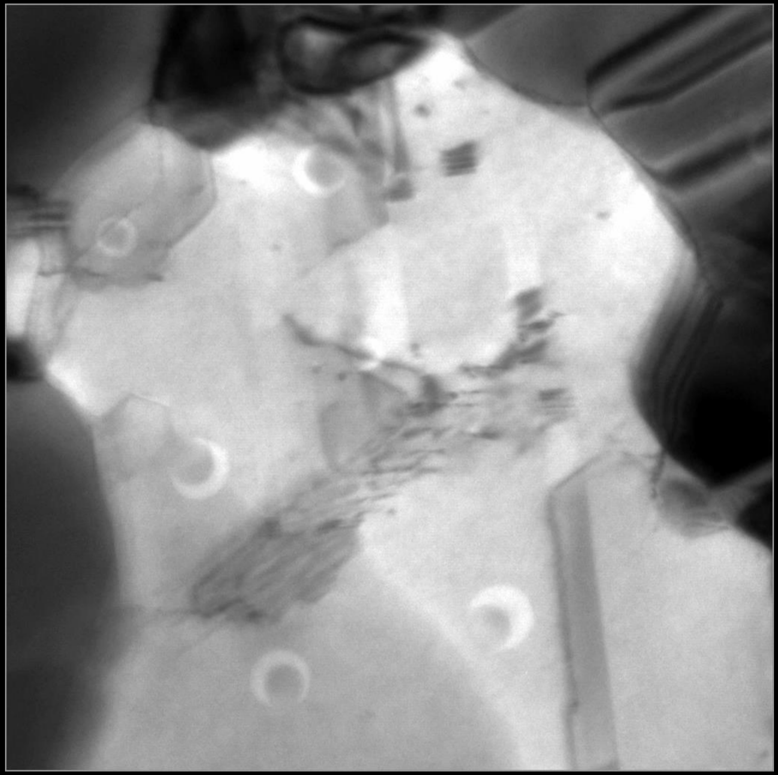


Sandia National Laboratories

Dose Rate Effects

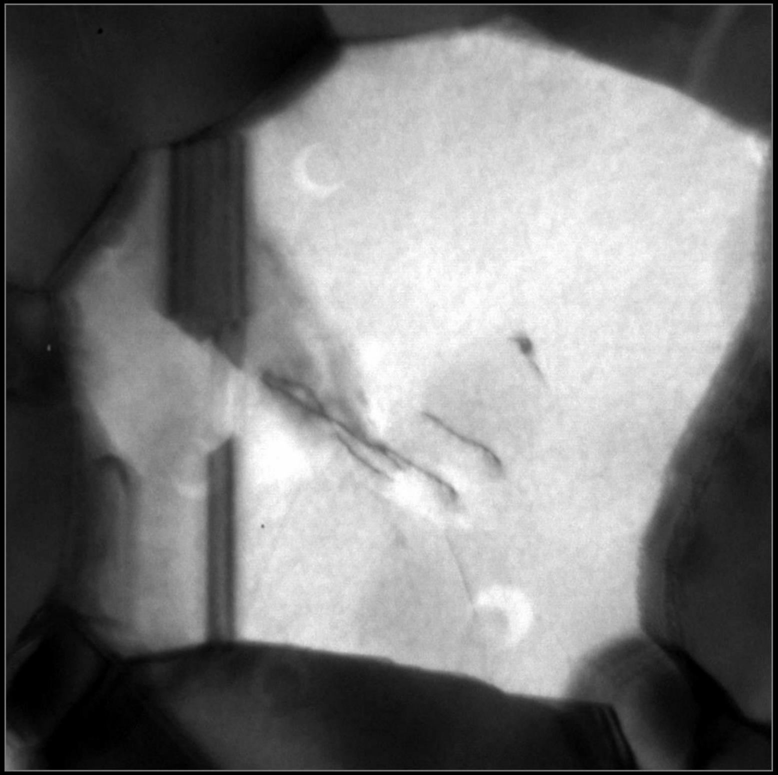
Collaborators: C. Chisholm , P. Hosemann, & A. Minor

7.9 x 10^9 ions/cm^2/s



VS

6.7 x 10^7 ions/cm^2/s

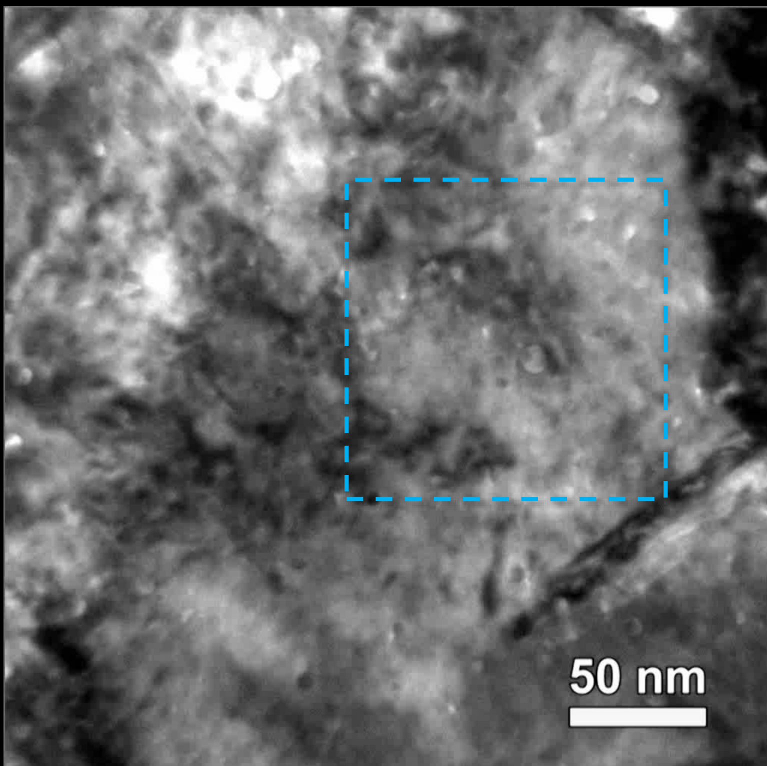


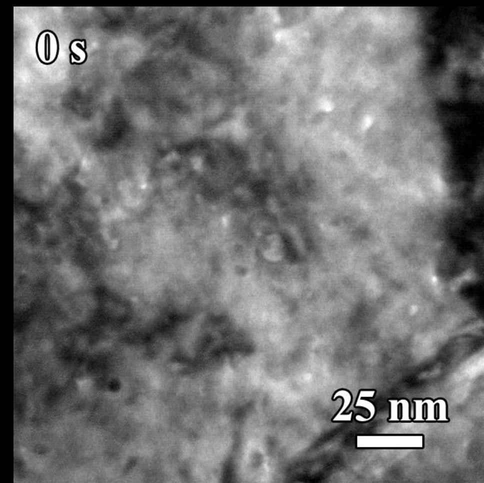
Improved vibrational and ion beam stability permits us to work at 120kx or higher permitting imaging of single cascade events

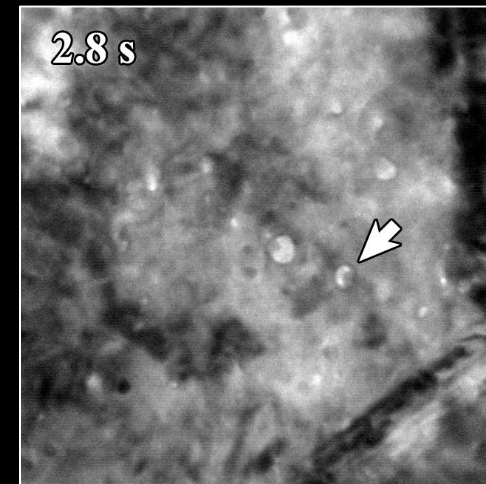
Simultaneous *In situ* TEM Triple Beam: 2.8 MeV Au^{4+} + 10 keV He^{+}/D_2^{+}

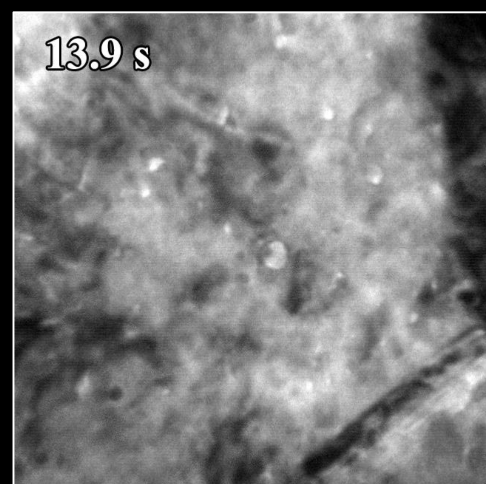
Collaborator: D.C. Bufford

Video playback speed x1.5.









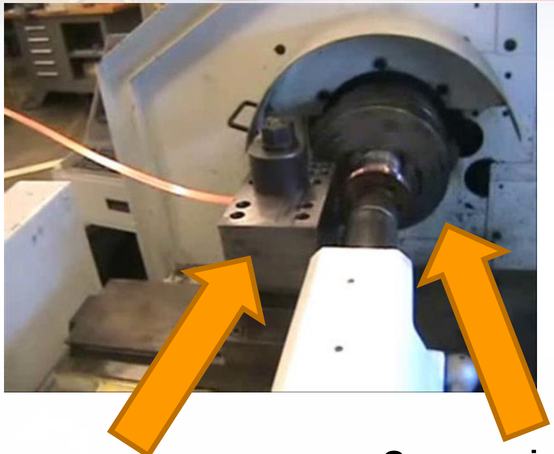
In-situ triple beam He, D_2, and Au beam irradiation has been demonstrated on Sandia's I^3TEM!

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

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

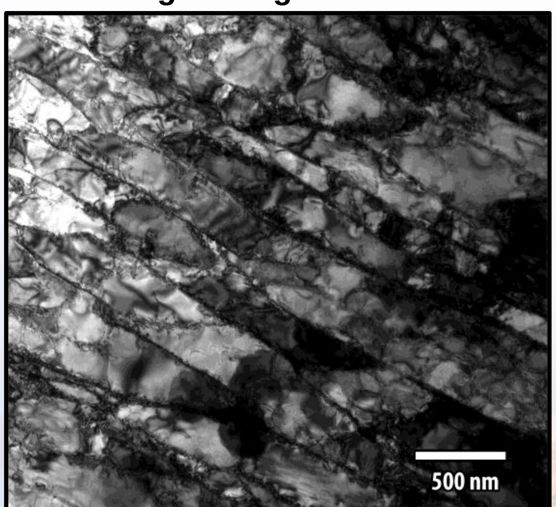
What Insight into Structural Stability is Gained from I³TEM Experiments?

Collaborators: O. El-Atwani, J. P. Allain, D. Buller, & J.A. Scott



Extrusion Machining tooling

Commercially available lathe

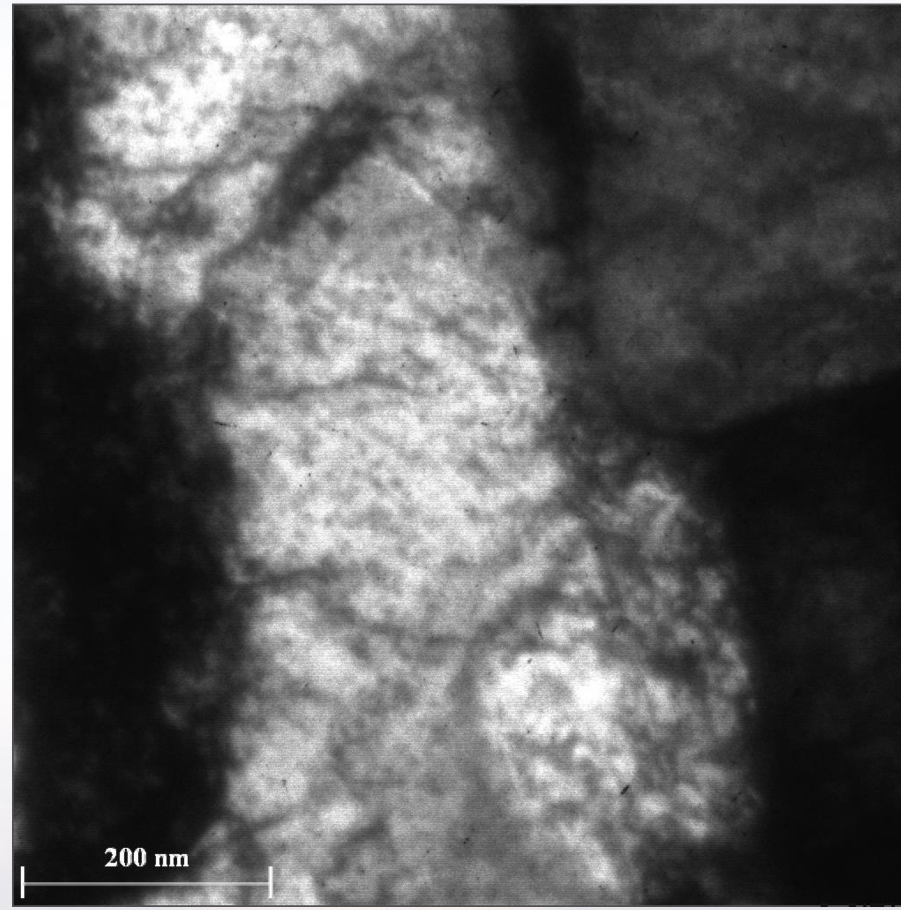


UFG Tungsten

From NW components through proposed NE cladding to waste storage:

Understanding Radiation Damage is Essential

- **I³TEM W irradiation and He implantation of SPD-W developed for ITER applications**



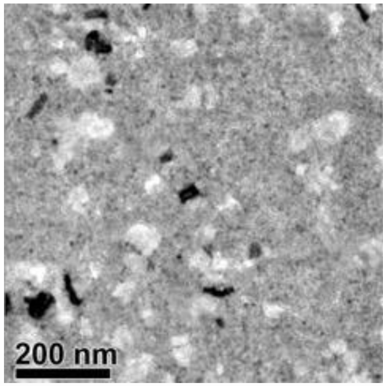
I³TEM is providing insight into:

1) Loop formation
2) Loop stability & migration
3) Rad & structural defect interactions

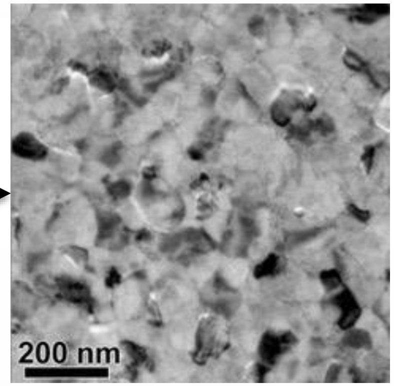
Scaling down to Nanocrystalline Tungsten Alloys

Collaborators: O.K. Donaldson, T. Kaub, G. Thompson, and J. Trelewicz

Nanocrystalline W

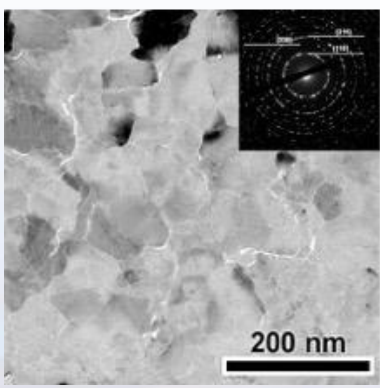


650 °C →

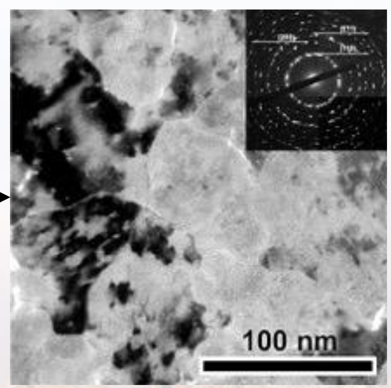


d_{avg} = 30 nm

d_{avg} = 86 nm



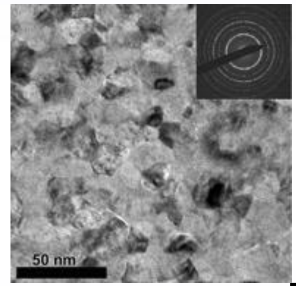
8.5 dpa →

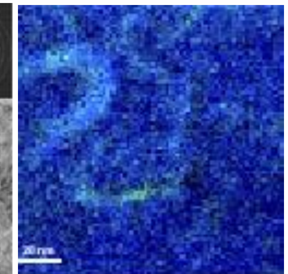


$\rho_{defect} = 2.5 \times 10^{22}\ m^{-3}$

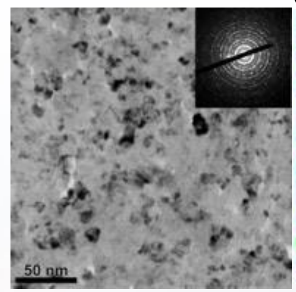
Alloying does not negatively effect radiation tolerance, while improving thermal and mechanical properties

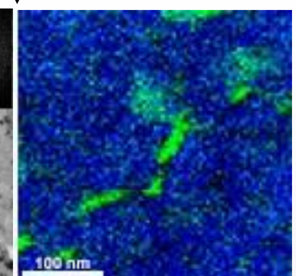
Nanocrystalline W-20at.%Ti





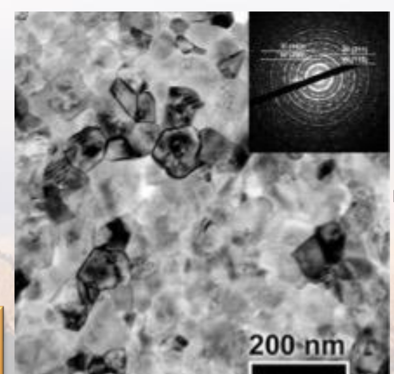
1000° C ↓ Anneal



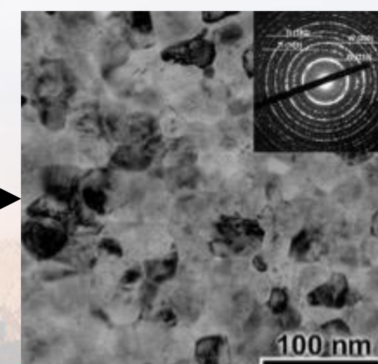


Grain growth is hampered by the addition of Ti

Ti solute is heterogeneously distributed after annealing



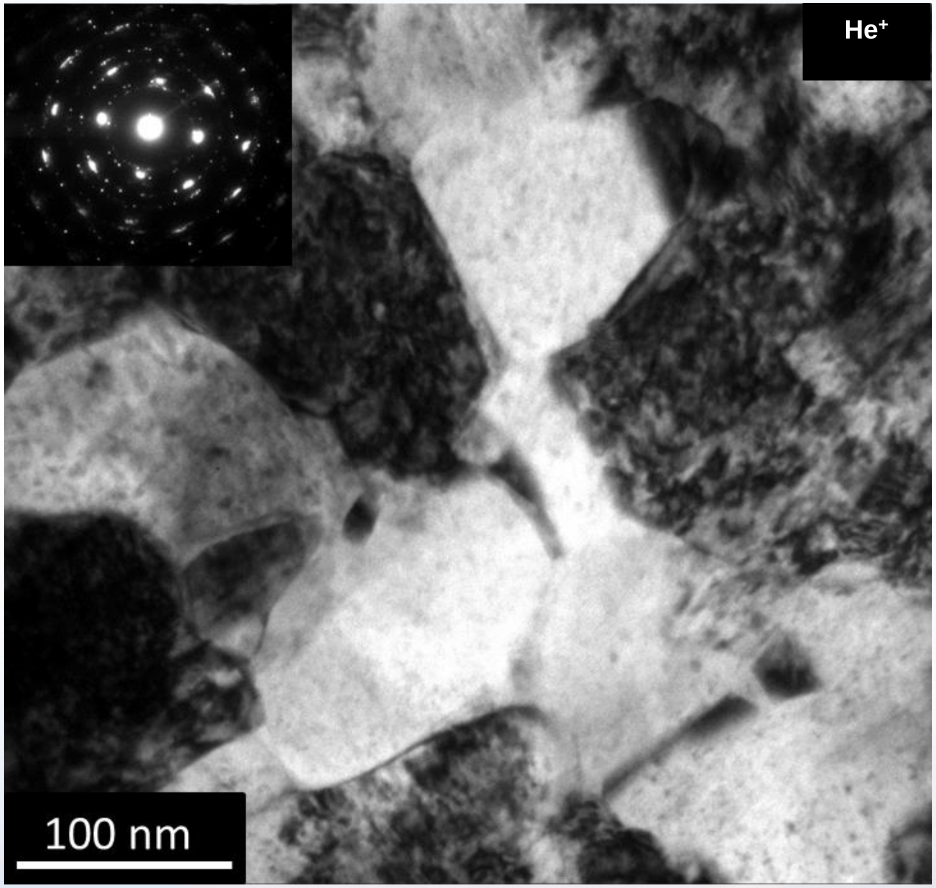
6.3 dpa →

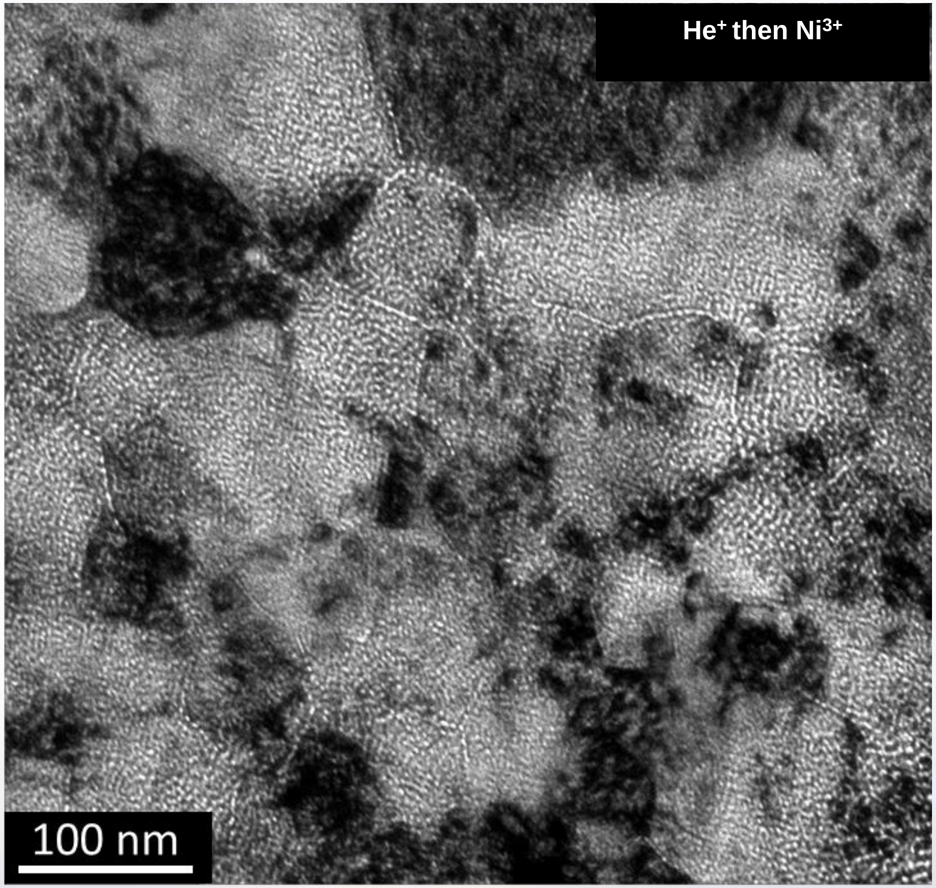


$\rho_{defect} = 2.8 \times 10^{22}\ m^{-3}$

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

Collaborator: B. Muntifering & J. Qu





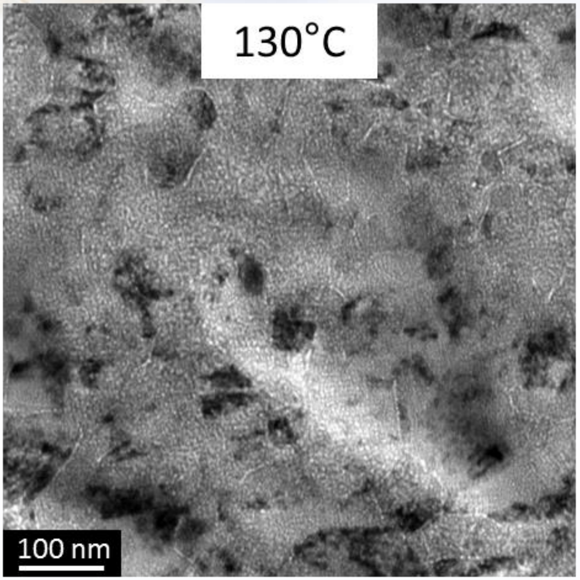
10^{17} He^+/cm^2

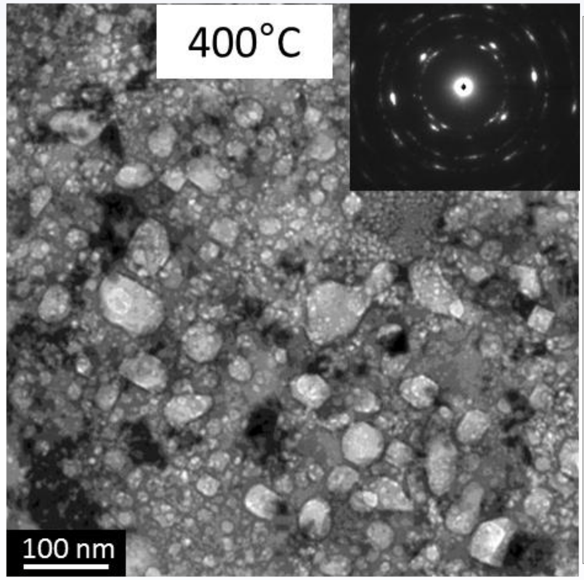
Visible damage to the sample

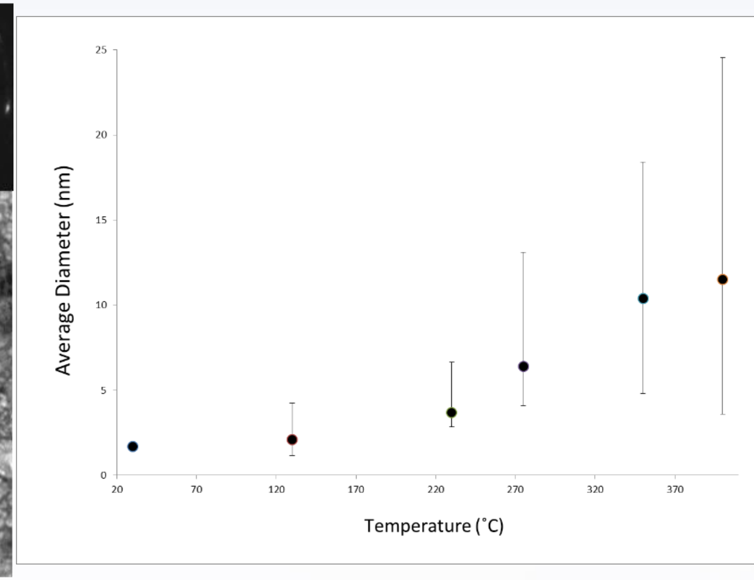
0.7 dpa Ni^{3+} irradiation

High concentration of cavities along grain boundaries

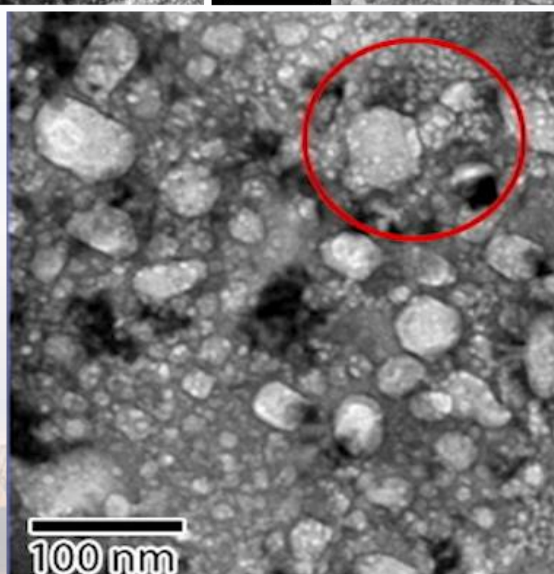
Cavity Growth during In-situ Annealing of 10 keV He^{+} Implanted and then 3 MeV Irradiated Ni^{3+}

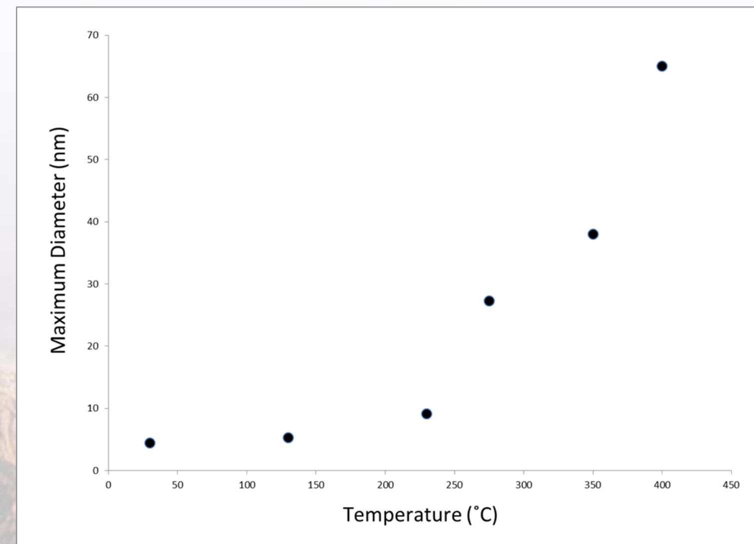






Bubble to cavity transition and cavity evolution can be directly studied



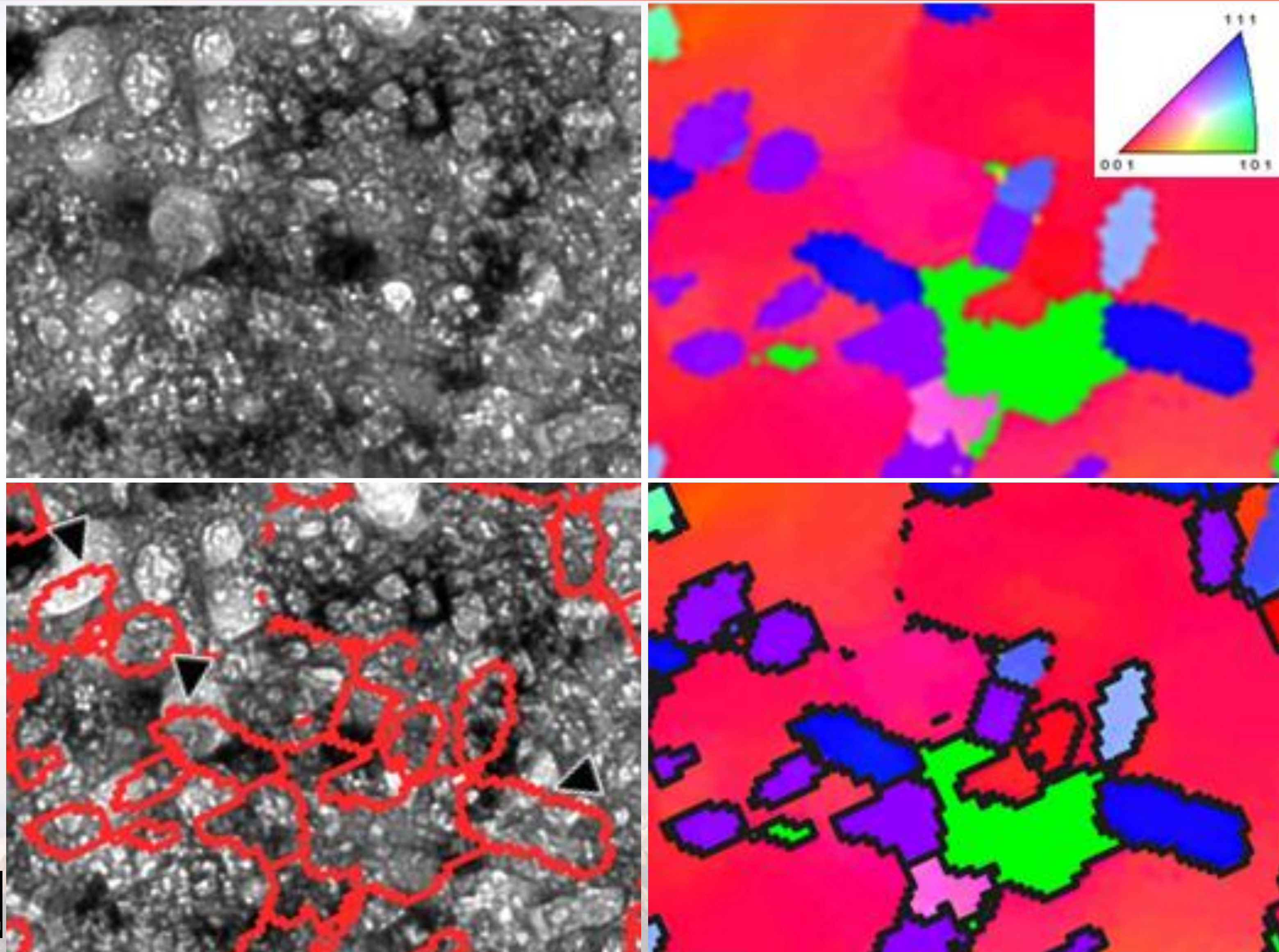


Precession Electron Diffraction Reveals Hidden Grain Structure

Cavities in helium implanted, self-ion irradiated,

nc nickel film annealed to 400 ˚C

Cavities span multiple grains at identified grain boundaries



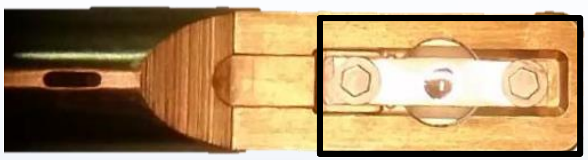
100 nm

In situ Qualitative Mechanical Testing

Gatan straining TEM Holder

- Minimal control over displacement and no “out-of-box” force information
- Successful in studies in observing dislocation-GB interactions/mechanisms
- Ideally both grains have kinematic BF 2-beam conditions: challenging in ST holder

Traditional Gatan Heating and Straining Holder

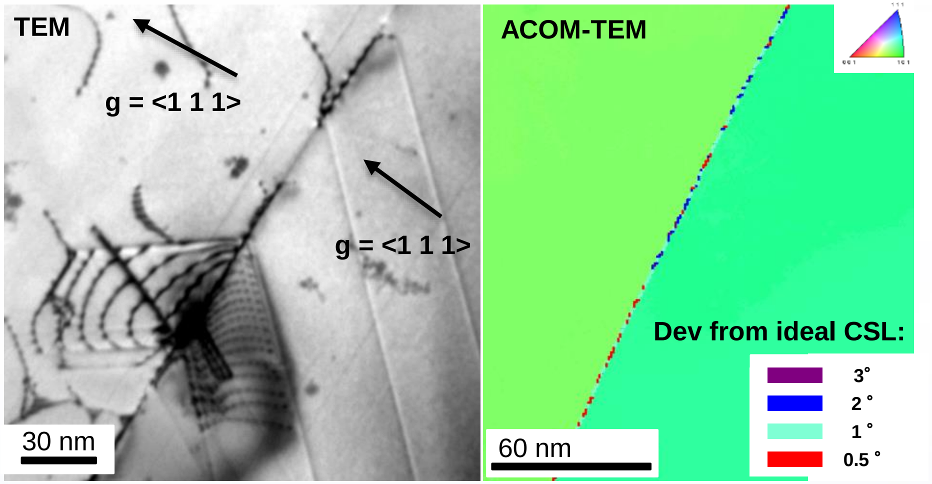


Thin film tension “jig”:

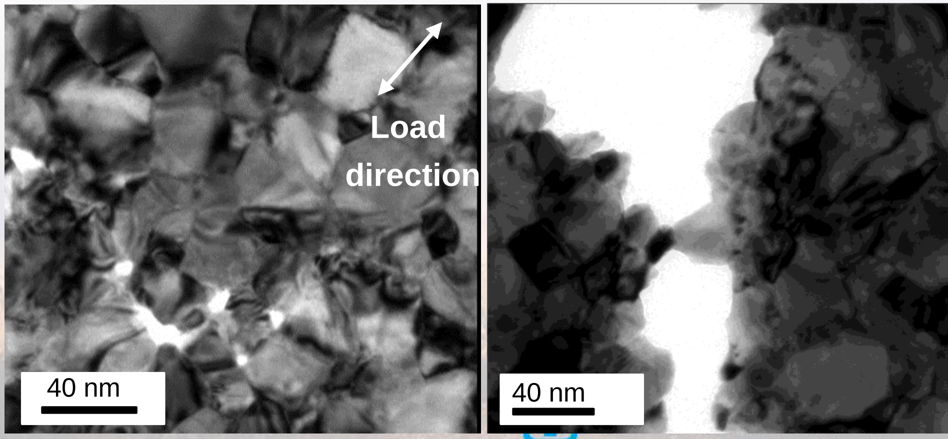
Traditional jet thinned disk

12 x 2.5 mm jigs, MEMs device, or jet thinned disk

Dislocation interactions as a function of GB character (Σ3 twin GB below):

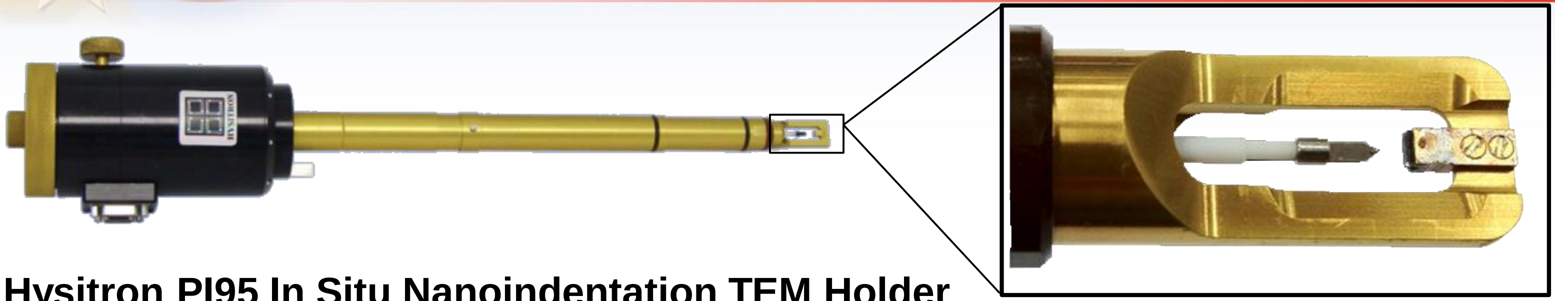


Observe deformation mechanisms in nanocrystalline metals during tensile straining:



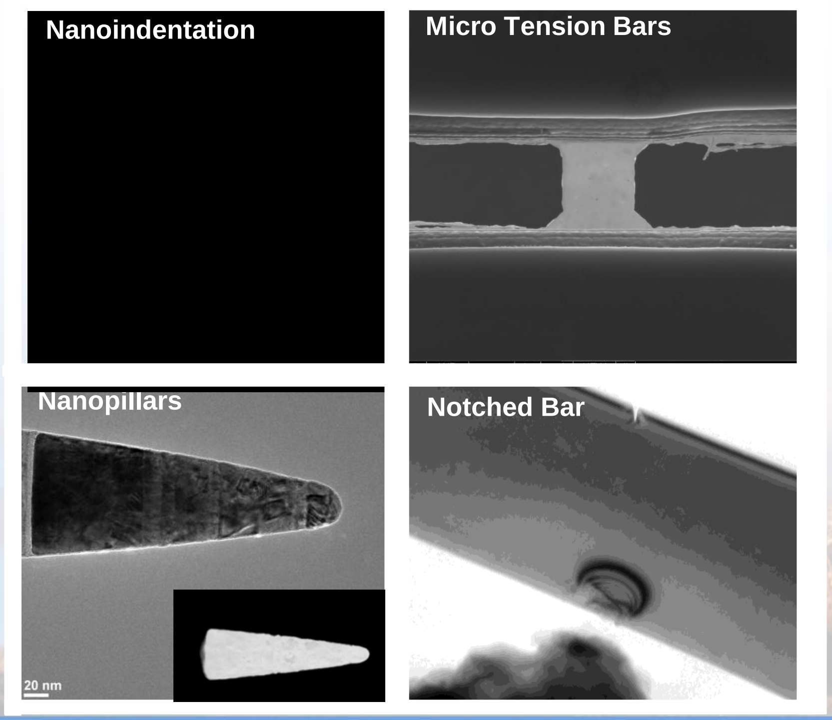
In situ Quantitative Mechanical Testing

Collaborators: Douglas Stauffer , Eric Hintsala, S.A. Syed Hysitron Bruker Inc.



Hysitron PI95 In Situ Nanoindentation TEM Holder

- **Sub nanometer displacement resolution**
- **Quantitative force information with μN resolution**
- **Concurrent real-time imaging**



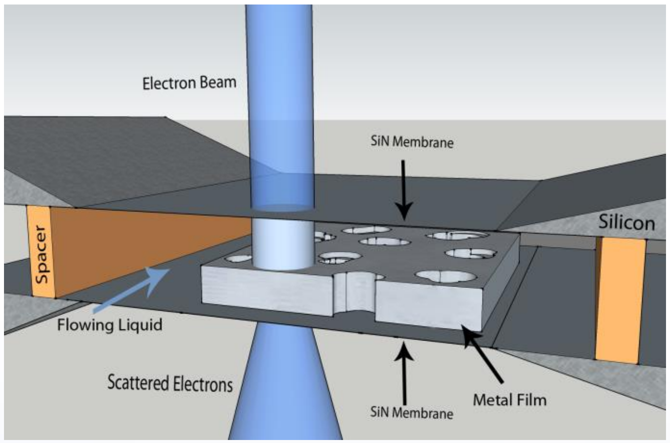
- **A variety of sample geometries**
- **Load functions examined at I^{3}TEM:**
  1) **Indentation**
  2) **Tension**
  3) **Fatigue**
  4) **Creep**
  5) **Compression**

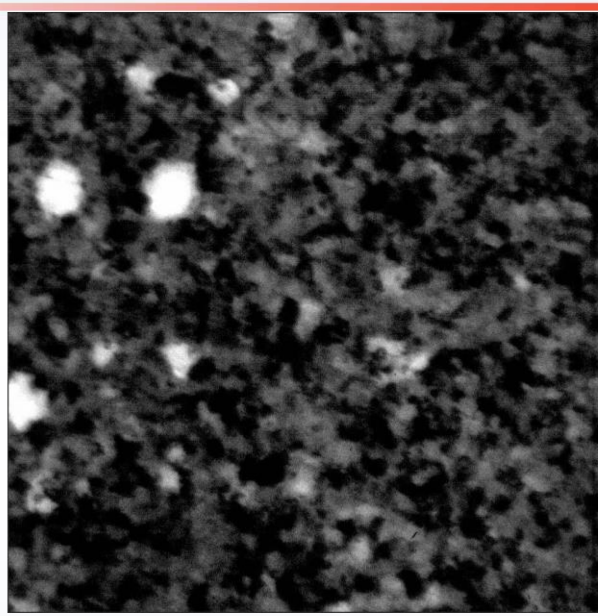
Can We Gain Insight into the Corrosion Process through *In situ* TEM?

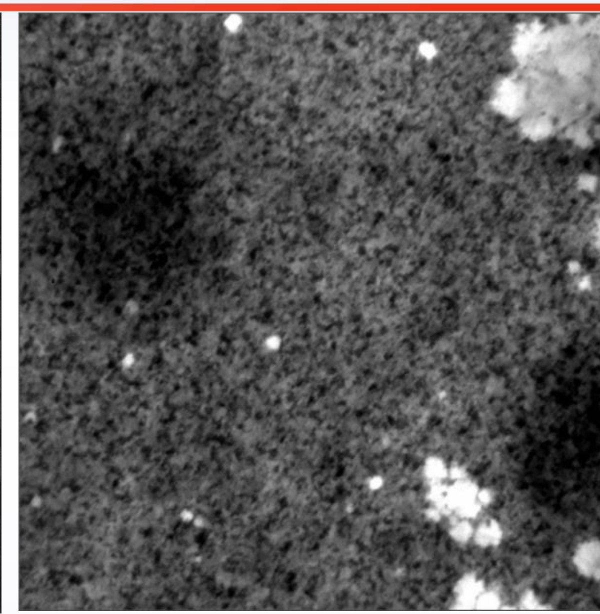
Contributors: D. Gross, J. Kacher, I.M. Robertson & Protochips, Inc.

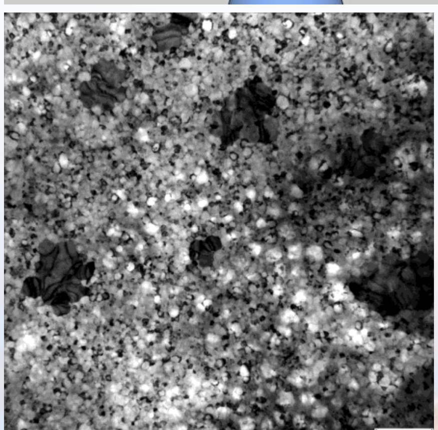
Microfluidic Stage

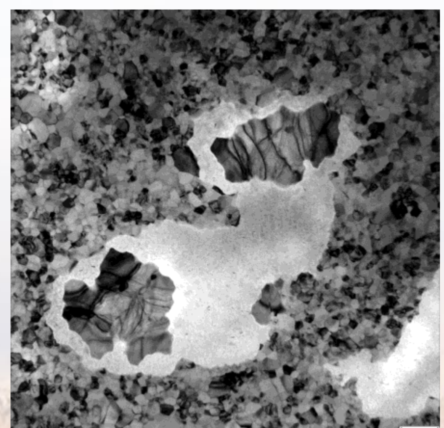
- Mixing of two or more channels
- Continuous observation of the reaction channel

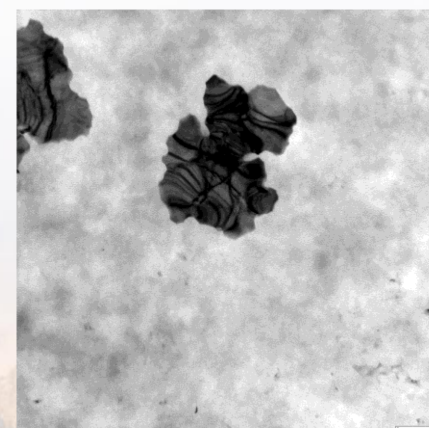










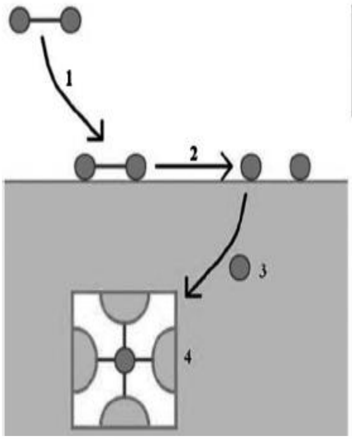


Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.

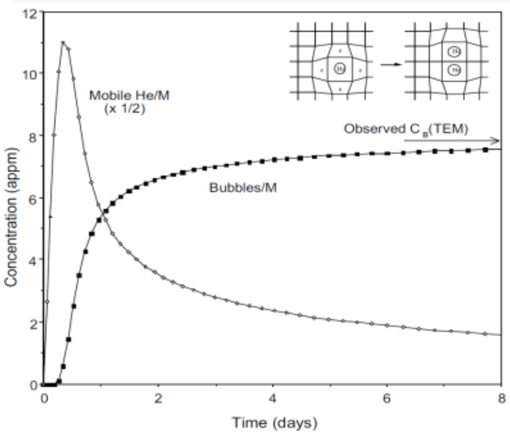
Large grains resulting from annealing appear more corrosion tolerant

In situ TEM Hydrogen Exposure

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



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

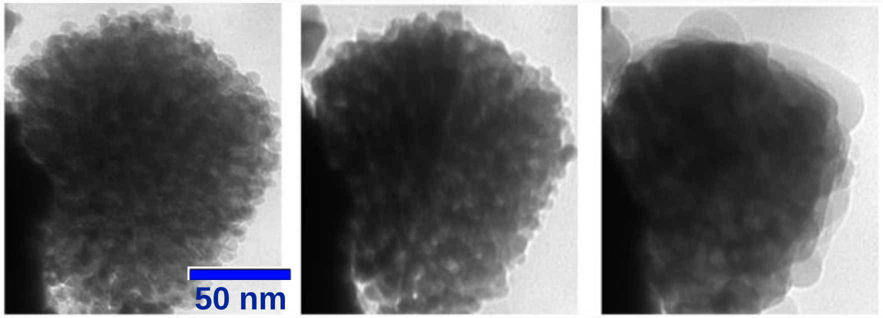


Cowgill, D., Fusion Sci. & Tech., 28 (2005) p. 539
Trinkaus, H. *et al*., JNM (2003) p. 229
Thiebaut, S. *et al*. JNM (2000) p. 217

Vapor-Phase Heating TEM Stage

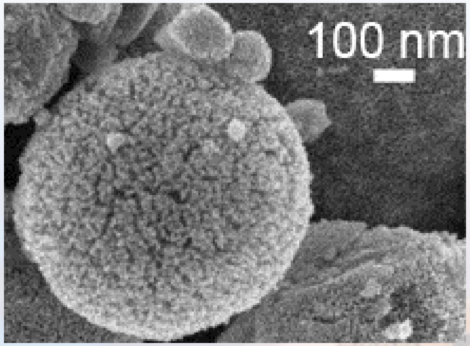
- Compatible with a range of gases
- *In situ* resistive heating
- Continuous observation of the reaction channel
- Chamber dimensions are controllable
- Compatible with MS and other analytical tools

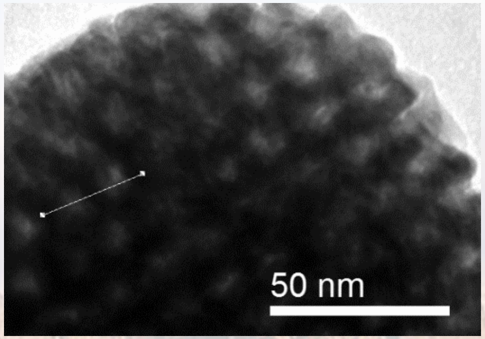
- 1 atm H_2 after several pulses to specified temp.



125° C 200° C 300° C

Harmful effects may be mitigated in nanoporous Pd





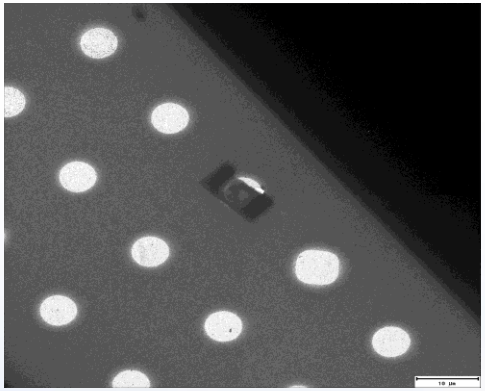
New *in situ* atmospheric heating experiments provide great insight into nanoporous Pd stability

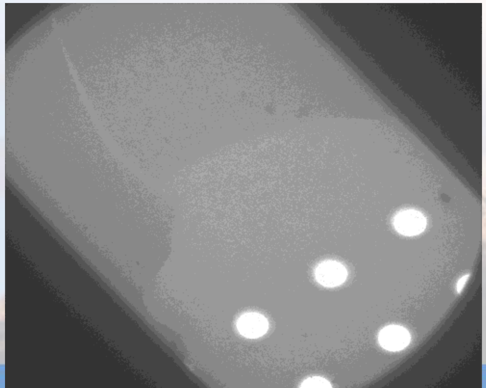
Feasibility of Studying Zircaloy 2 at Nominally 1 atm

Collaborators: S. Rajasekhara and B.G. Clark

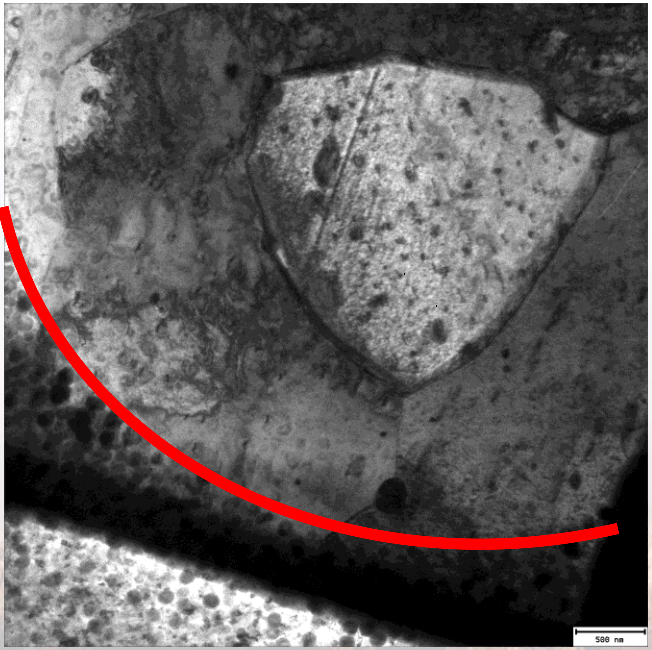
Vapor-Phase Heating TEM Stage

- 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

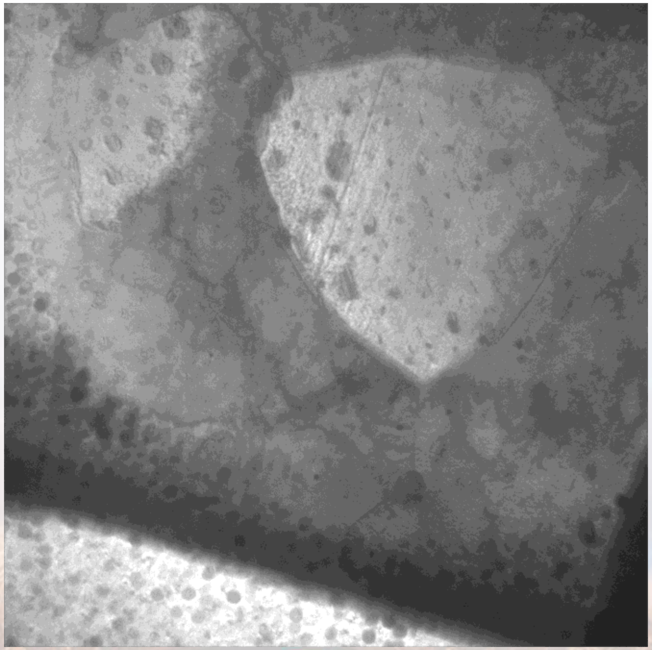




Vacuum & Single Window



Nominally 1 atm H_2 & Two Windows



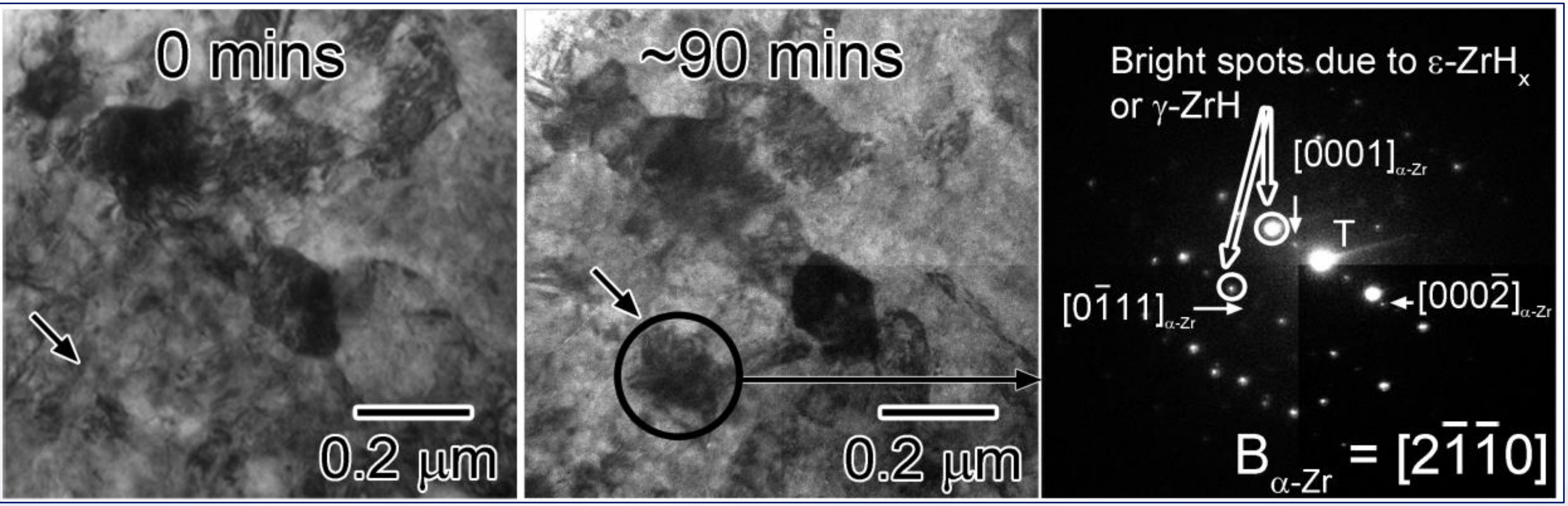
Most features are observed in both despite the decreased resolution resulting from the additional SiN window and 5 µm of air

In situ Observation of Hydride Formation in Zirlo

Collaborators: S. Rajasekhara and B.G. Clark

Absolute hydrogen pressure: 327 torr (~ 0.5 atm),

Ramp rate: 1 ^{o}C/s, Final temperature: ~ 400 C, Dwell time: ~ 90 mins

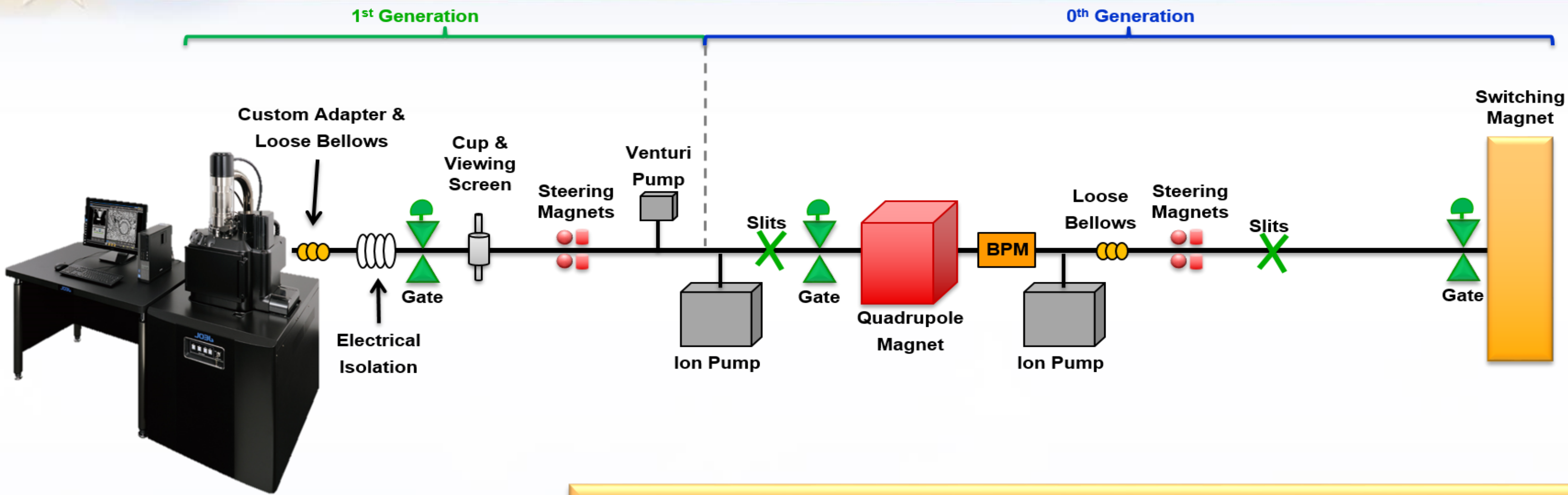


Hydride formation shown, for the first time by use of a novel TEM gas-cell stage, at elevated temperature and hydrogen pressure

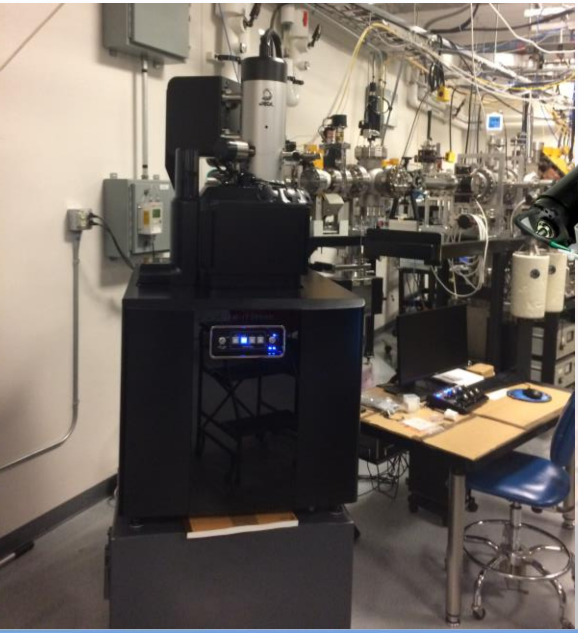
Schematic of the *In situ* SEM Beamline

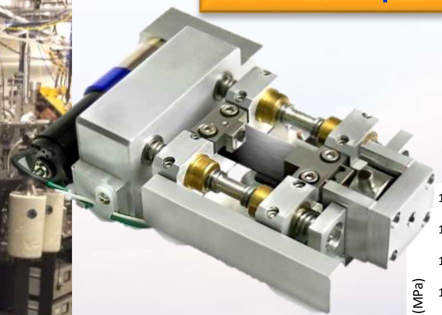
Collaborators: D.L. Buller & S. Briggs

8/24/2017

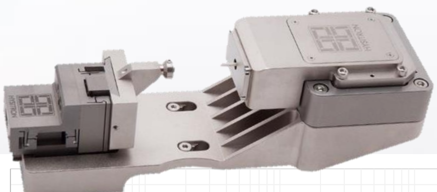


Beam Line planned for the *in situ* SEM will be developed in phases. Ultimate plan is for multiple accelerators being attached for dual or triple beam experiments.

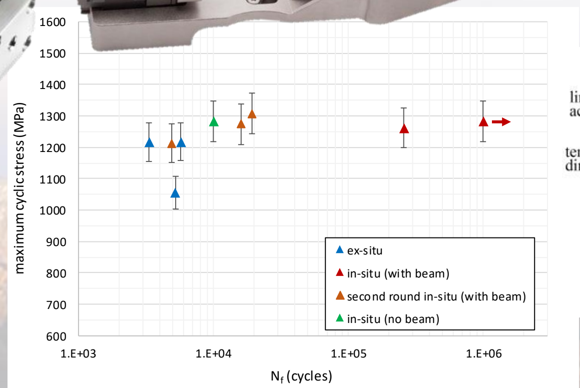




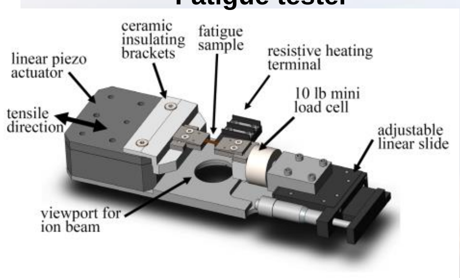
MTI Fullam Straining Heating



Hysitron PI85 Nanoindenter

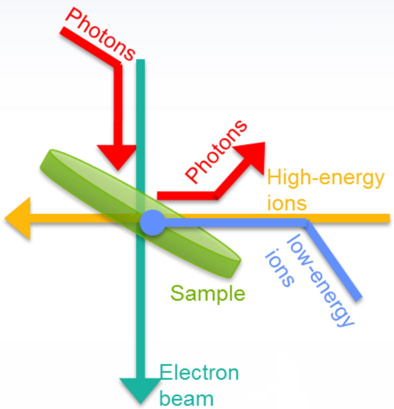


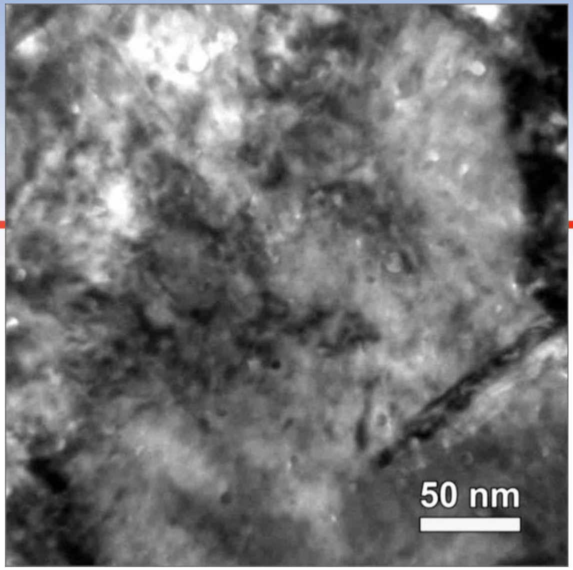
Custom-built Piezo Fatigue tester



Summary

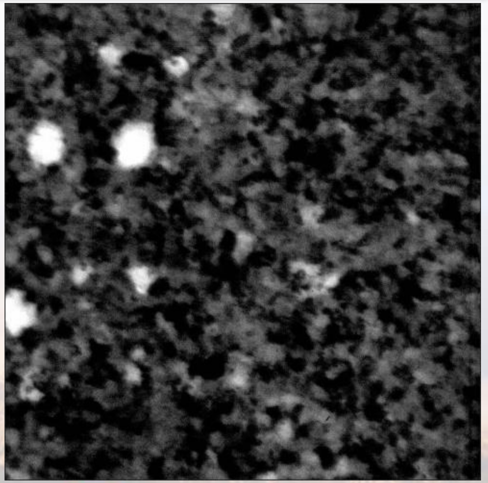
- Sandia's I³TEM capabilities:
  - *In situ* high energy ion irradiation from H to Au
  - *In situ* gas implantation
  - Heating up to 1,000 ˚C
  - Quantitative and bulk straining
  - Two-port microfluidic cell
  - Gas flow/heating stage
  - Electron tomography
  - Precession Electron Diffraction





- Currently applying the current I³TEM capabilities to various material systems in combined and harsh environmental conditions
- Sandia's I³TEM future capabilities being developed:
  - In situ ion irradiation TEM in liquid or gas (currently capable)
  - DTEM: Nanosecond resolution (laser optics being developed)
  - Beamline: Add 1 MV NEC Tandem & convert 90˚ magnet to bend beams 45˚

Sandia's I³TEM although still under development is providing a wealth of interesting initial observations and harsh environments



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

Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC., a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

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