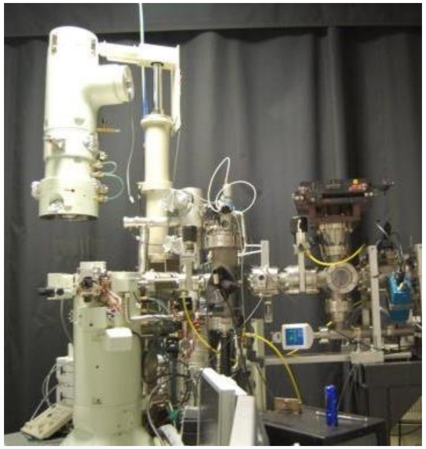
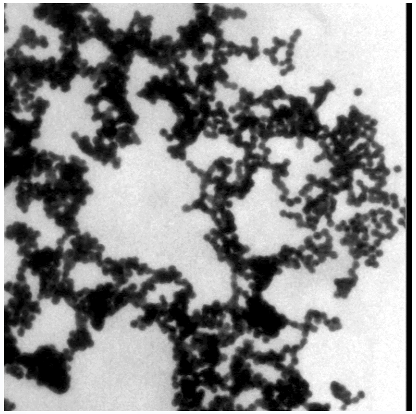
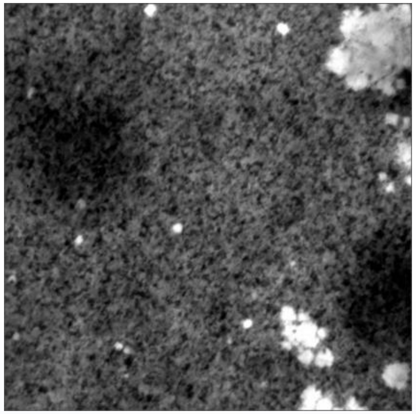
Exploring Materials in Extreme Environments with Nanometer Resolution

SAND2017-13003PE

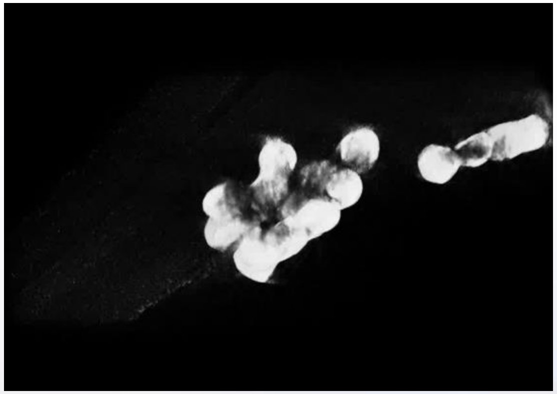
K. Hattar
Sandia National Laboratories
November 14, 2017







***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, B.L. Doyle, S. H. Pratt, & M.T. Marshall**
- **Sandia: B. Boyce, T.J. Boyle, P.J. Cappillino, J.A. Scott, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, E. Carnes, J. Brinker, D. Sasaki, J.A. Sharon, T. Nenoff, W.M. Mook**
- **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 supported by the US Department of Energy, Office of Basic Energy Sciences.

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Potential Evolution of System Design

Use the Nearest Stone

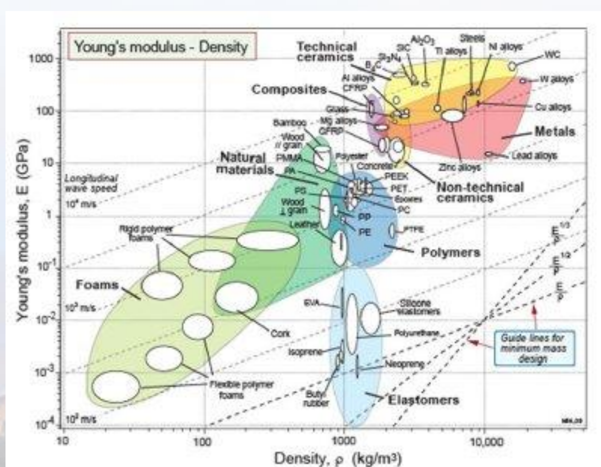
to

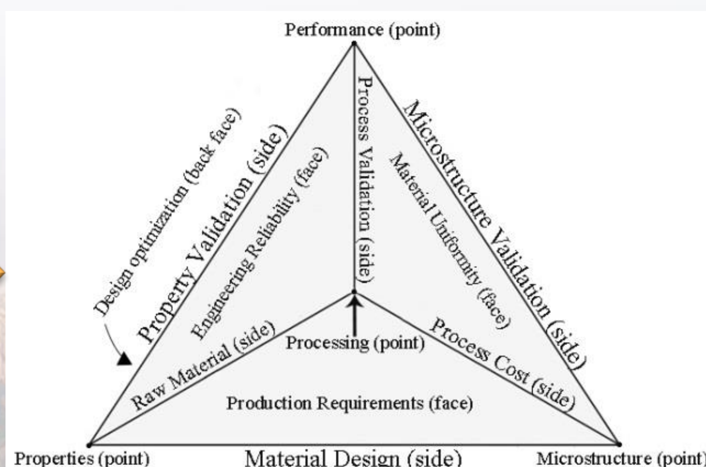
- Radar charts and Ashby plots of current material
- Accelerated and field testing
- **Scientist create a new materials. Engineers find an application.**

Materials by Design

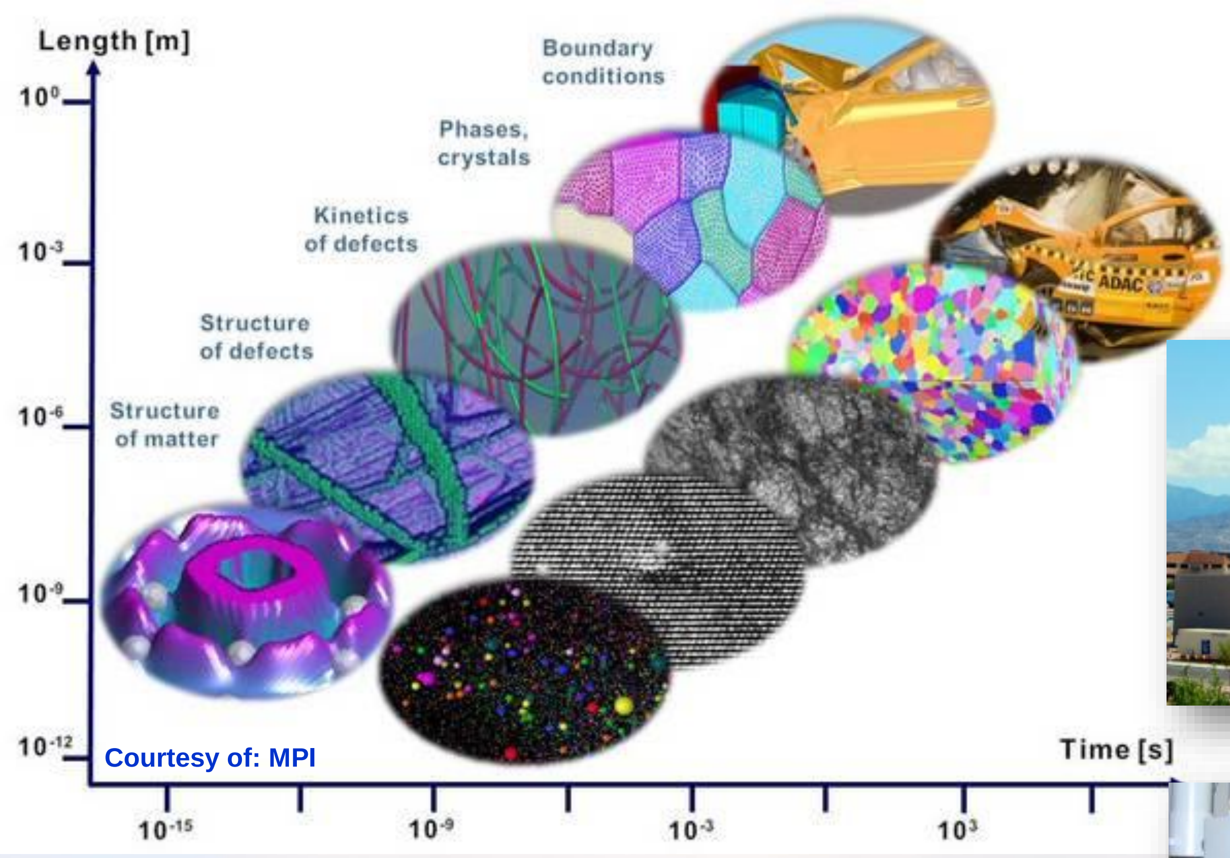
- **Physics-based approach**
- **Requires multiscale modeling**
- **Engineers require given properties, Scientists tailor the chemistry and microstructure to achieve it.**

Great vision! We are making strides, but we are not there yet





Investigating the nm Scale to Understand the km Scale Response of Materials in the Extremes



Ion Beam Lab (IBL)

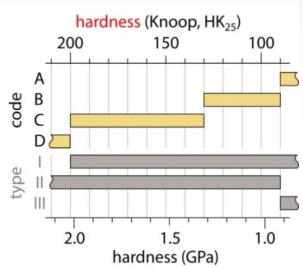
In situ Ion Irradiation TEM (I^{3}TEM)

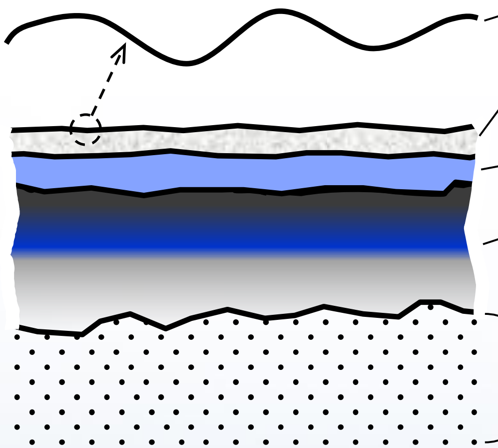
To develop predictive physics-based models, a fundamental understanding of the structure of mater, defects, an the kinetics of structural evolution in the environments of interest are needed

Nature of Metallic Surfaces

Electronics Applications (connectors, switches and relays):

- Gold desired because it is chemically Inert and has low electrical resistance
- Pure gold is soft and has unacceptable amount of friction and wear
- Gold is typically hardened by alloying with small amounts of Ni or Co





No surface is perfectly flat

A

B

C

A: Physisorbed/Chemisorbed

B: Oxides (Chemically Reacted)

C: Deformed layers

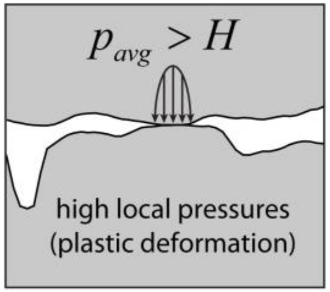
Undeformed Base Metal/Alloy

From ASTM B488-11 / MIL-DTL-45204D:

	type	suggested applications (ASTM)
> 99.7% Au	I	general-purpose, high-reliability electrical contacts
(hardest) > 99.0% Au	II	general-purpose, wear resistance; low temperature only
(softest) > 99.9% Au	III	soldering; limits impact of oxidation of codeposited material
	IIIA	semiconductor components, nuclear eng., high temperature

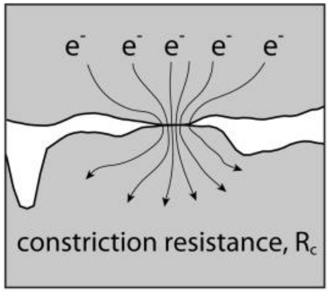
Asperity Contacts, Constriction, Asperity Contacts and Surface Films

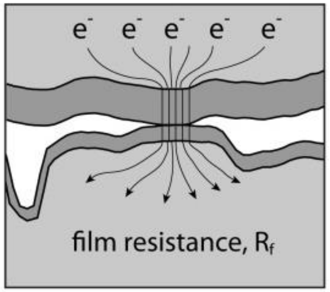
areal sum of asperity contacts and surface films define electrical contact resistance



... for metal contacts the real area is a function of hardness and contact force (Bowden & Tabor, 1939):

$$A_r \cong \frac{F_n}{H}$$





... ECR is a function of the constriction and film resistances:

$$ECR = \sum_i \left(R_{c,i} + R_{f,i}\right)$$

Archard, JAP (1953) 24:981
R. Holm, Electrical Contacts Handbook (1958)
Greenwood & Williamson, P. roc. Royal Society (1966)
T.W. Scharf & S.V. Prasad, JMS (2013) 48:511-531

Can We Get Away from Electrochemical Hard Au?

Electroplating:

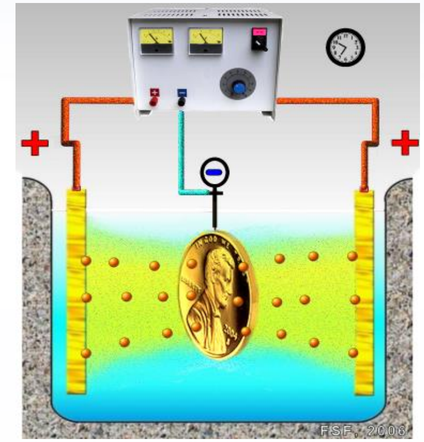
Hard Gold is typically plated from a cyanide precursor

$KAu(CN)_2$

Can contain bath modifiers of As, Pb, Cd or Tl

Small amounts hardening agent such as Ni or Co

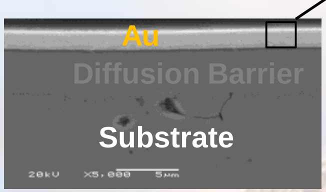
Nickel barrier layer applied first

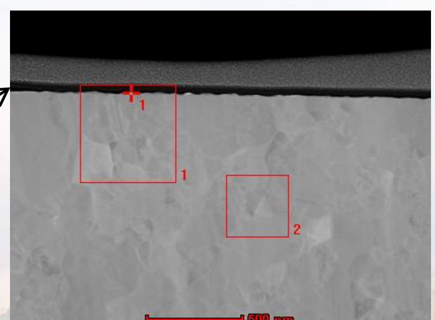


Diffusion and Surface Oxidation:

Diffusion and segregation of Ni or Co hardeners to the surface over time, ECR degradation

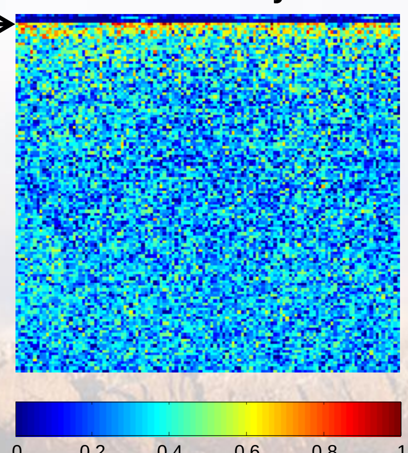
Electroplated Hard Au does not Age well and can be Toxic!



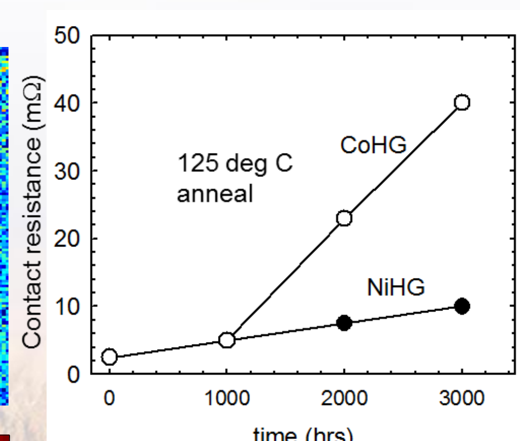


Surface NiO layer

Relative Ni concentration After 35+ years



Cross-sectional TEM and Spectral Imaging

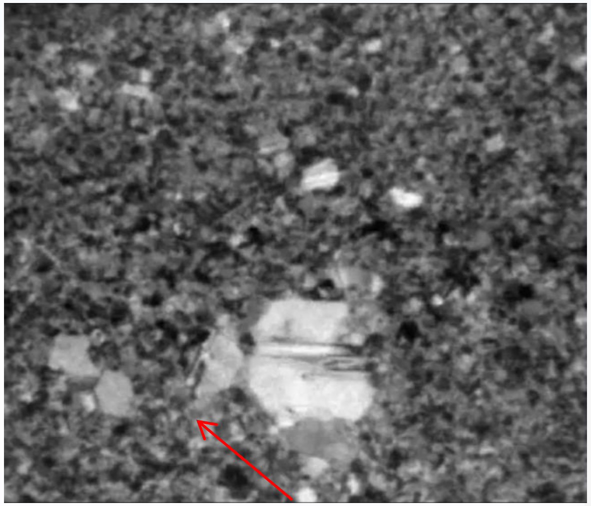


*Y. Okinaka and M. Hoshino, Gold Bulletin, **31**(1), 3 (1998).*

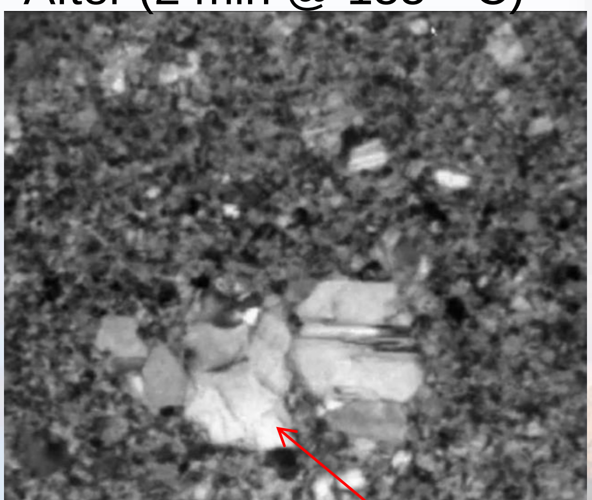
Exploring the Thermal Stability of PVD Hard Gold

Collaborators: J-E Mogonye, S.V. Prasad, J. Machael, R. Goeke, and P. Kotula

Before

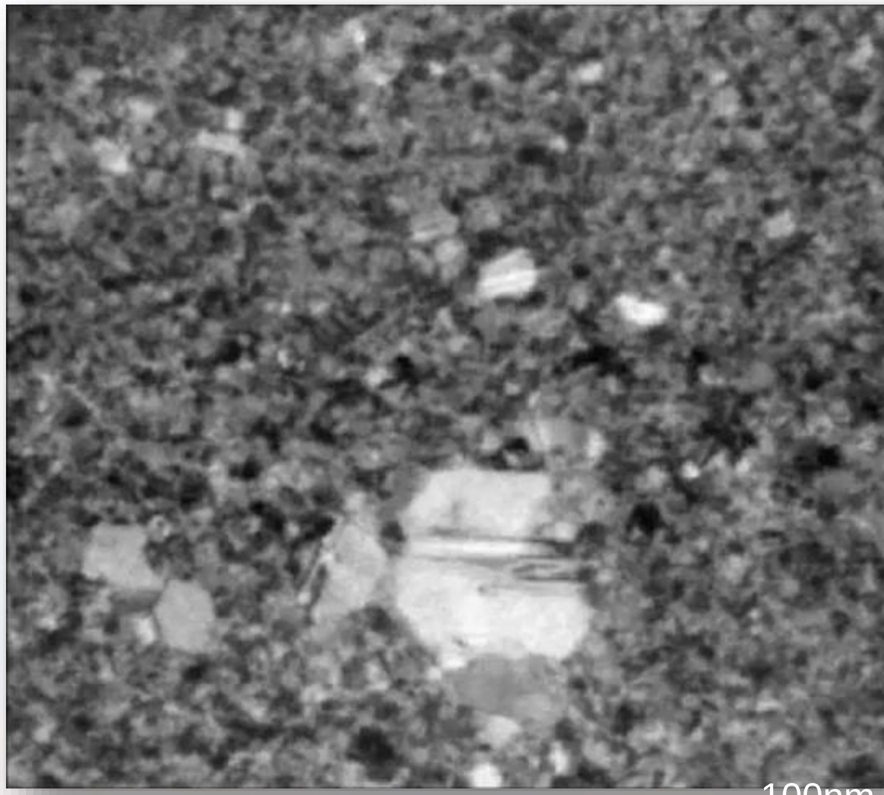


After (2 min @ 155° C)



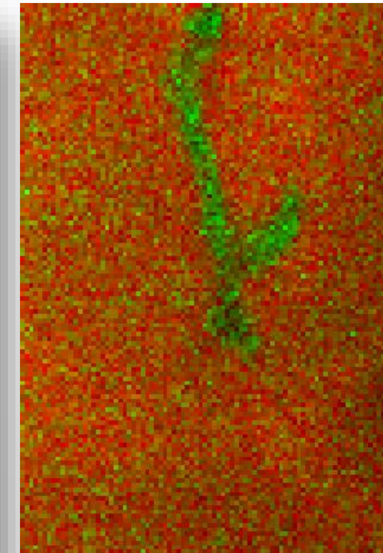
Abnormal grain growth dominates the thermal stability of Au+ZnO

Au + ZnO 5%

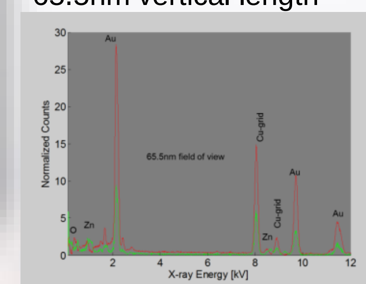


Speed in 16X real time

Zn/Au ratio



65.5nm vertical length



- *Au+ZnO can be co-deposition by PVD (e-beam evaporation or sputtering)*
- *Au+ZnO shows good tribology properties*
- *PVD is environmentally friendly*

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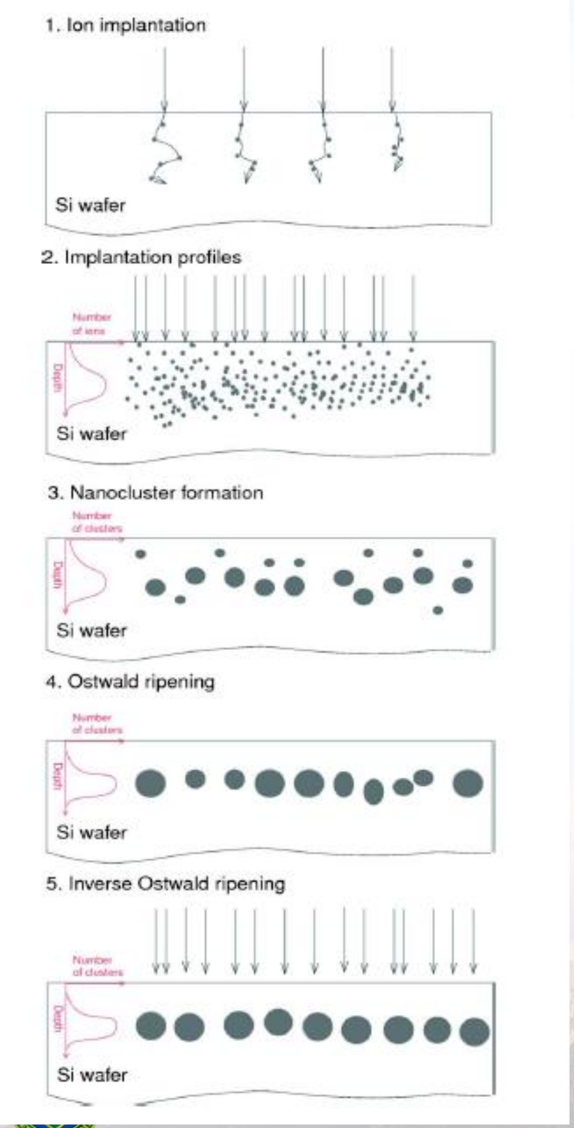
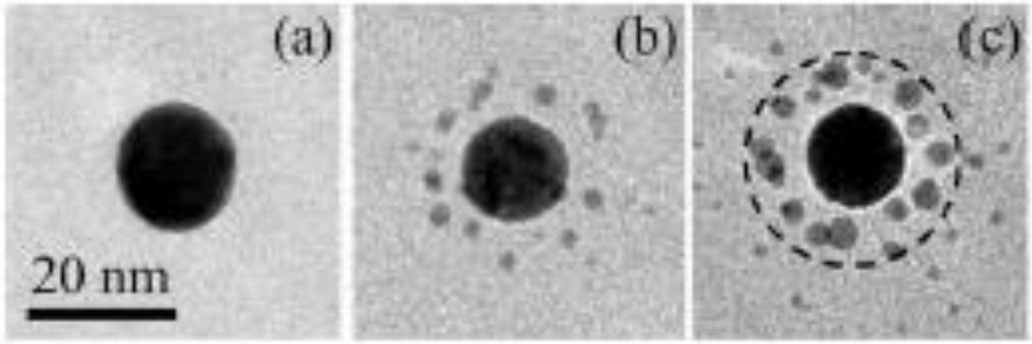
Previous Studies Investigated Embedded Particles



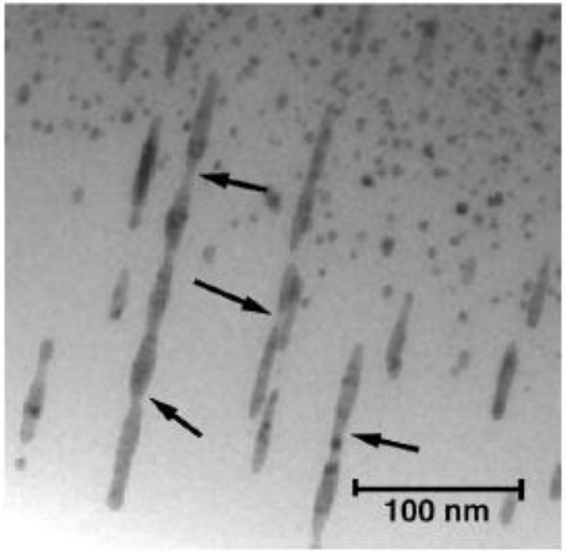
A.V. Krasheninnikov and K. Nordlund. *J. Appl. Phys.* 107 (2012) 071301.



G. Rizza et. al. *Phys. Rev. B 76* (2007) 245414.

Au NCs in SiO_2 matrix irradiated at increasing fluences with 4 MeV Au ions at room temperature. Two generations of satellites observed.

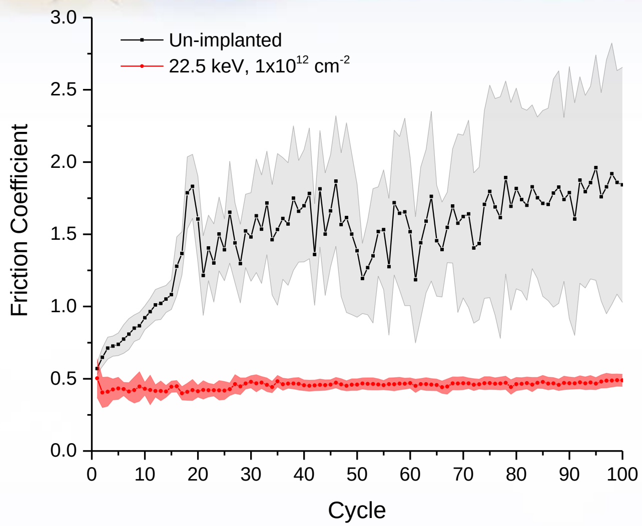
Ion tracks can be formed in SiO_2 matrices and nanoparticle elongation and nanowire formation has been observed. Pt NP are shown, irradiated with 185 MeV Au ions. Arrows point to nanorod fragments.

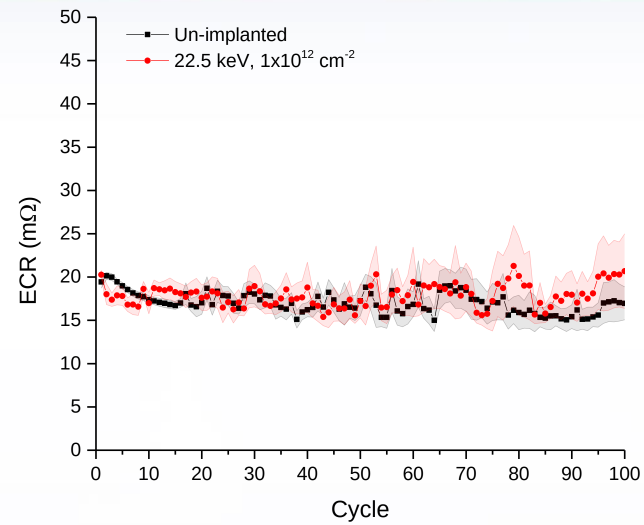


M.C. Ridgway et. al. *NIM B 267* (2009) 931.

He Implantation Greatly Improves Wear Properties

Collaborators: J-E Mogonye & S.V. Prasad

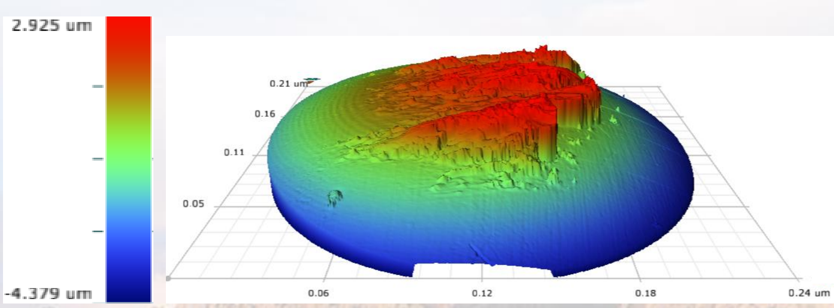




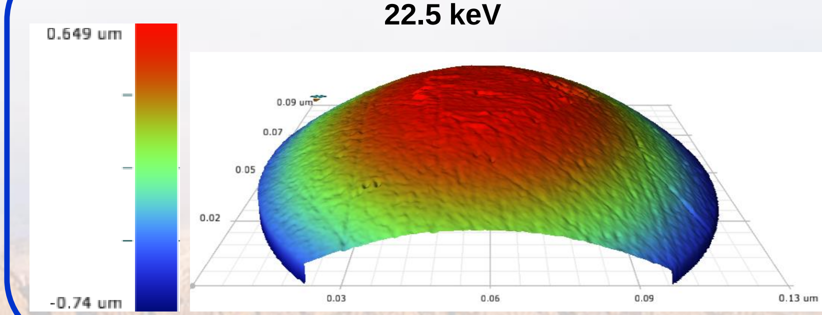
Friction is significantly reduced with ^{3}He implantation while maintaining ECR performance

Scanning white light interferometer topographical construction of riders after 100 cycles

Rider after 100 cycles against Un-implanted Au



Rider after 100 Cycles against Au implanted to 1E12 cm^{-2} @ 22.5 keV



Wear is significantly reduced with minimal effect in ECR

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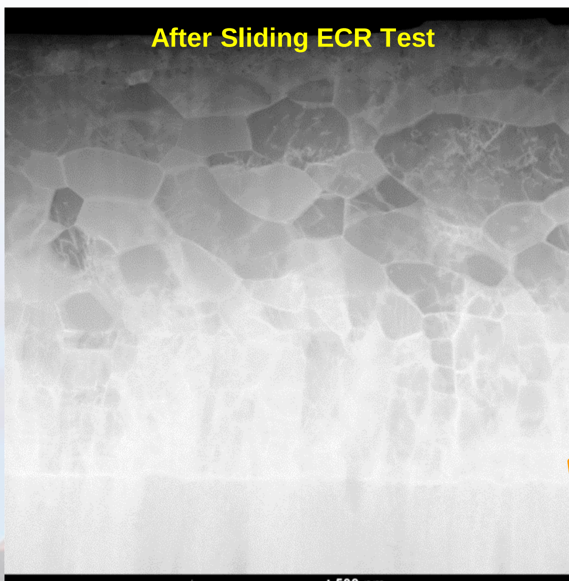
STEM Images of Sub-surfaces Provides Insight

Collaborators: P. Kotula, J-E Mogonye & S.V. Prasad

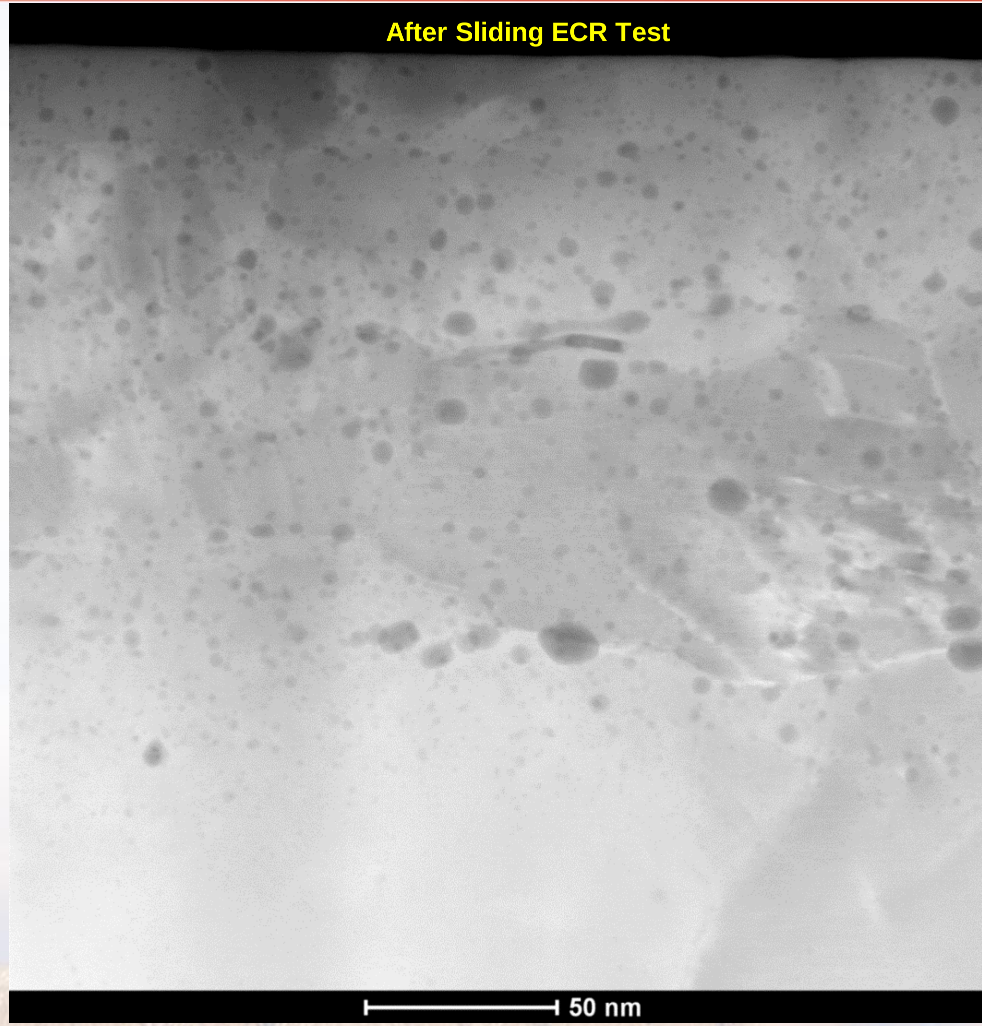


22.5 keV

1 x 10^{12} cm^{-2}

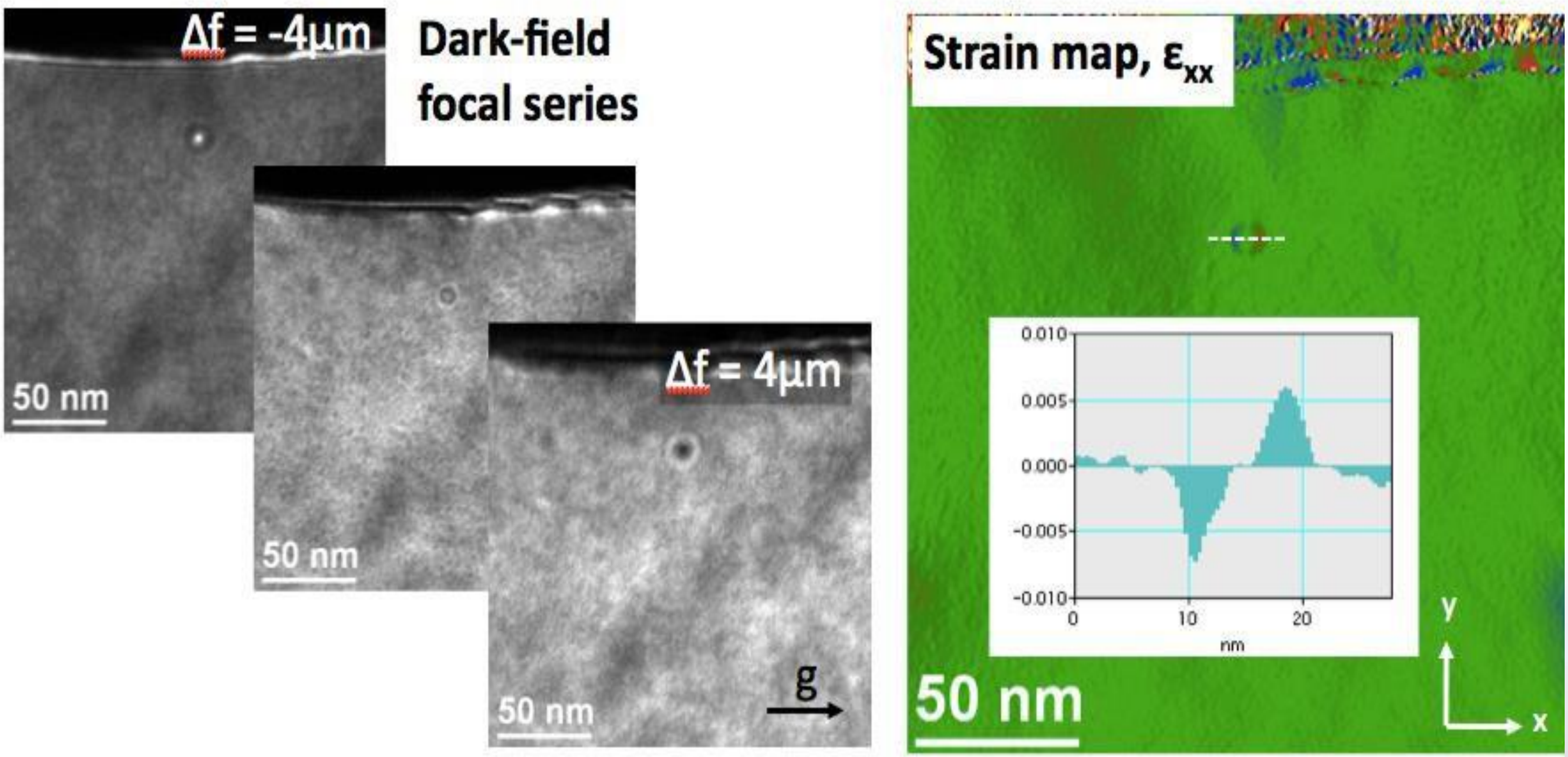
Recrystallization is observable after 100 cycles

Au – Pt Interface



An increase in both observable density and diameter of He bubbles, suggests wear induced He coalescence from interstitial and previously un-observable He

Strain mapping at a He bubble in Au



In-line Holography Suggests that Significant Local Strain May Contribute to Enhanced GB Stability and Subsequent Wear Properties

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

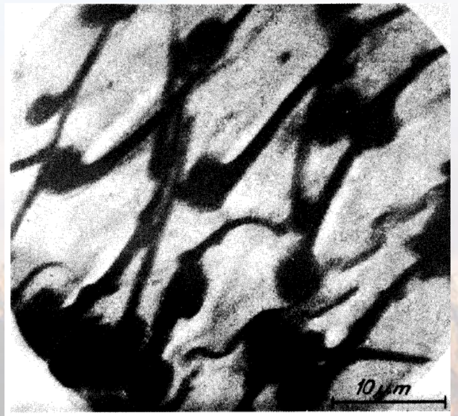


Fig. 6: Wing surface of the house fly.
(First internal photography, U = 60 kV, M_e = 2200)
(Driest, E., and Müller, H.O.: Z. Wiss. Mikroskopie 52, 53-57 (1935)

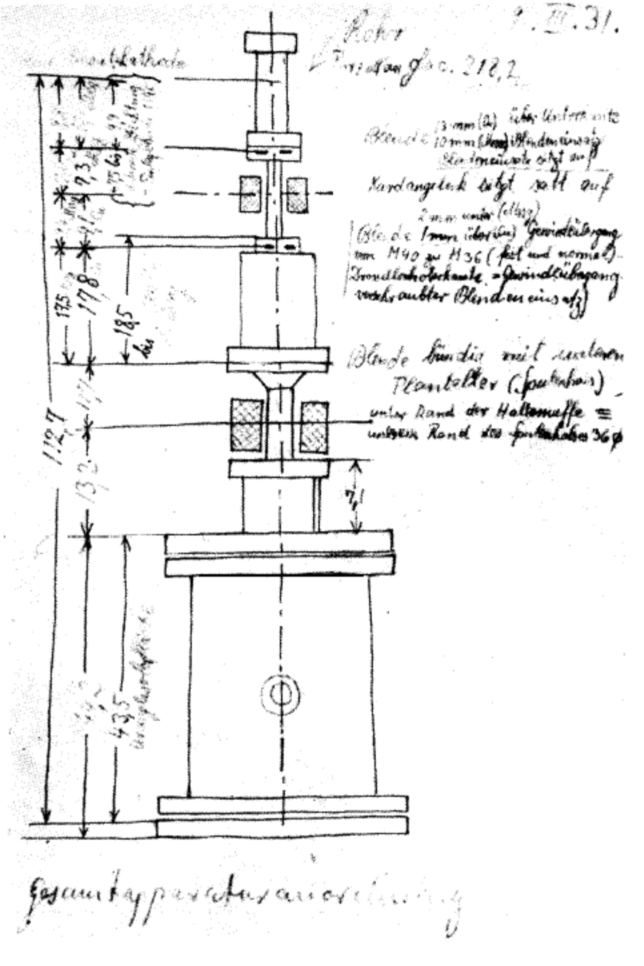
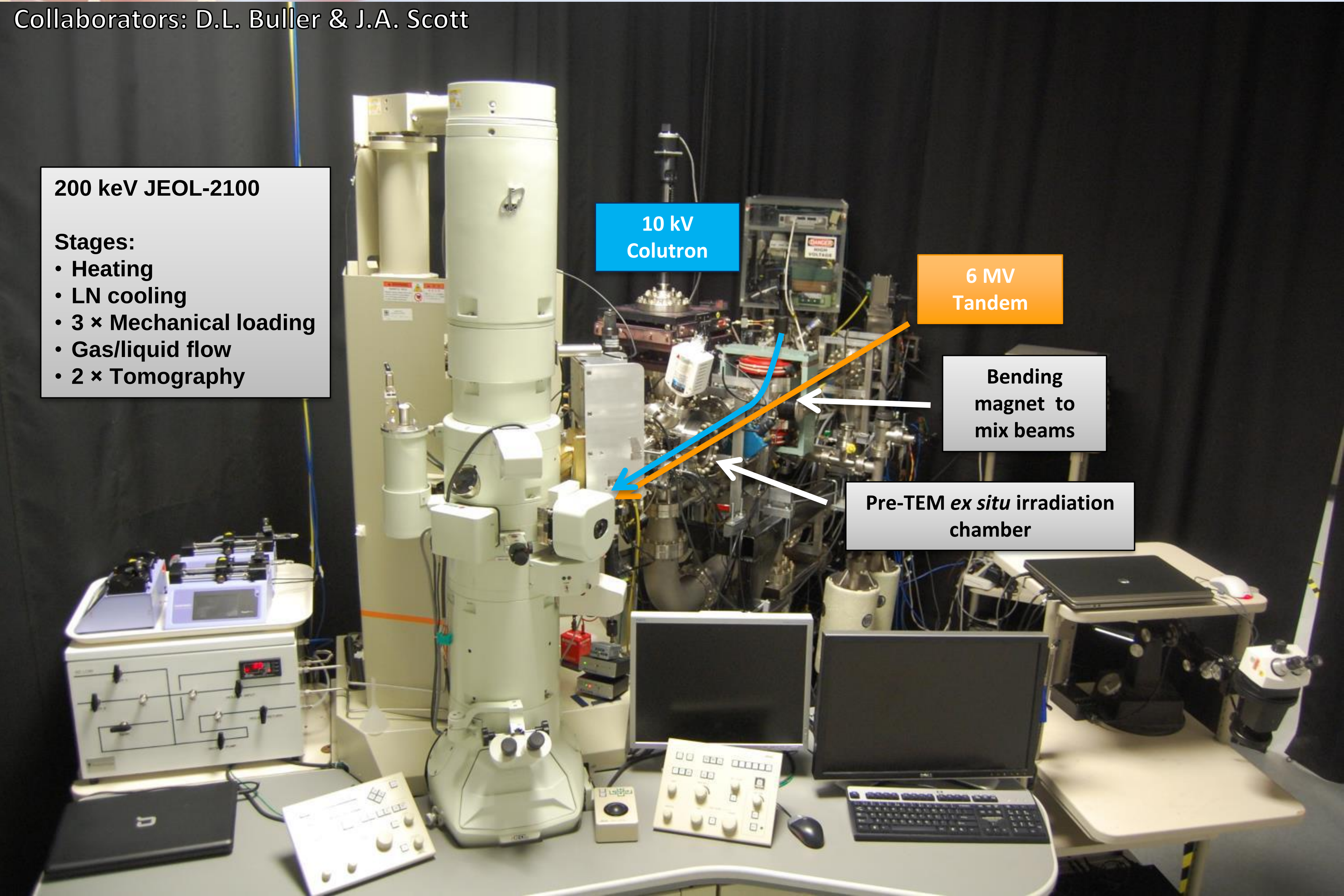


Fig. 2: Sketch by the author (9 March 1931) of the cathode ray tube for testing one-stage and two-stage electron-optical imaging by means of two magnetic electron lenses (electron microscope) [8].

E. Ruska Nobel lecture (1986)

In situ Ion Irradiation TEM Facility

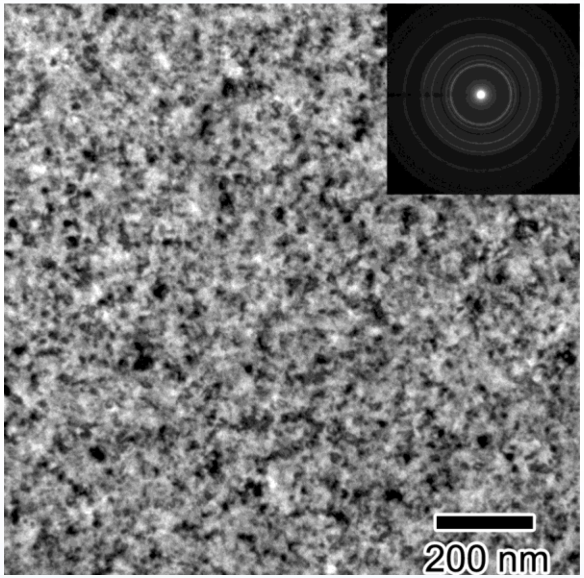
Collaborators: D.L. Buller & J.A. Scott

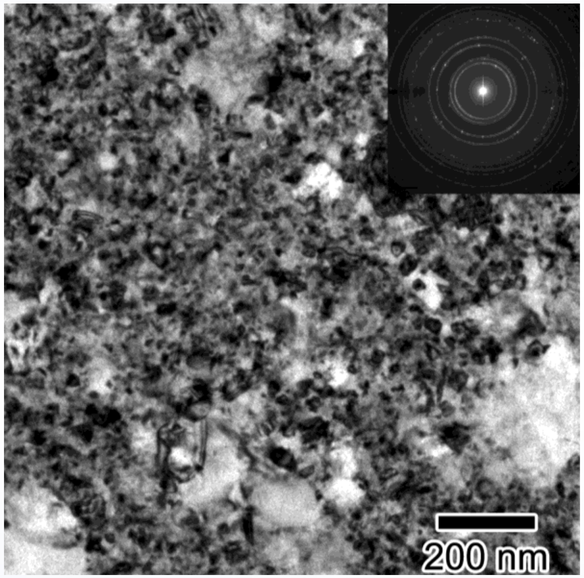


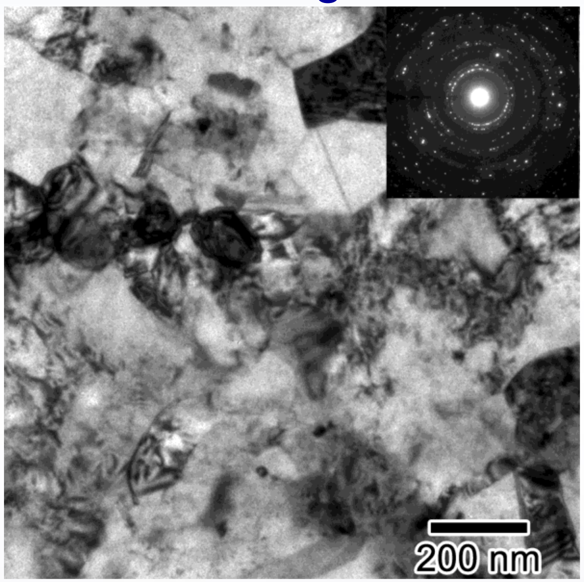
Thermal Stability of Nanocrystalline Materials

Collaboration with: J.A. Knapp, D.M. Follstaedt, and I.M. Robertson

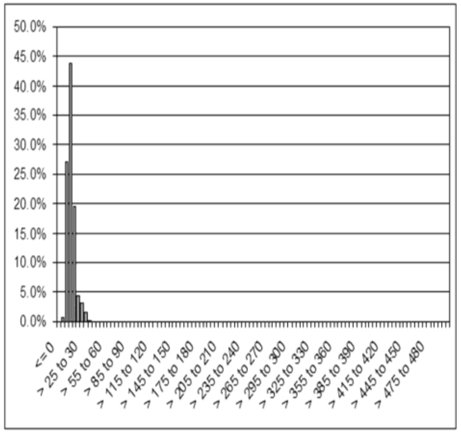
Nanograined	Bimodal	Ultra-fine grained

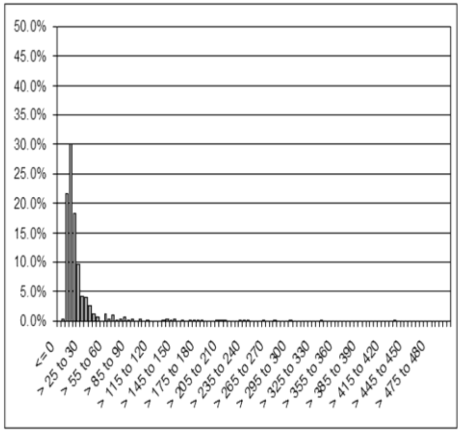


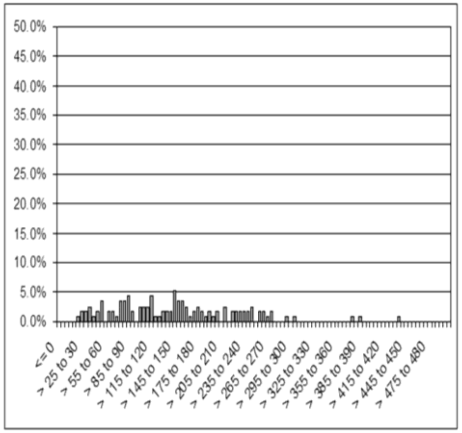




80 nm-thick As-deposited on Si	1 hr at 275 ° C	1 hr at 375 ° C



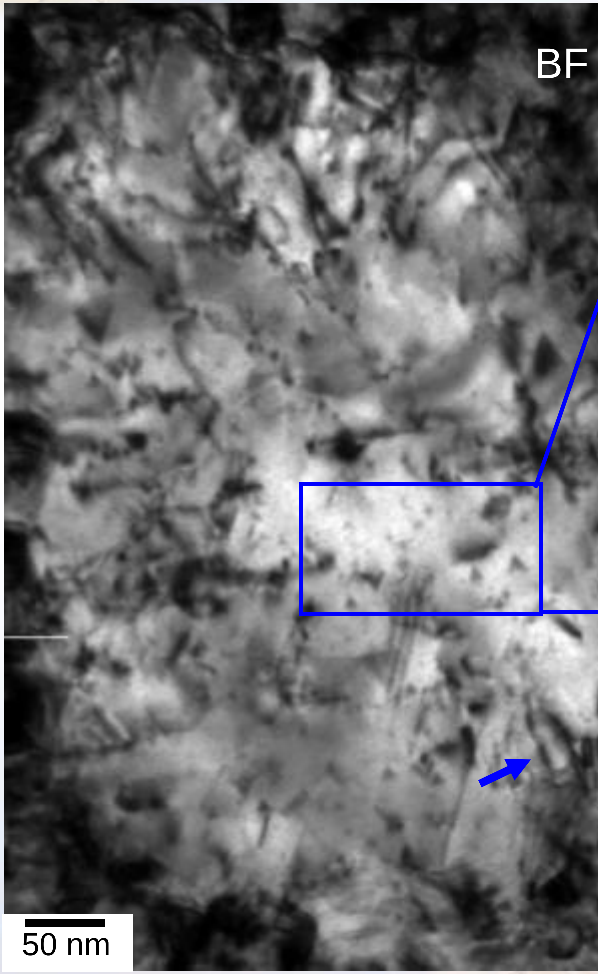




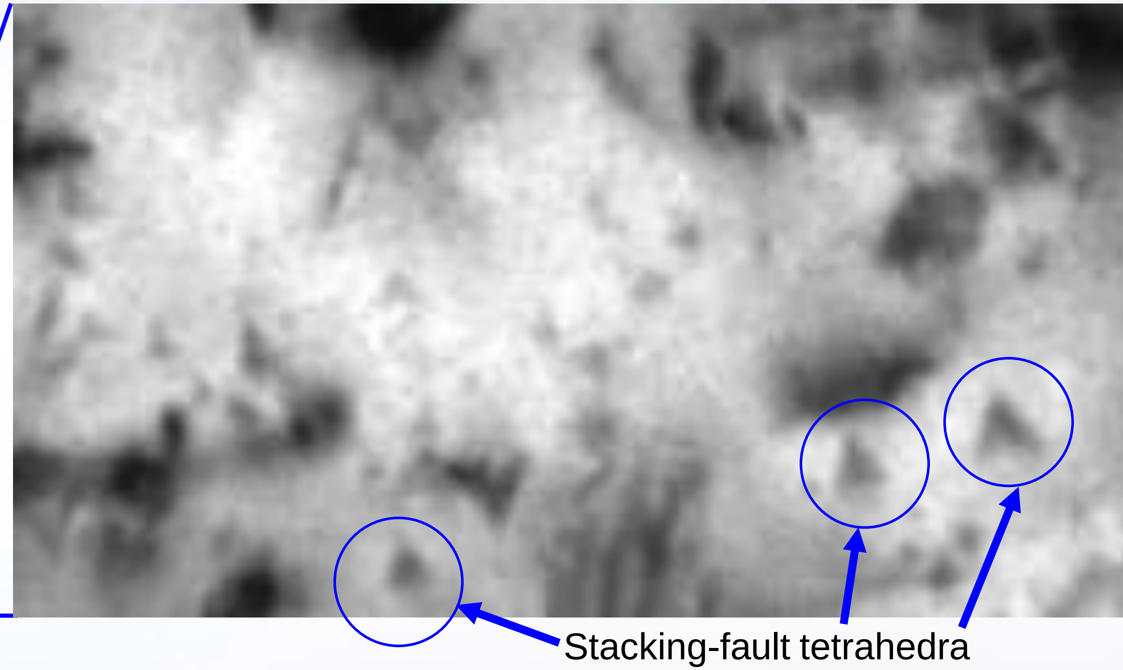
A few select grains grow at the expense of the remaining matrix

A Variety of Unexpected Defect Structures in Ni

Collaboration with: J.A. Knapp, D.M. Follstaedt, and I.M. Robertson

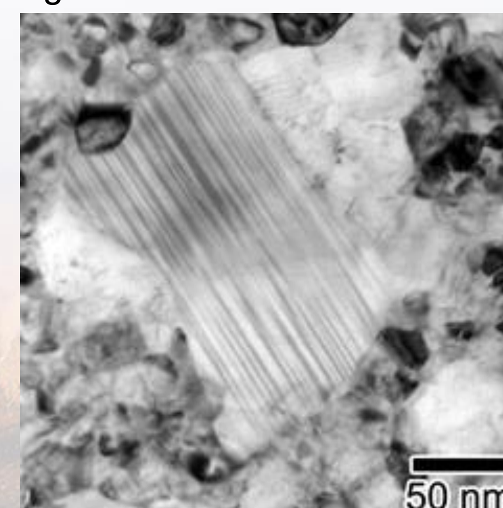


230 nm thick PLD Ni Annealed at 225 ºC for 14 hrs.



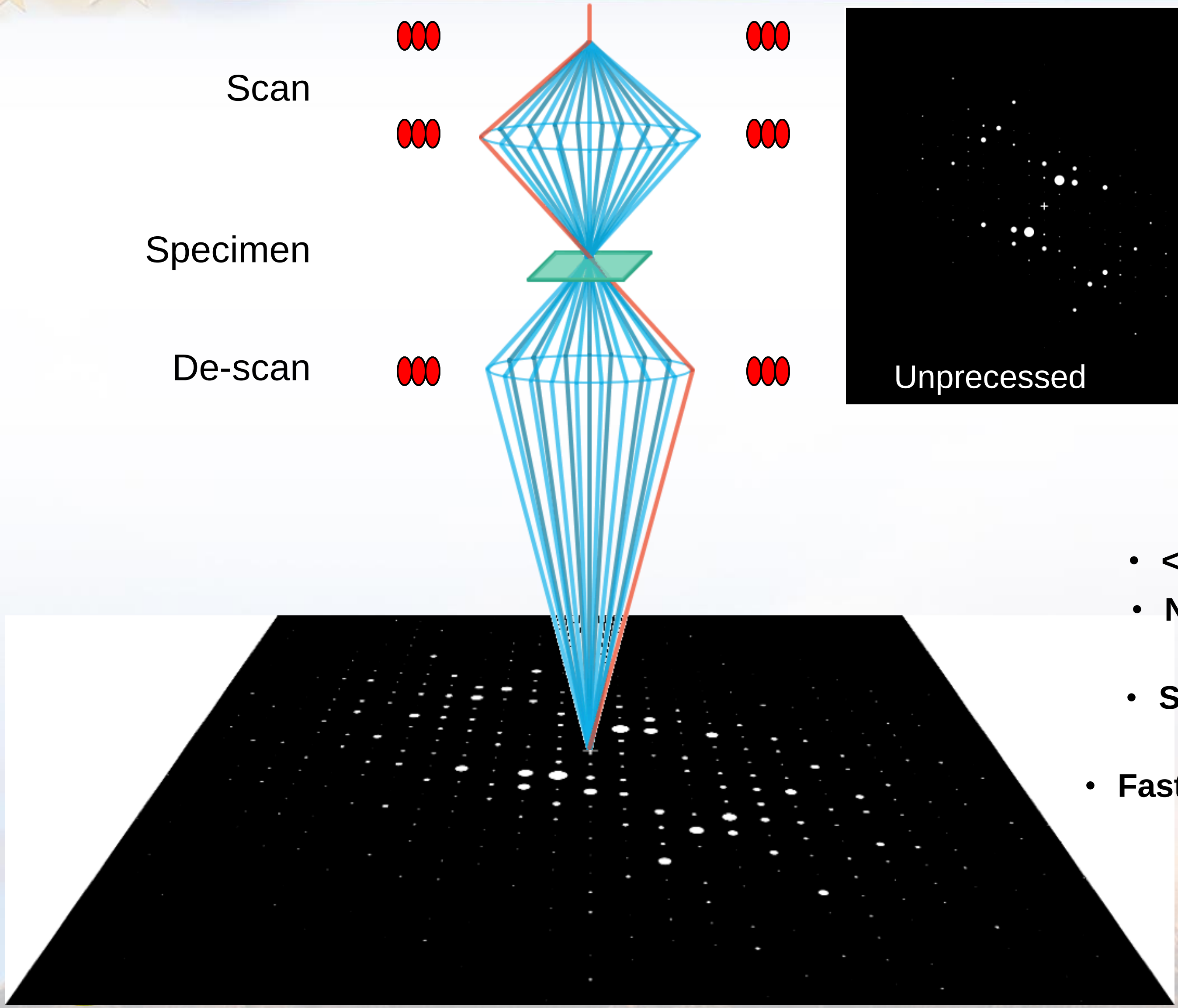
Multitude of defects in annealed PLD Ni

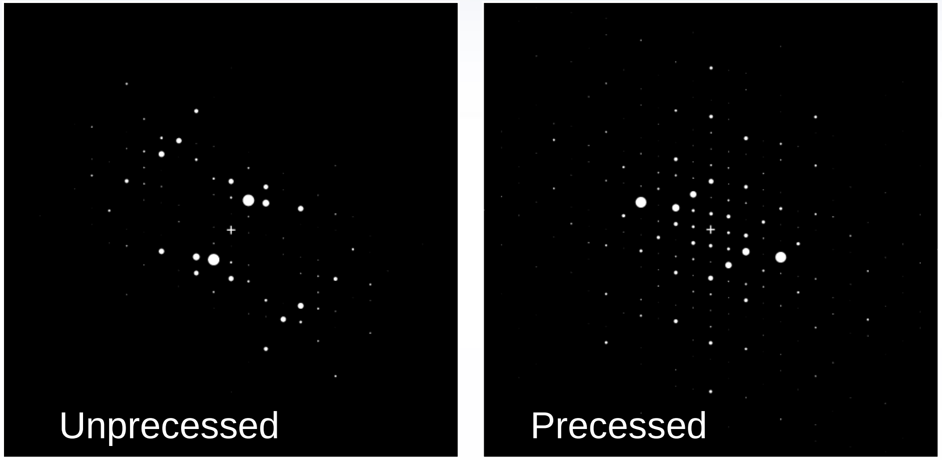
- SFT at temperature
- Stable microstructure for over 15 months
- SFT **not** due to irradiation, quenching, high strain rate
- SFT are theorized to be formed by rapid grain growth through the high free-volume at the initial grain boundaries



Precession Electron Diffraction (PED) Microscopy

Collaborators: K.J. Ganesh, S. Rajasekhara, & P.J. Ferreira





Advantages

- **< 10 nm spatial resolution**
- **Near kinematical electron diffraction**
- **Symmetry ambiguities are resolved**
- **Fast and automated acquisition**
  - **~200 grains in 15 min.**

Chris Own, PhD Dissertation, Northwestern University, 2004

Slide courtesy: K. J. Ganesh, K. Hattar

In Situ Irradiation of Nanocrystalline Au

- Au foil during bombardment with 10 MeV Si^{3+}
- ~22 s of 4000s total experiment time

In situ ion irradiation
TEM: 10 MeV Si into
nanocrystalline Au.

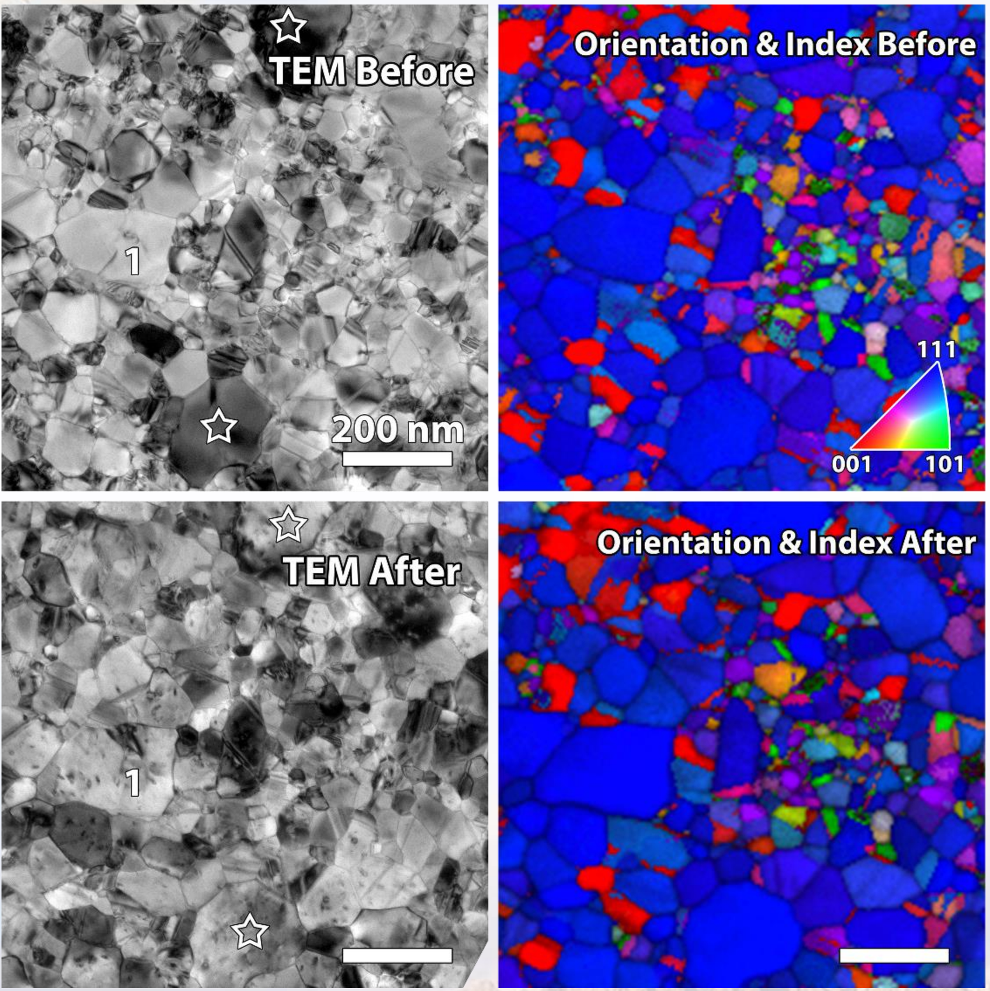
Playback at 2 × real time.

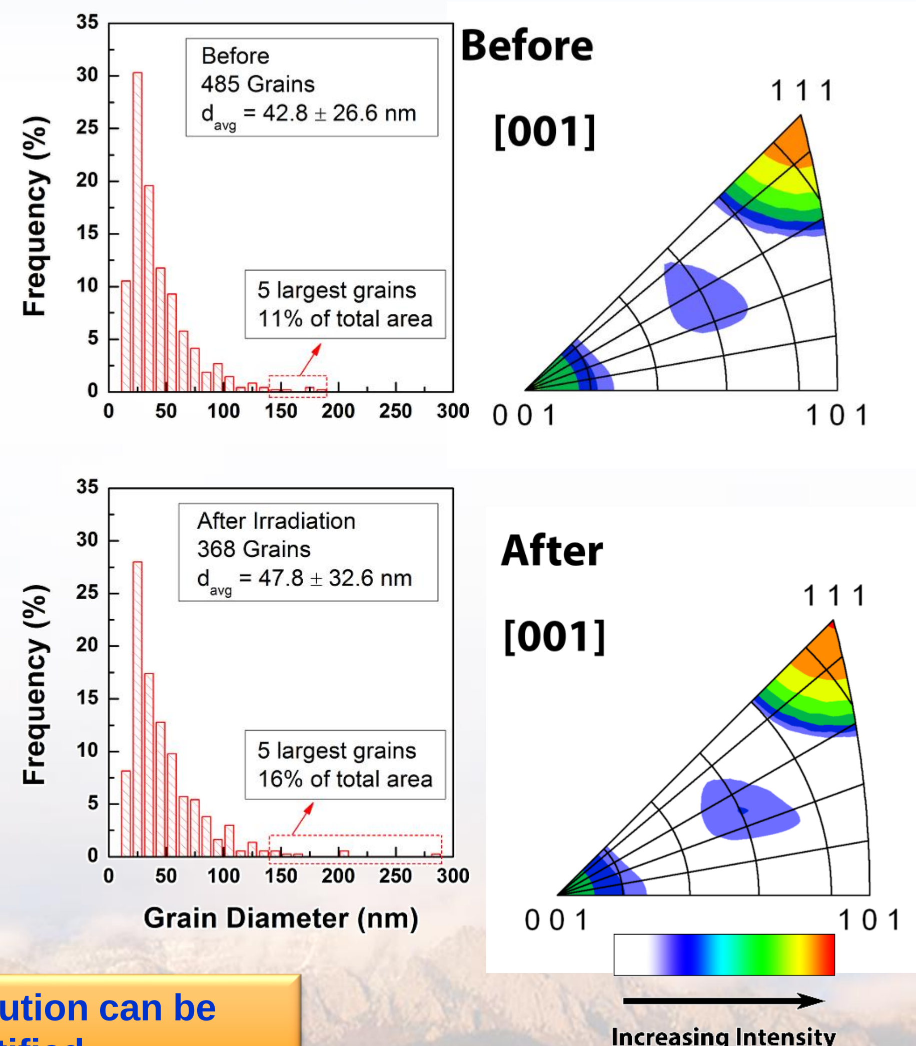
Locations of single ion strikes and resulting microstructural change captured.

2× real time

Quantifying Stability of Nanocrystalline Au during 10 MeV Si Ion Irradiation

Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles





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

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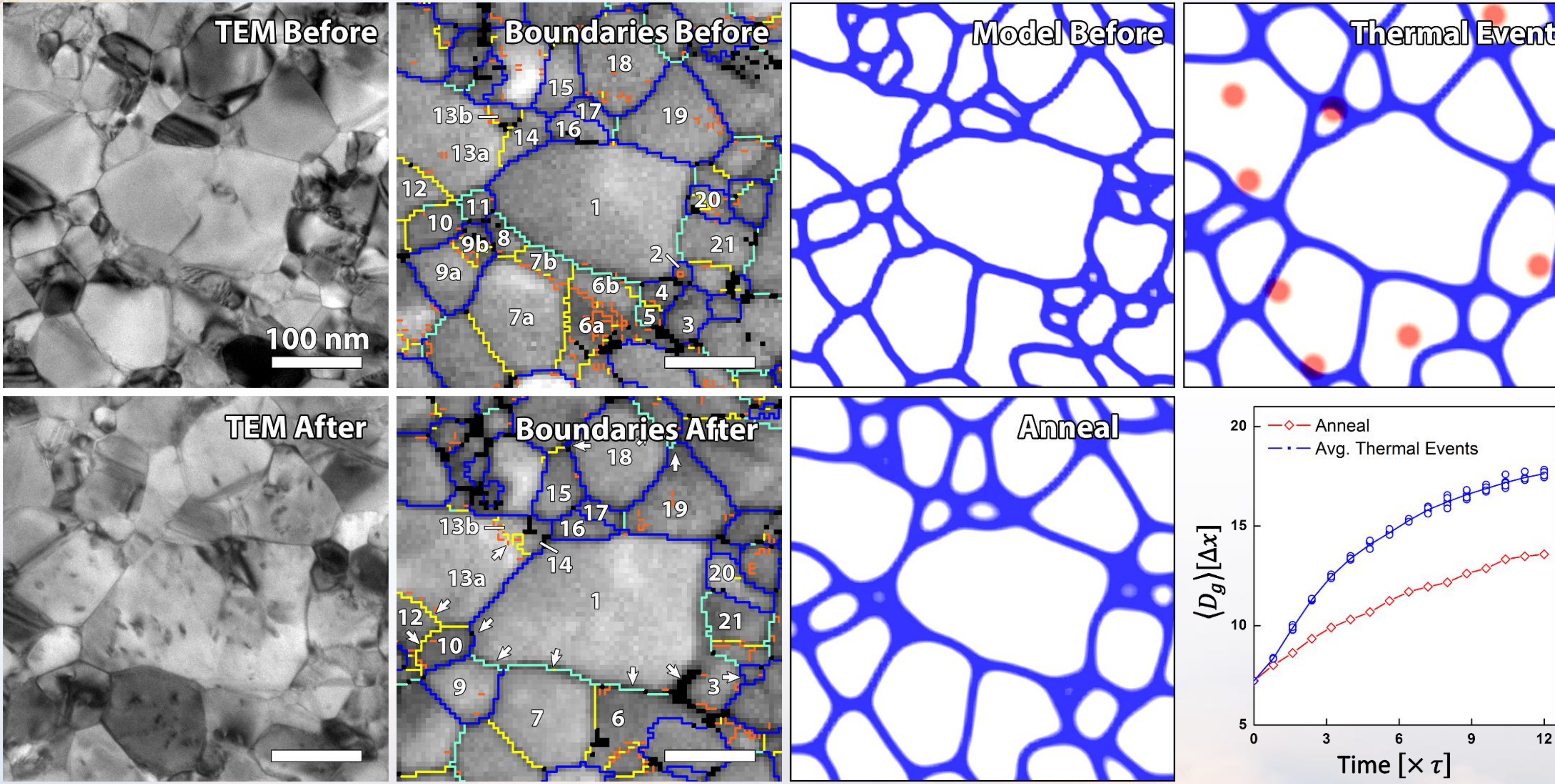
Direct Comparison to Mesoscale Modeling

Collaborators: D.C. Bufford, F. Abdeljawad, & S.M. Foiles



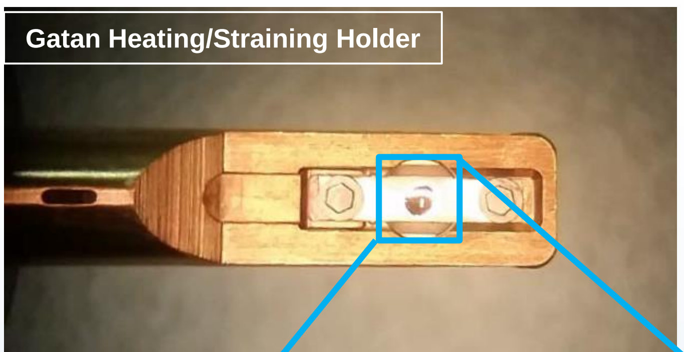
3° ≤ φ < 15°
15° ≤ φ < 30°
30° ≤ φ

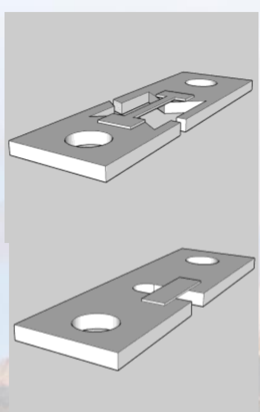
Because of the matching length scale, the initial microstructure can serve as direct input to either MD of mesoscale models & subsequent structural evolution can be directly compared.

In situ Qualitative Mechanical Testing

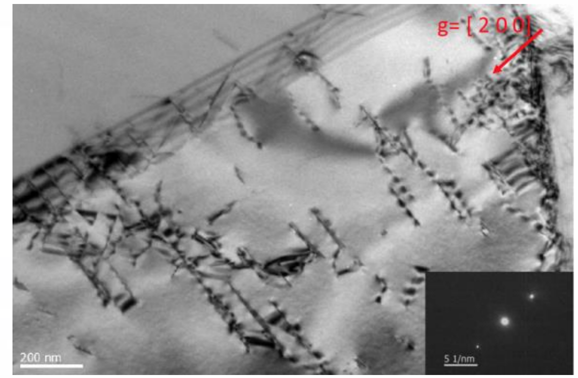
In Situ Straining TEM Holder

- Minimal control over displacement
- No out-of-box force information
- Ability to observe dislocation-GB interactions

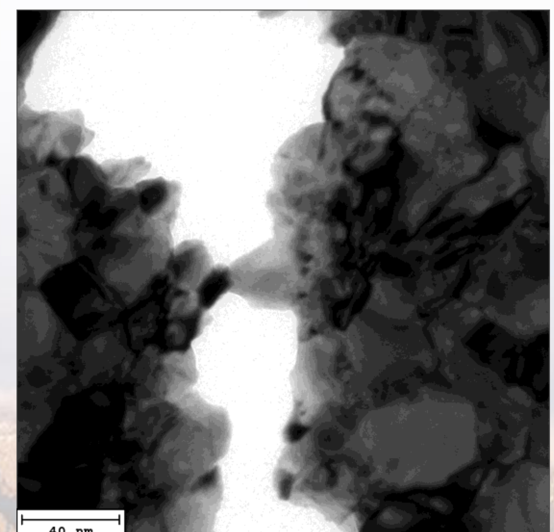




Observe dislocation and pile-up interactions as function of GB character



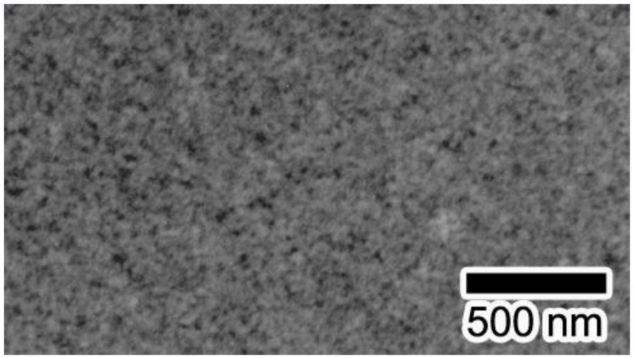
Observe deformation mechanisms in nanocrystalline metals during tensile straining

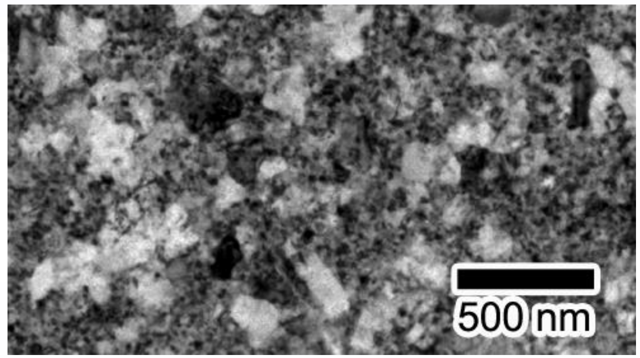


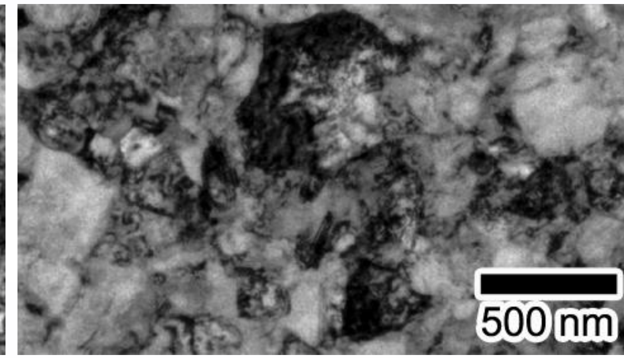
Failure Analysis of Strained PLD Ni

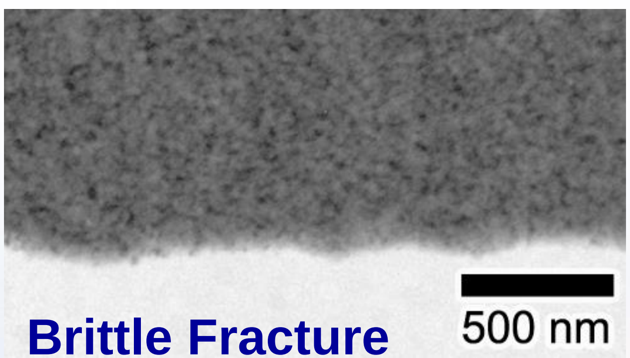
Collaboration with: J.A. Knapp, D.M. Follstaedt, and I.M. Robertson

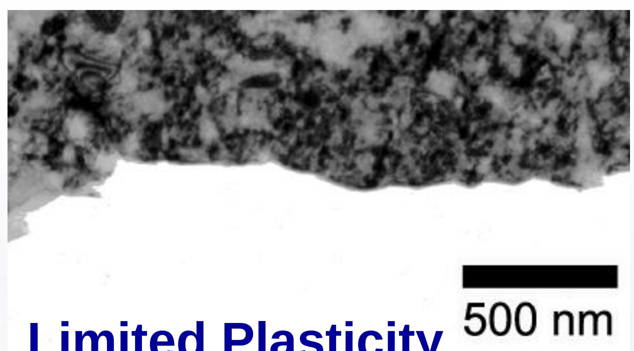
Nanograined	Bimodal	Ultra-fine grained











Brittle Fracture

- No observation of global plasticity

Limited Plasticity

- Dislocation pile-up
- Local shear

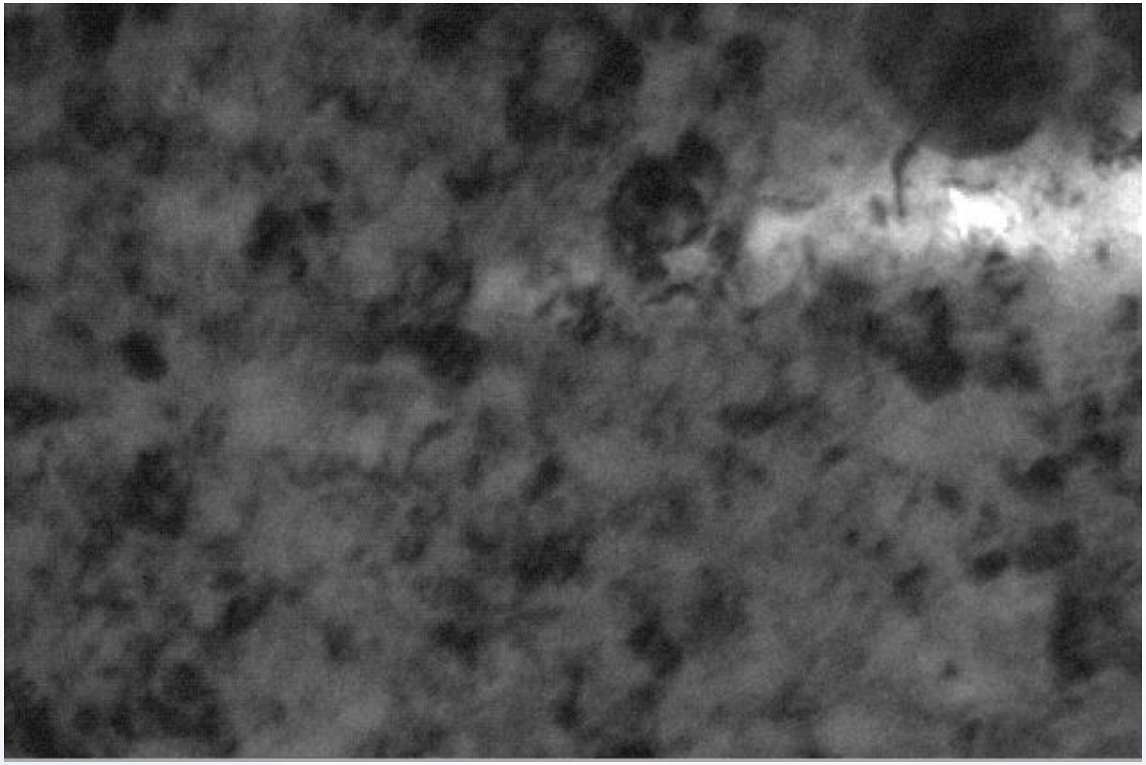
Shear Failure

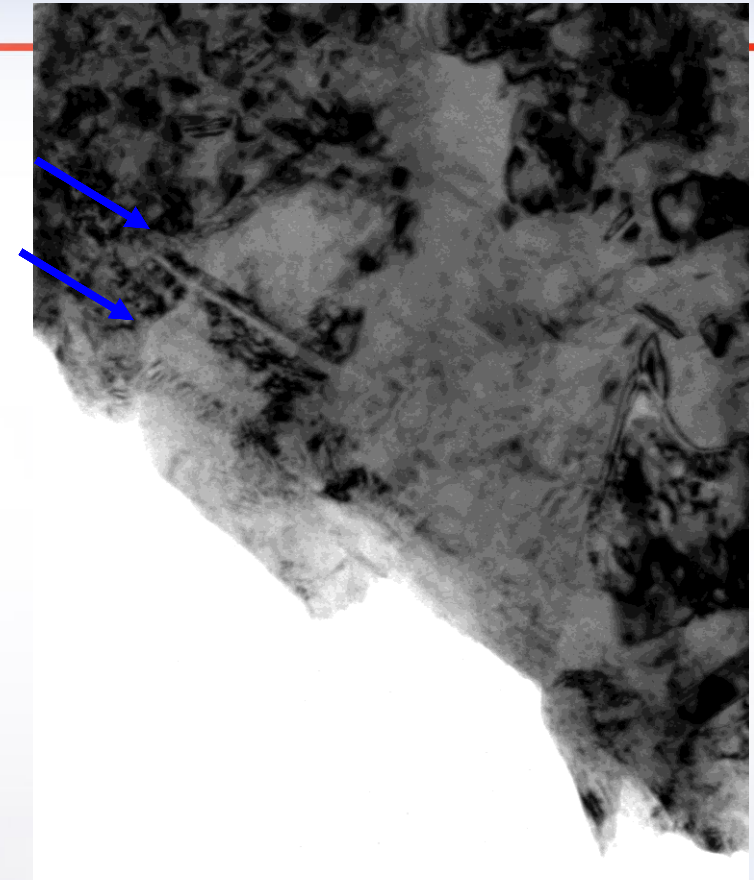
- Shear teeth
- Dislocation structure

Fracture surfaces provide insight to deformation processes

Deformation and Failure in Bimodal Ni

80 nm-thick PLD Ni annealed at 275 ºC





Throughout the film

- Elastic strain
- Limited dislocation slip

In the plastic zone

- Extensive dislocations slip
- Twinning

At Crack Tip

- Necking
- Grain agglomeration

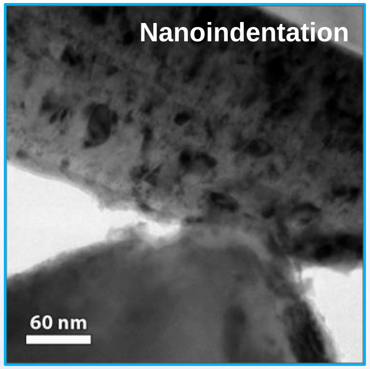
We have some insight into the unique thermal and mechanical mechanisms and properties. What is the initial nanostructure that causes this?

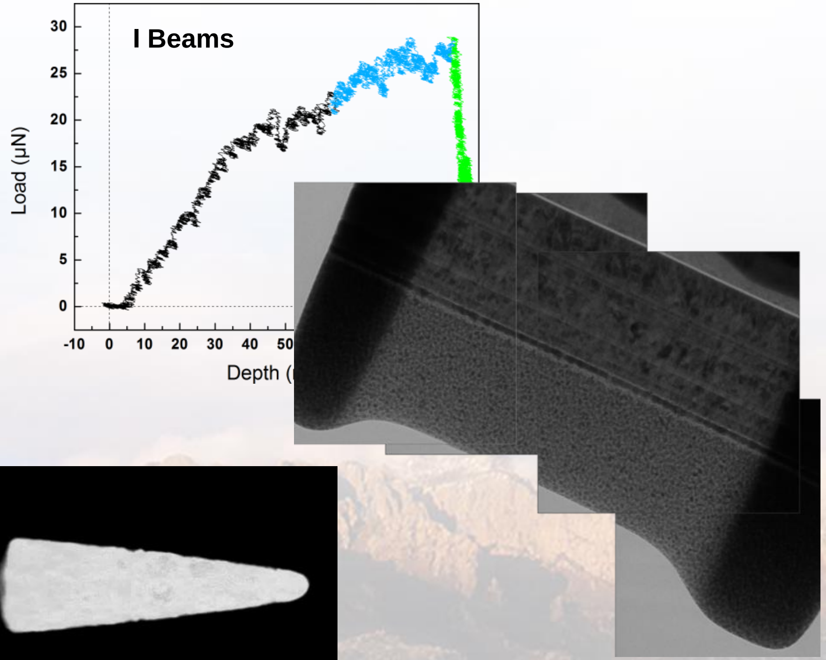
In situ Quantitative Mechanical Testing

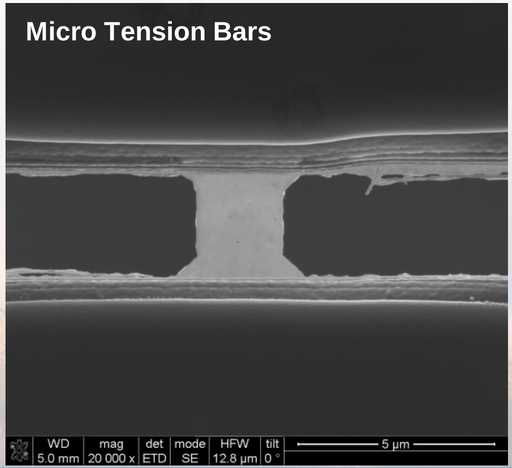
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

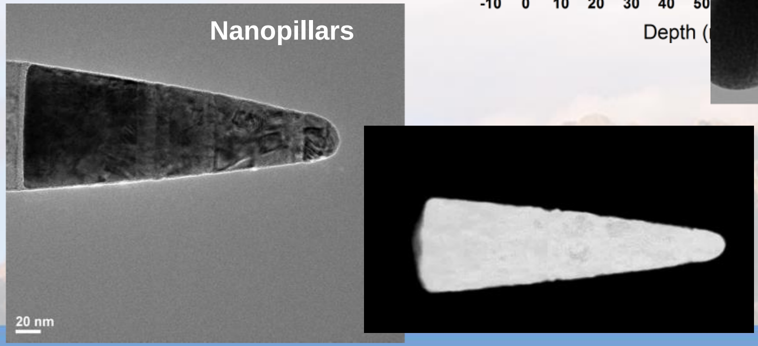
Hysitron PI95 *In Situ* Nanoindentation TEM Holder

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

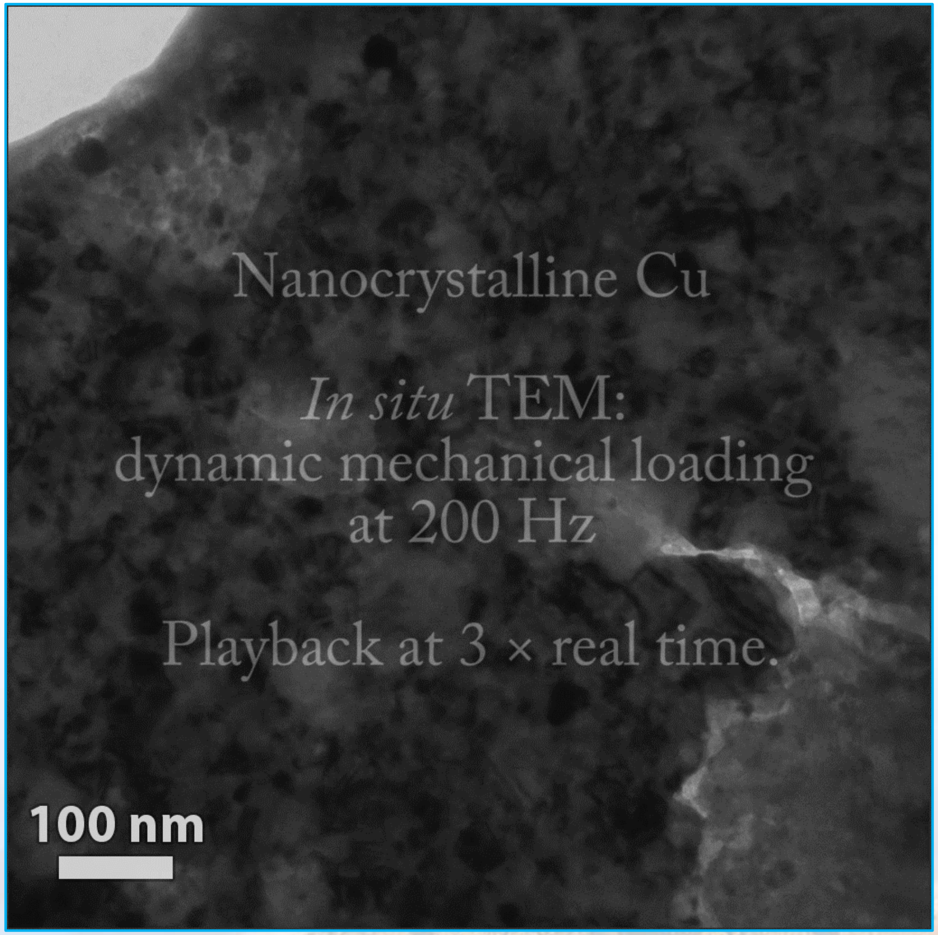




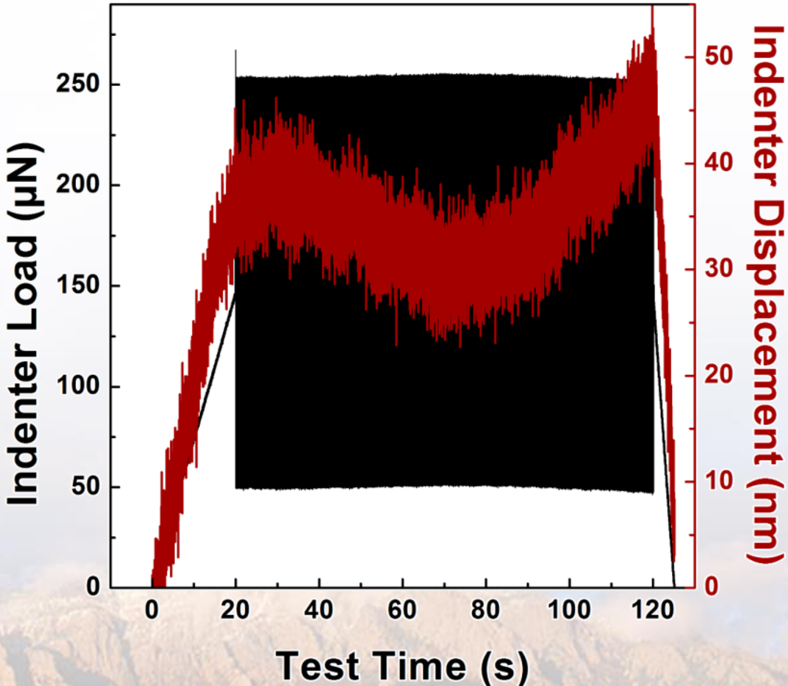




High Cycle Fatigue: Experimental Observations



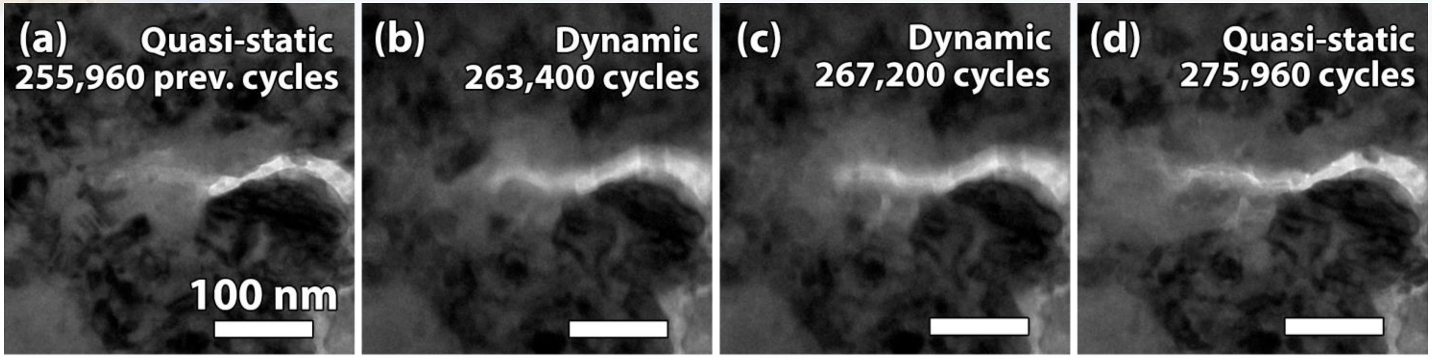
- Cyclic loading at 200 Hz
- Structural change at crack tip captured

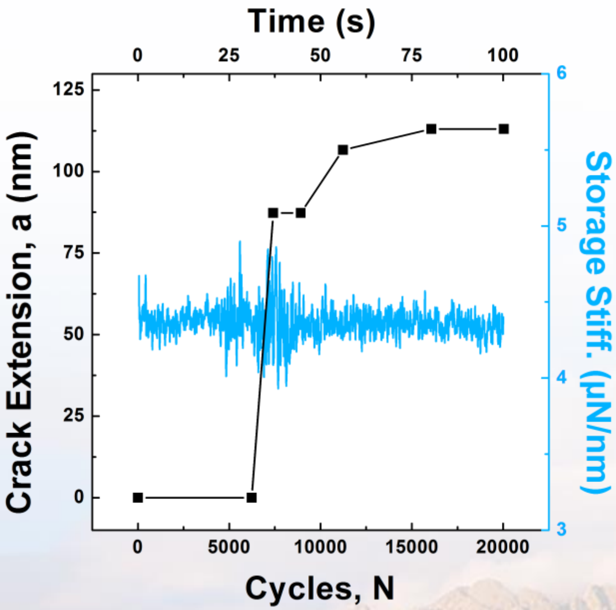


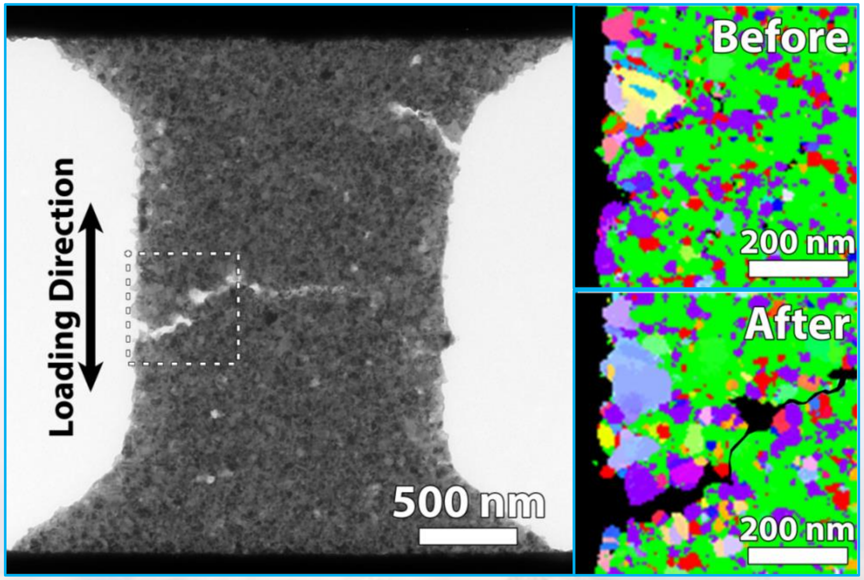
Video playback ×3

First high cycle fatigue experiments with continuous observation by TEM!

High Cycle Fatigue: Crack Growth Quantified

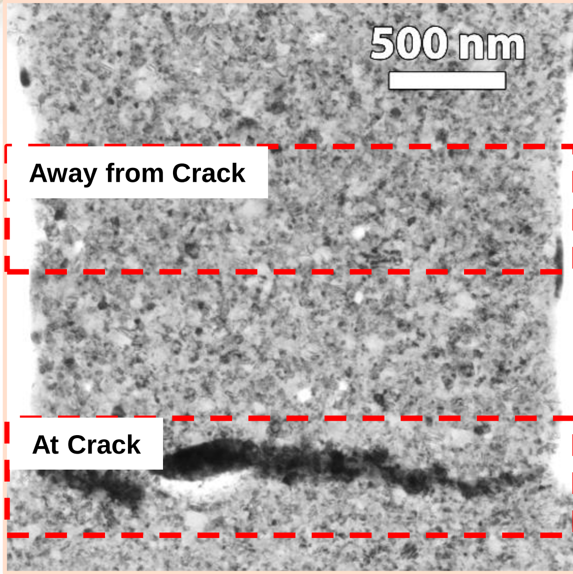


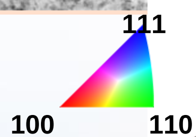


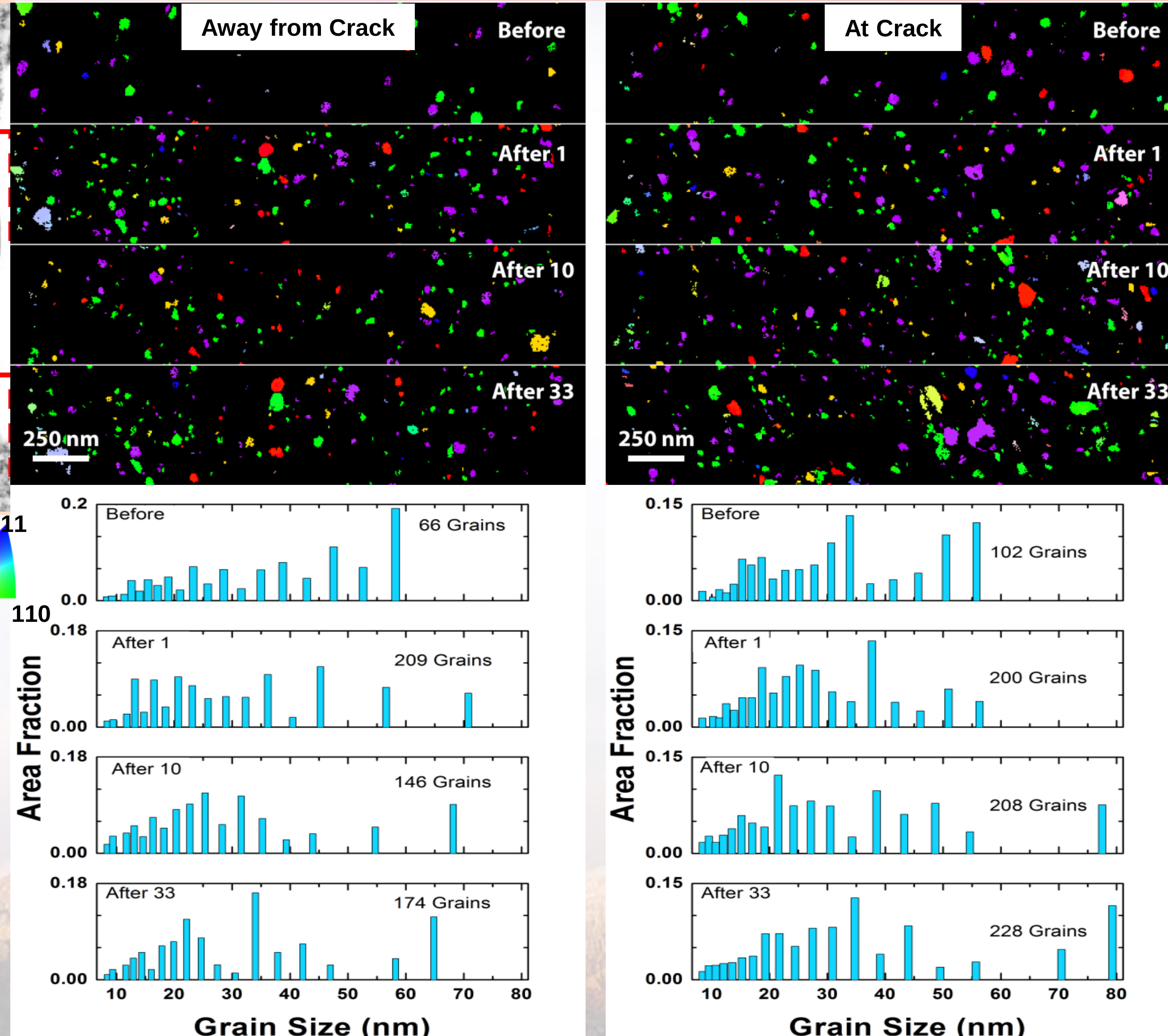


- Picometer-per-cycle measured crack growth rate
- Evidence of fatigue-induced grain growth.

Comparison of Grain Size Near and Far from the Fracture Surface





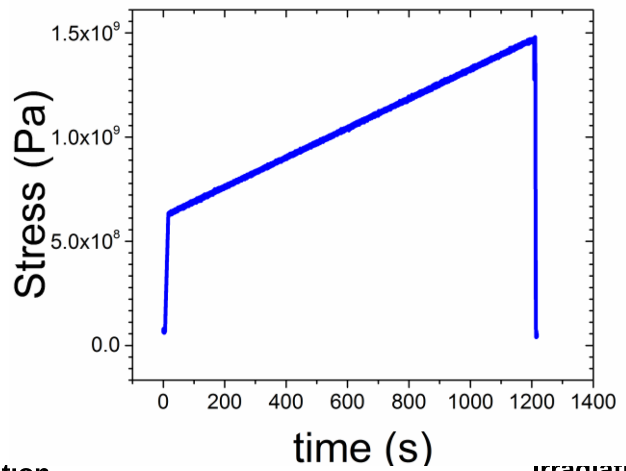


Texture and Grain size analysis can target:

- Various regions
- Quality of PED patterns
- Particular orientations
- Set of grain sizes
- Etc.

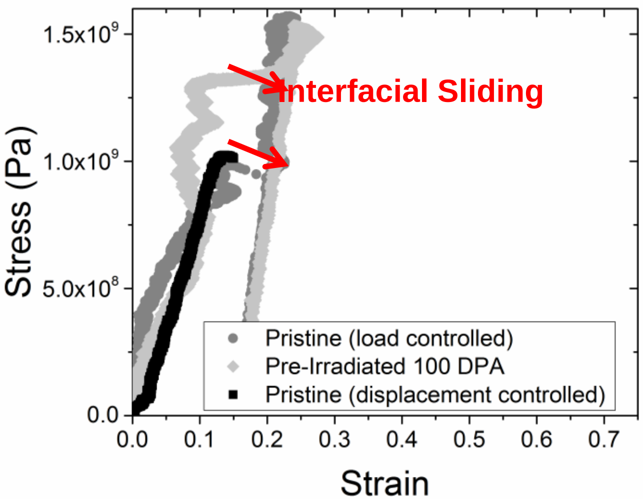
Irradiation Creep (4 MeV Cu^{3+} 10^{-2} DPA/s)

Controlled Loading Rate Experiments



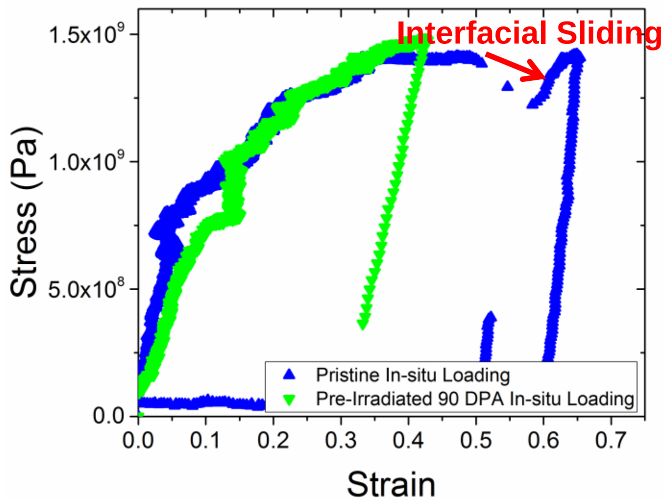
No Irradiation

(Loading rate 0.6 Mpa s^{-1})



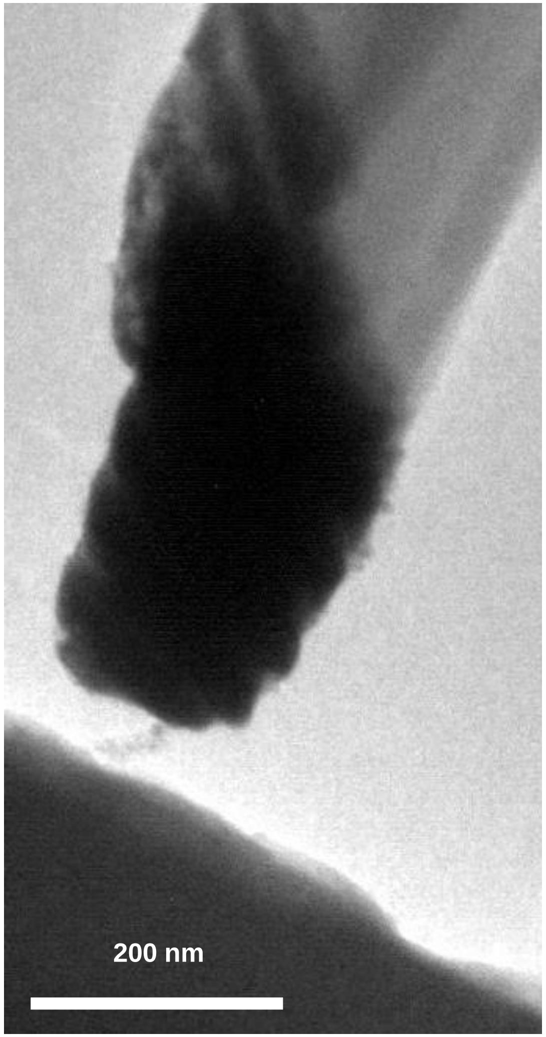
Irradiation Creep

(Loading rate 0.6 Mpa s^{-1})



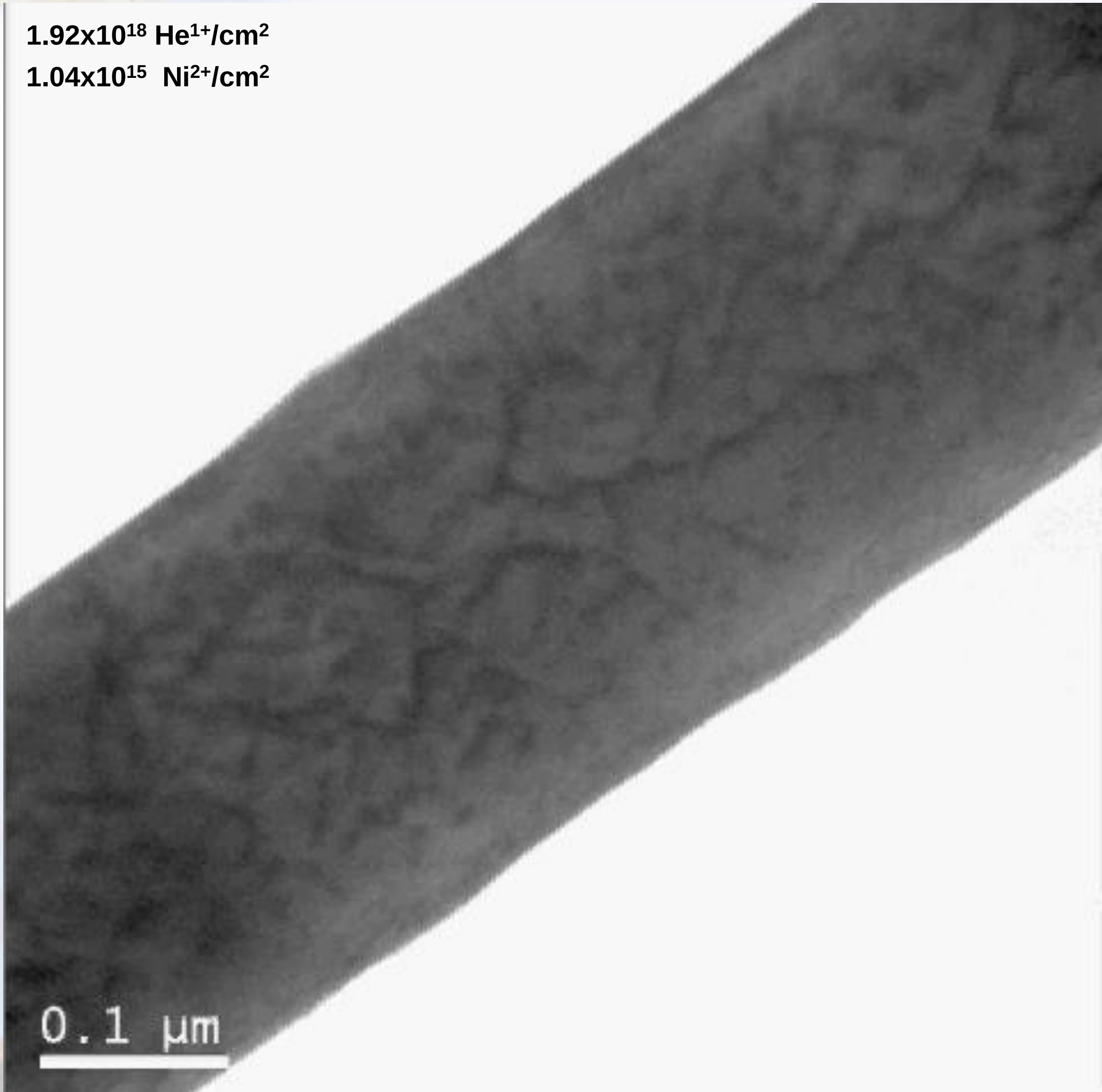
50 nm Cu-W multilayer

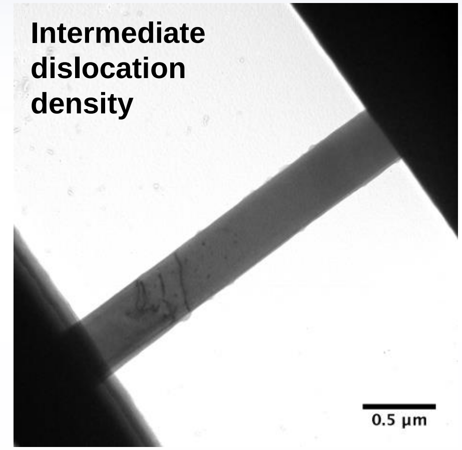
20 Min

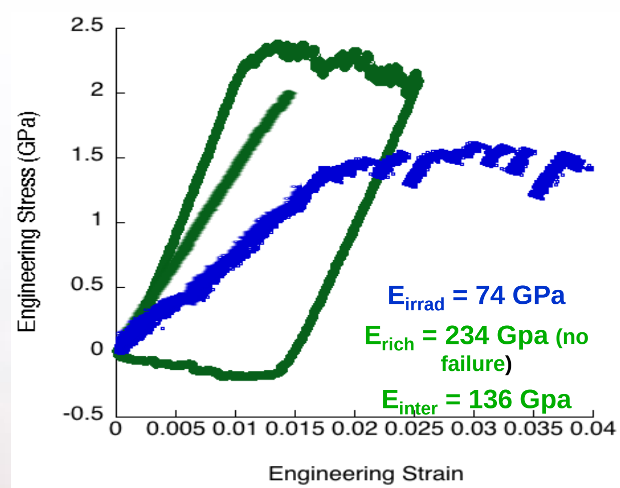


In situ TEM Tensile Testing of Ion Irradiated Mo-alloy Wires

Contributors: C. Chisholm, H. Bei, E.P. George, P. Hosemann, & A.M. Minor

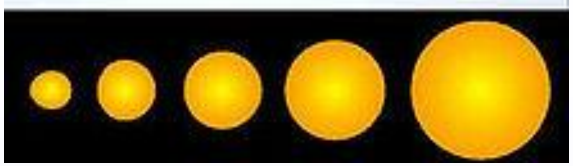






Dislocation structure and subsequent deformation and failure mechanisms through controlled ion beam modification

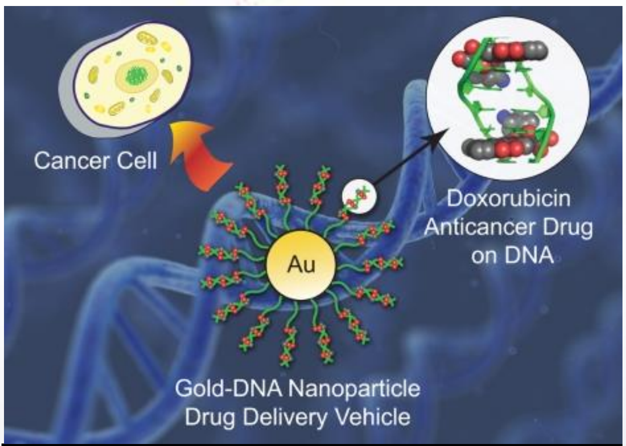
Nanoparticles in Extreme Environments

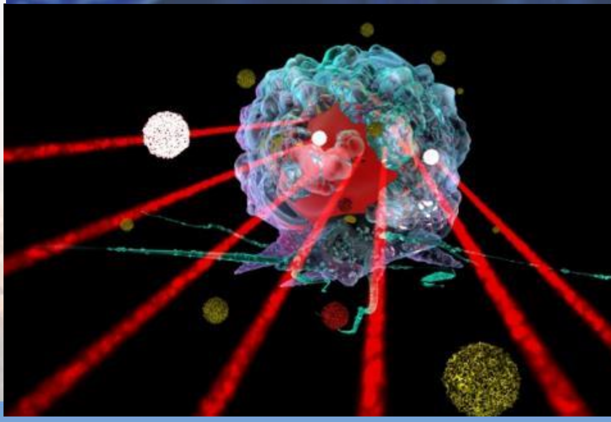


Nenoff et. al. *SNL News Release* (2007).

Au nanoparticles (Au NP) are of interest due to their unique optical, electronic, molecular-recognition, and catalytic properties

- **Photothermal and laser ablation cancer therapies – concentrate IR into heat**
- **Tumor targeting and drug carriers – enhance dose delivery**
- **Catalysts for air-pollution control**





Do the unique properties of the NP withstand extreme radiation?

Cumulative Effects of Ion Irradiation as a Function of Ion Energy and Au Particle Size

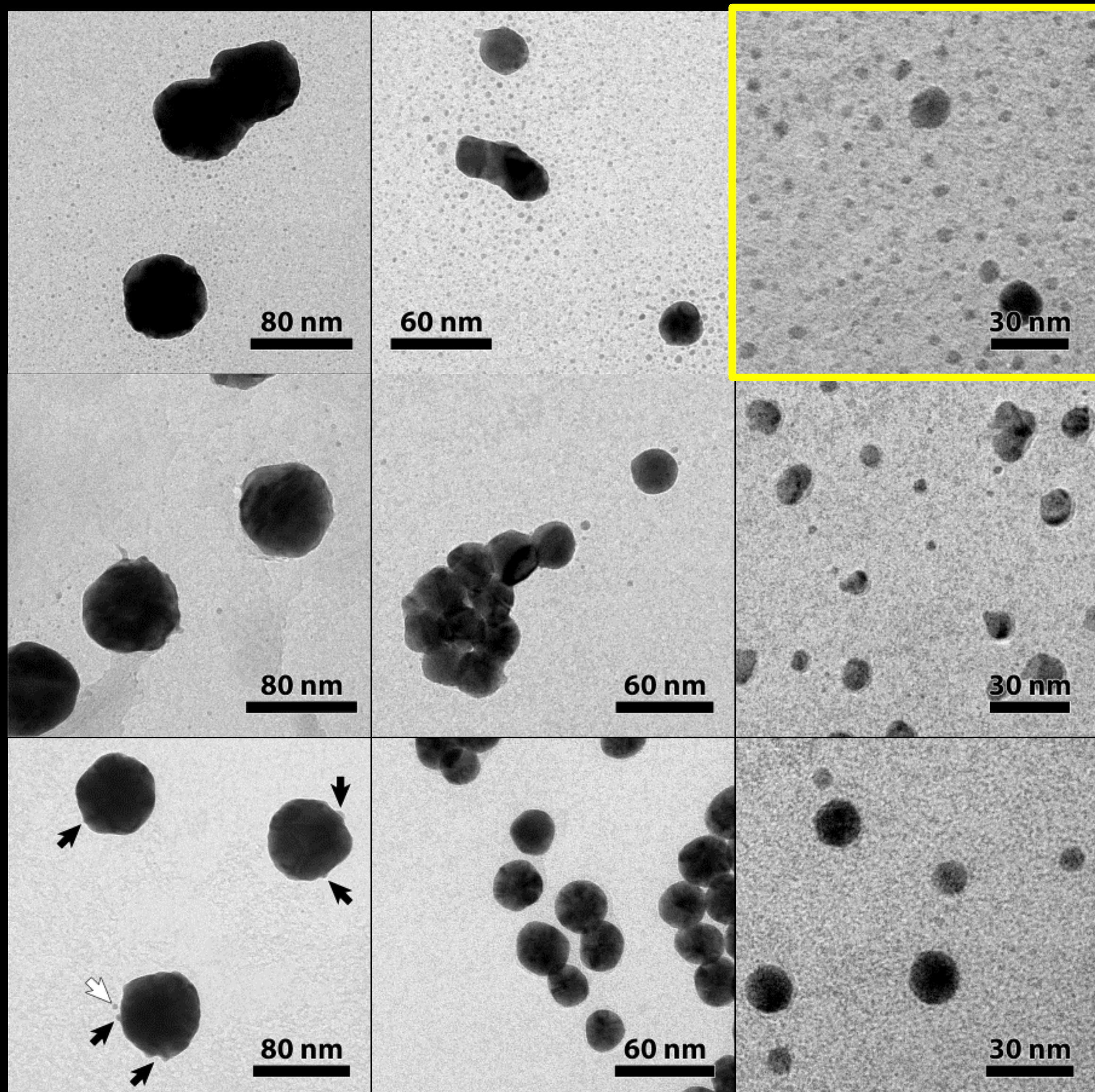
Collaborator: D.C. Bufford

60 nm | 20 nm | 5 nm

46 keV Au^{1-}
3.4×10^{14} /cm²

2.8 MeV Au^{4+}
4×10^{13} /cm²

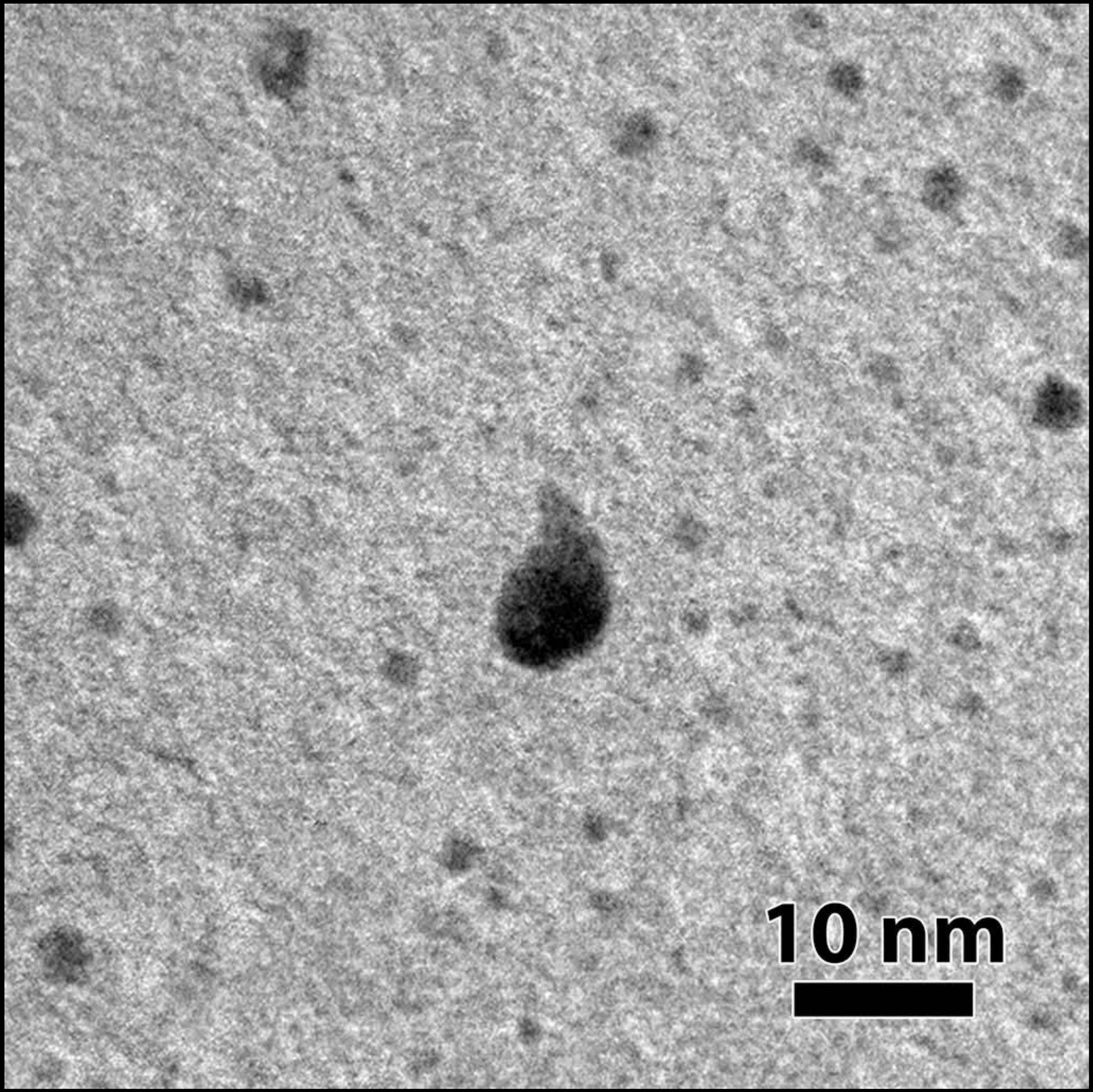
10 MeV Au^{8+}
1.3×10^{12} /cm²

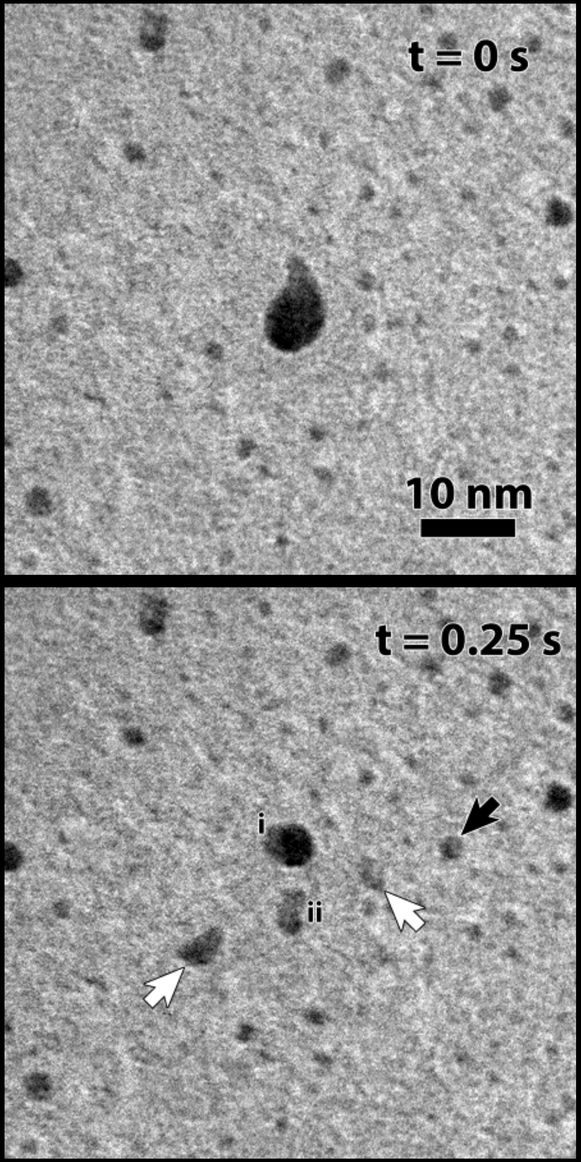


Particle and ion energy dictate the ratio of sputtering, particle motion, particle agglomeration, and other active mechanisms

Single Ion Effects with 46 keV Au^{1-} ions: 5 nm

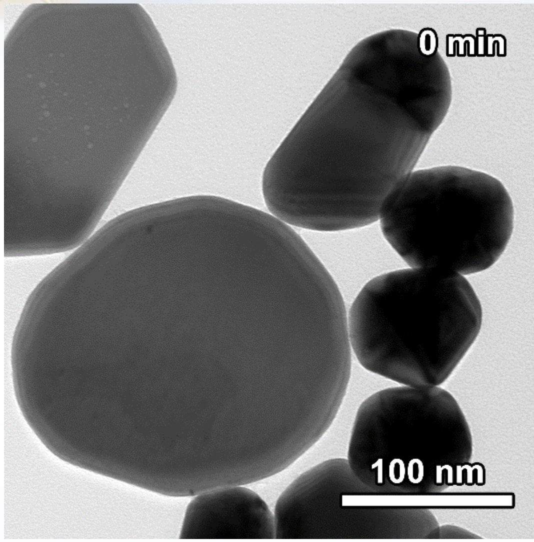
Collaborator: D.C. Bufford

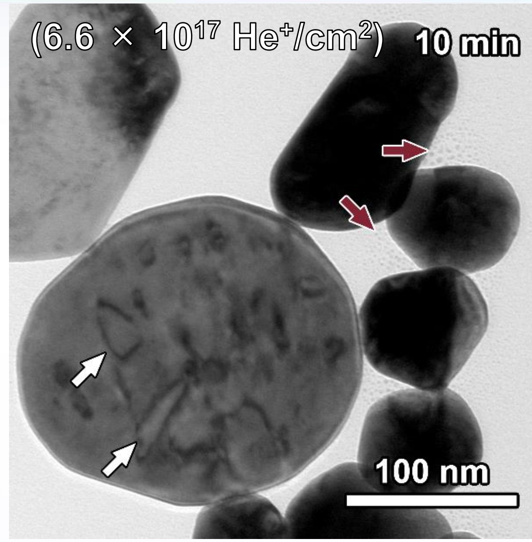


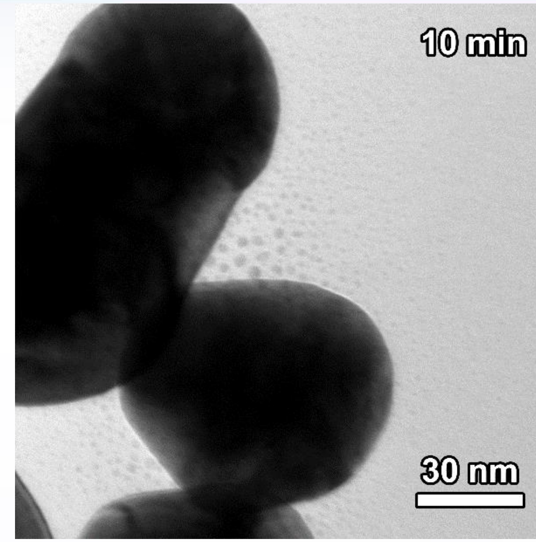


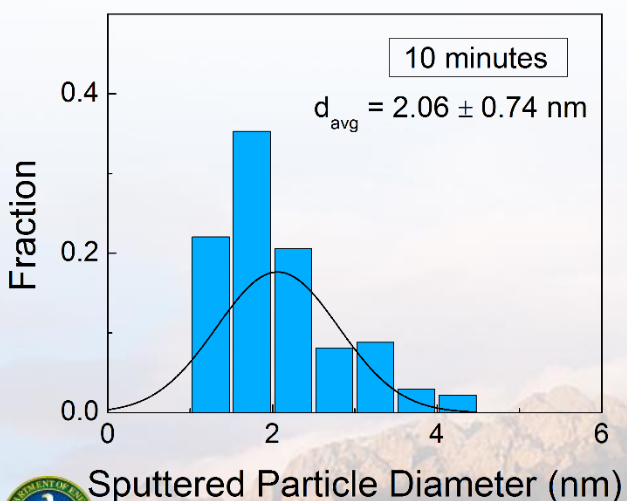
Formation of Dislocation Loops & Sputtered Particles due to He implantation

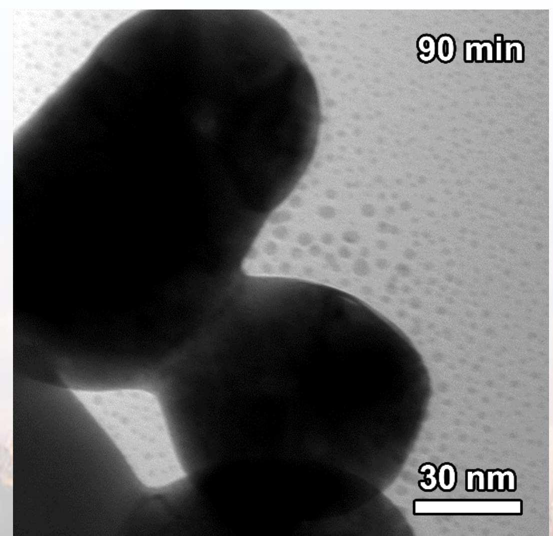
Collaborators: D.C. Bufford, S.H. Pratt & T.J. Boyle

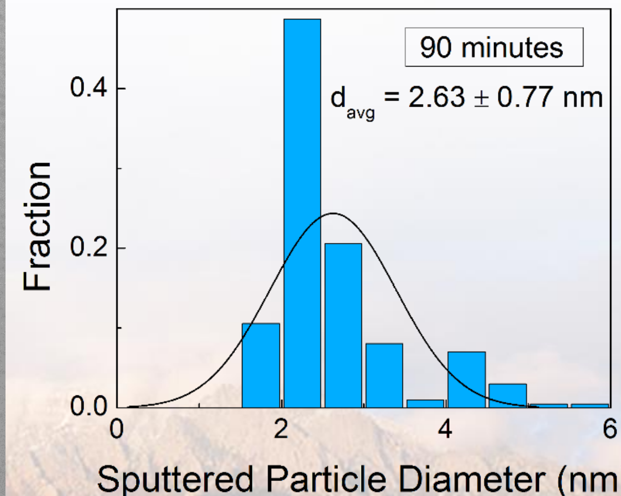










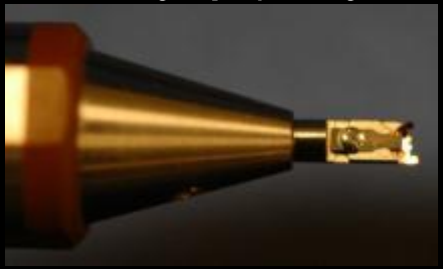


Advanced Microscopy Techniques Applied to Nanoparticles in Radiation Environments

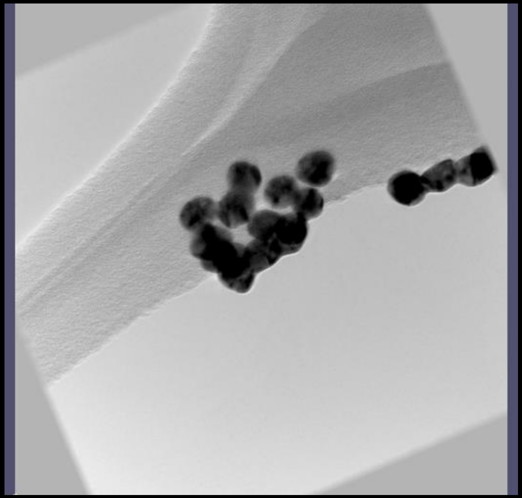
Collaborators: S.M. Hoppe & T.J. Boyle

In situ Ion Irradiation TEM (I³TEM)

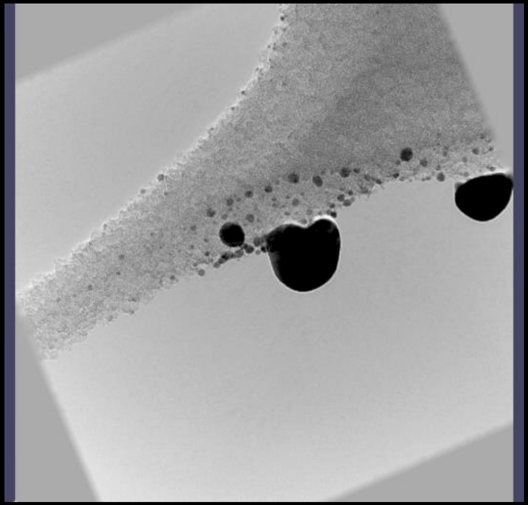
Hummingbird tomography stage



Aligned Au NP tilt series - unirradiated

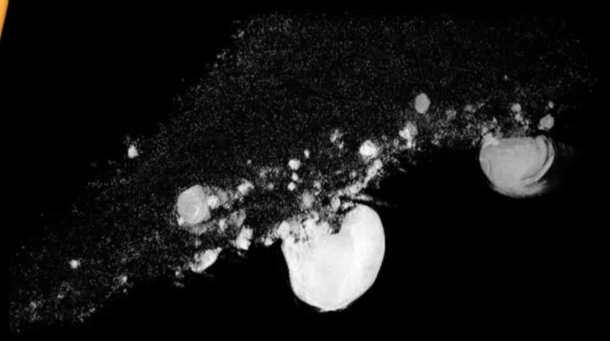


Aligned Au NP tilt series - irradiated



Unirradiated Au NP model

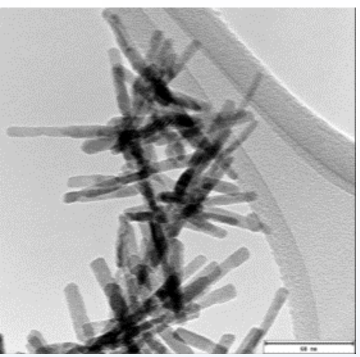
Irradiated Au NP model



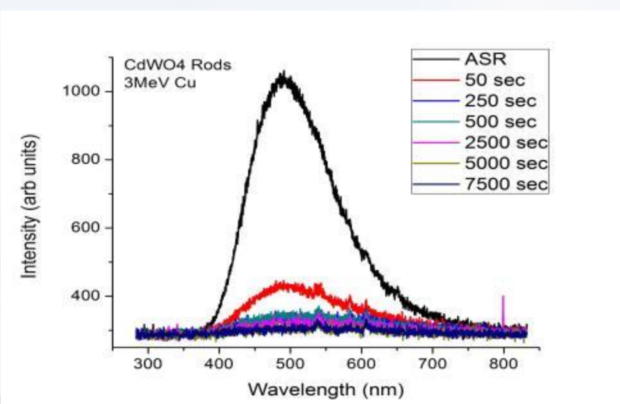
The application of advanced microscopy techniques to extreme environments provides exciting new research directions

Radiation Tolerance is Needed in Advanced Scintillators for Non-proliferation Applications

Contributors: S.M. Hoppe, B.A. Hernandez-Sanchez, T. Boyle

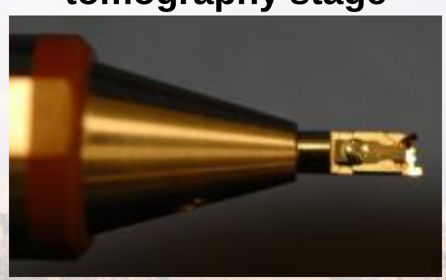


High-Z nanoparticles ($CdWO_4$) are promising, but are radiation sensitive



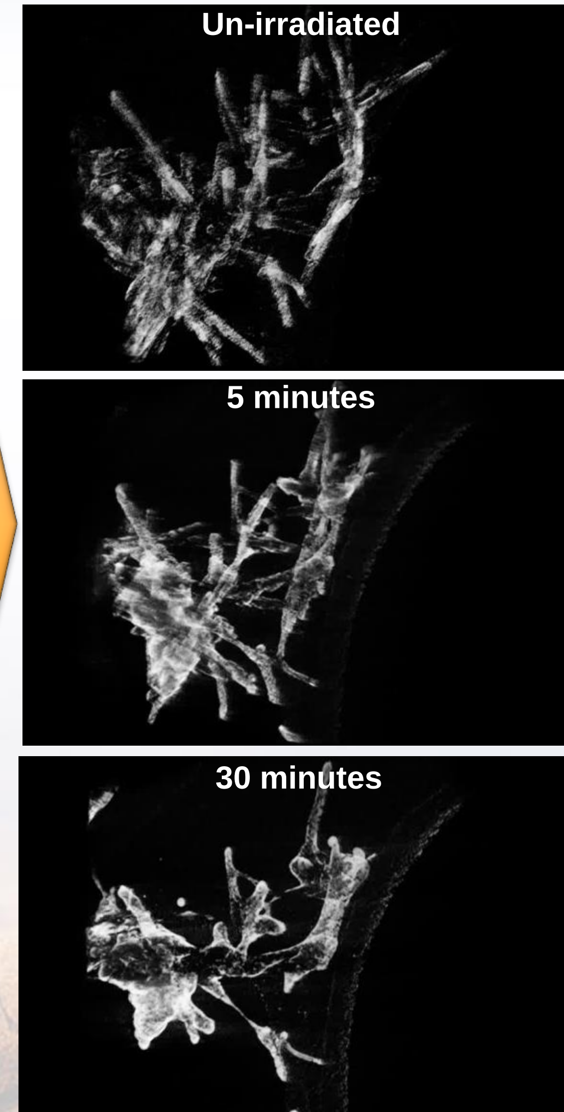
In situ Ion Irradiation TEM (I³TEM)

Hummingbird tomography stage



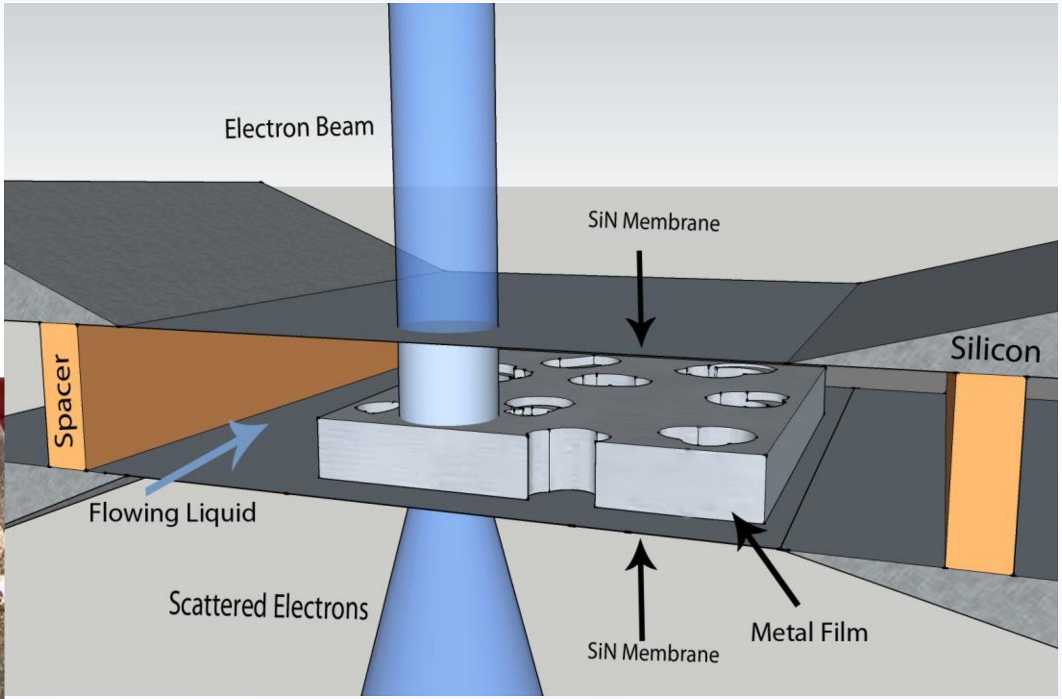
Tomography of Irradiated $CdWO_4$:

3 MeV Cu^{3+} at ~30 nA



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

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

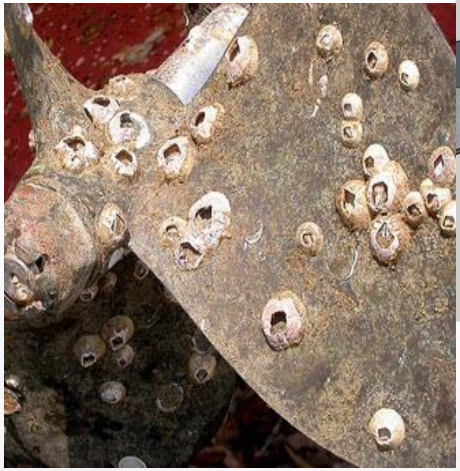


Biocorrosion

Drag

Introduces corrosive species

Decreased performance



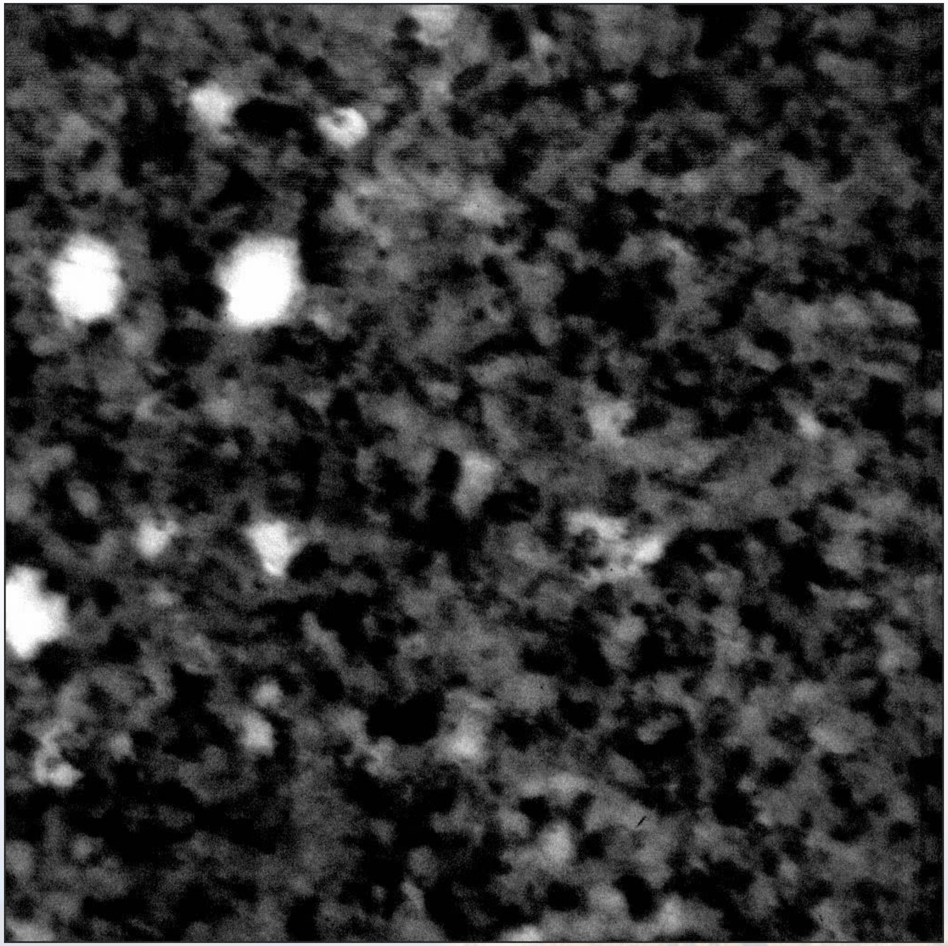
Microfluidic Stage

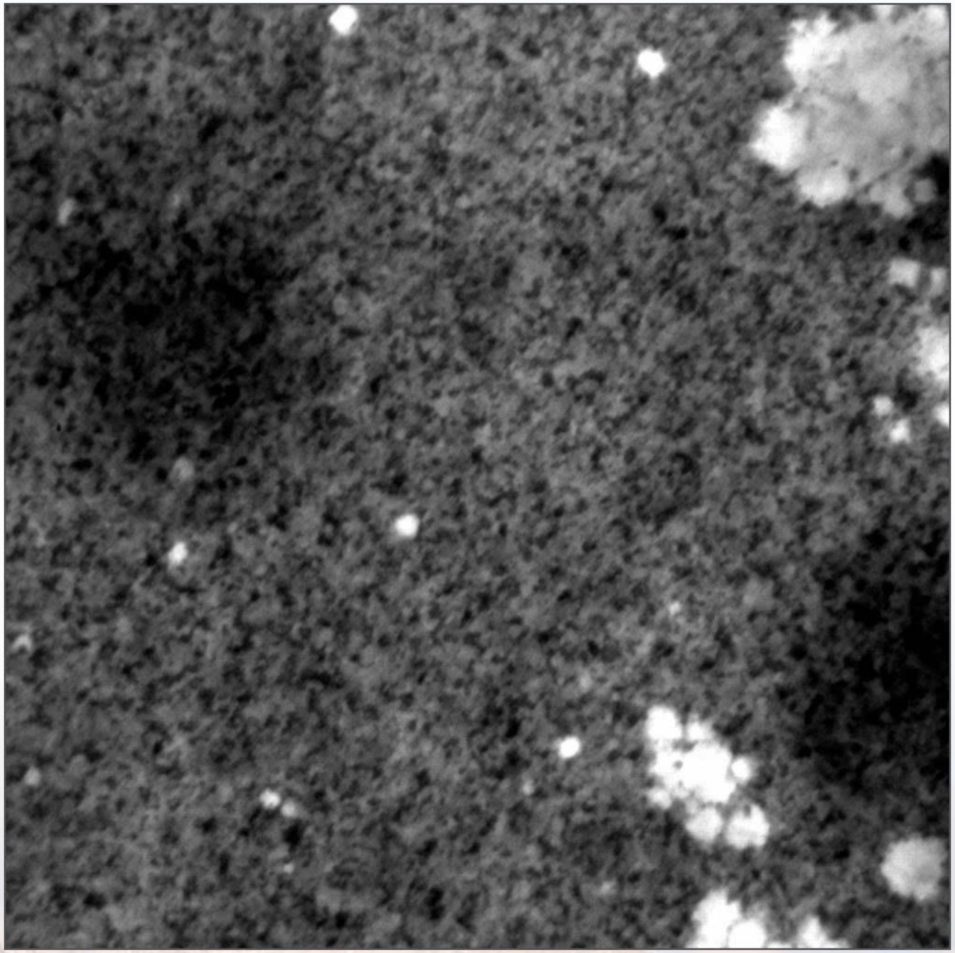
- **Mixing of two or more channels**
- **Continuous observation of the reaction channel**
- **Chamber dimensions are controllable**
- **Films can be directly deposited on the SiN membrane**

More than $5.7 billion is spent annually on control and prevention

Acetic Acid Corroding Nanocrystalline Iron

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



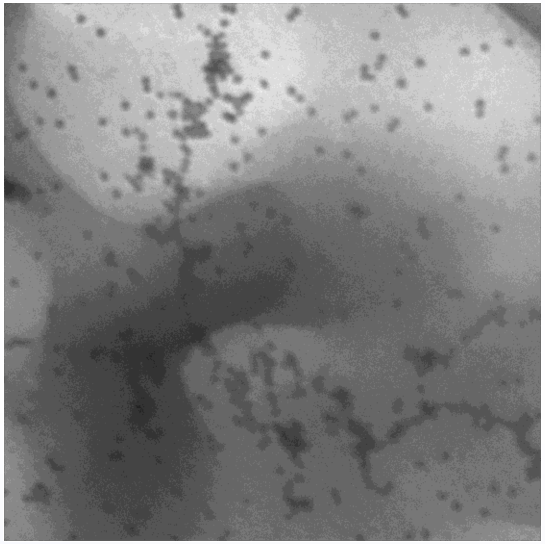


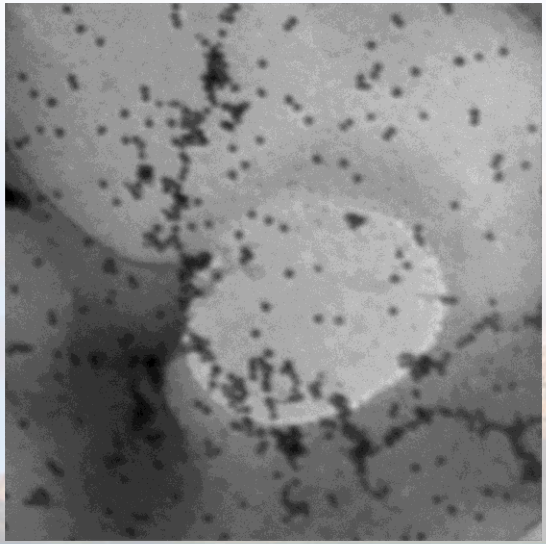
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.

Sandia National Laboratories

In Situ TEM observation of three-phase mixture

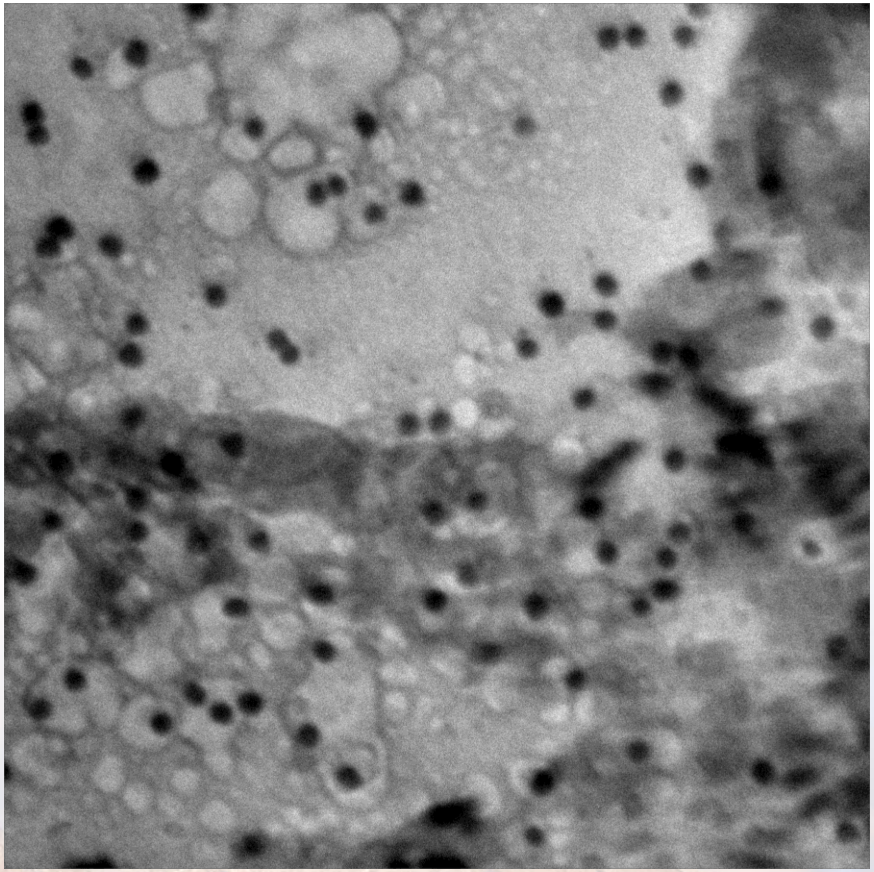
Collaborator: Sarah Hoppe





A pair of images before and after radiolysis from focusing the electron beam

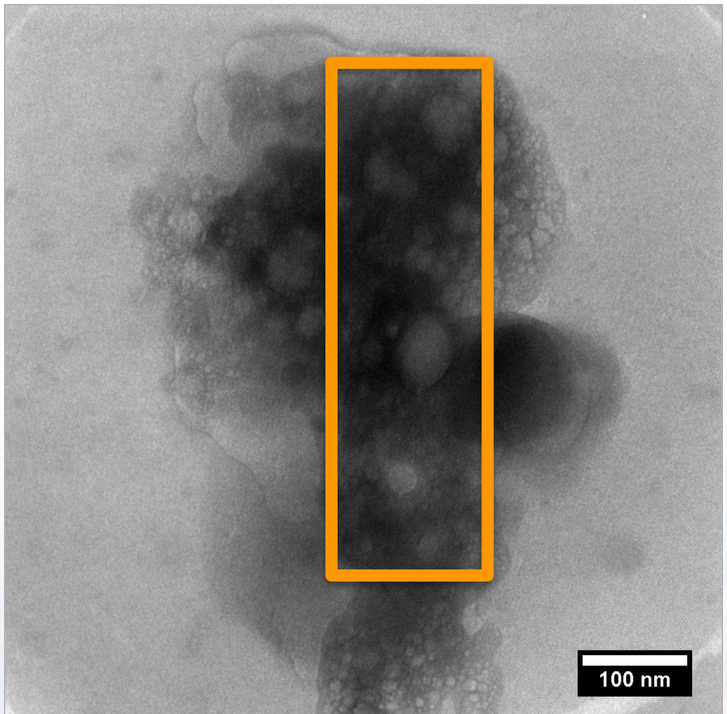
Au nanoparticles, aqueous solution, and gas bubbles



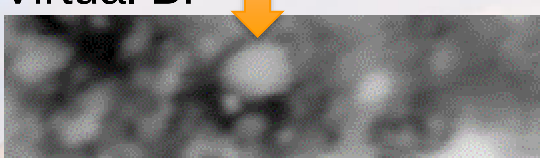
PED in Liquid Cell Environment

$LaCl_3 \cdot 7H_2O : 10\ H_2O$: PED

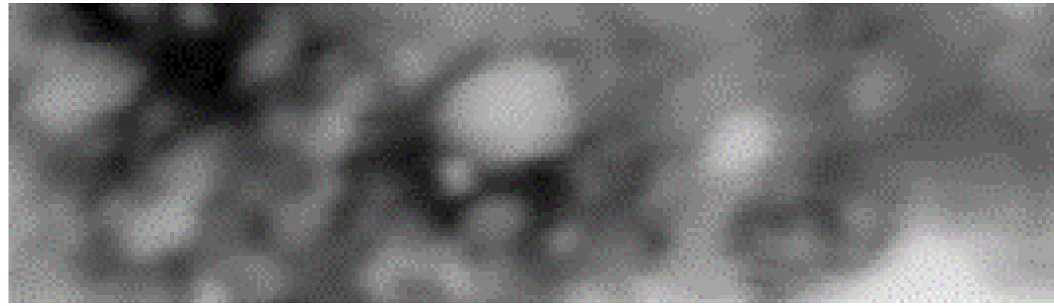
TEM Image



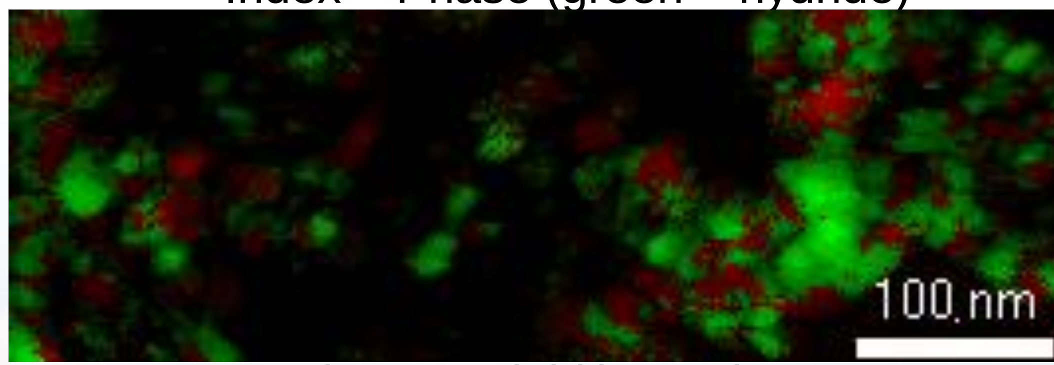
Virtual BF



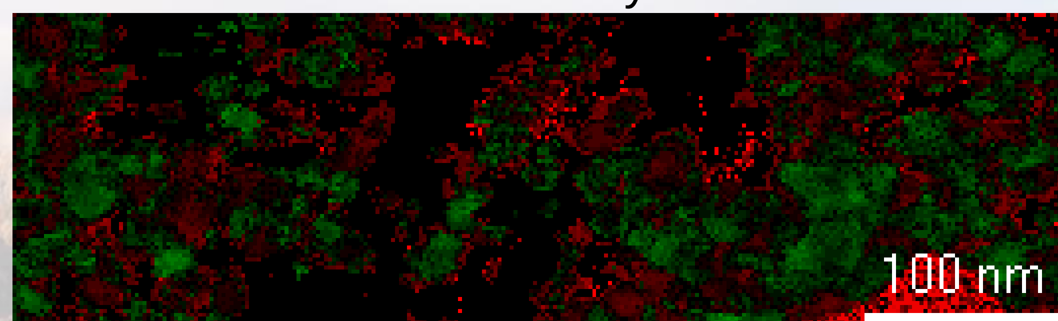
Virtual BF



Index + Phase (green = hydride)



Phase Reliability + Phase

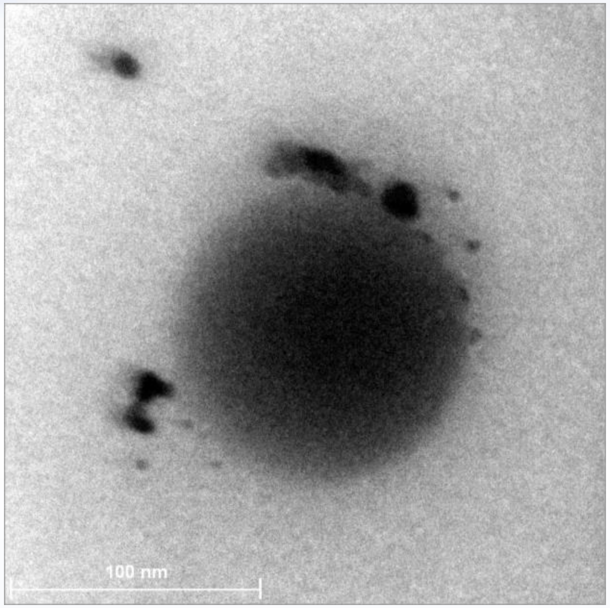


Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

S. Hoppe,
E. Carnes,
J. Brinker

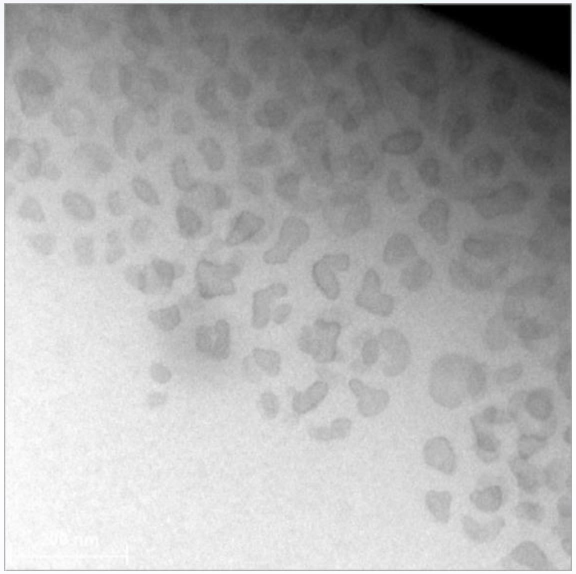
Liposome encapsulated Silica destroyed by the electron beam



BSA Crystallization

S. Hoppe

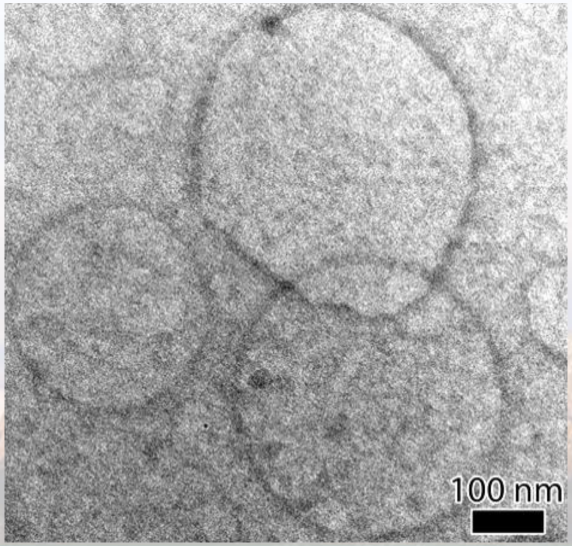
Crystallization of excess Bovine Serum Albumen during flow



Liposomes in Water

S. Hoppe,
D. Sasaki

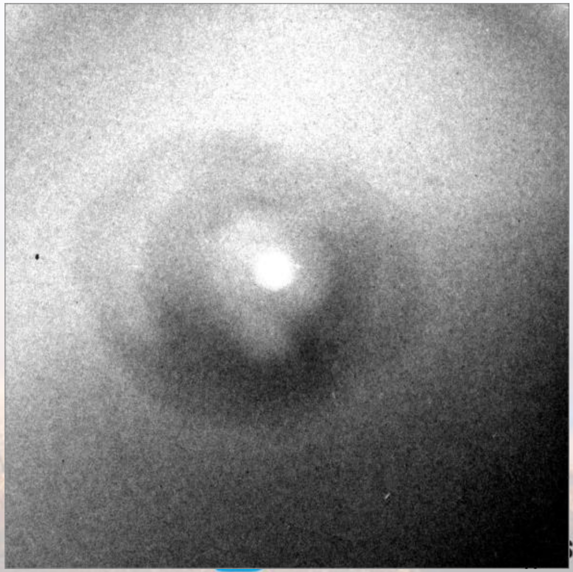
Liposomes imaged in flowing aqueous channel



La Structure Formation

S. Hoppe,
T. Nenoff

La Nanostructure form from $LaCl_3$ H_2O in wet cell due to beam effects

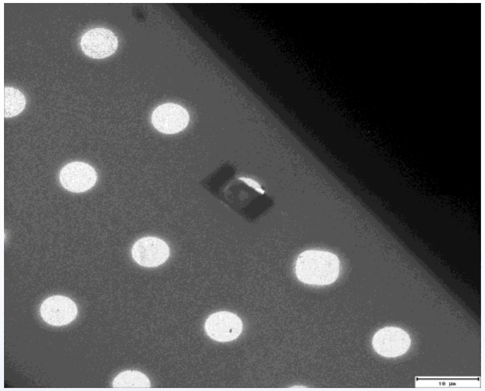


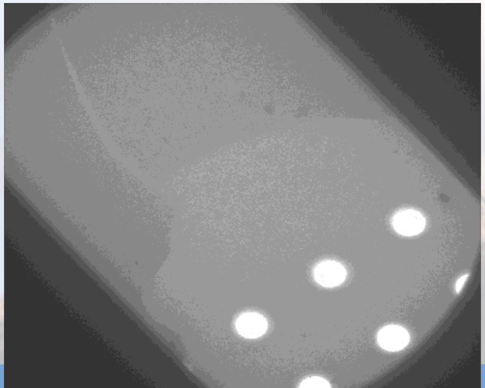
Feasibility of Studying Zircaloy 2 at Nominally 1 atm

Collaborators: S. Rajasekhara and B.G. Clark

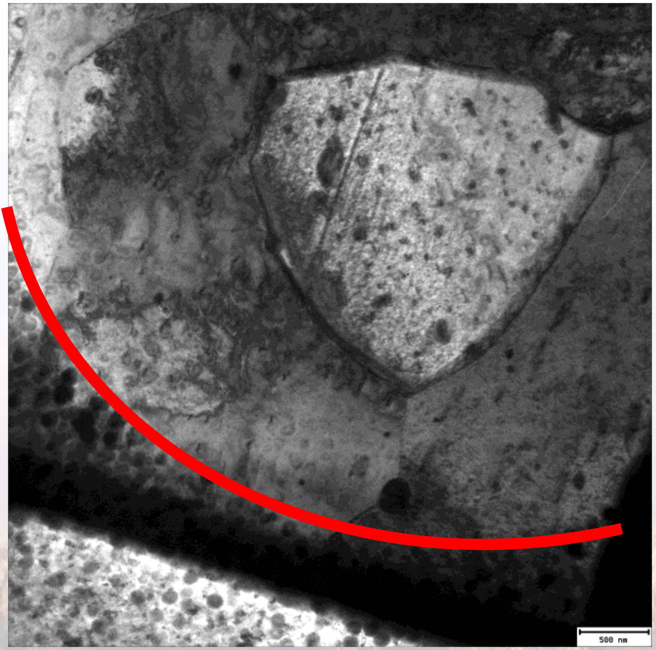
Vapor-Phase Heating TEM Stage

- 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

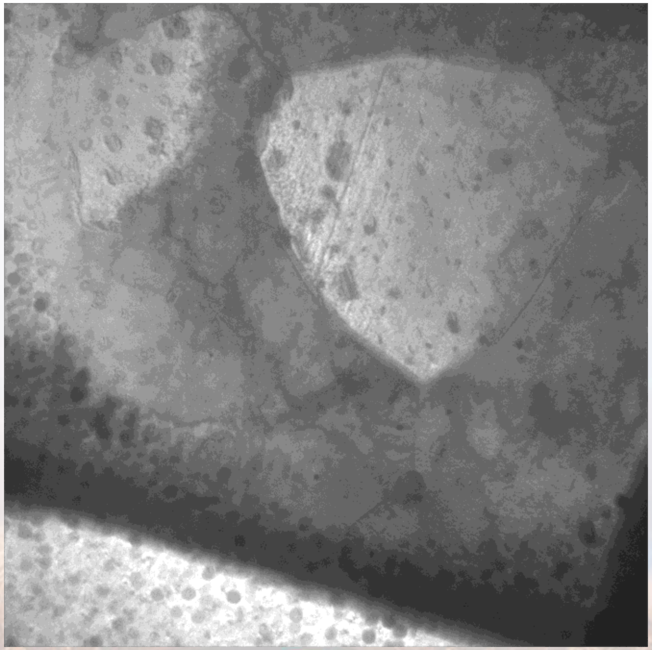




Vacuum & Single Window



Nominally 1 atm H_2 & Two Windows

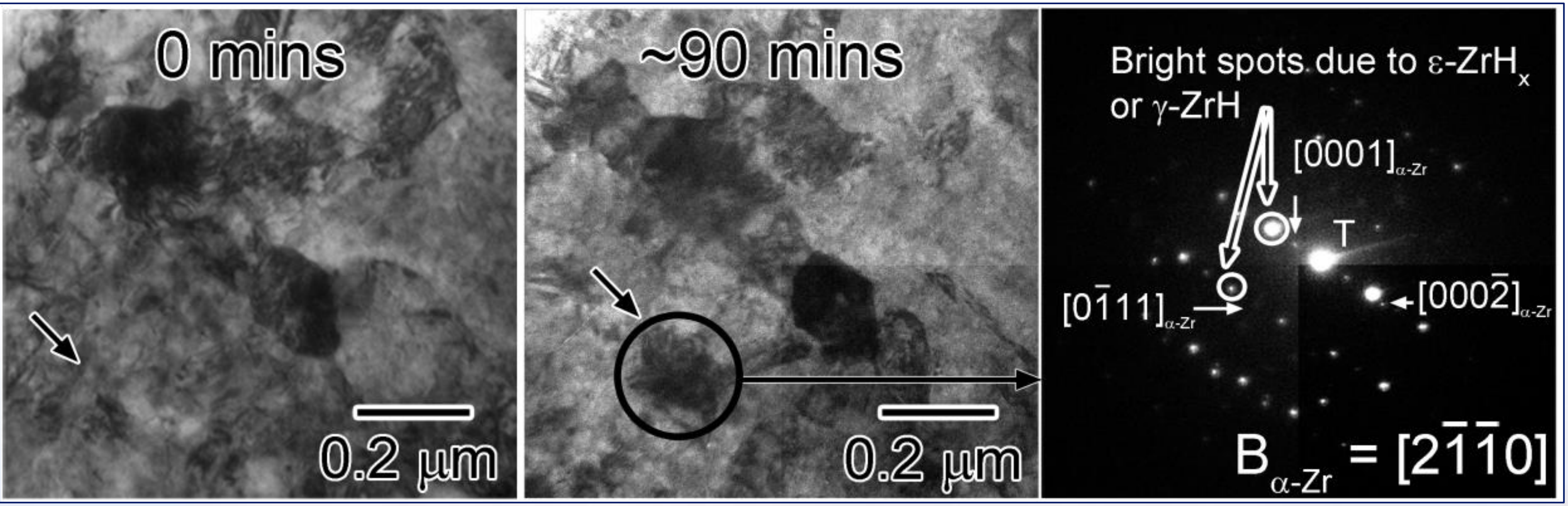


Most features are observed in both despite the decreased resolution resulting from the additional SiN window and 5 µm of air

In situ Observation of Hydride Formation in Zirlo

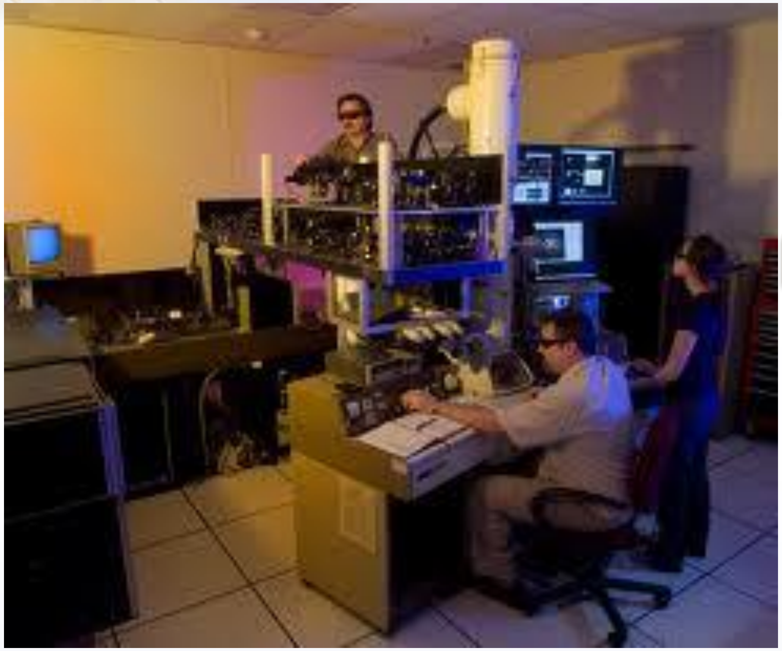
Collaborators: S. Rajasekhara and B.G. Clark

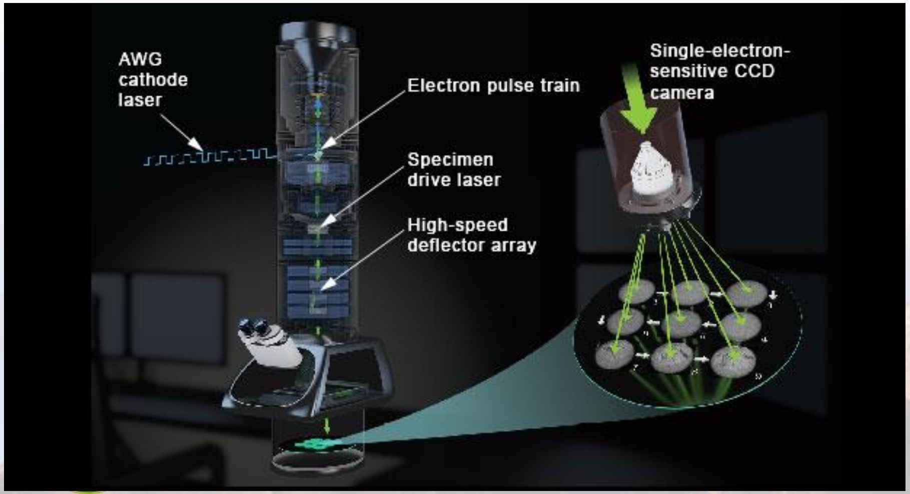
Absolute hydrogen pressure: 327 torr (~ 0.5 atm),
Ramp rate: 1 ^{o}C/s, Final temperature: ~ 400 C, Dwell time: ~ 90 mins



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

Can I^{3}TEM and DTEM systems be combined?

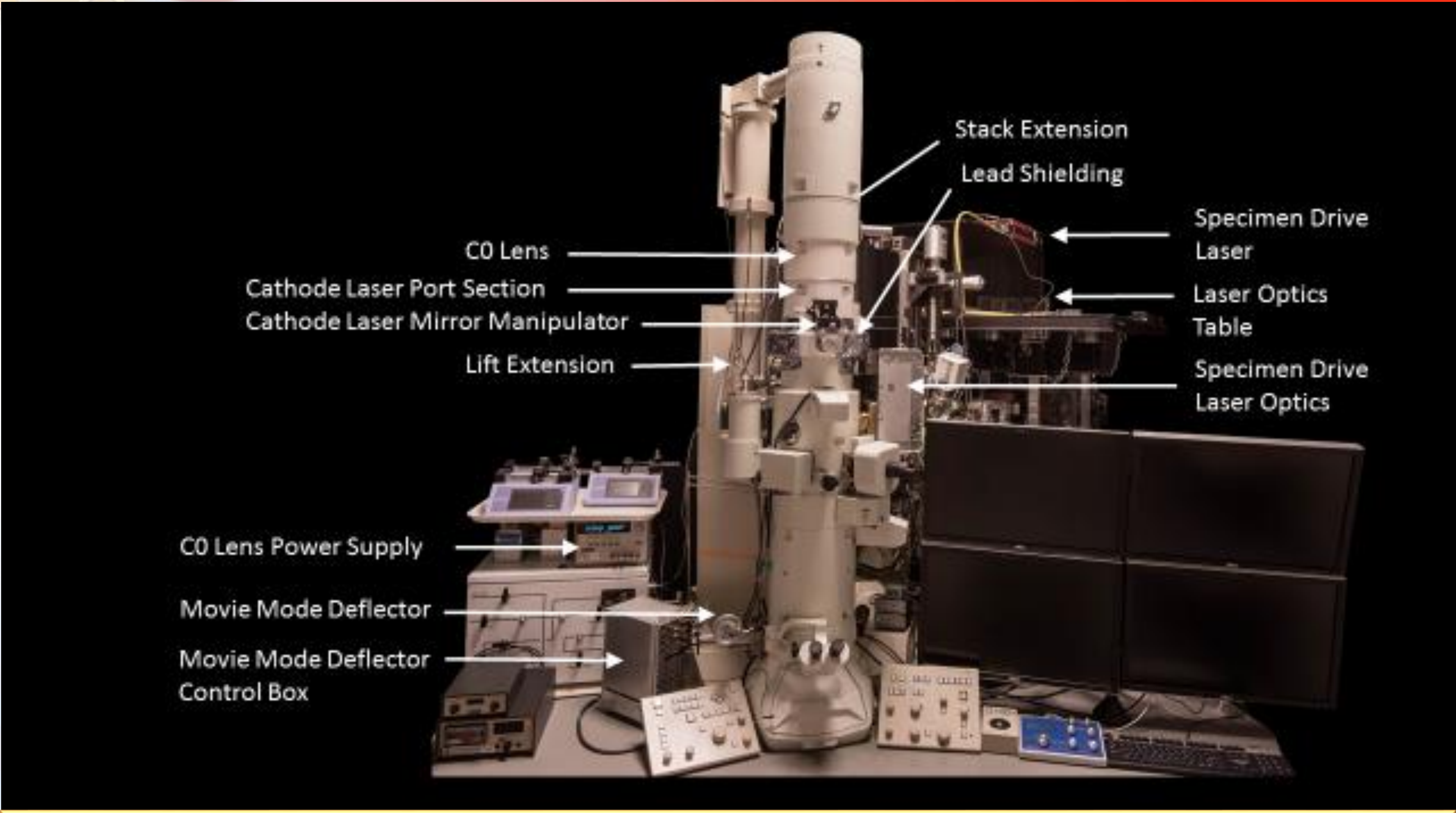




Goal:

To combine the state-of-the-art in microscopy of DTEM and I^{3}TEM to elucidate the response of extreme overlapping environments with adequate spatial and temporal resolution.

Current Status of Laser Addition and DTEM Conversion



UV laser has been purchased, the safety modifications and paperwork is complete, and optics are under construction. The Ta cathode is under development. Hope for single shot observations soon!

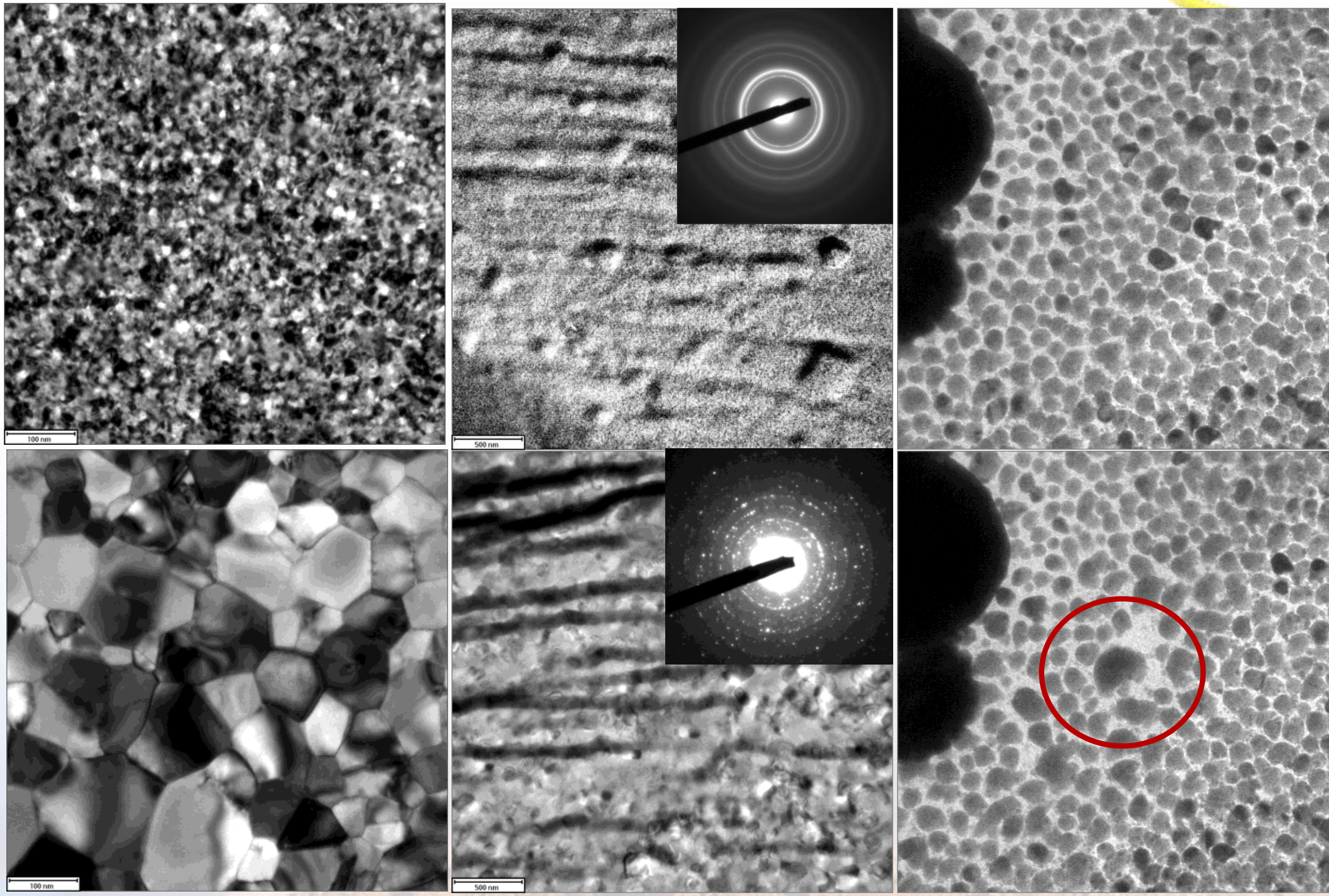
Initial Laser Heating Observations

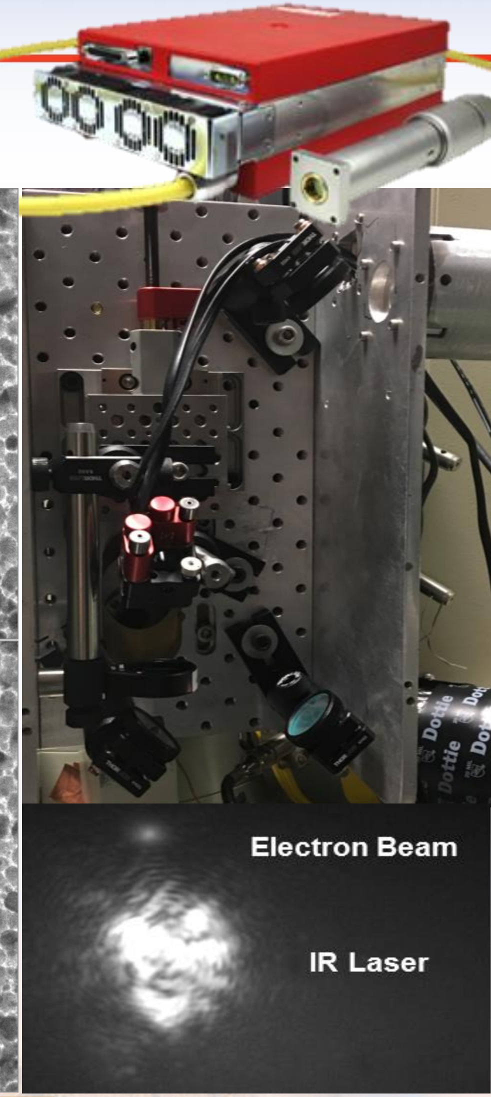
Collaborator: C.M. Barr, D. Adams, M. Abere

Pt Grain Growth | Reactive Multilayer Films | Nanoparticle Sintering

Before

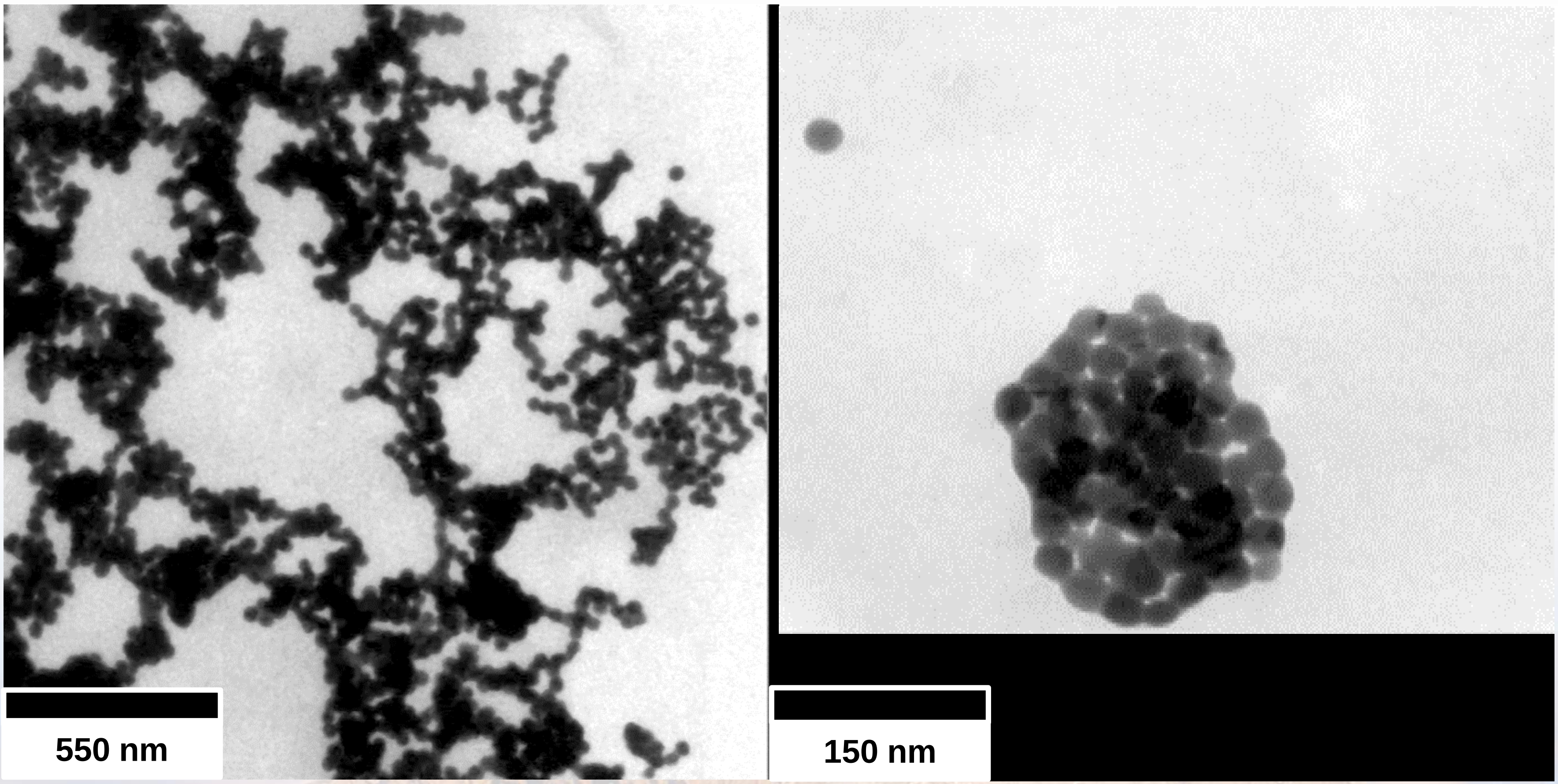
After





We can now introduce rapid thermal heating with any TEM stage or ion beam conditions

Complex Interaction Au NPs Exposed to Laser Irradiation

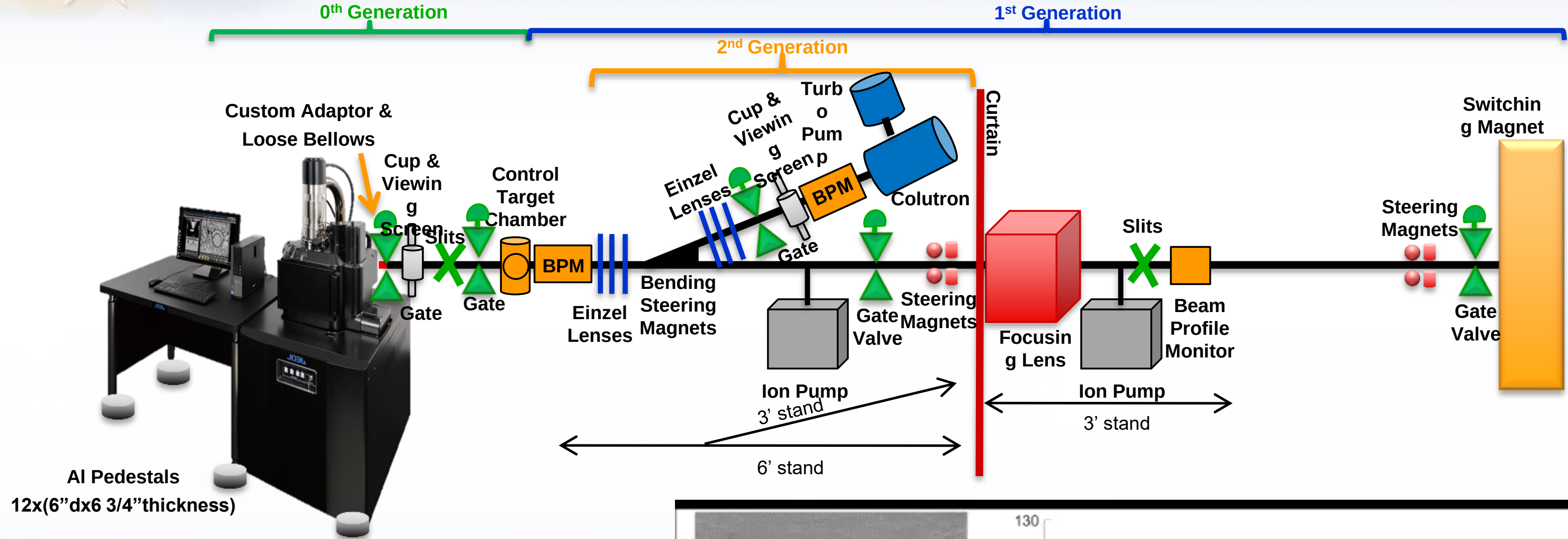


Speed = 2.5x

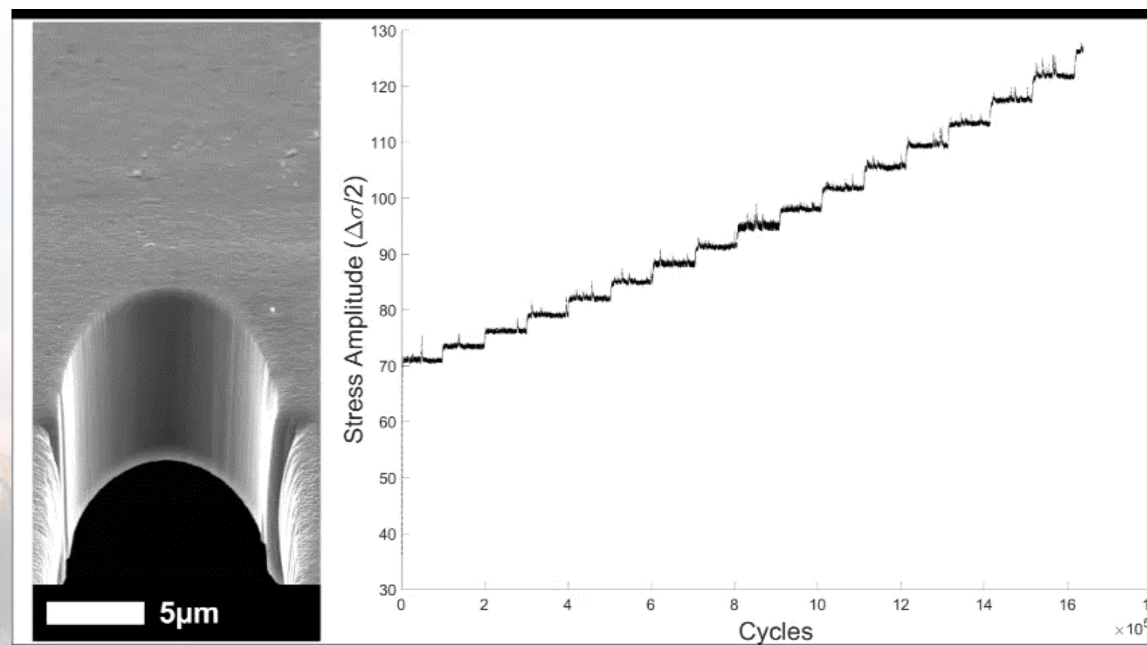
A Complex Combination of Sintering, Reactions, and Ablation Occurs

Scaling Back Up:
In situ Ion Irradiation SEM (I^3SEM)

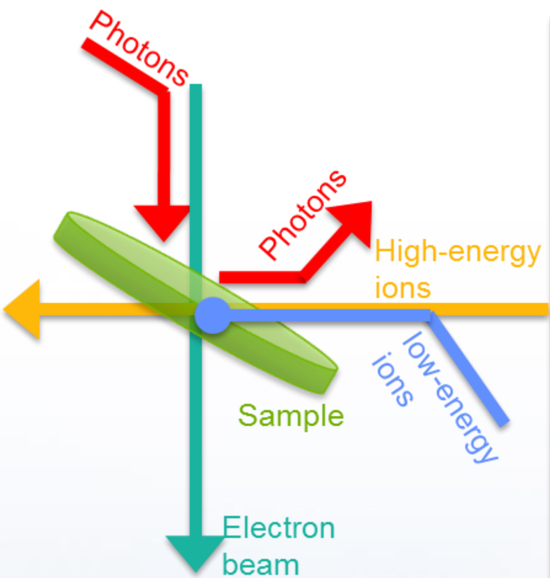
Collaborators: N. Heckman, D. Buller, B. Boyce, J. Carroll, P. Price, C. Taylor, B. Muntifering, S. Briggs



We are designing this to be the world's best *in situ* SEM for overlapping extreme environments



Summary



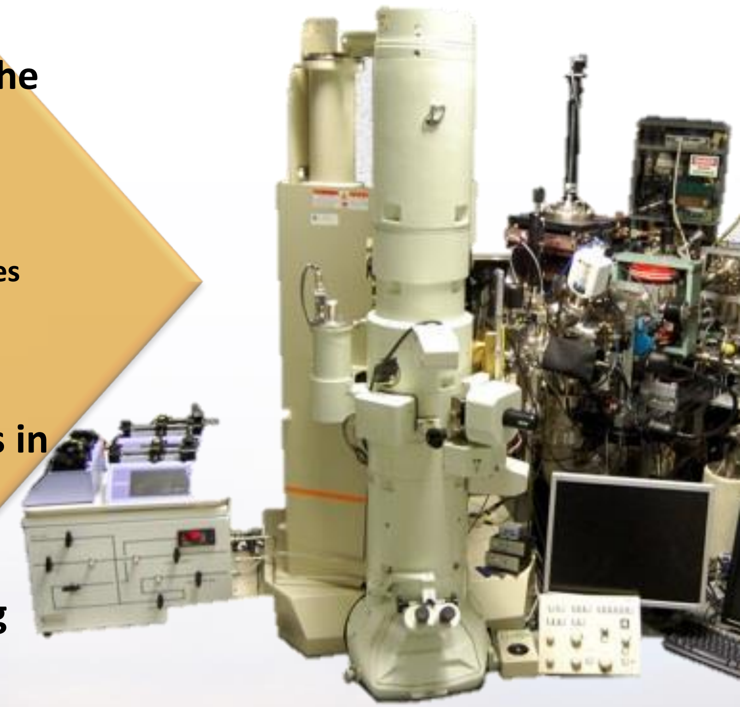
The Ion Beam Lab at Sandia National Laboratories applies a variety of nanoscale tools to a wealth of problems

Sandia's I³TEM is one of a few in the world

- ***In situ* irradiation from H to Au**
- ***In situ* gas implantation**
- **Combinations of in-situ techniques**

I³TEM can provide fundamental understanding to key mechanisms in a variety of extreme conditions

The I³TEM capability are still being expanded…



Collaborators:

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

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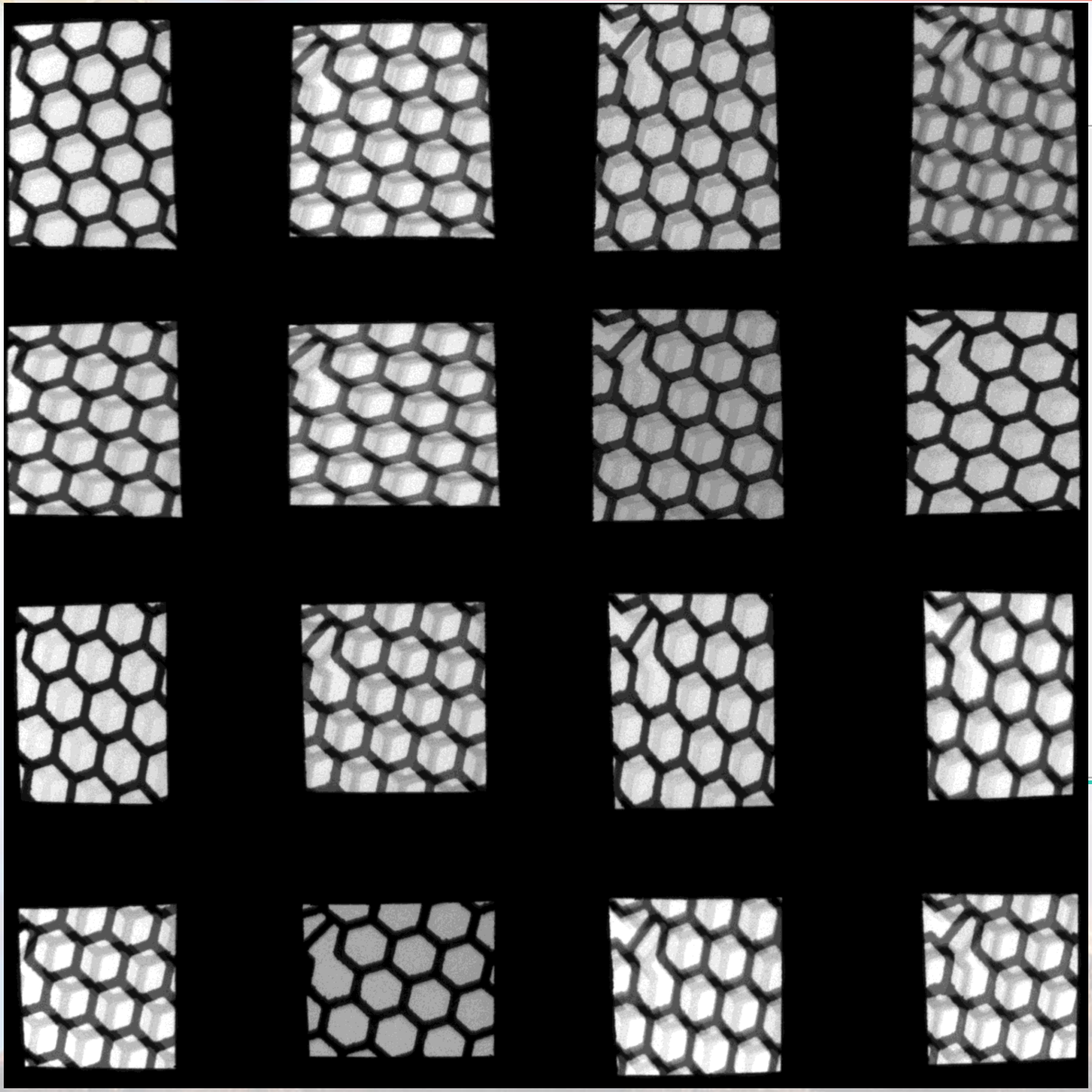
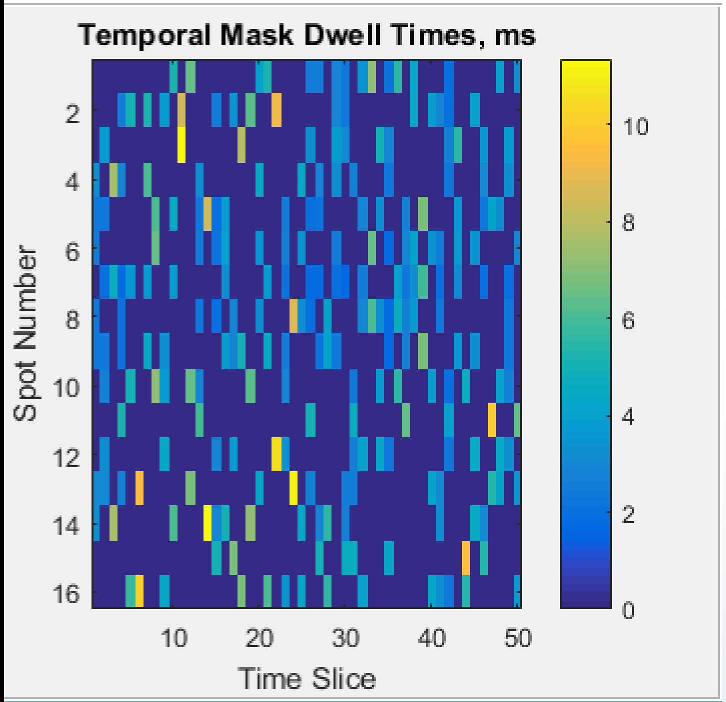
Back-up

Demonstration of Movie Mode Deflectors

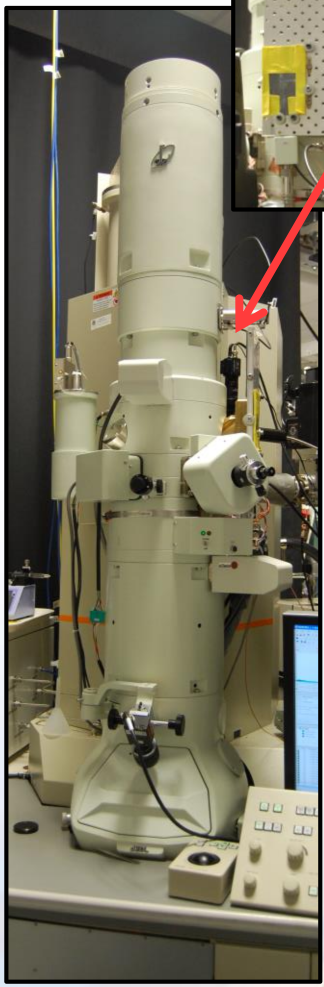


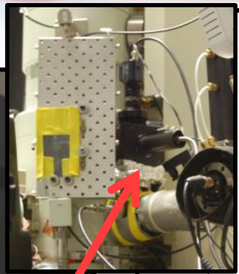


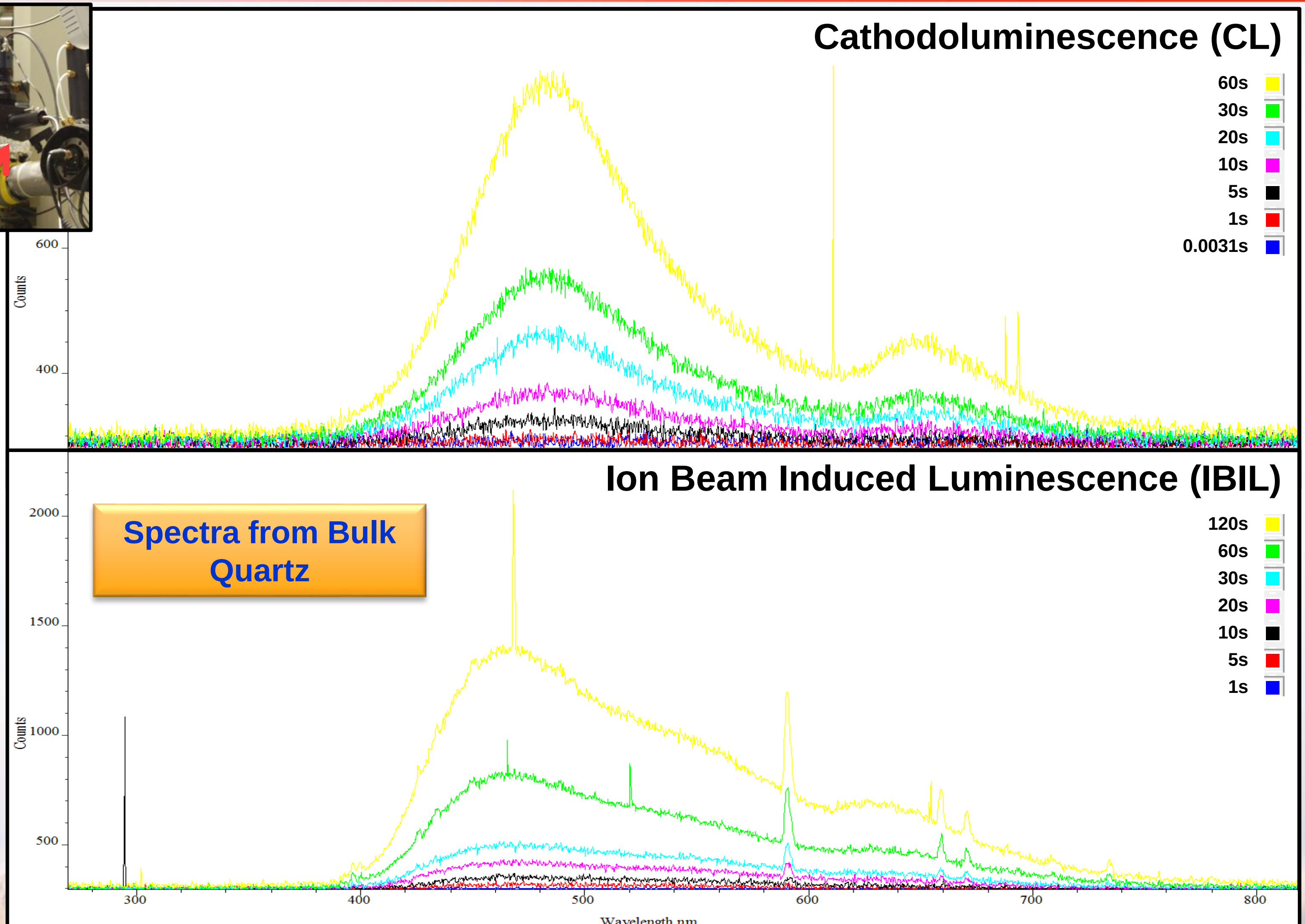
Demonstration of Relativity done 12/14/2016.

Future Direction: *In situ* TEM
Ion beam Induced Luminescence (IBIL)

Collaborator: J. Gutierrez-Kolar







Significant optimization is still needed; potential is promising

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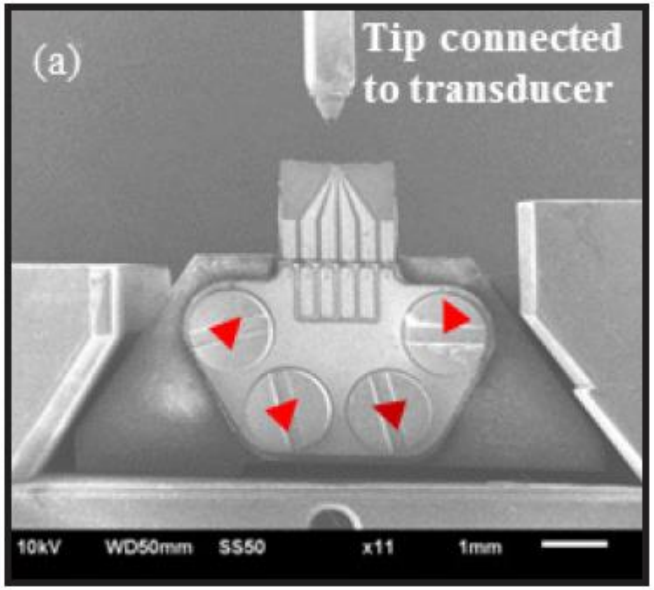
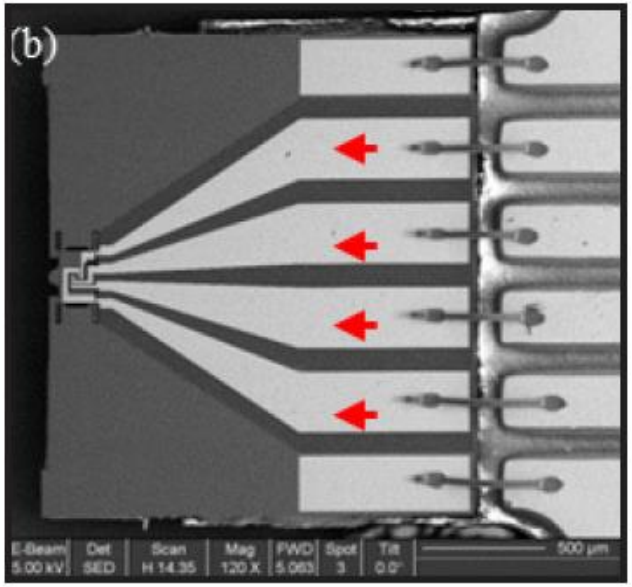
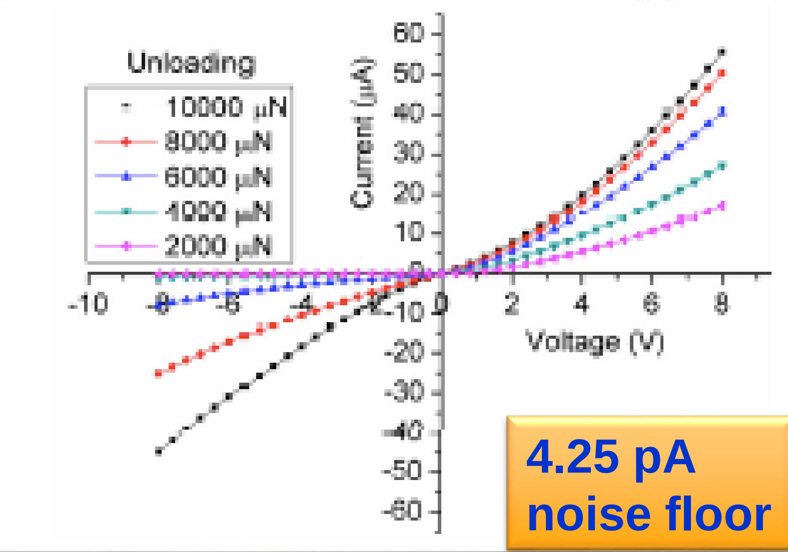
Controlling the Sample through TEM Stage Upgrades

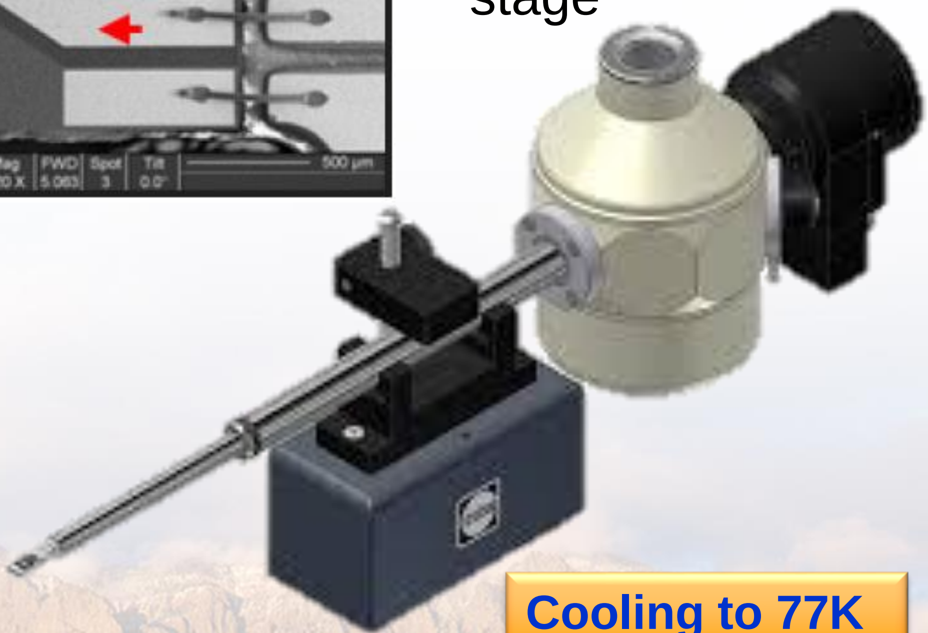






4.25 pA noise floor

- Electromechanical upgrade to Hysitron PI-95
- Gatan double-tilt liquid nitrogen stage



Cooling to 77K

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