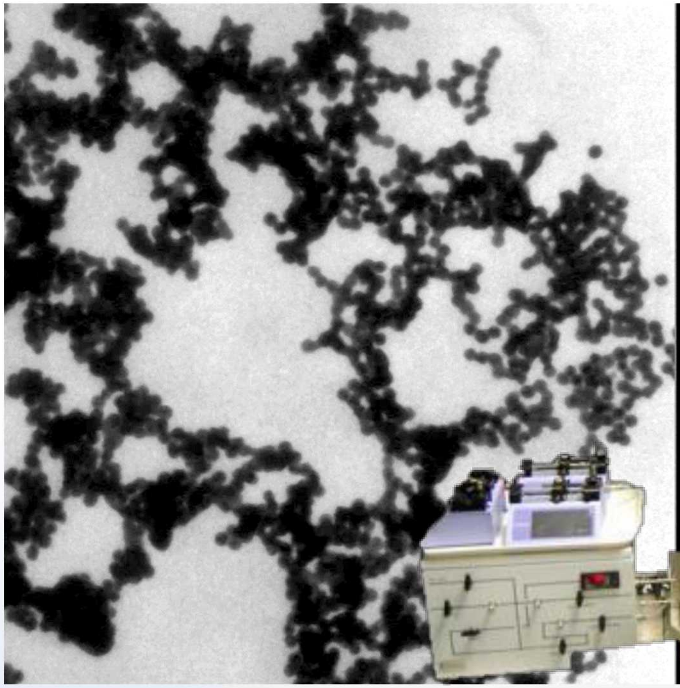
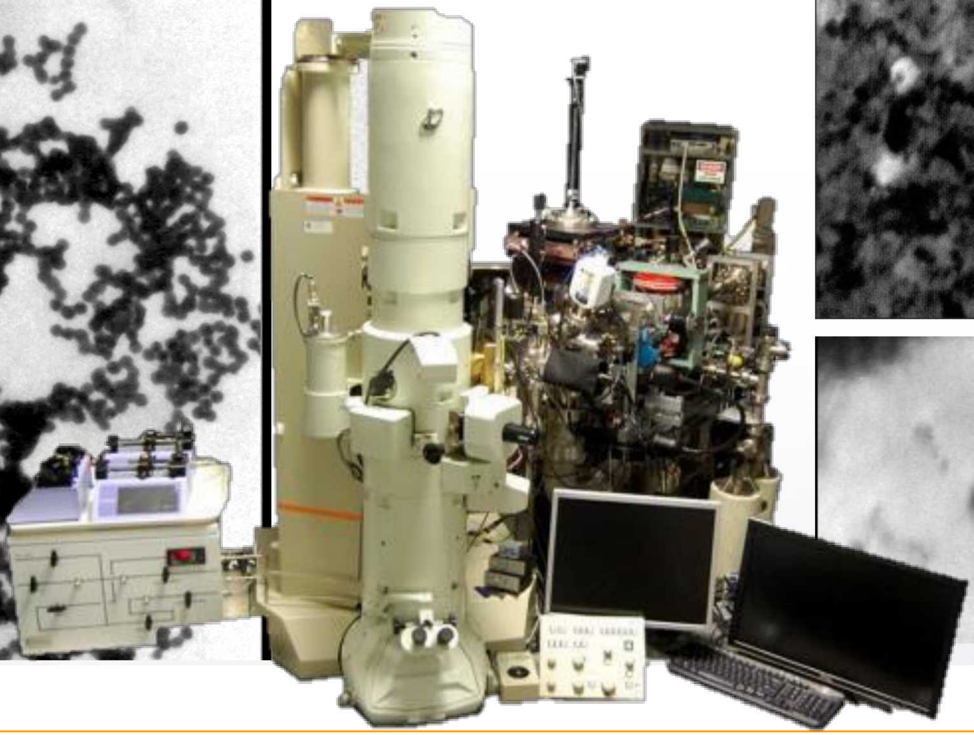
Exploring Extreme Environments with Adequate Temporal Resolution

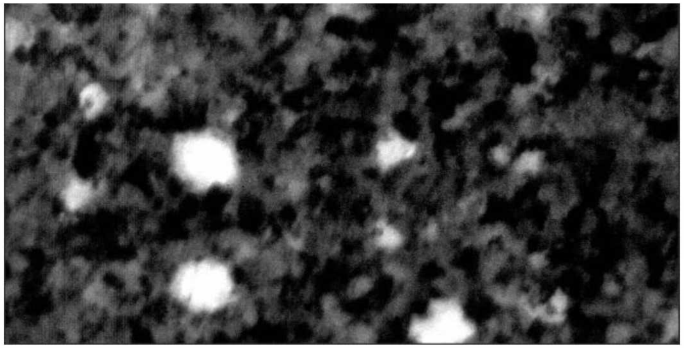
SAND2019-4654C

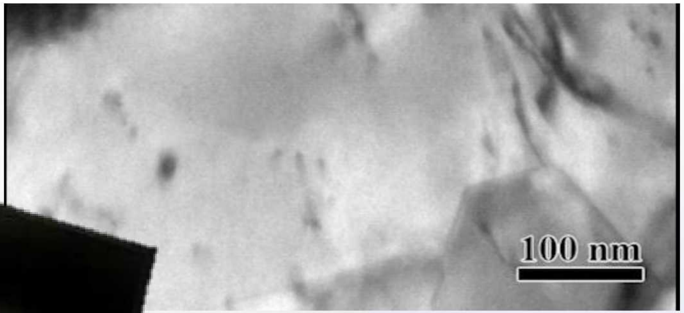
K. Hattar
Sandia National Laboratories

April 26th, 2019









Collaborators:

- D.L. Buller, D.C. Bufford, S.H. Pratt, T.J. Boyle, B.A. Hernandez-Sanchez, S.J. Blair, B. Muntifering, C. Chisholm , P. Hosemann, A. Minor, J. A. Hinks, F. Hibberd, A. Ilinov, D. C. Bufford, F. Djurabekova, G. Greaves, A. Kuronen, S. E. Donnelly, K. Nordlund, F. Abdeljawad, S.M. Foiles, J. Qu, C. Taylor, J. Sugar, P. Price, C.M. Barr, D. Adams, M. Abere, L. Treadwell, A. Cook, A. Monterrosa, IDES Inc, J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, W. Mook, Hysitron Inc., G.S. Jawaharram, S. Dillon, R.S. Averback, N. Heckman, J. Carroll, S. Briggs, E. Carnes, J. Brinker, D. Sassaki, T. Nenoff, B.G. Clark, P.J. Cappillino, B.W. Jacobs, M.A. Hekmaty, D.B. Robinson, L.R. Parent, I. Arslan, & Protochips, Inc.

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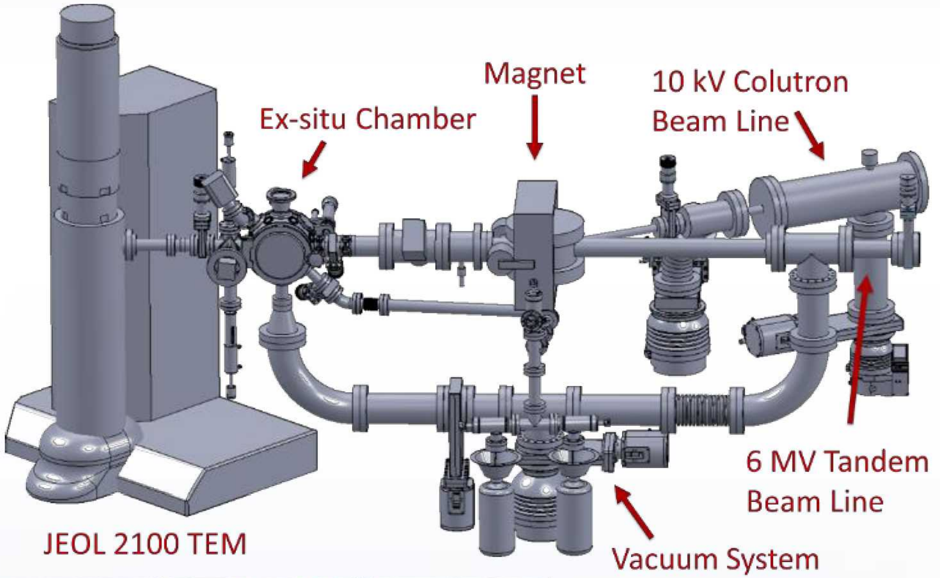
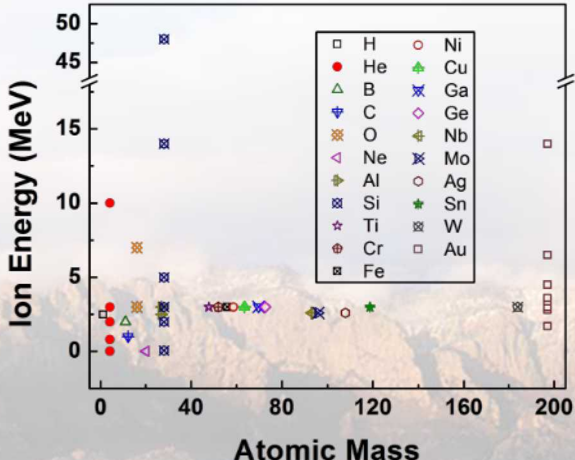
Sandia's Concurrent *In situ* Ion Irradiation TEM Facility

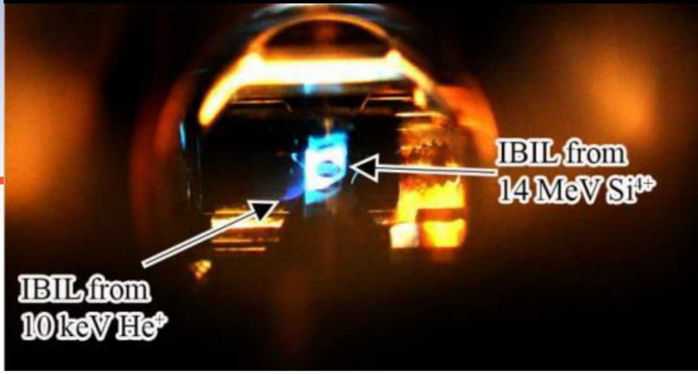
Collaborator: D.L. Buller

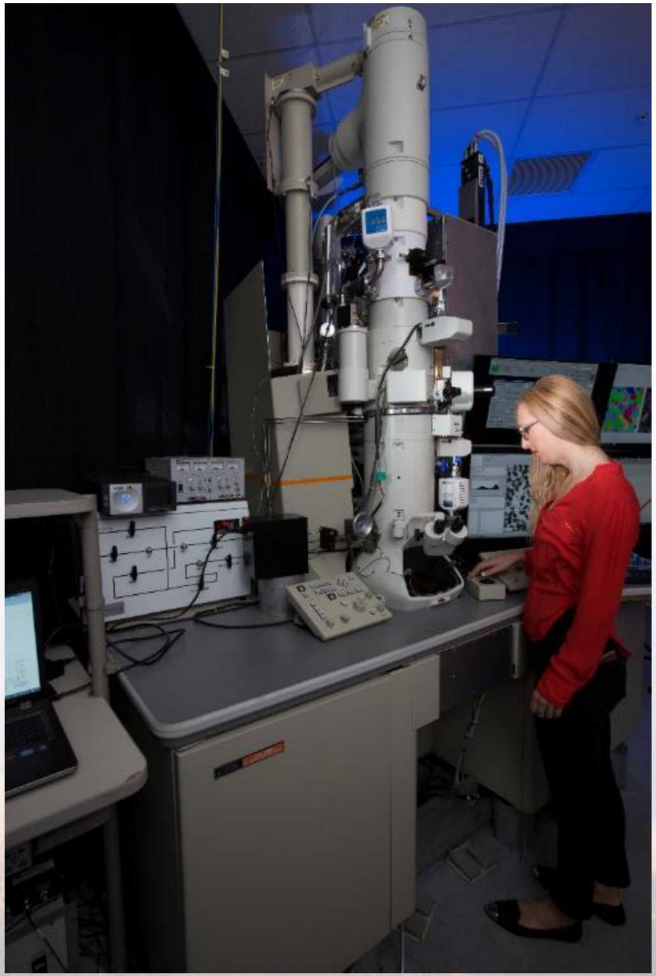
10 kV Colutron - 200 kV TEM - 6 MV Tandem





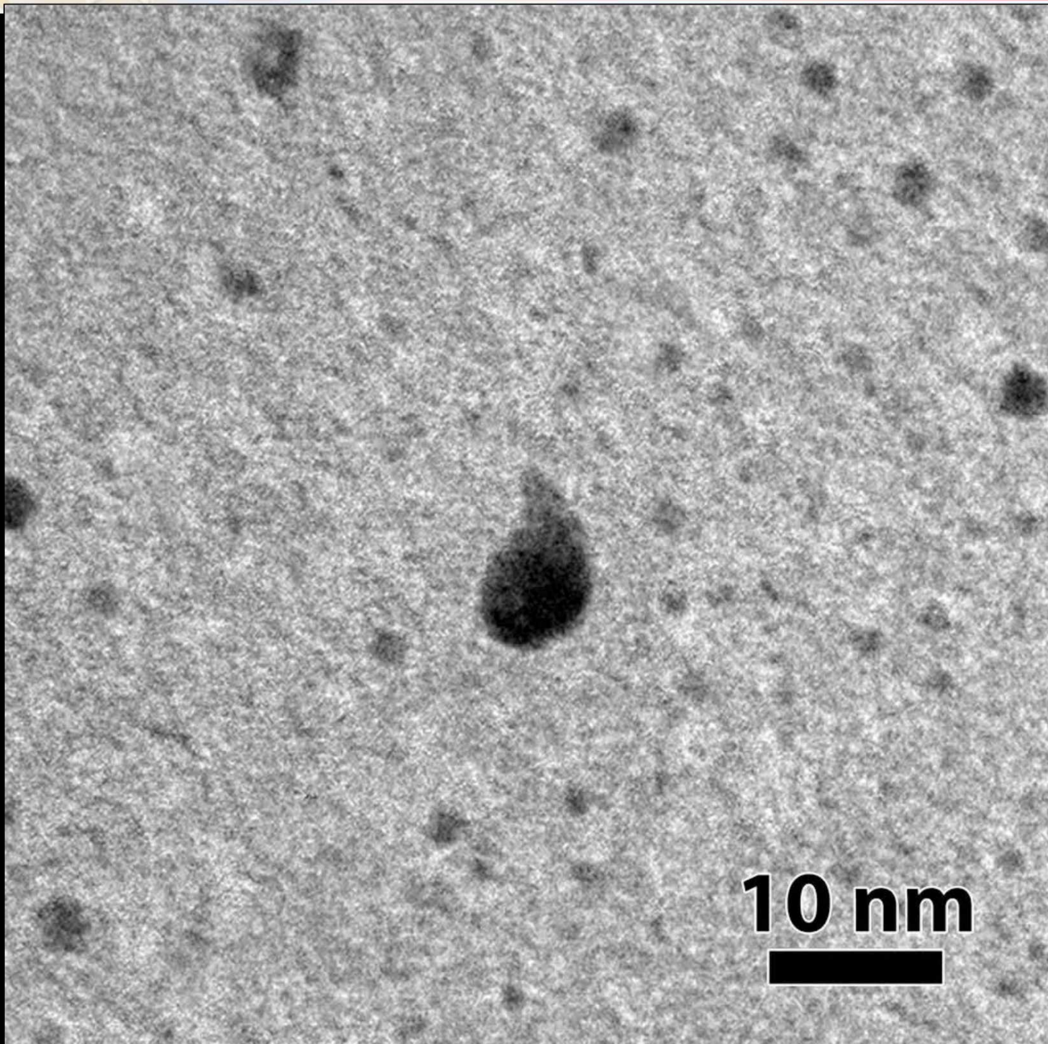
IBIL from a quartz stage inside the TEM

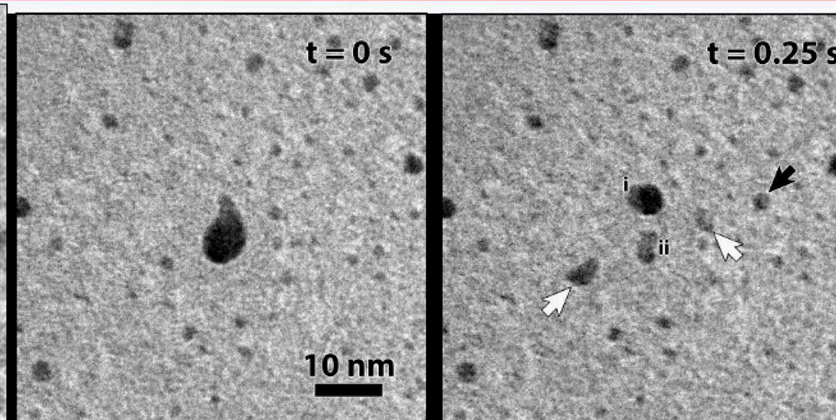


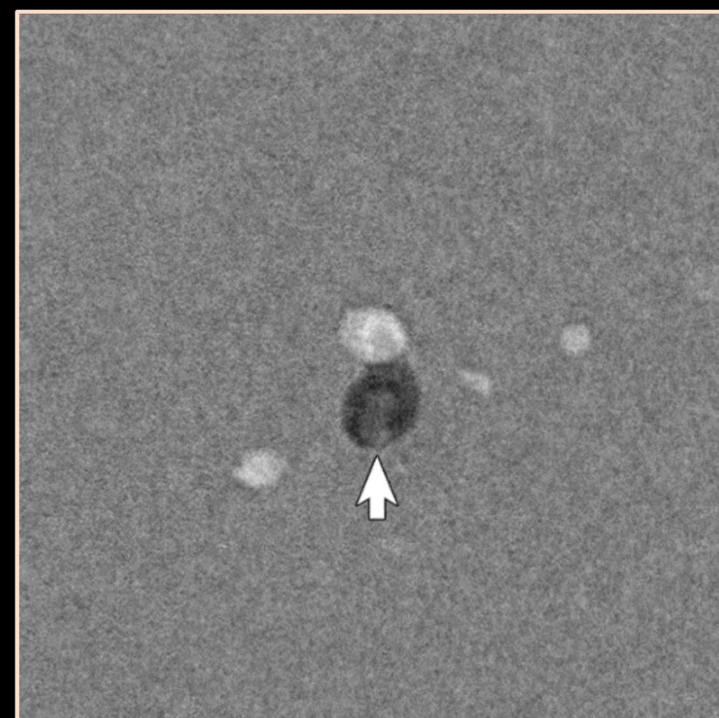


Single Ion Strikes:
46 keV Au^{1-} ions into 5 nm Au nanoparticles

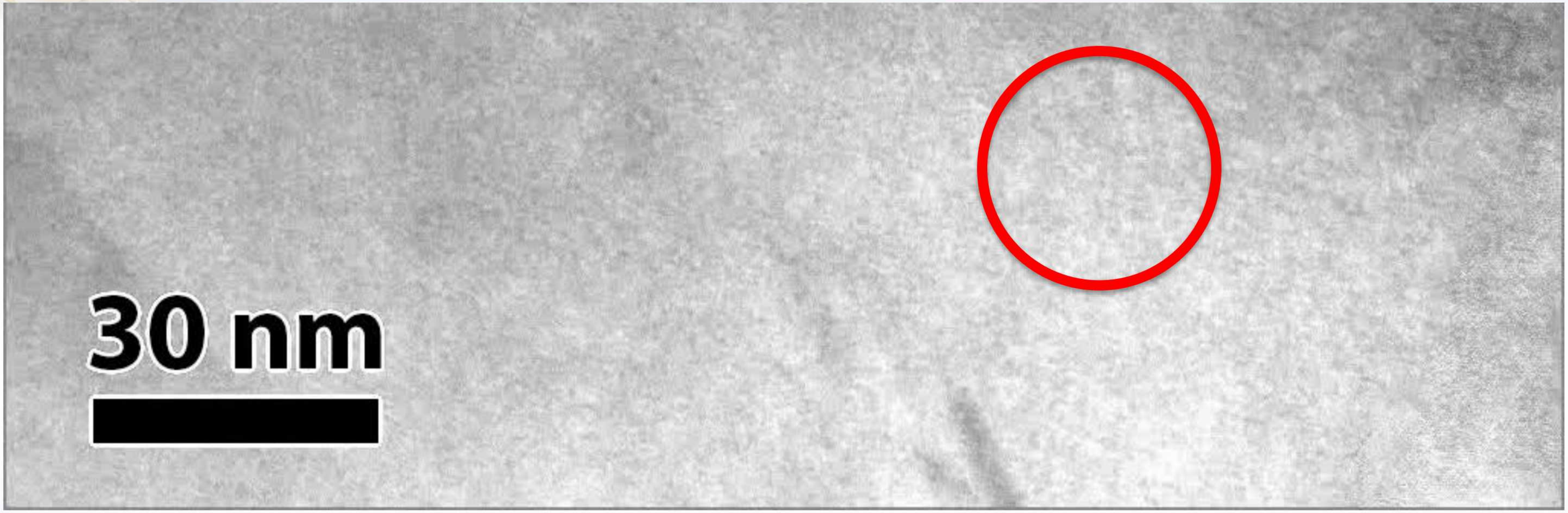
Collaborator: D.C. Bufford

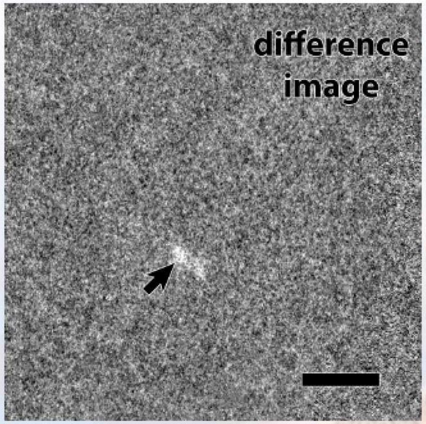






Motivation: In-Situ Single Ion Strikes in Si





- 1.7 MeV Au into single crystal Si
- Single ion strikes can be observed in semiconductors
- Non-symmetric structure in contrast to the spherical approximation

Can we go beyond this to observe:

- Important aspects of structural evolution (ns to hrs.)?
- Evolution in more complex systems (GaAs)?
- Directly correlate it to key model parameters?

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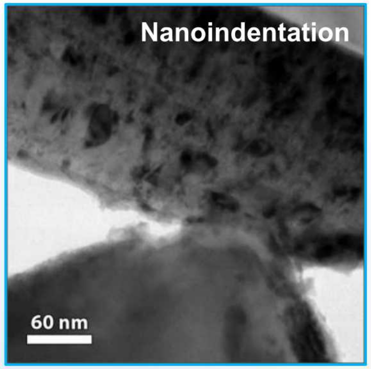
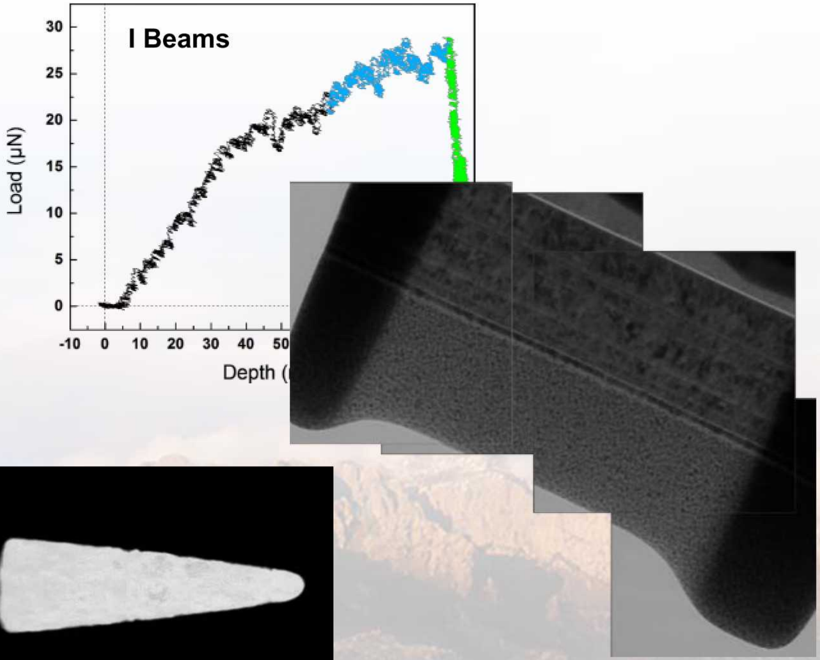
In situ Quantitative Mechanical Testing

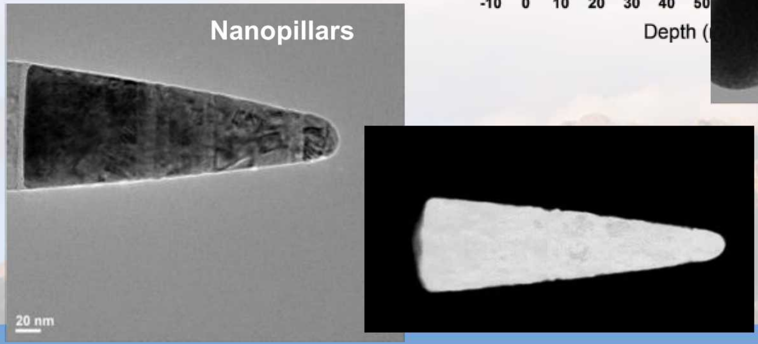
Contributors: J. Sharon, B. L. Boyce, C. Chisholm, H. Bei, E.P. George, P. Hosemann, A.M. Minor, & Hysitron Inc.

Hysitron PI95 *In Situ* Nanoindentation TEM Holder

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

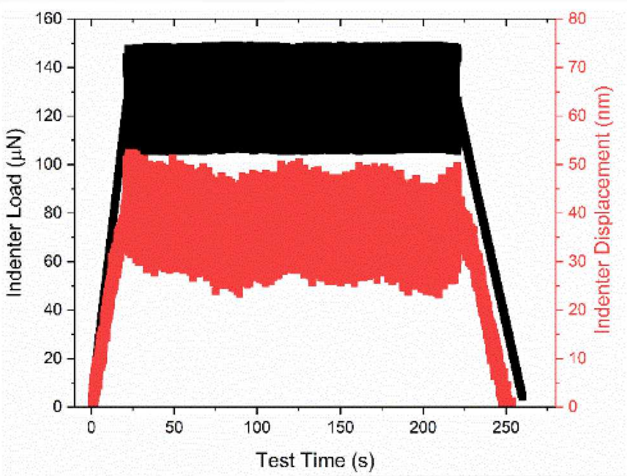






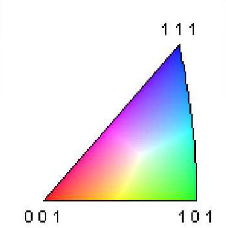
Cyclic Loading Coupled with ACOM

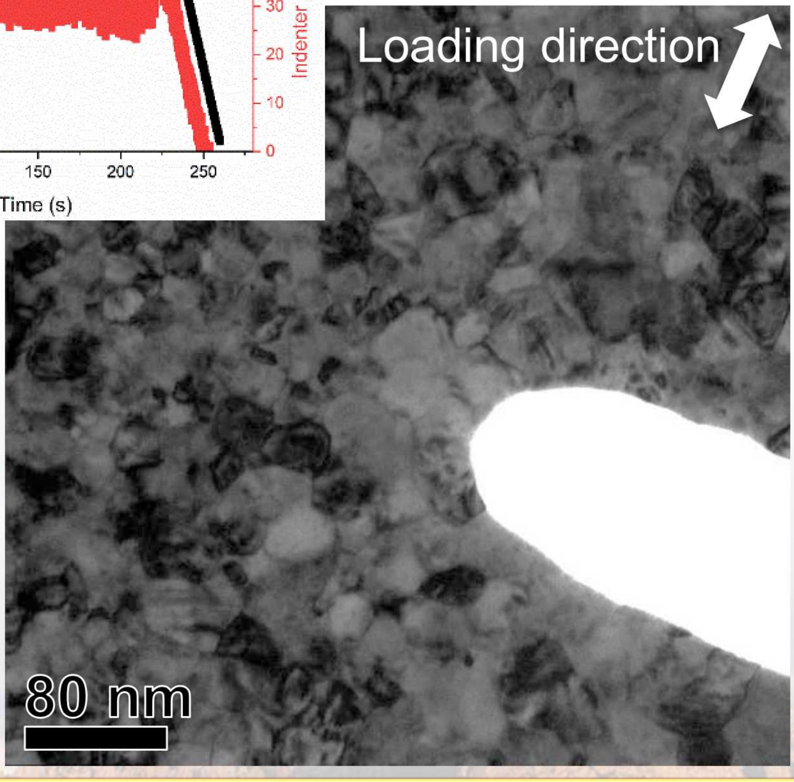
Collaborators: C. Barr & W. Mook

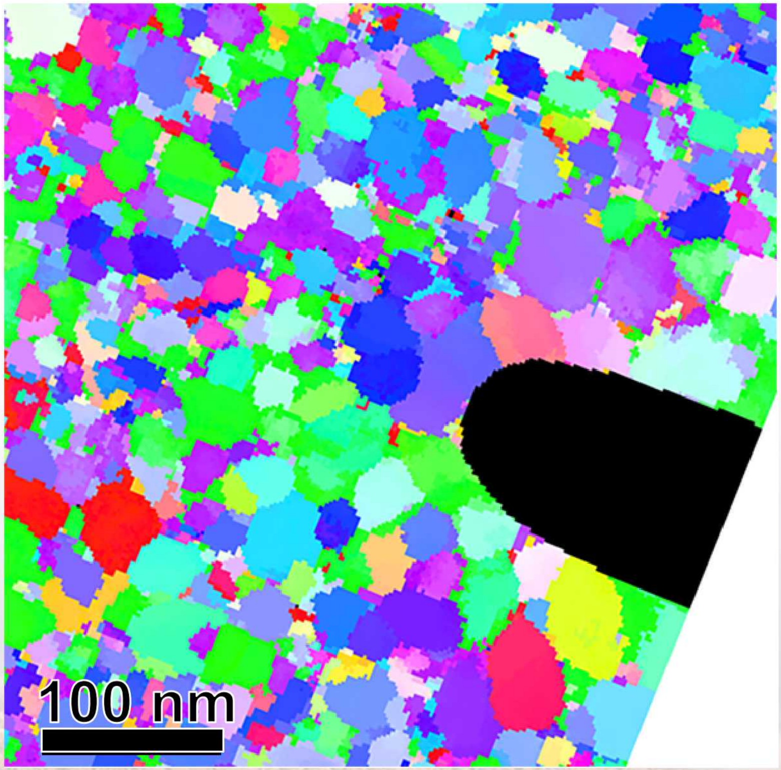


Mean load (P_{mean}) = 135 uN

Amplitude load (P_{amp}) = 35 uN



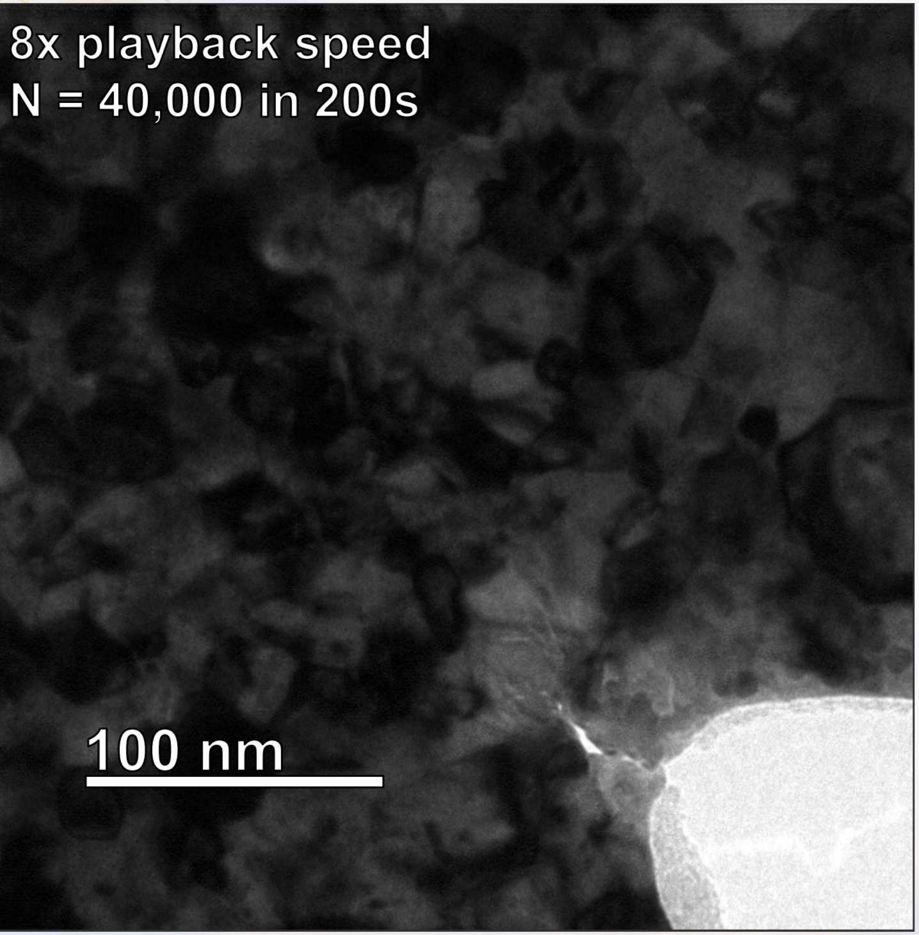


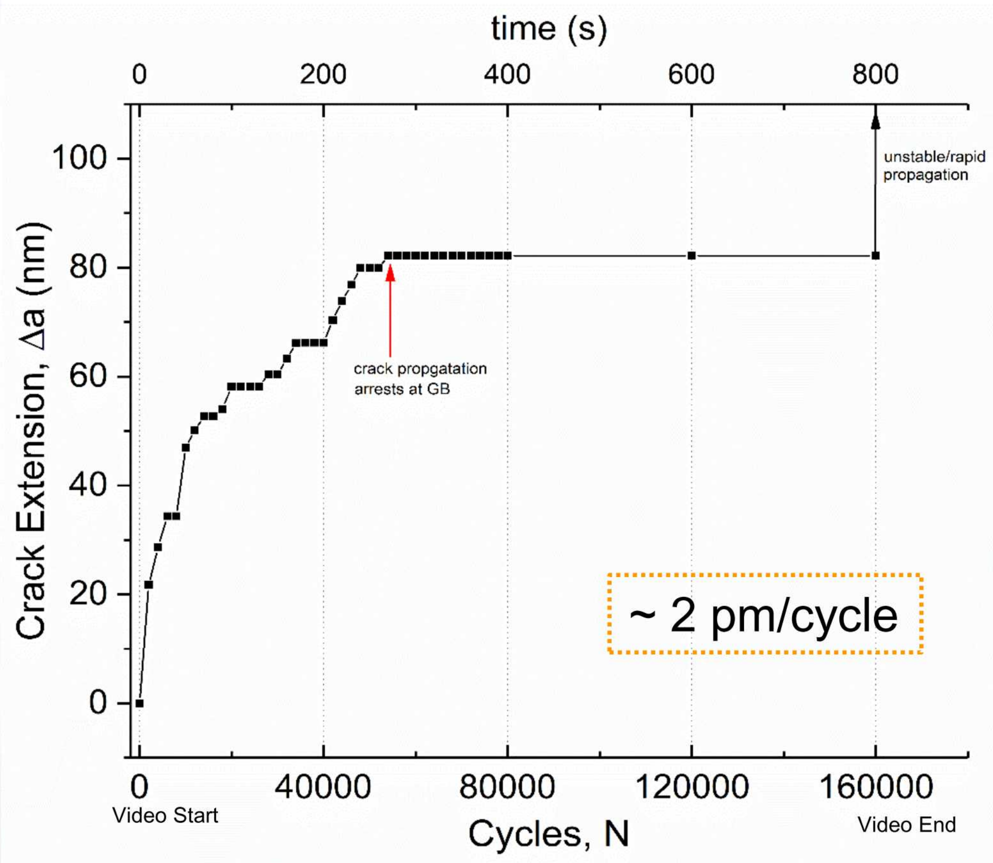


Orientation maps pre-, intermediate, and post- in-situ mechanical test can assist in deconvoluting possible mechanisms during cyclic loading

Cyclic Loading: Complex Crack Propagation

Collaborators: C. Barr & W. Mook





- Mean load: 135 uN; Amplitude load: 35 uN
- 200 Hz, 200s test (15 fps 1k x 1k camera)

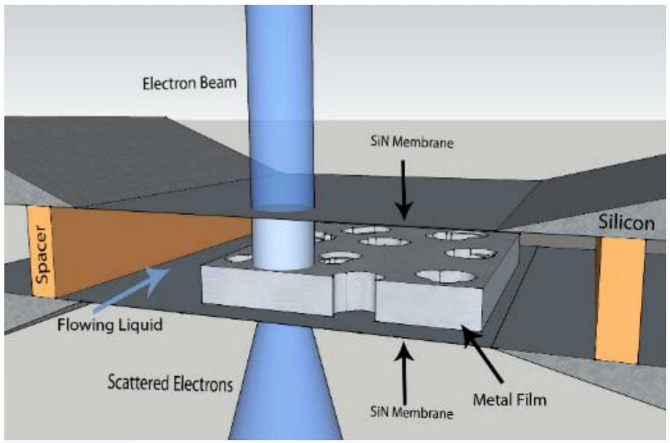
- da/dN = 1.7×10^{12} m/cycle
- Non-linear crack extension rate
- Crack propagation path changes "direction"

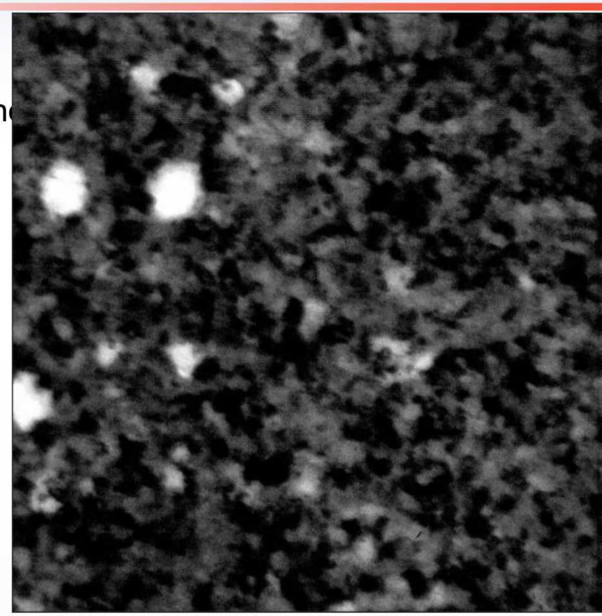
Can We Gain Insight into the Corrosion Process through *In situ* TEM?

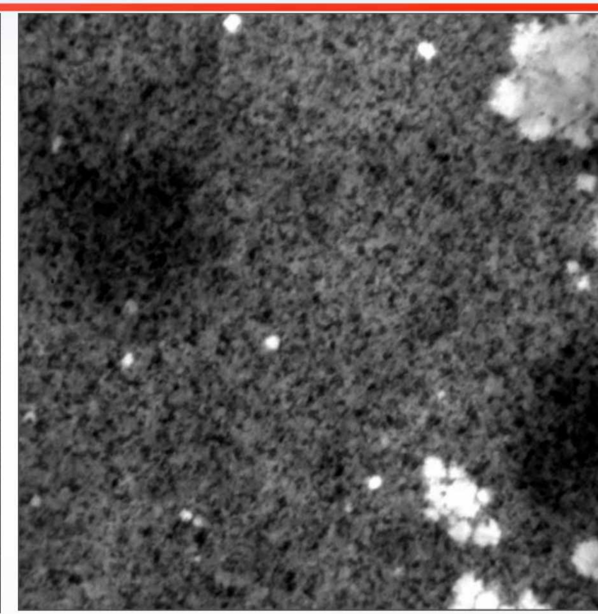
Contributors: D. Gross, J. Kacher, I.M. Robertson & Protochips, Inc.

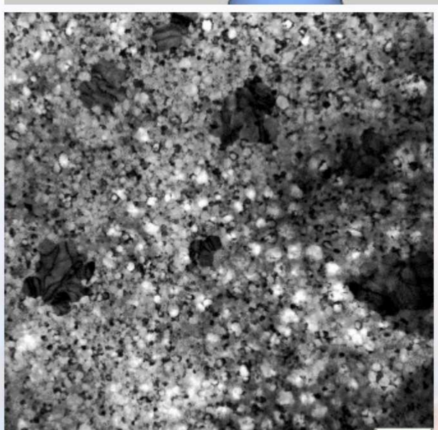
Microfluidic Stage

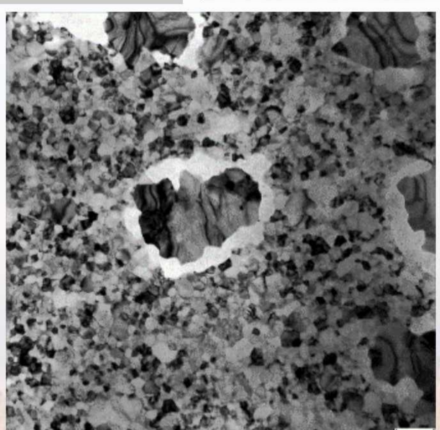
- Mixing of two or more channels
- Continuous observation of the reaction chann

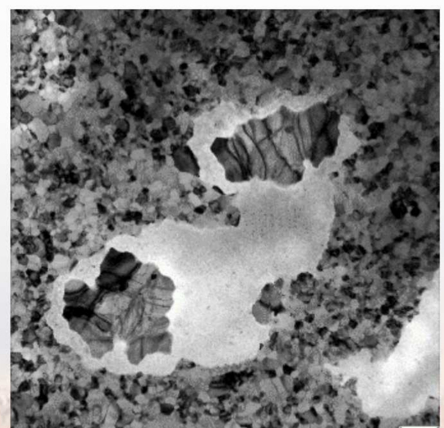












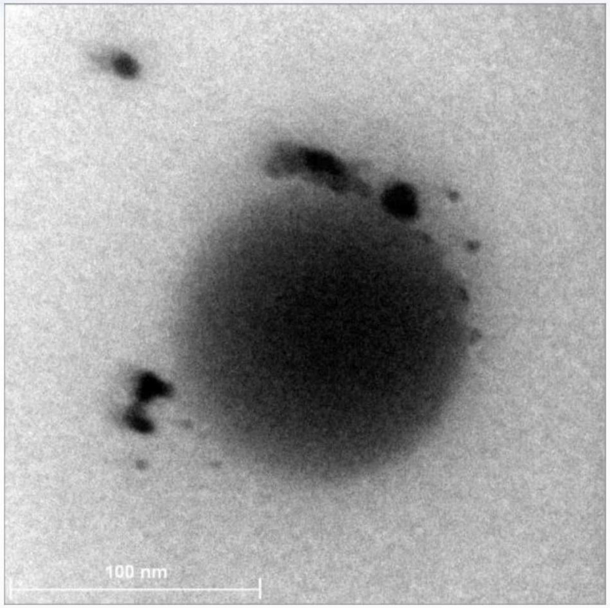
Pitting mechanisms during dilute flow of acetic acid over 99.95% nc-PLD Fe involves many grains.
Large grains resulting from annealing appear more corrosion tolerant

Other Fun Uses of Microfluidic Cell

Protocell Drug Delivery

S. Hoppe,
E. Carnes,
J. Brinker

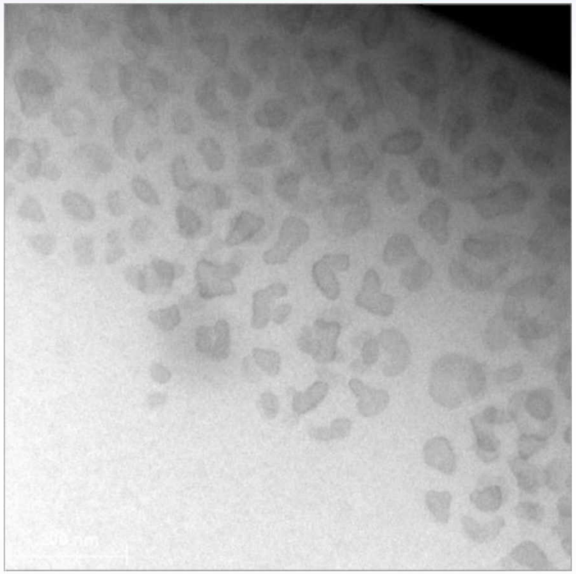
Liposome encapsulated Silica destroyed by the electron beam



BSA Crystallization

S. Hoppe

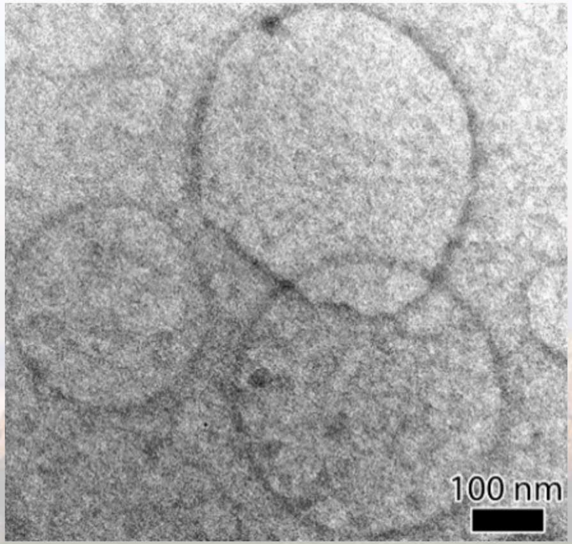
Crystallization of excess Bovine Serum Albumen during flow



Liposomes in Water

S. Hoppe,
D. Sasaki

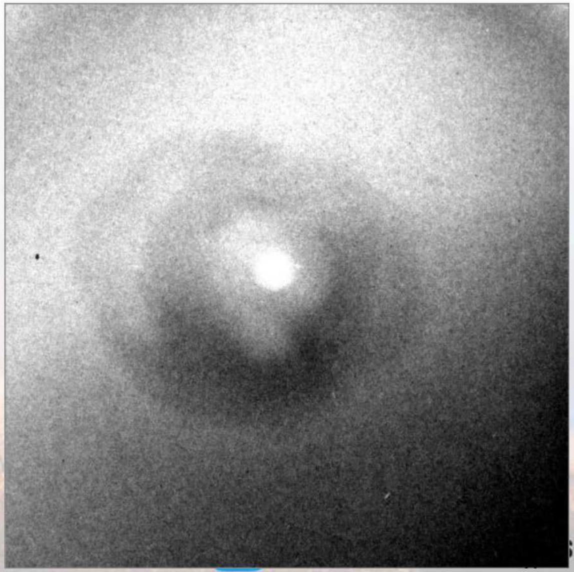
Liposomes imaged in flowing aqueous channel



La Structure Formation

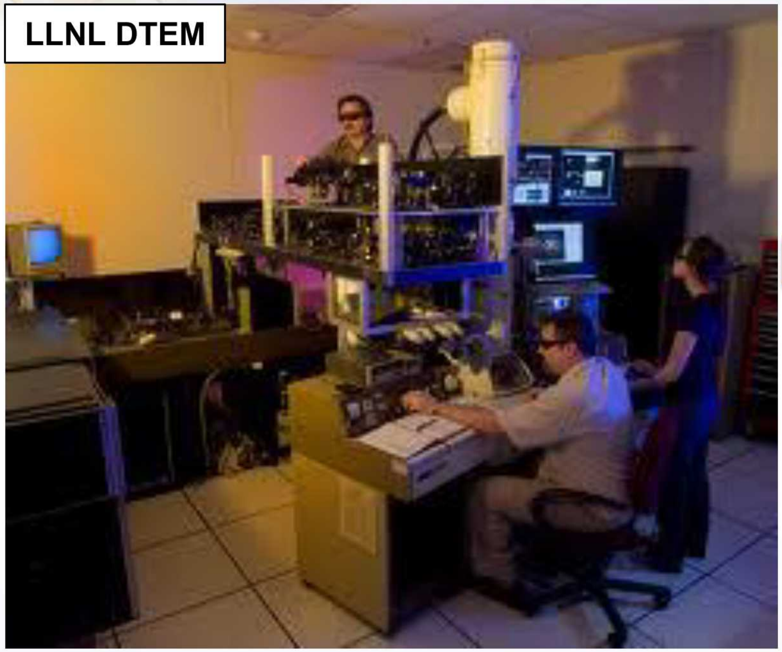
S. Hoppe,
T. Nenoff

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

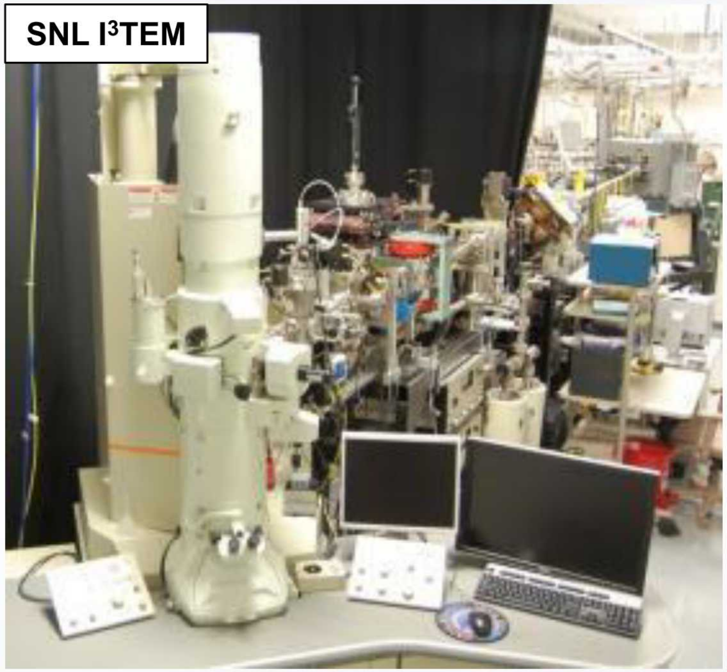


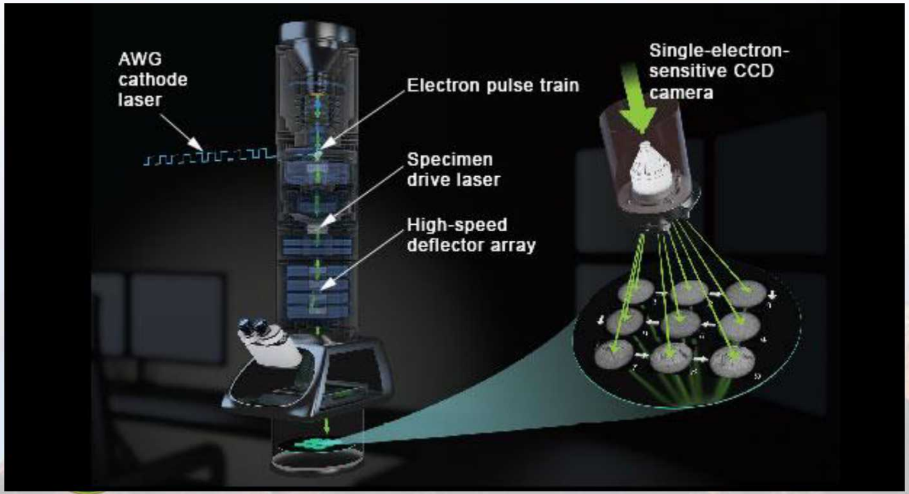
Can I³TEM and DTEM systems be combined?

LLNL DTEM



SNL I³TEM

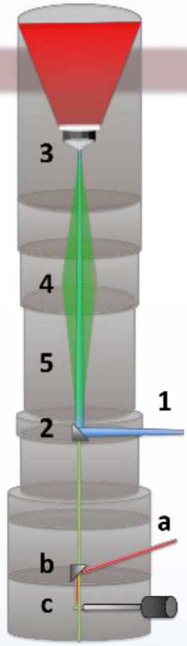


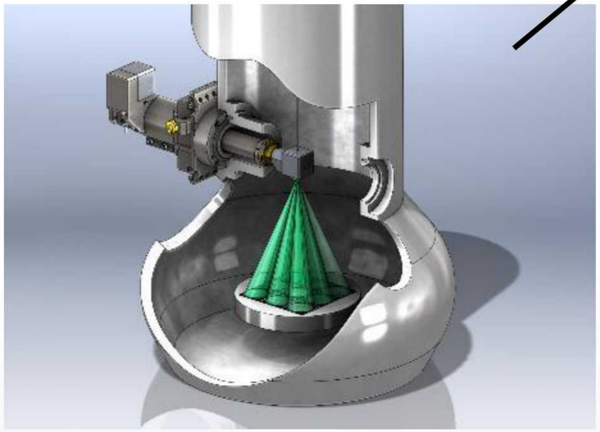


Goal:

Combine the state-of-the-art in microscopy of DTEM and I³TEM to elucidate the response of extreme overlapping environments with adequate spatial and temporal resolution.

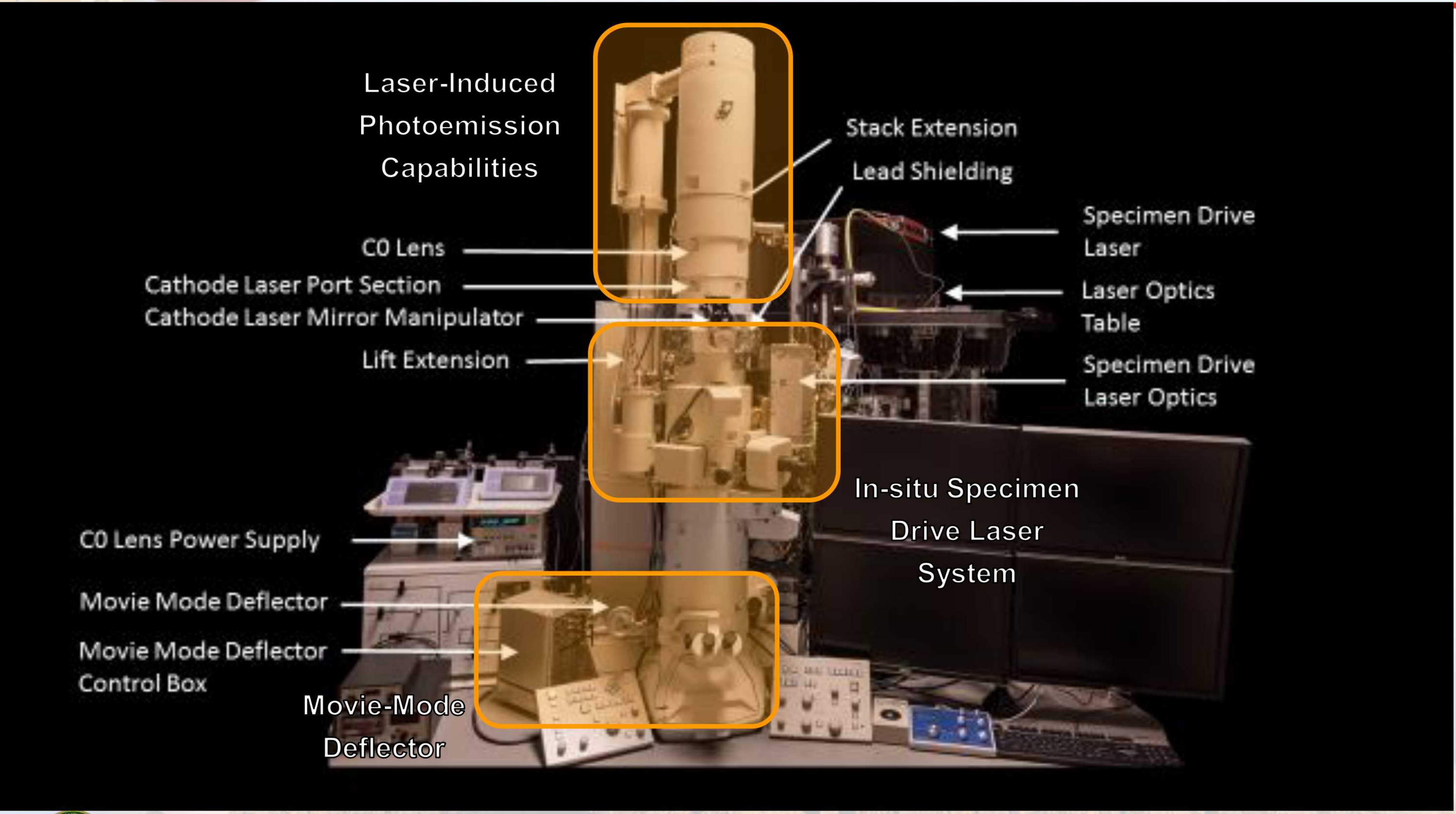
Increasing Temporal Resolution



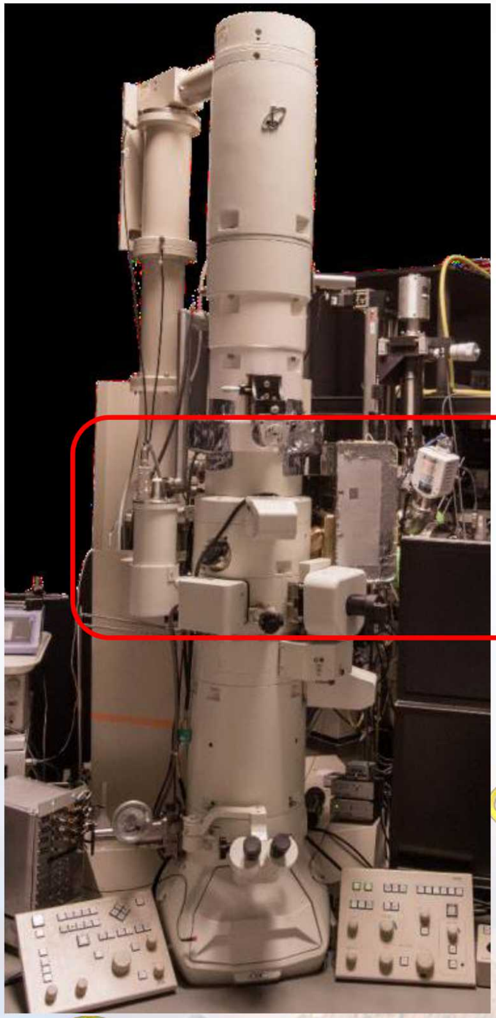


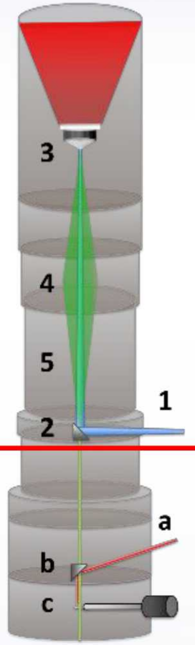
- **DTEM**
  - Laser induced photoemission of electrons is needed to achieve sufficient current density to produce an image
  - Provides nanosecond imaging of irreversible process
- **Deflector System**
  - Multiple images acquired on single frame
  - Microsecond imaging possible
  - Virtually no missing data (nanosecond gaps)
- **Standard 1K TVIPS camera**
  - Due to camera read out rate few images can be acquired
  - 10-20fps maximum
  - Missing data during camera readout

Current Status of Laser Addition and DTEM Conversion



In-situ Specimen Drive Laser System





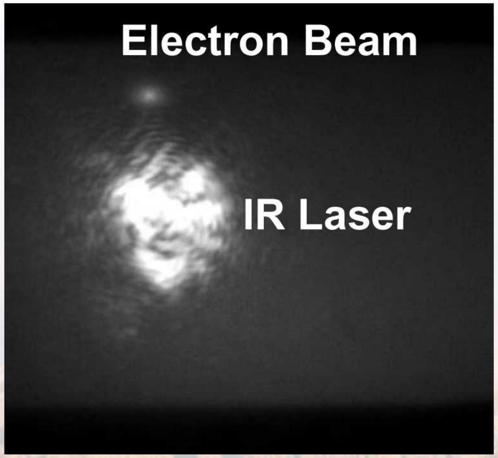
Specimen Drive Laser

a. **Adjustable power 1064 nm infrared specimen (IR) drive laser**

b. **IR laser is reflected directly onto the specimen with metal mirror**

c. **Heat specimens in *in situ* holders, which otherwise would not be possible**

- **Laser capabilities:**
  - **2-20 Watts**
  - **Pulsed or continuous operation**
  - **50 μm diameter spot size**
  - **Positioning mirror, which can be used during laser operation**



Laser Alignment TEM Holder

- **Phosphor screen**
- **Borescope**
- **CCD camera**
- **Precise alignment of the laser to the electron beam**

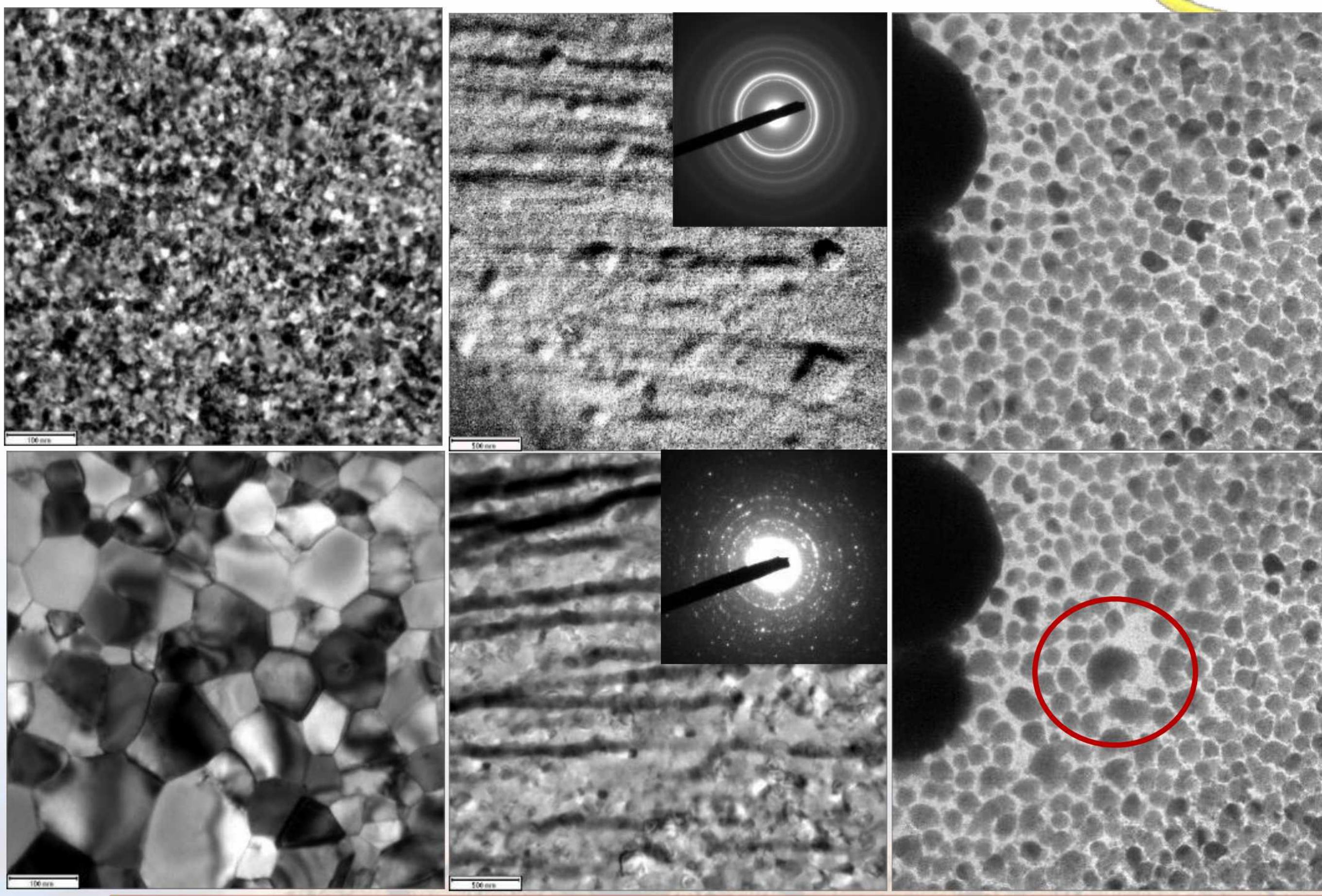
Initial Laser Heating Observations

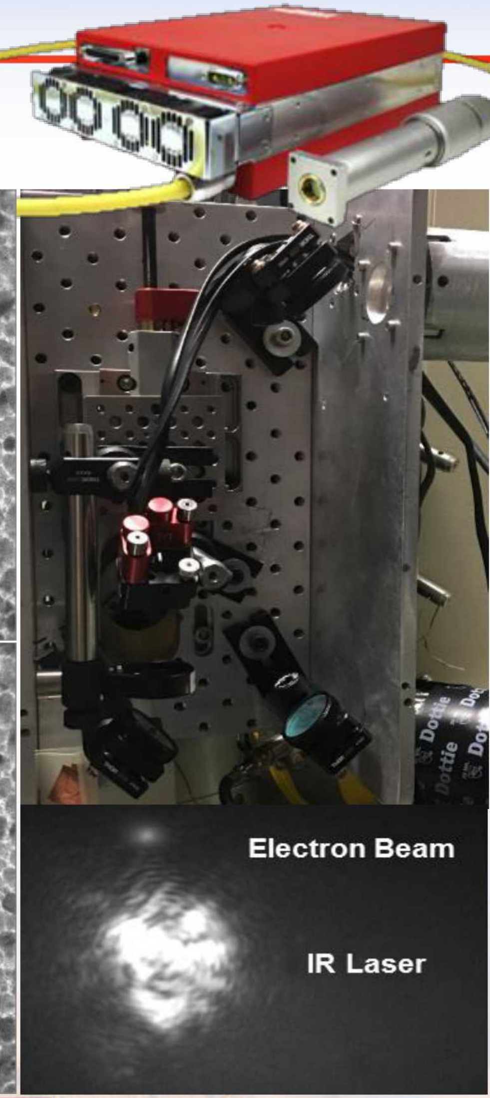
Collaborator: P. Price, C.M. Barr, D. Adams, M. Abere

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

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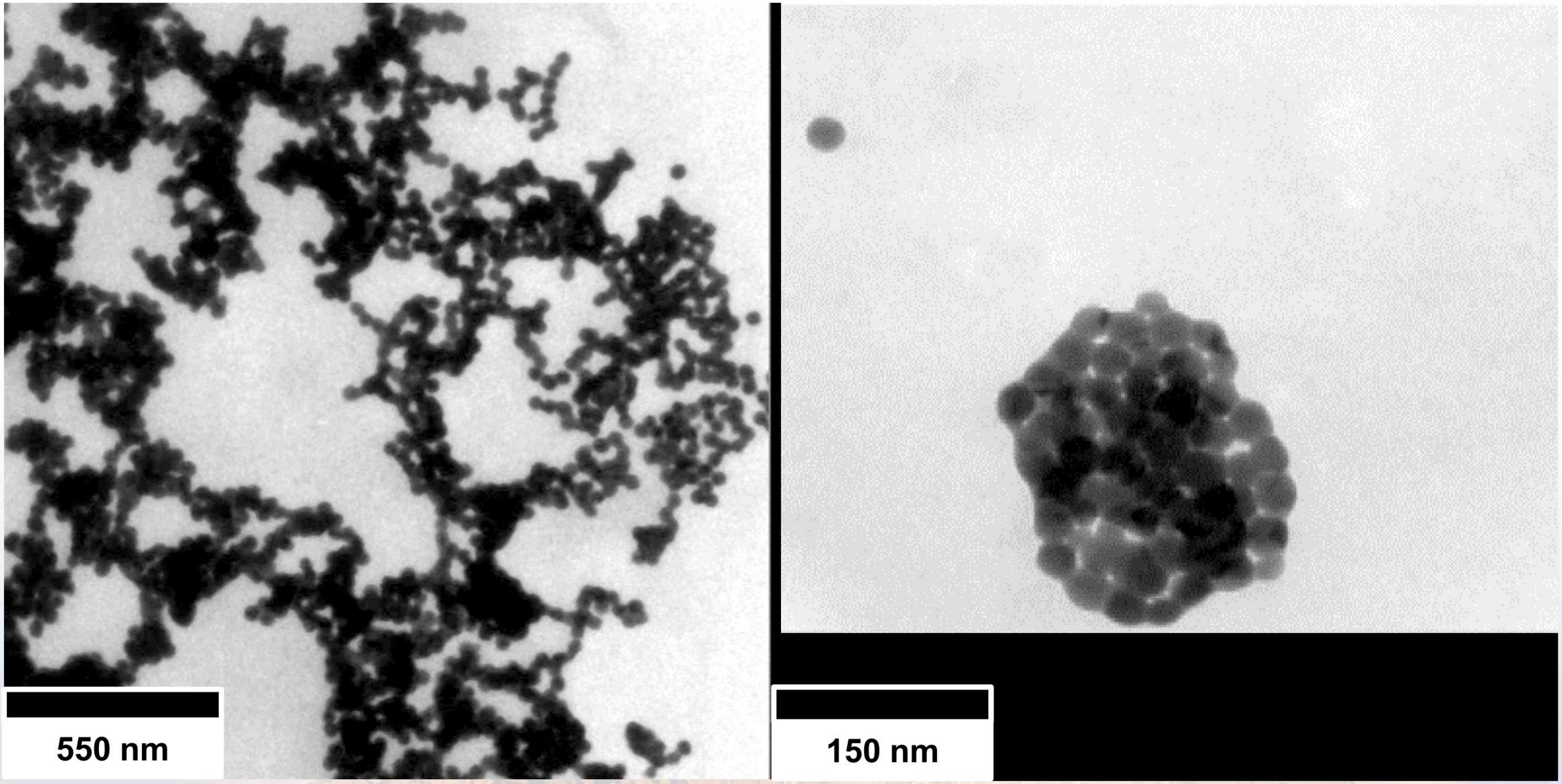
Complex Interaction Au NPs Exposed to Laser Irradiation

Contributors: P. Price, L. Treadwell, A. Cook



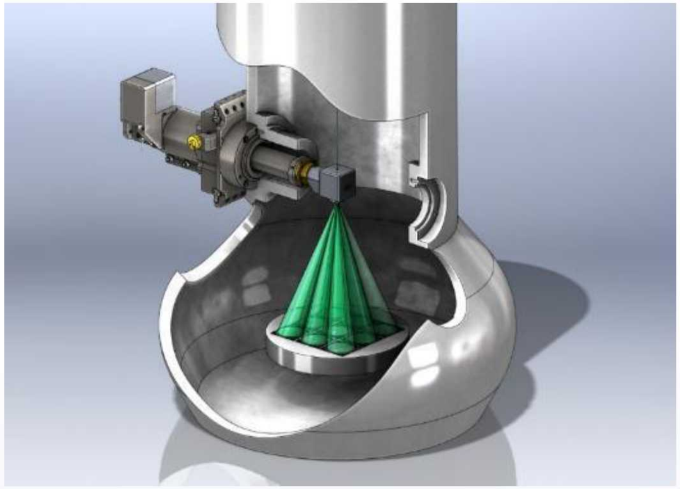
Speed = 2.5x

A Complex Combination of Sintering, Reactions, and Ablation Occurs

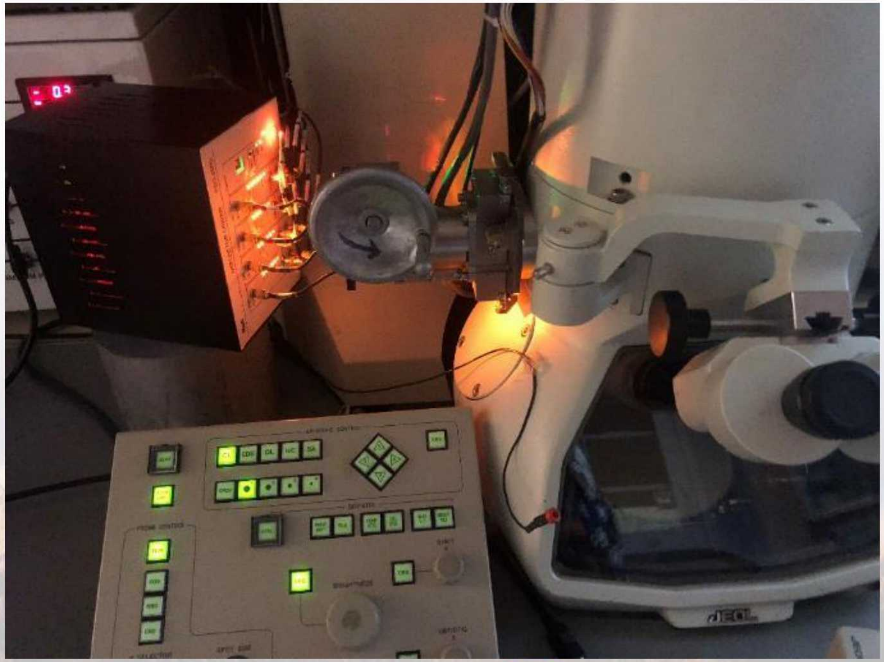
μs Resolution with a Standard Camera

Collaborator: P. Price, A. Monterrosa, D. Adams, M. Abere, & IDES Inc.

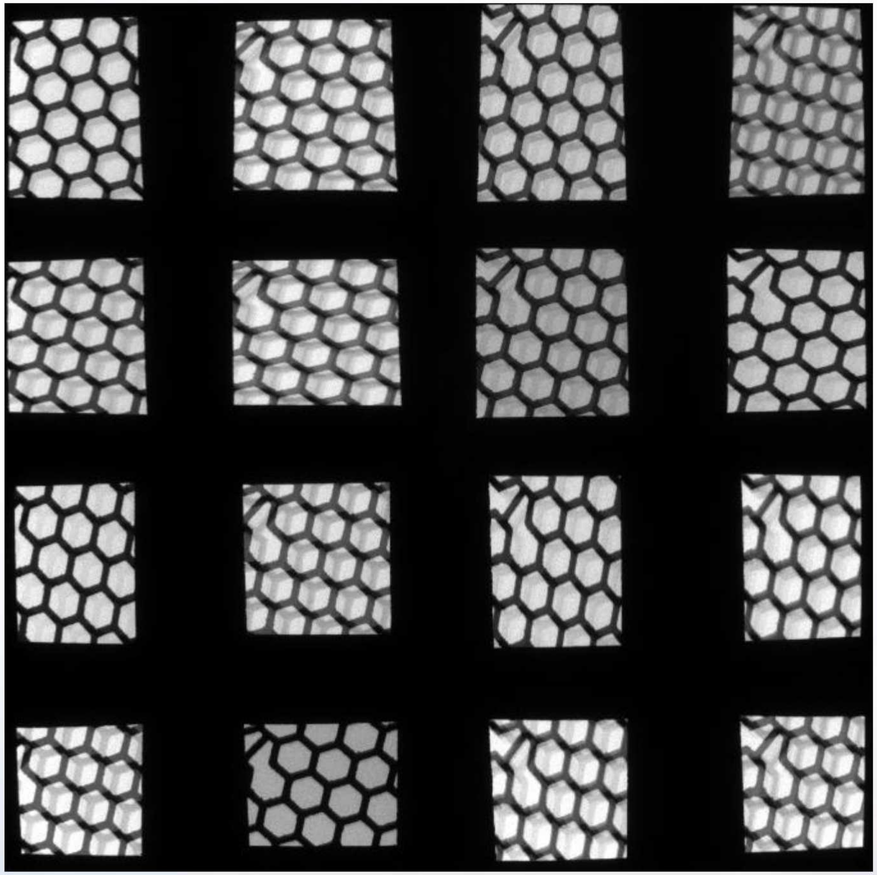
fs ps ns **μs** **ms** s



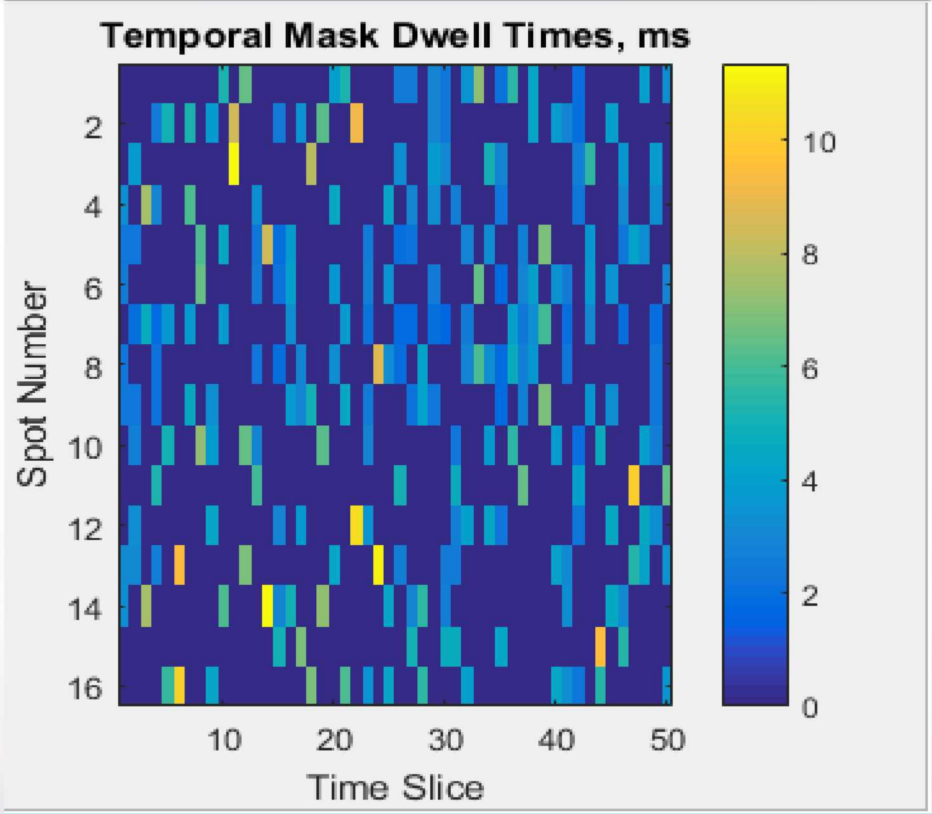
- Electrostatic deflection of electrons
- 4, 9, or 16 images per frame, spread over a large camera
- Any exposure time up to the limits of the camera
  - Ultimate limit is beam current/brightness



Movie Mode & Temporal Compressive Sensing



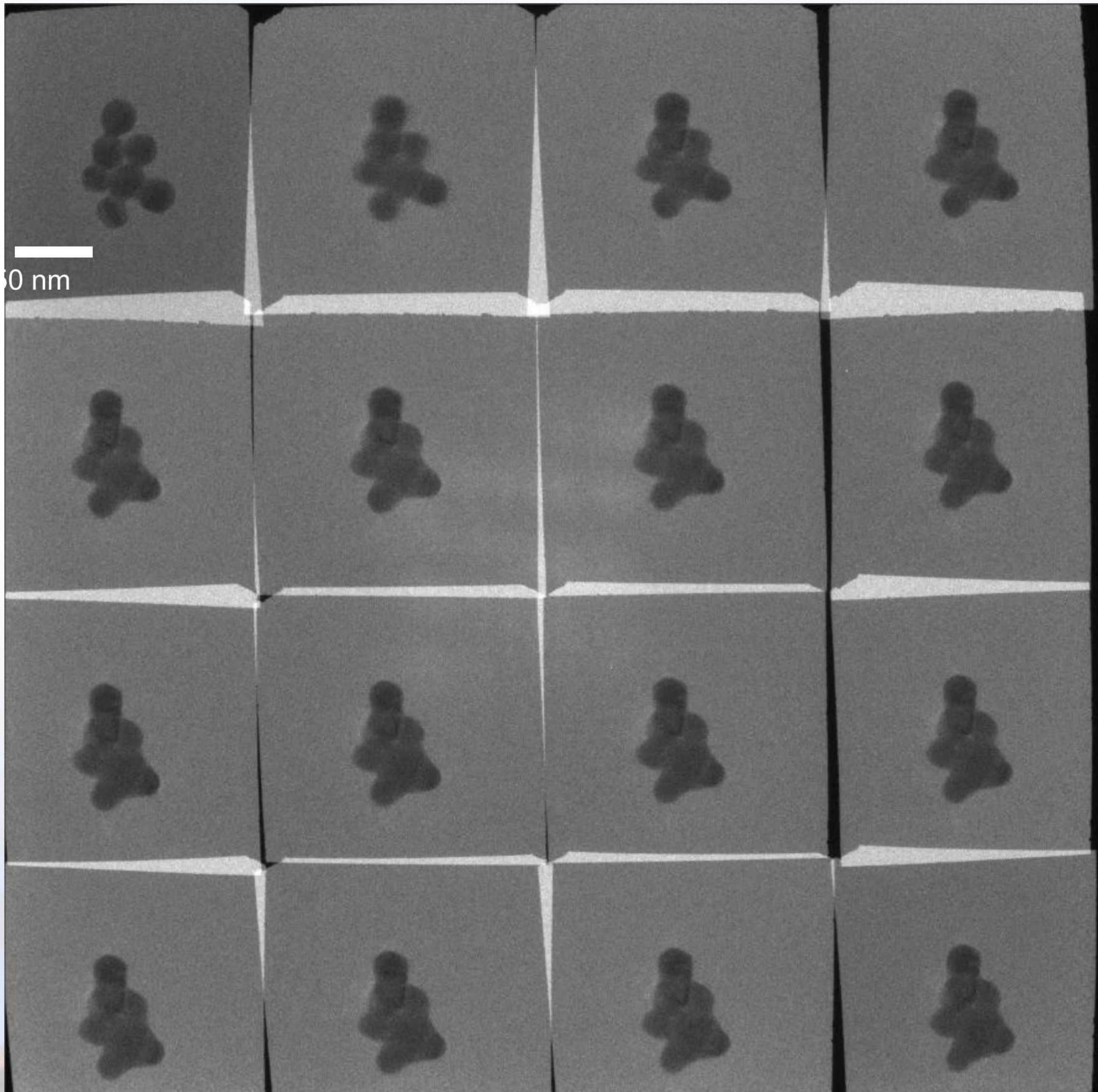
Up to 100 exposures can be acquired on a single camera frame giving *μs* temporal resolution

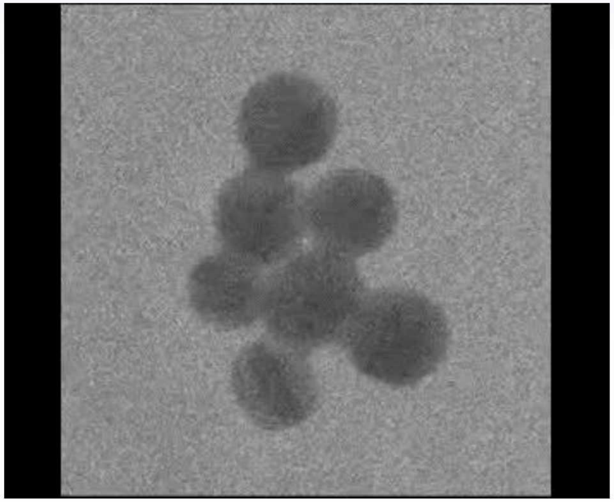


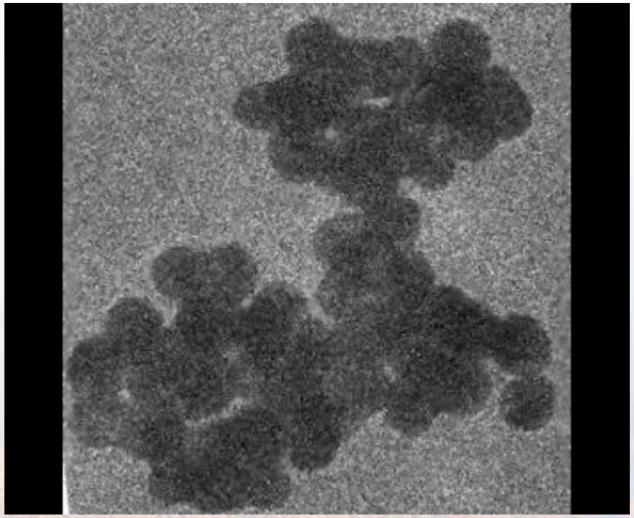
- **Electron intensity of a single exposure is randomly distrusted to multiple images within a single frame**
- **Record of random mask used to distribute exposure intensity is later used deconvolute images**

1-to-1 Frame Capture (<5 ms per frame)
Sintering of 20 nm Au Nanoparticles

Collaborator: P. Price, A. Monterrosa, & IDES Inc.



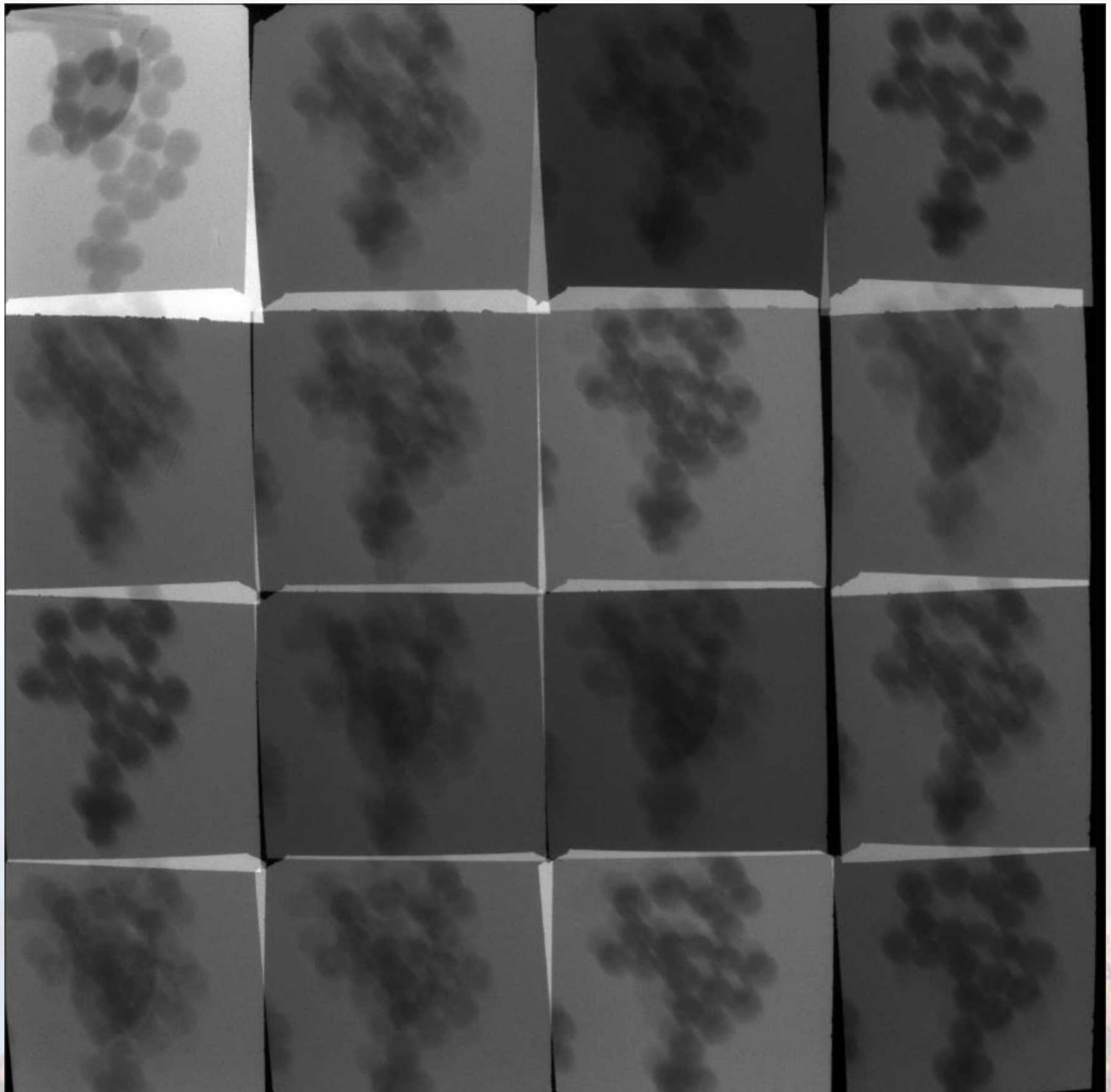




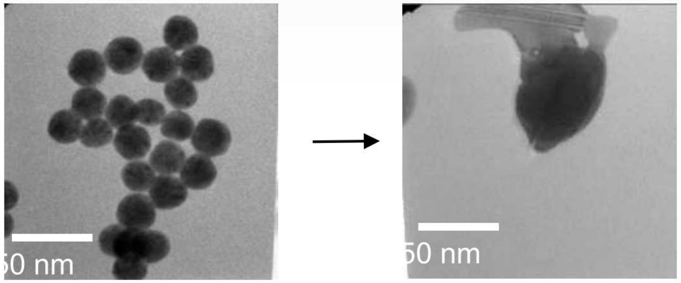
16 frames captured with <5 ms exposure per frame

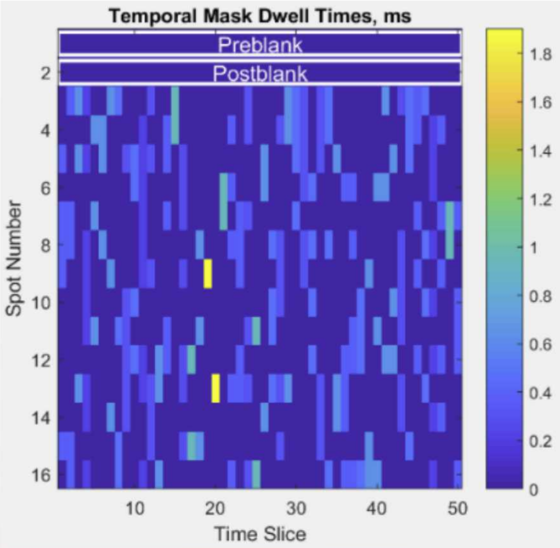
Temporal Compressive Sensing to Improve Temporal Resolution

Collaborator: P. Price, A. Monterrosa, & IDES Inc.



Before After



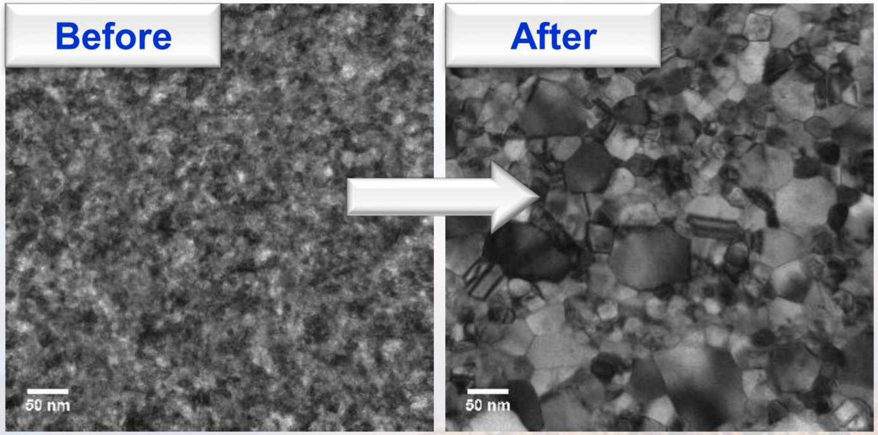


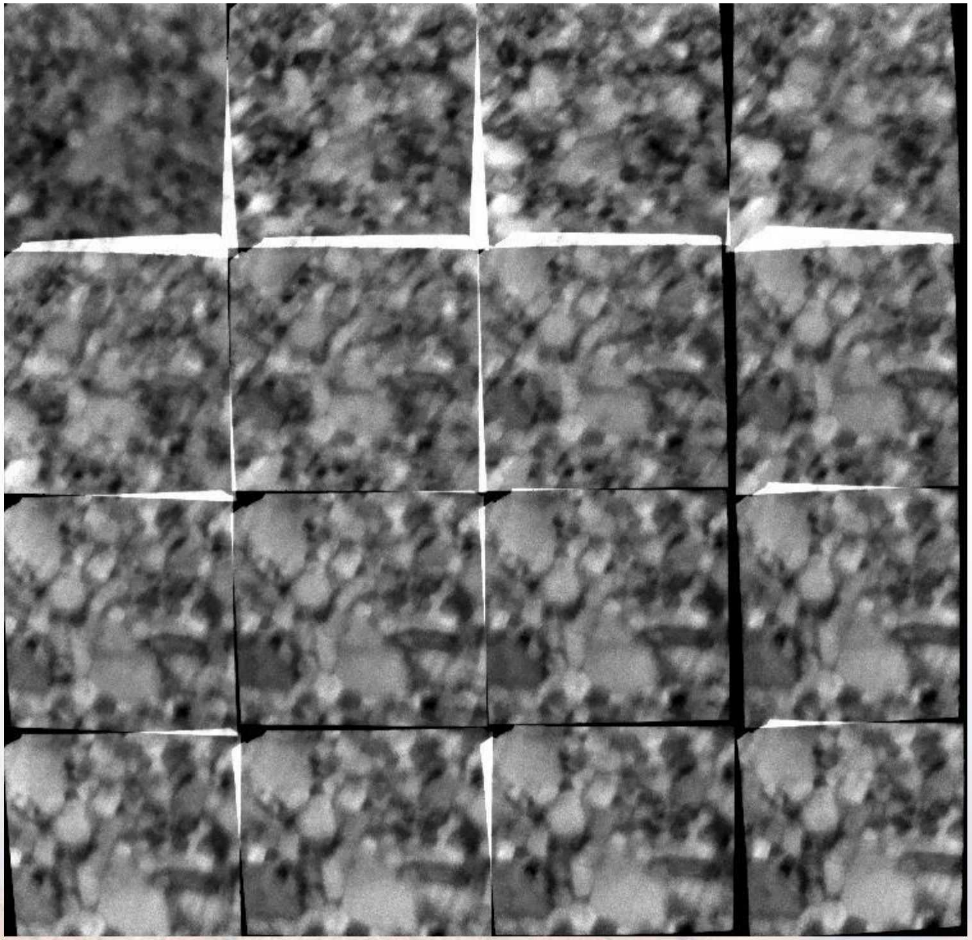
A pseudorandom exposure pattern can produce more than 16 frames within the same exposure time

Motivation: In-situ Microstructure Evolution

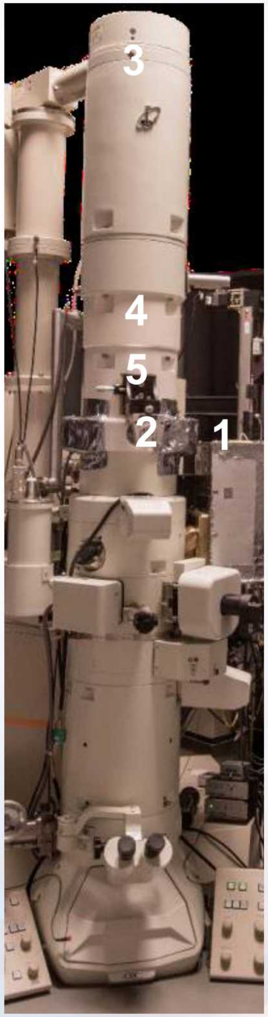
- In-situ TEM images of IR laser driven grain growth in Pt foil
- Characterize grain boundary and grain evolution
- Rapid heating & cooling controlled by specimen drive laser

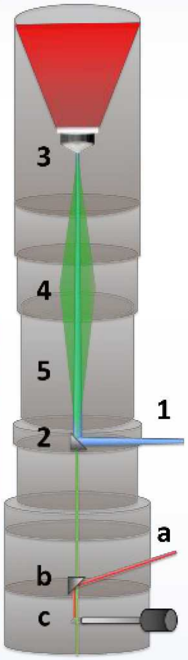
Deflector System: Evolution of Pt microstructure with ~10ms exposures shown on the right

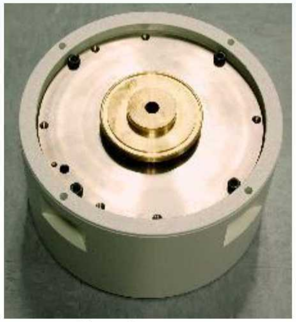




Conversion of a Standard JEOL 2100 TEM to DTEM: Increase Current Density







C_0 Lens

Drift Section

UV Laser

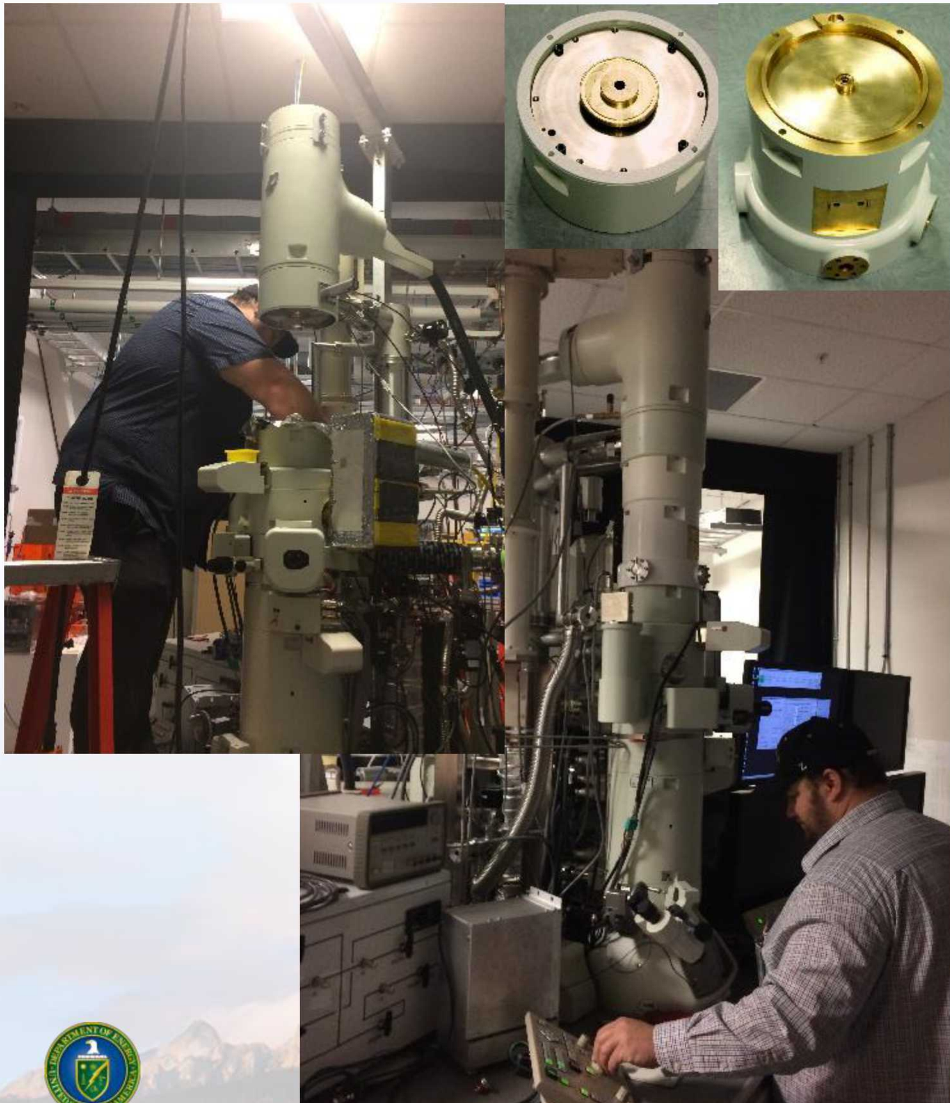
A standard LaB_6 TEM has on average 1 or 2 electrons in the column at a given time!

Laser induced photoemission of electrons can increase nanosecond electron current by 6-8 orders of magnitude

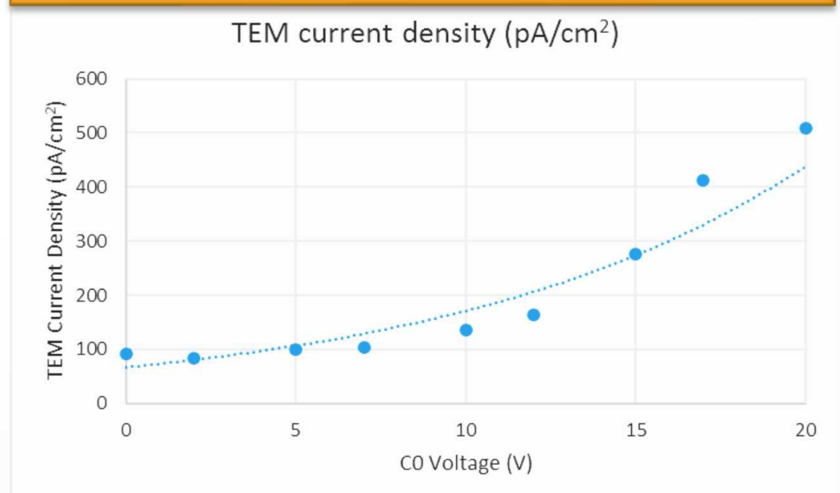
DTEM Development

1. Ultraviolet laser and optics system capable of producing nanosecond pulses
2. Adjustable molybdenum mirror assembly to reflect the UV laser up the column
3. Tantalum cathode disc filament
4. Addition of a C_0 lens and power supply to gather electrons increasing current to the specimen
5. Addition of a drift section to condense electrons from the C_0 lens
6. Lead shielding as needed to ensure safe operation of the instrument

Increased Current Density & added 18” to TEM Column



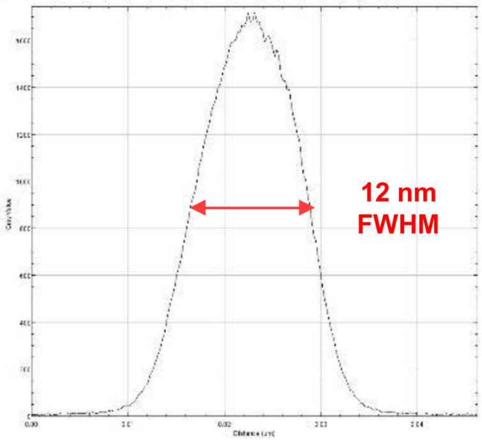
Addition of C_0 lens and drift section give a 5X increase in current density

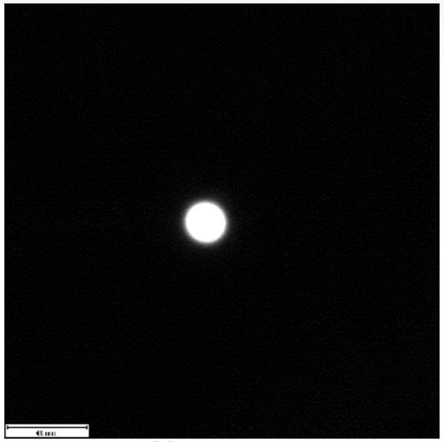


- **Disc cathode produces wider distribution of electrons than LaB_6**
- **Current density can be increased by adding C_0 lens to condense electrons to a smaller probe**
- **Strength of C_0 lens is controlled by external adjustable power supply**
  - Trade off between current density and resolution
- **Drift section gives more time for electrons to condense after the C_0 lens. C_0 can be weaker**

Tantalum Filament Exchange

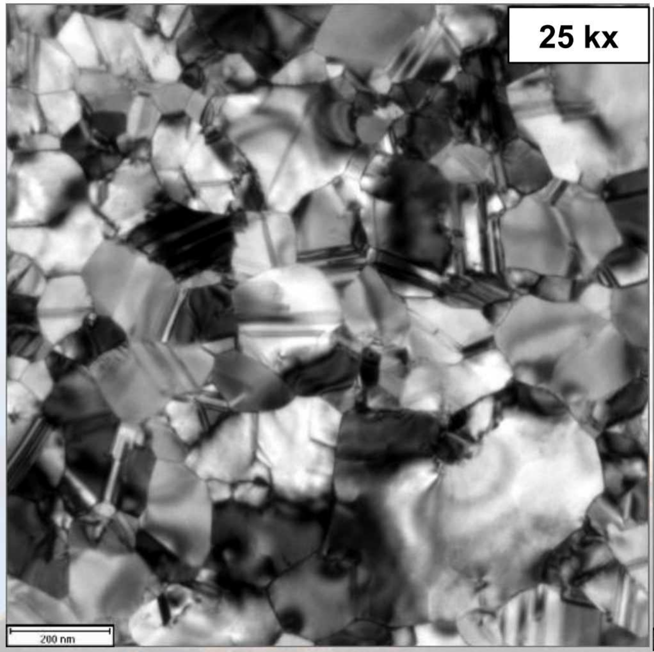
- **Changes with C_0 lens setting of 0V**
  - 50% reduction in electron
    - LaB_6 ~ 50 pA/cm^2
    - Ta ~ 30 pA/cm^2
  - Higher beam current at from source
    - LaB_6 ~ 7 μA
    - Ta ~ 23 μA
  - Increase in minimum spot size for precession
    - LaB_6 ~ 8 nm FWHM
    - Ta ~ 12 nm FWHM

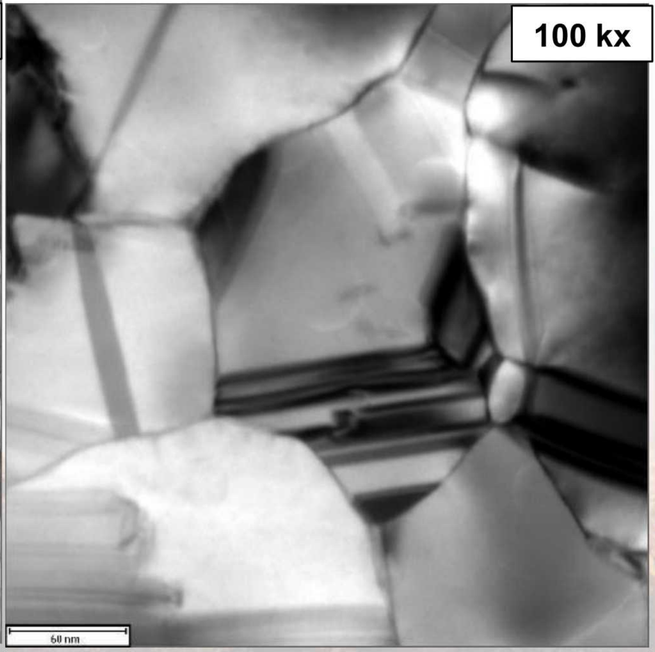




Above:

Smallest achievable spot size for precession with Ta cathode





Left:

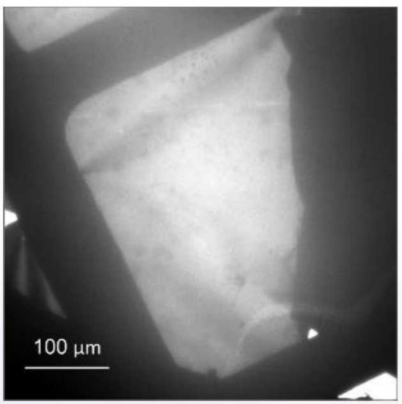
Bright field TEM images of nanocrystalline gold taken with Ta filament

Easily switch between DTEM and thermal emission with good resolution in minutes!

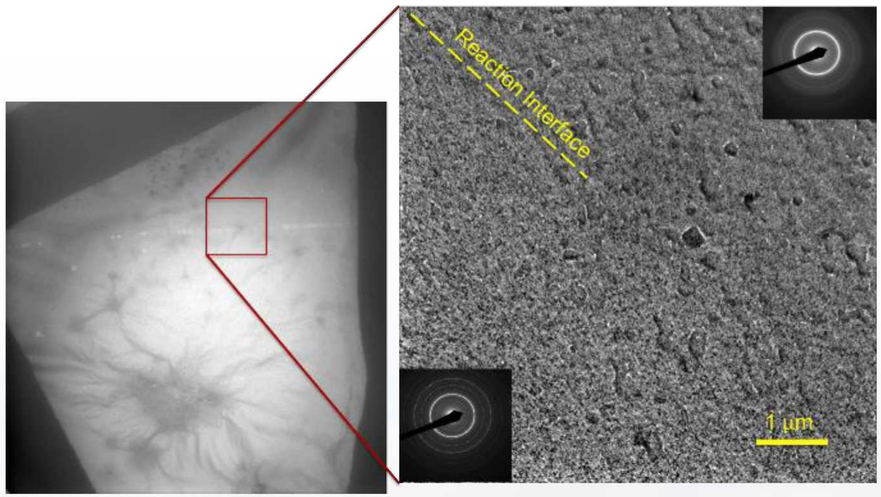
Filament exchange in as little as 90 min.

DTEM of Reactive Multilayers

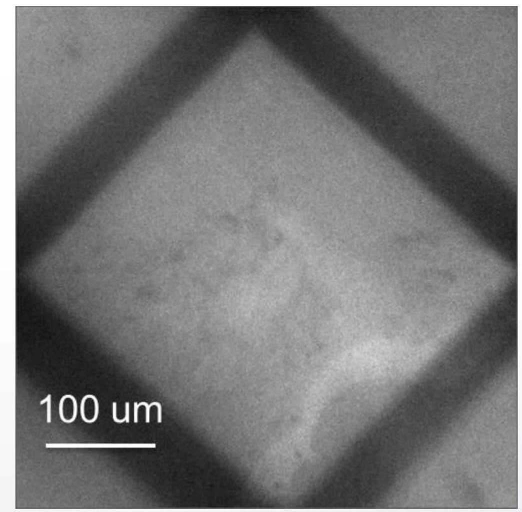
Reactive multilayers provide an excellent example of the capabilities of DTEM. Al/Co multilayer samples are ignited by the IR laser, and convert to a CoAl phase. The reaction front propagates across the sample at ~10 m/s. With a fast enough time resolution this wavefront can be captured.



Al/Co multilayer sample **before IR laser shot**



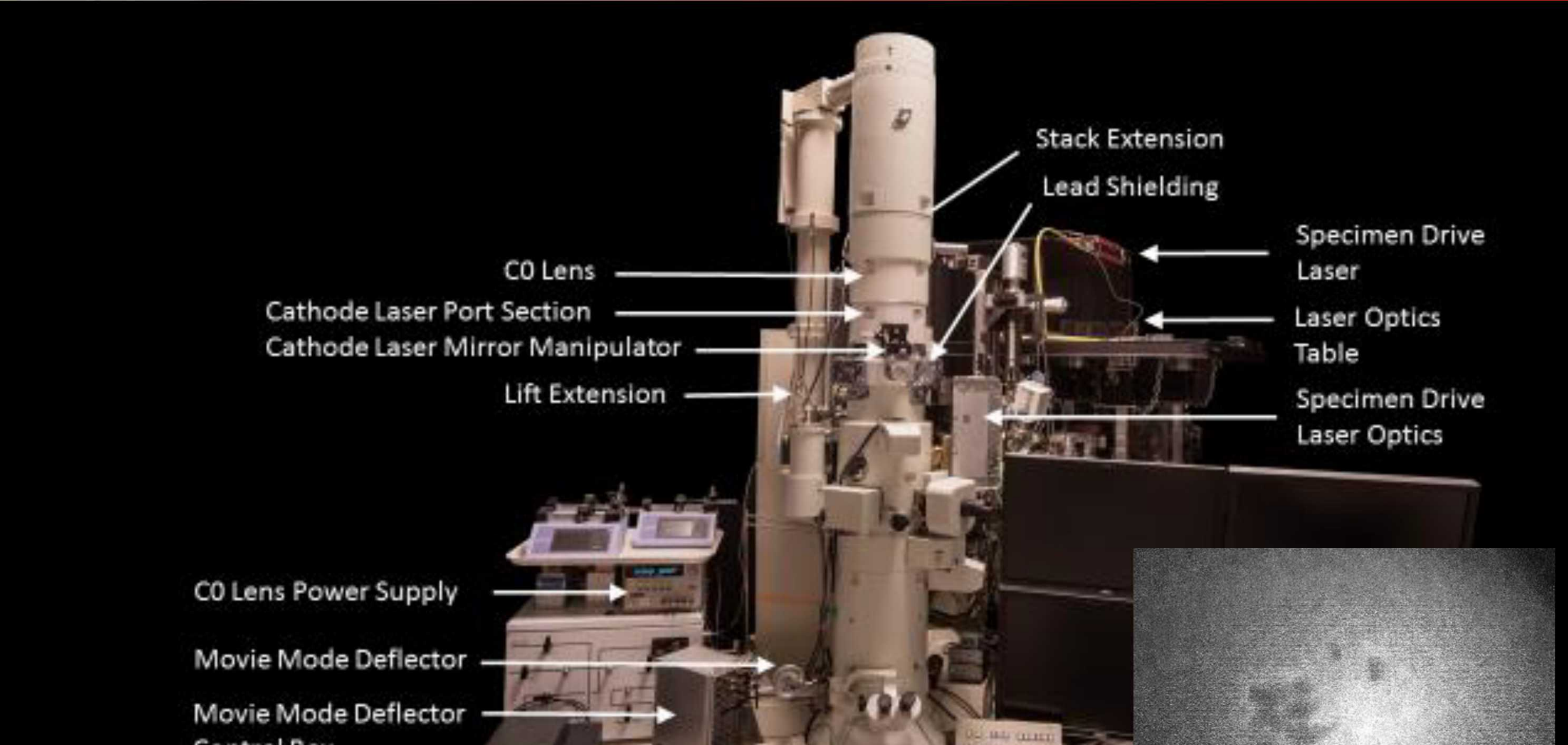
Al/Co multilayer sample **after IR laser shot** and closeup of reaction interface

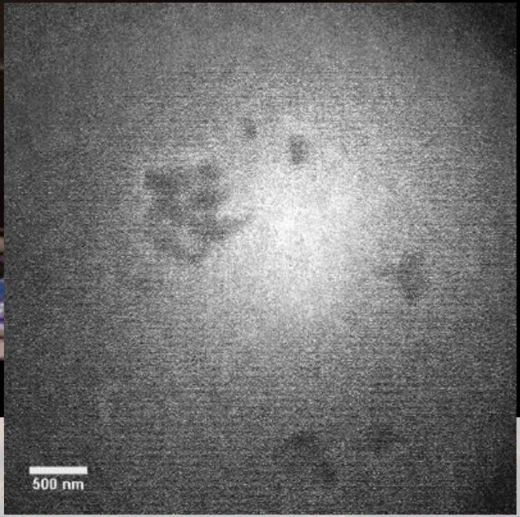


Single shot DTEM of unreacted Al/Co multilayer sample

Current Status of DTEM Conversion

Collaborator: P. Price, A. Monterrosa, C.M. Barr, D. Adams, M. Abere, & IDES Inc.

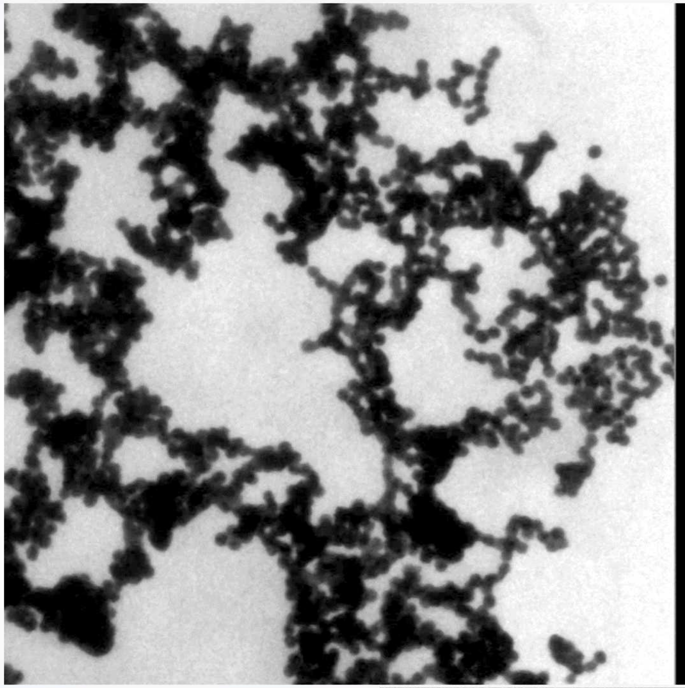


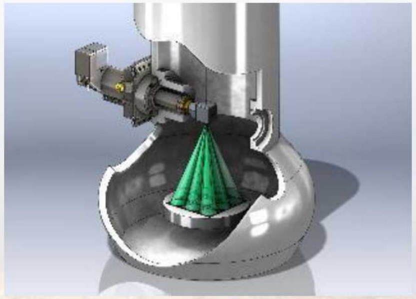


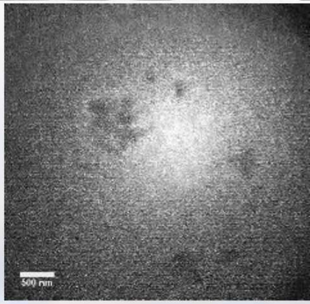
- 266 nm UV laser induced photoemission has been achieved!
- 6 ns single-shot DTEM image of P47

Unconventional *In situ* Microscopy Creates a wealth of Possibilities

- Fundamental TEM modifications for DTEM, movie mode, and compressive sensing have progressed.
- Maintained functionality of the TEM using LaB_6 thermal emission with minimal impact
- Possible to switch between LaB6 and DTEM in just a few hours
- Compressive sensing permits microsecond temporal resolution
- Laser induced photoemission of electrons has been achieved with in single shot 6 ns temporal resolution
- Advanced temporal resolution on the I^3TEM combined with the wide range of available in-situ capacities provide a wealth of experimental control







<u>Collaborators:</u>

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Sandia's USER Capabilities

D. Hanson, W. Martin, M. Wasiolek

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- Spring and Fall proposals for 18 months
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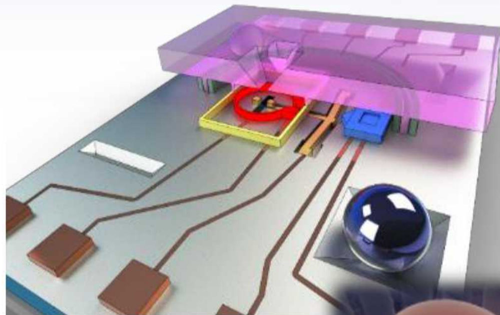

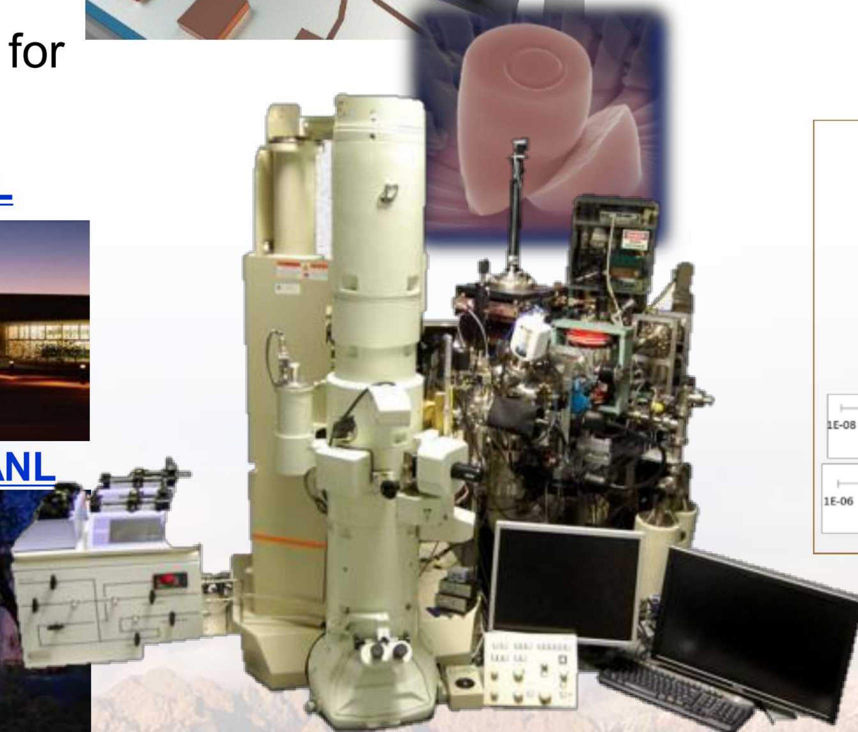

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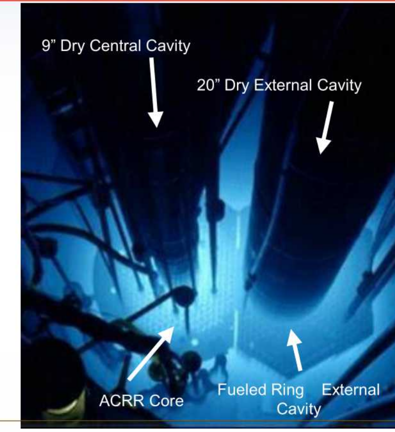
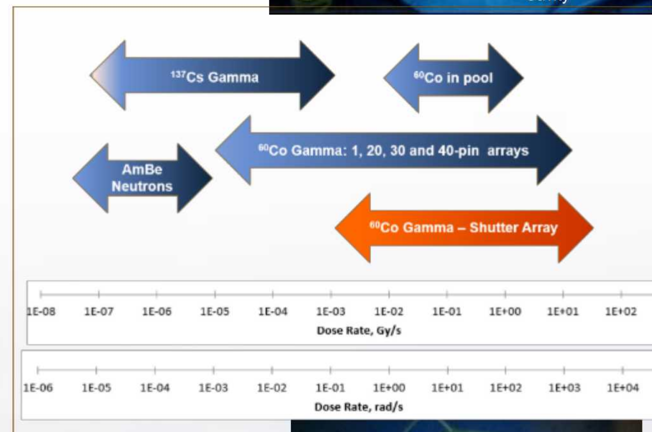
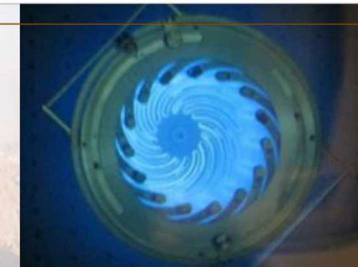
- Three proposal a year for 9 months





Core Facility - SNL

Gateway Facility - LANL



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