Development of and Initial Results from the First In-Situ Ion Irradiation Dynamic TEM

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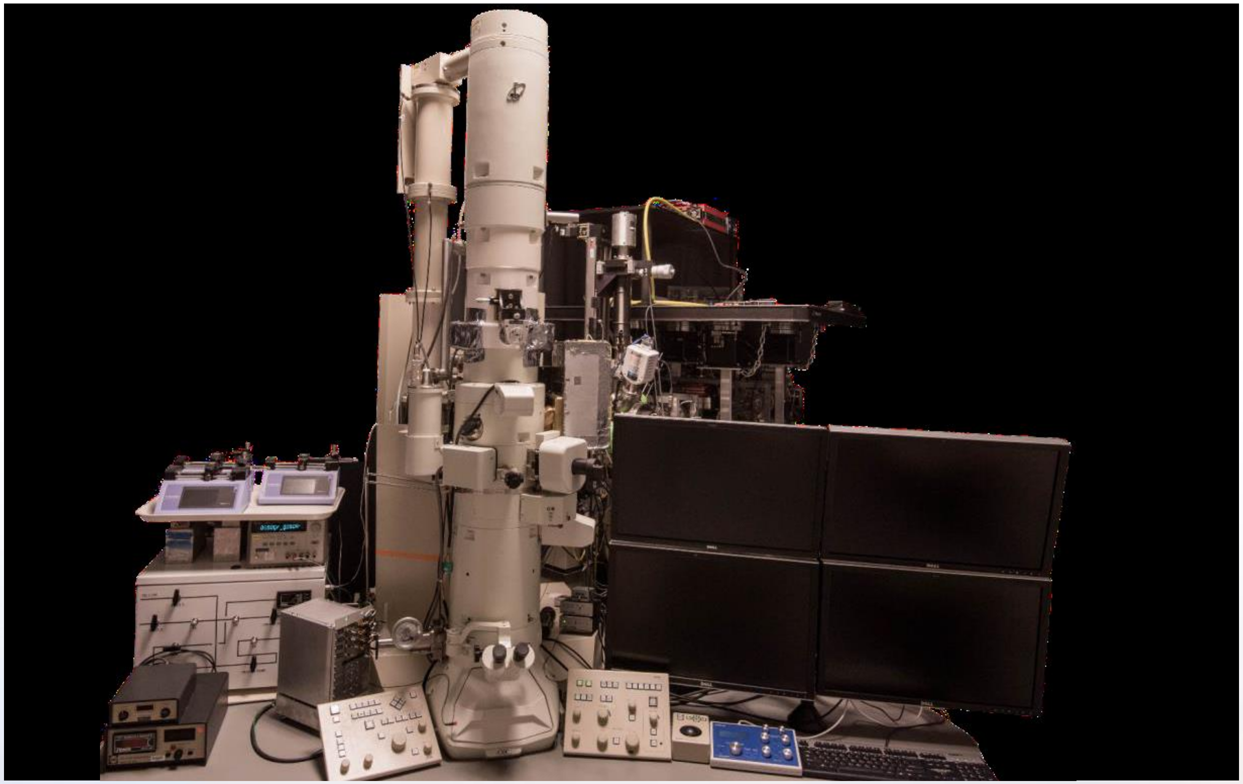
December 1, 2017



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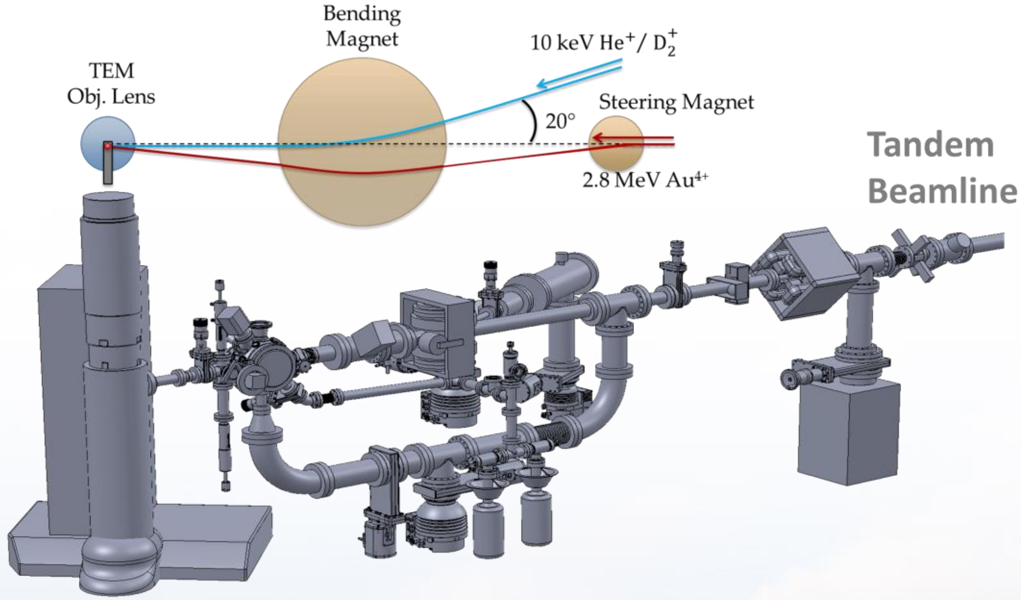
IDES INTEGRATED DYNAMIC ELECTRON SOLUTIONS

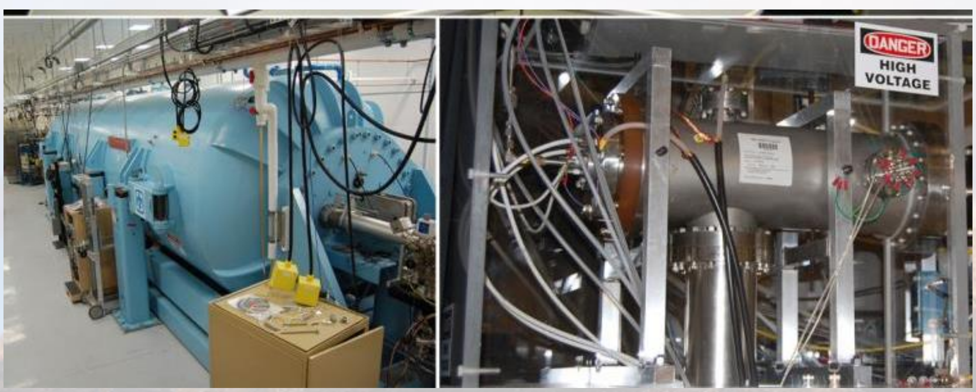
Sandia National Laboratories

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

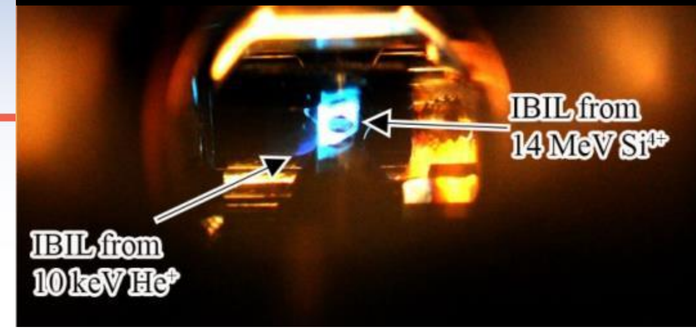
Collaborator: D.L. Buller

10 kV Colutron - 200 kV TEM - 6 MV Tandem



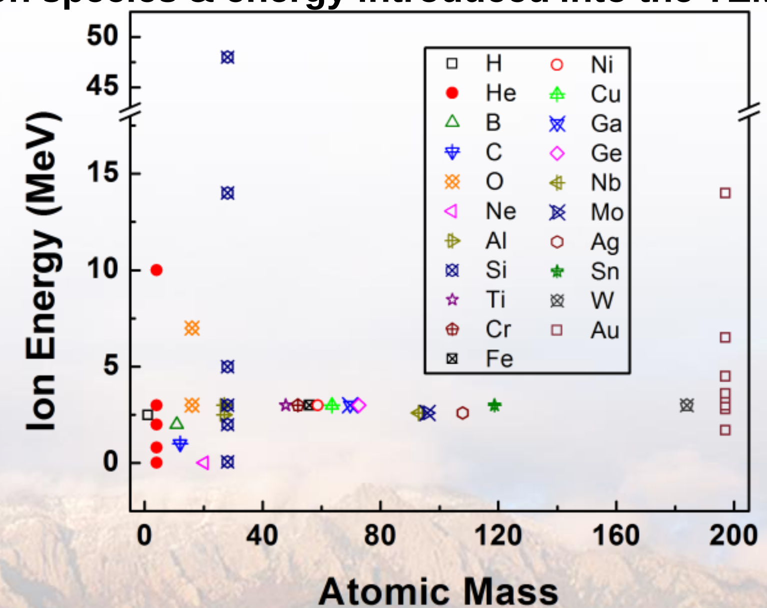


Ion beam-induced luminescence (IBIL) from a quartz stage inside the TEM



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

Ion species & energy introduced into the TEM



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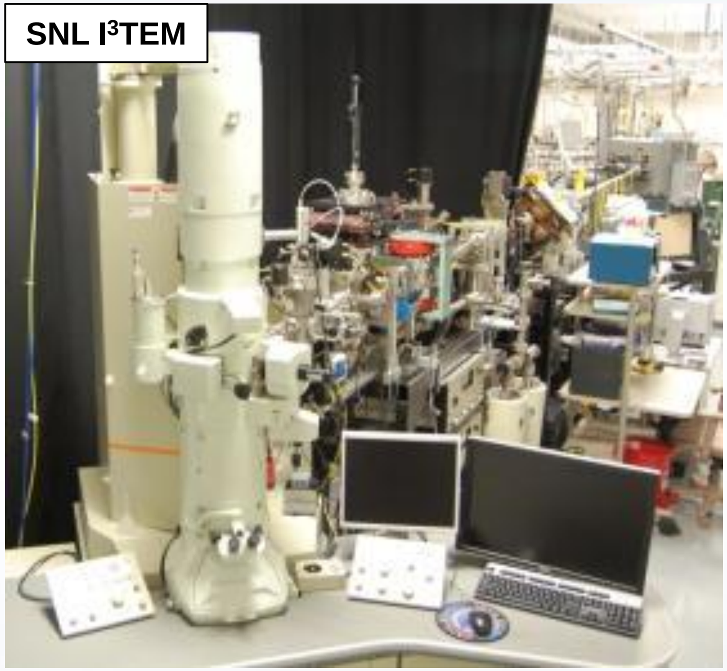
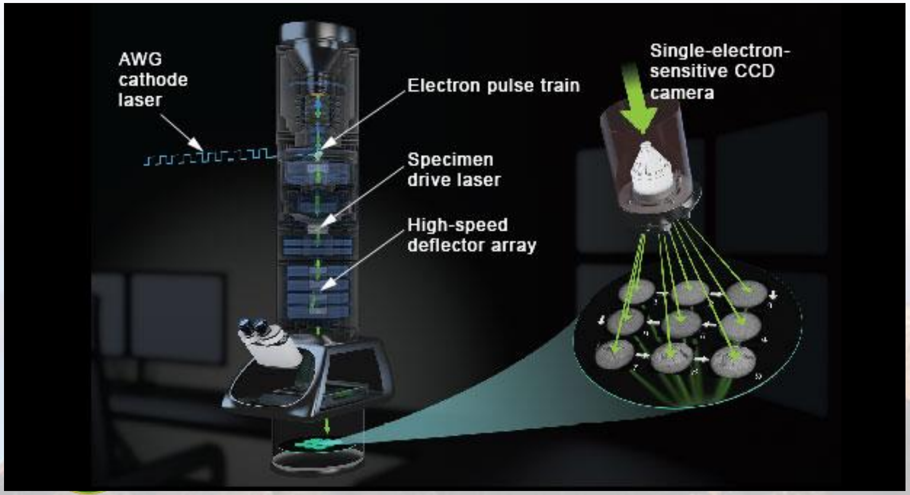
Can I^{3}TEM and DTEM systems be combined?

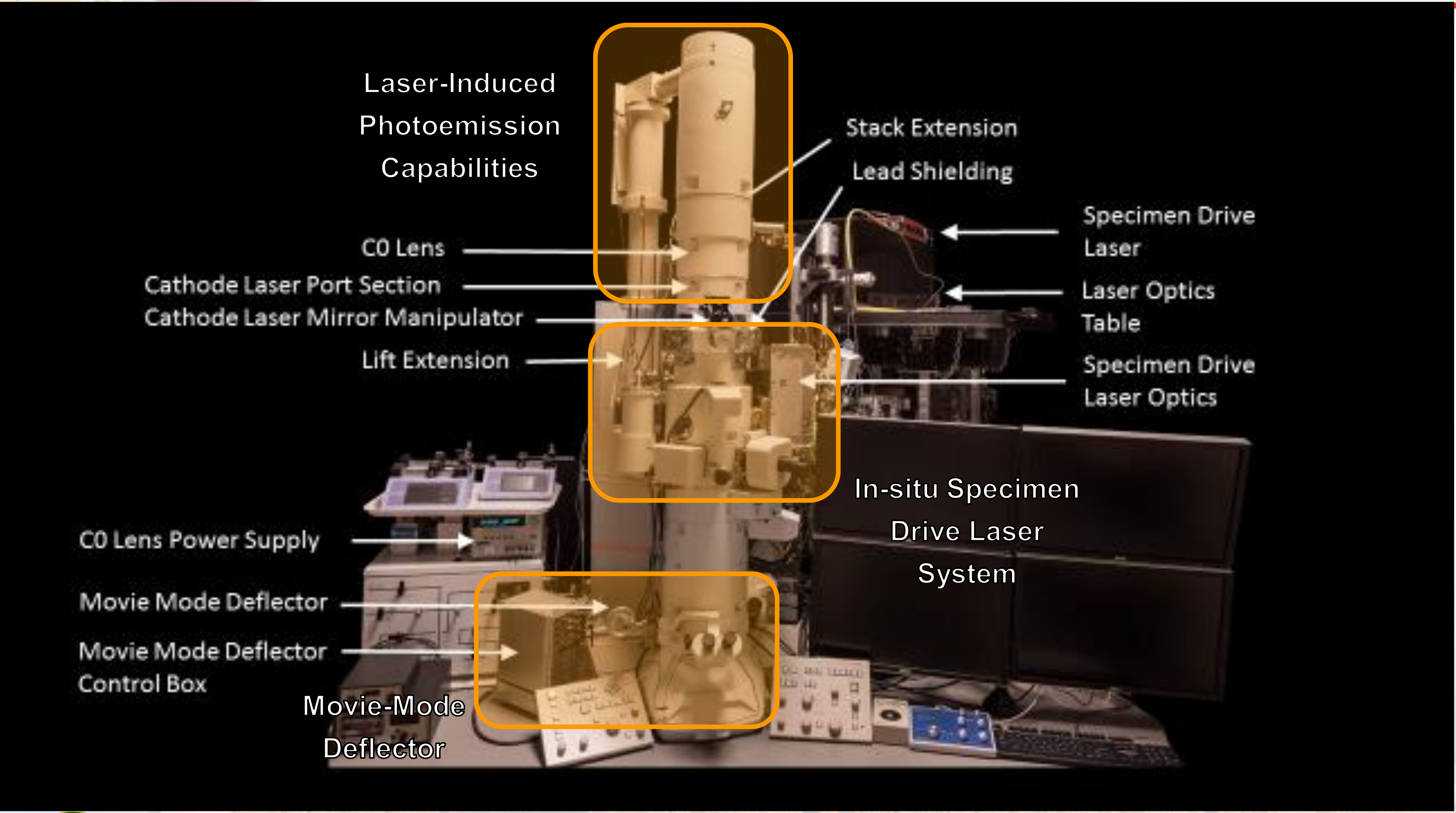




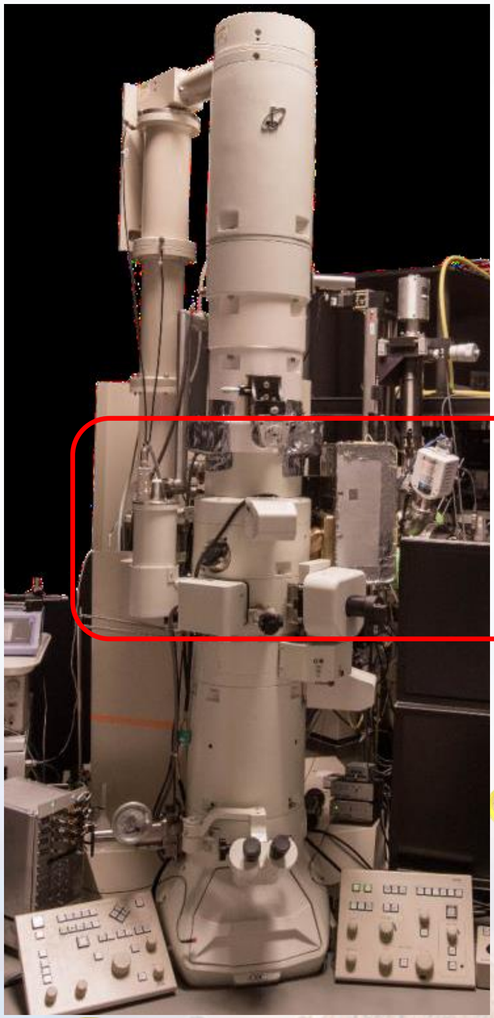
Goal:

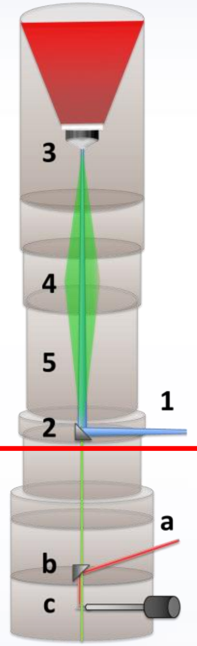
Combine the state-of-the-art in microscopy of DTEM and I^{3}TEM to elucidate the response of extreme overlapping environments with adequate spatial and temporal resolution.

Current Status of Laser Addition and DTEM Conversion



In-situ Specimen Drive Laser System





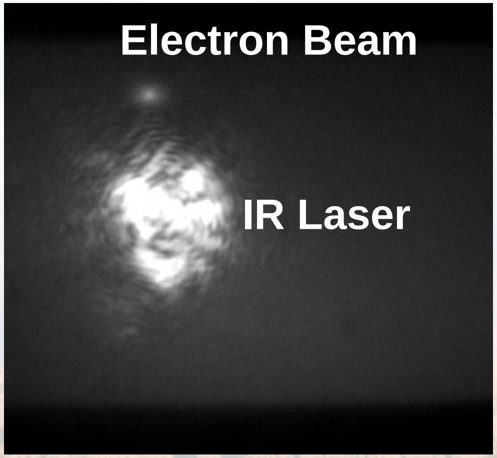
__Specimen Drive Laser__

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

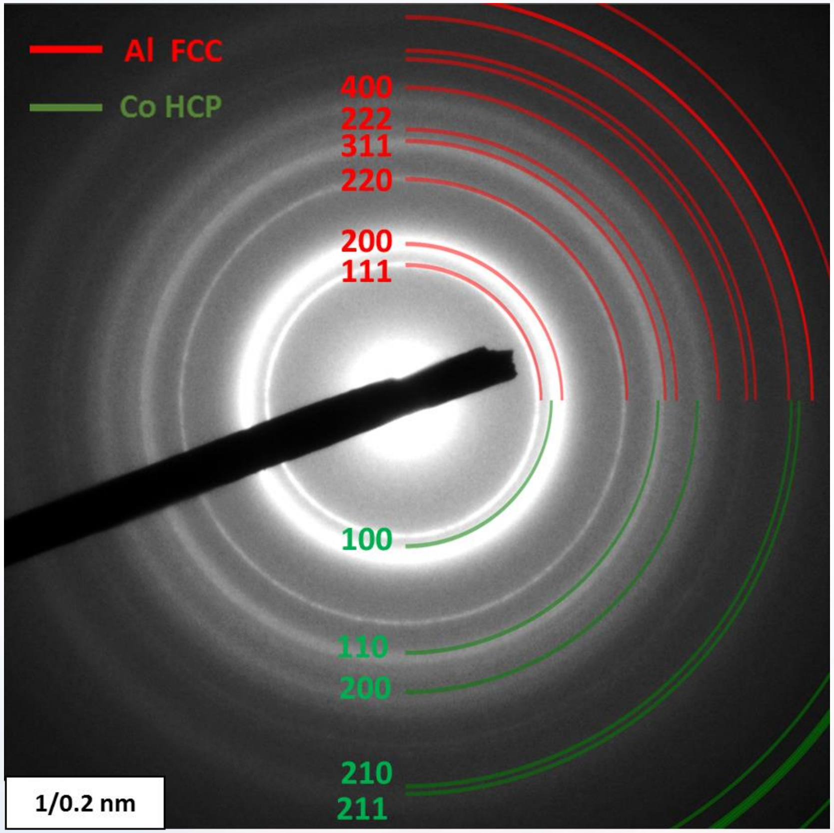
- Laser capabilities:
  - 2-20 Watts
  - Pulsed or continuous operation
  - 50 μm diameter spot size
  - Positioning mirror, which can be used during laser operation



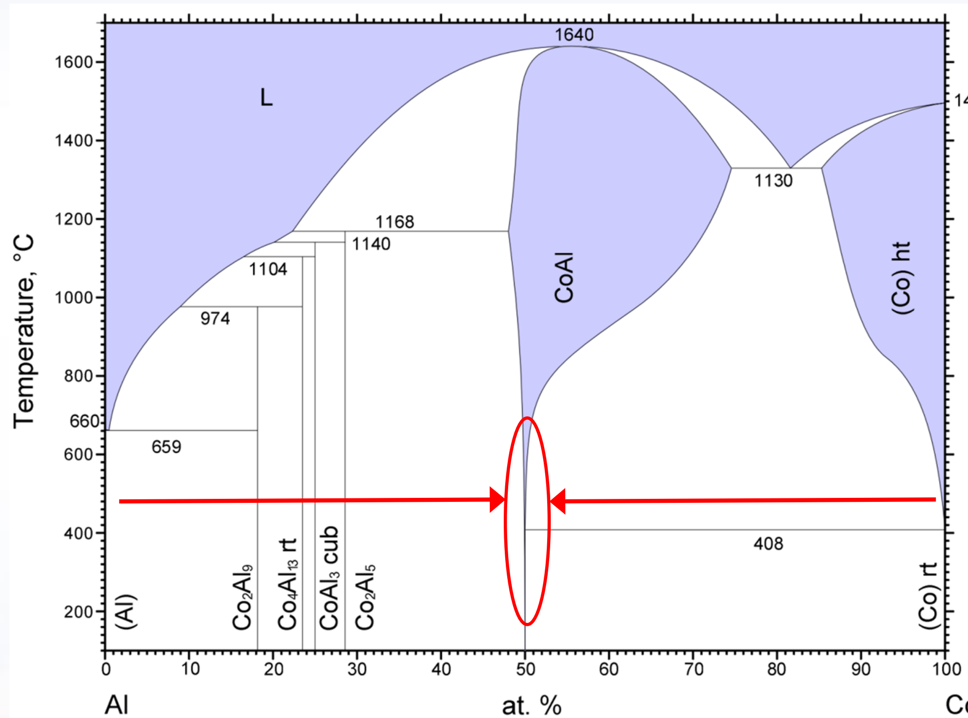
__Laser Alignment TEM Holder__

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

Motivation: Laser, Ion, and Electron Interactions in Multilayer Reactive Foils



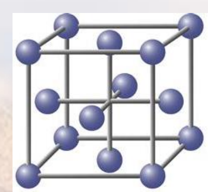
Above: Indexed selected area electron diffraction pattern of the two phases in the pre-reacted film



β Phase (50:50)

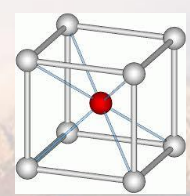
FCC Structure

$Fm\bar{3}m$



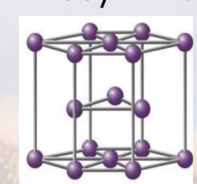
Cubic CsCl Structure

$Pm\bar{3}m$

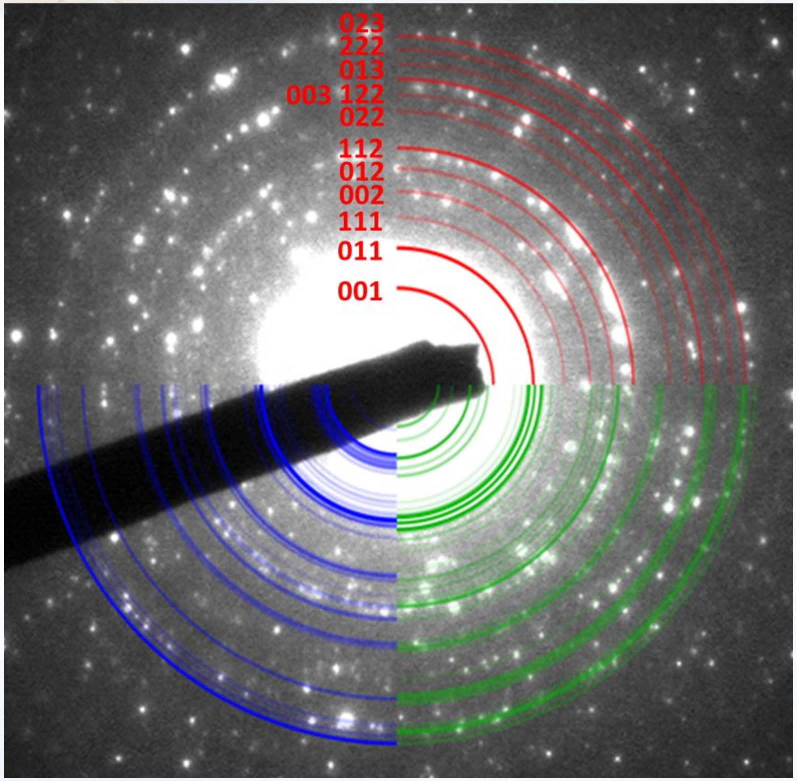


HCP Structure

$P63/mmc$



Motivation: Laser, Ion, and Electron Interactions in Multilayer Reactive Foils

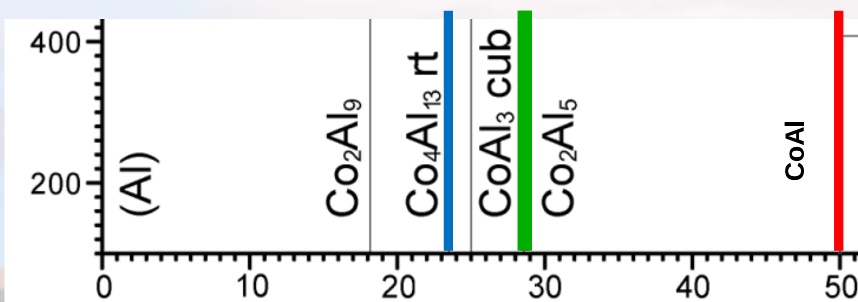


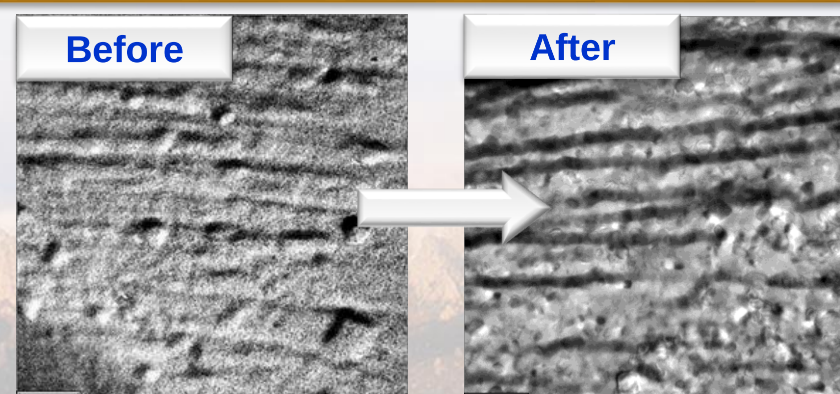
- Equilibrium phase: Cubic β CoAl (line compound)
- Other non-equilibrium phases may form due to local chemical variations
- Three possible line compound phases
  - CoAl (β) — $Pm\bar{3}m$ — Cubic Structure
  - Co_2Al_5 — $P63/mmc$ — Hexagonal Structure
  - Co_4Al_{13} — $Pmn21$ — Orthorhombic Structure
- A significant fraction of the intensity in the diffraction pattern is from the β CoAl phase shown in red

Laser Initiation of MLF has been initiated with a 1064 nm IR laser

The effects of laser, ion, and electron irradiation can all be probed in the same TEM

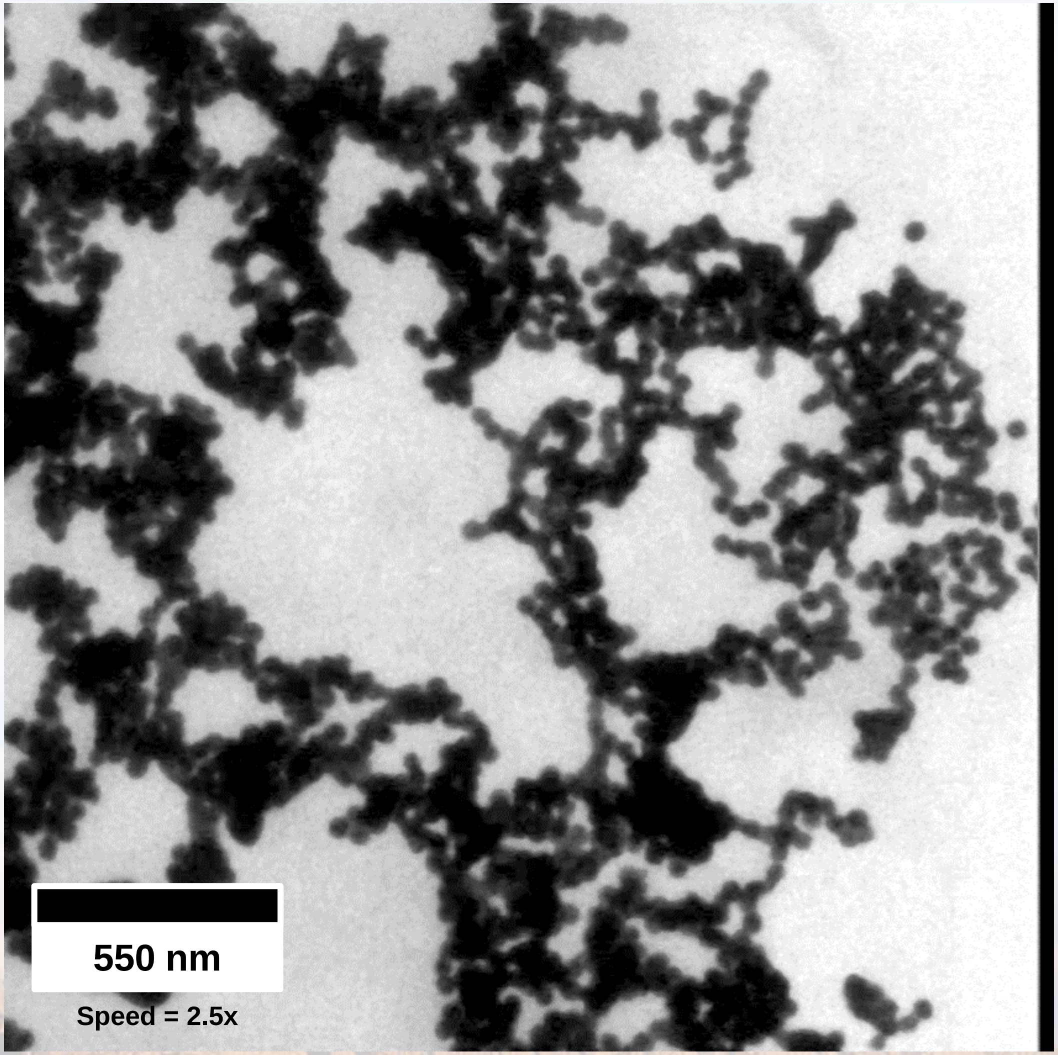
Line compounds in the phase diagram





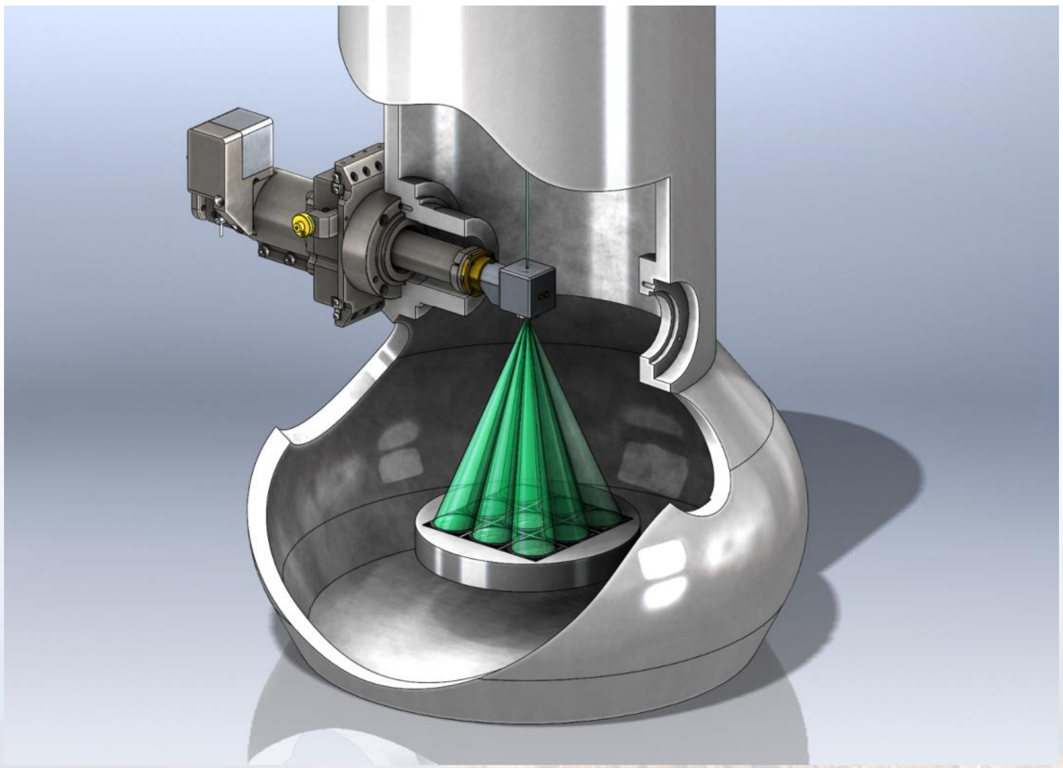
Motivation: Interaction of NPs During Laser Irradiation

- **Observe laser-nanoparticle interactions with sufficient temporal and spatial resolution to see relevant dynamics**
- **Stabilized video shows behavior of Au nanoparticles during laser irradiation with increasing laser power**
- **Investigate sintering and consolidation mechanisms with increased temporal resolution**
- **Possible applications to increase understanding of additive manufacturing systems**

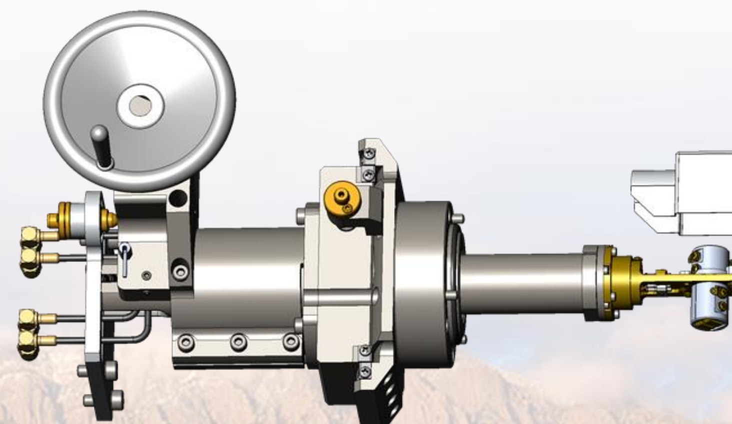


A Complex Combination of Sintering, Reactions, and Ablation Occurs

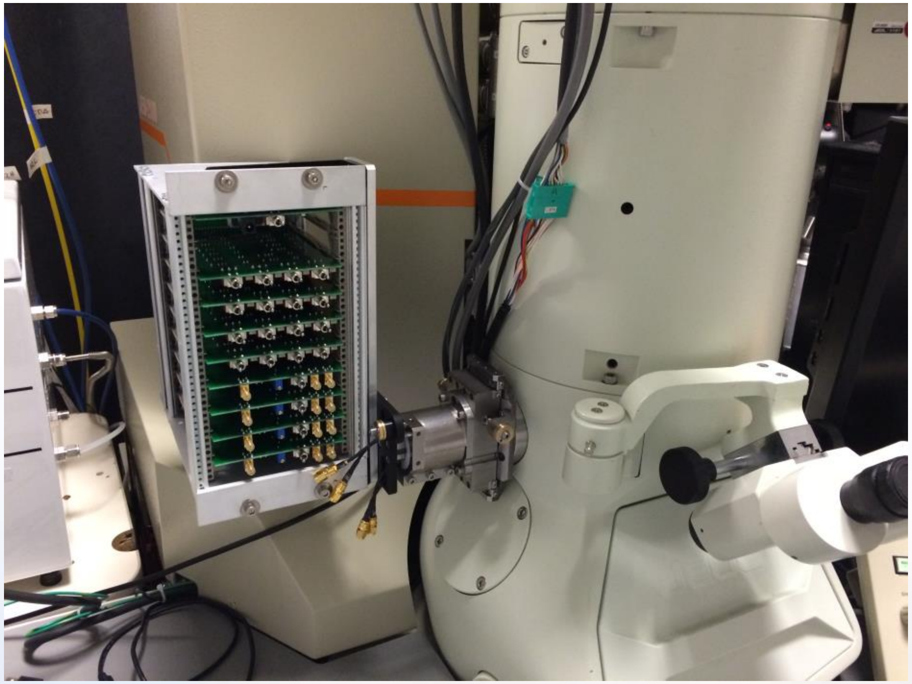
Movie-Mode deflector : μs Resolution with a Standard Camera

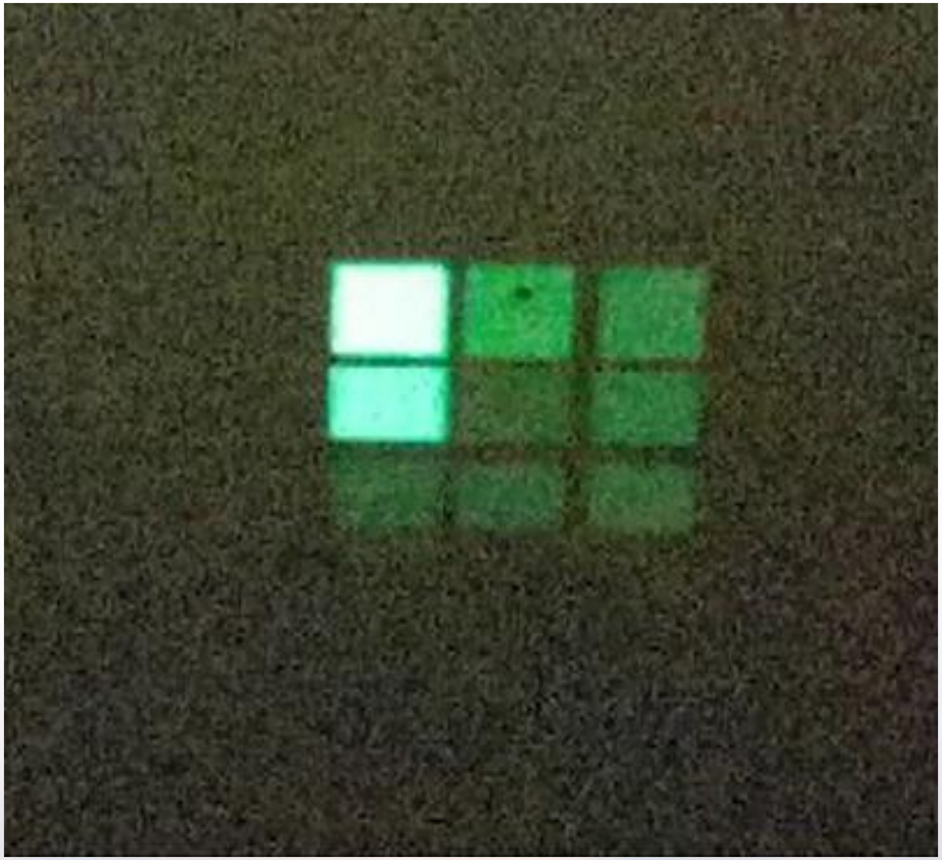


- Electrostatic deflection of electrons
- 4, 9, or 16 images per frame, spread over a large camera
- Any exposure time up to the limits of the camera
  - Ultimate limit is beam current/brightness

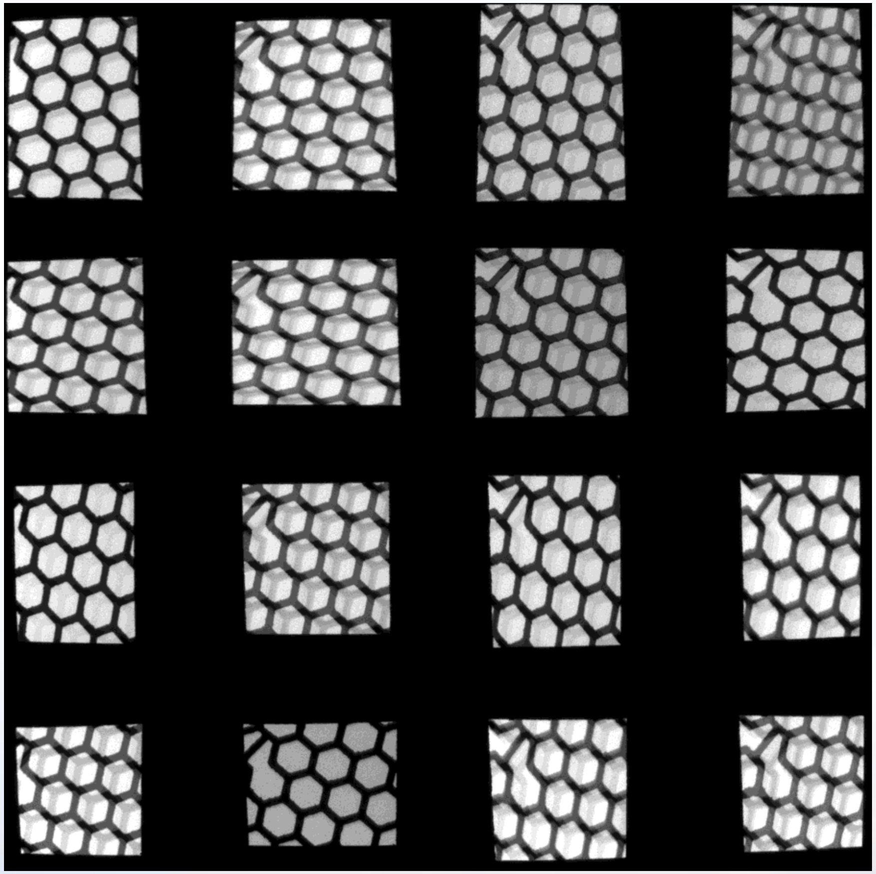


Demonstration of Movie-Mode Deflectors at Sandia

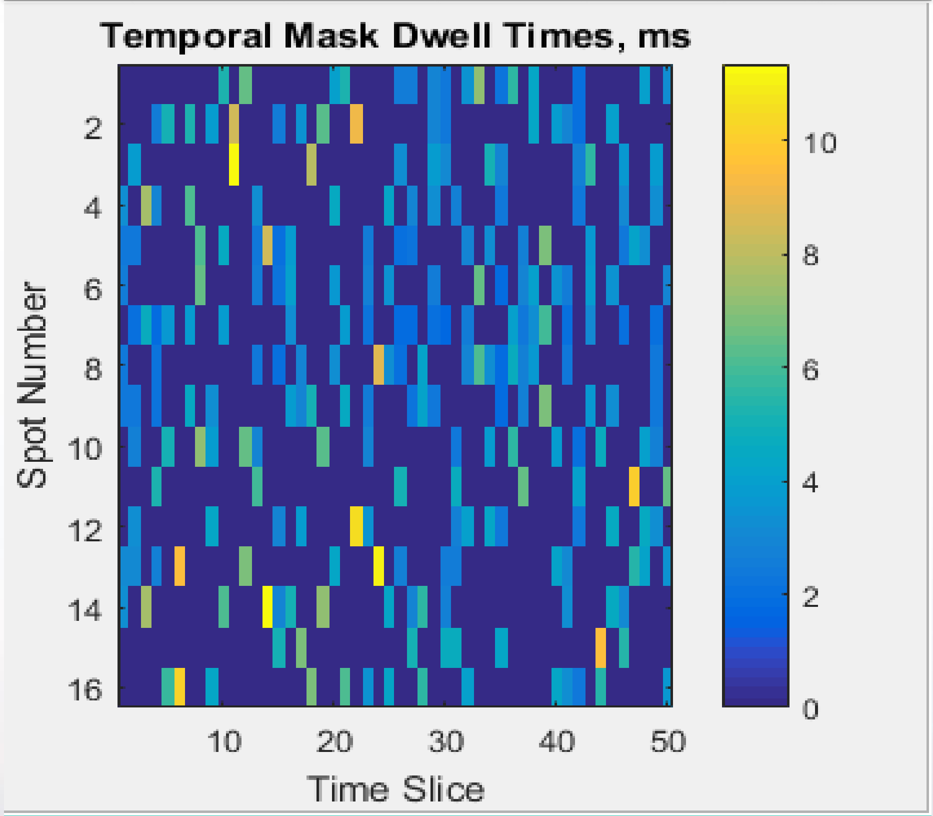




Movie Mode & Temporal Compressive Sensing



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

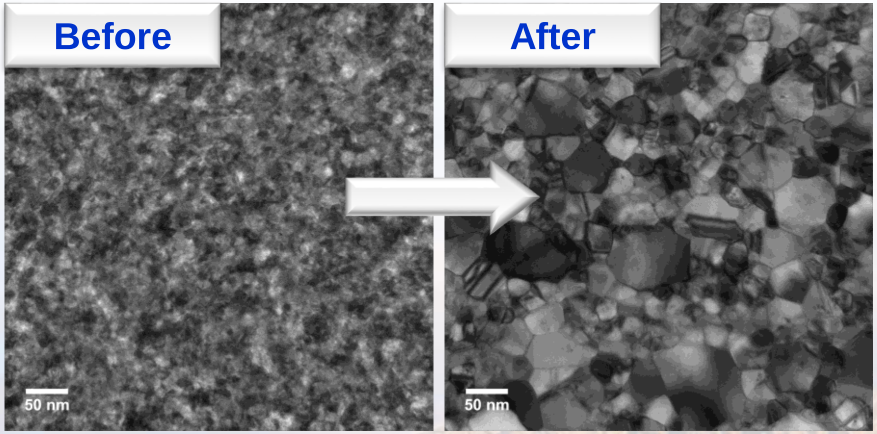


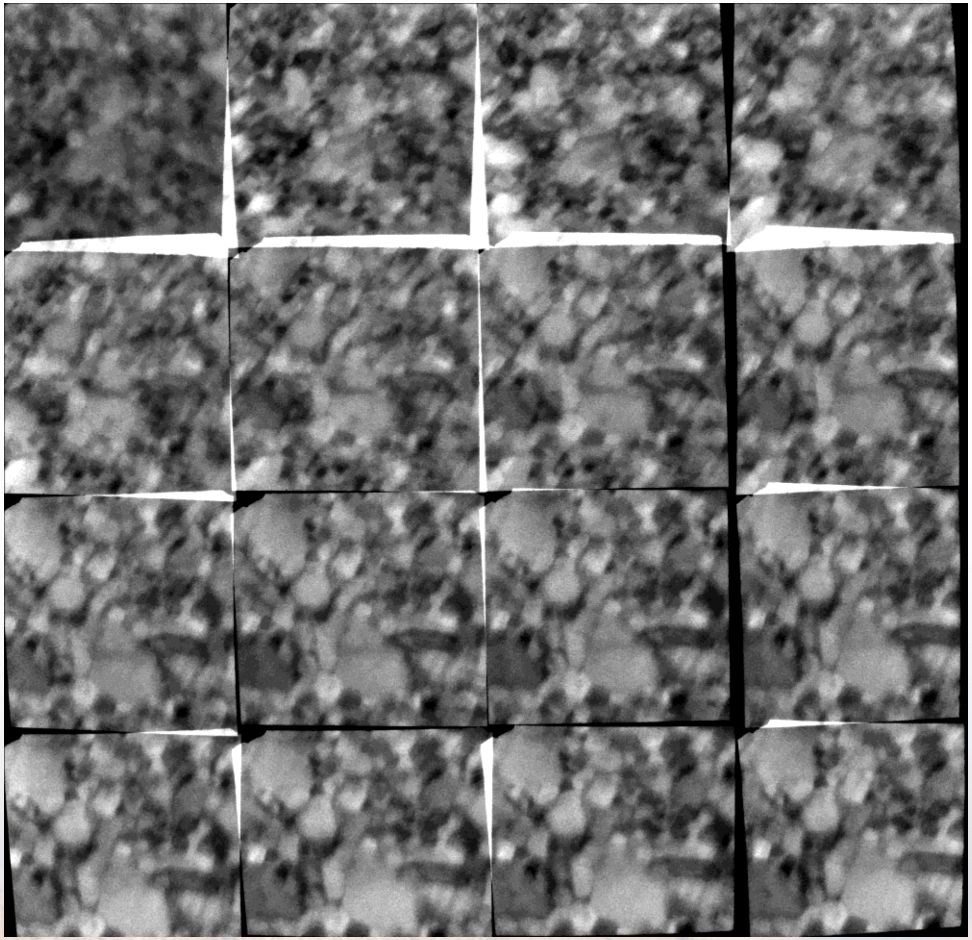
- **Electron intensity of a single exposure is randomly distrusted to multiple images within a single frame**
- **Record of random mask used to distribute exposure intensity is later used deconvolute images**

Motivation: In-situ Microstructure Evolution

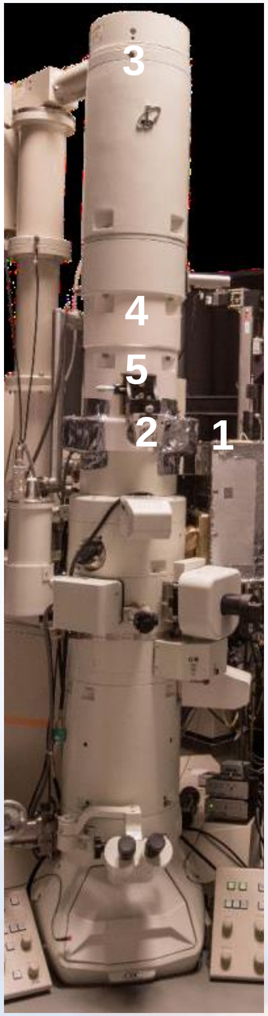
- In-situ TEM images of IR laser driven grain growth in Pt foil
- Characterize grain boundary and grain evolution
- Rapid heating & cooling controlled by specimen drive laser

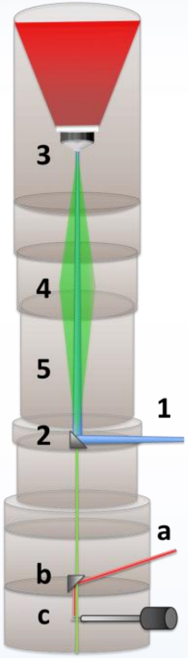
Deflector System: Evolution of Pt microstructure with ~10ms exposures shown on the right

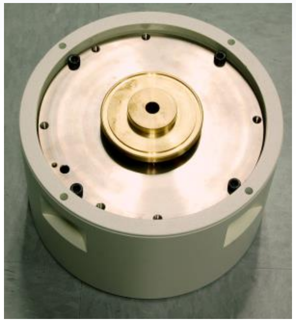




Conversion of a Standard JEOL 2100 TEM to DTEM: Increase Current Density







C_0 Lens

Drift Section

UV Laser

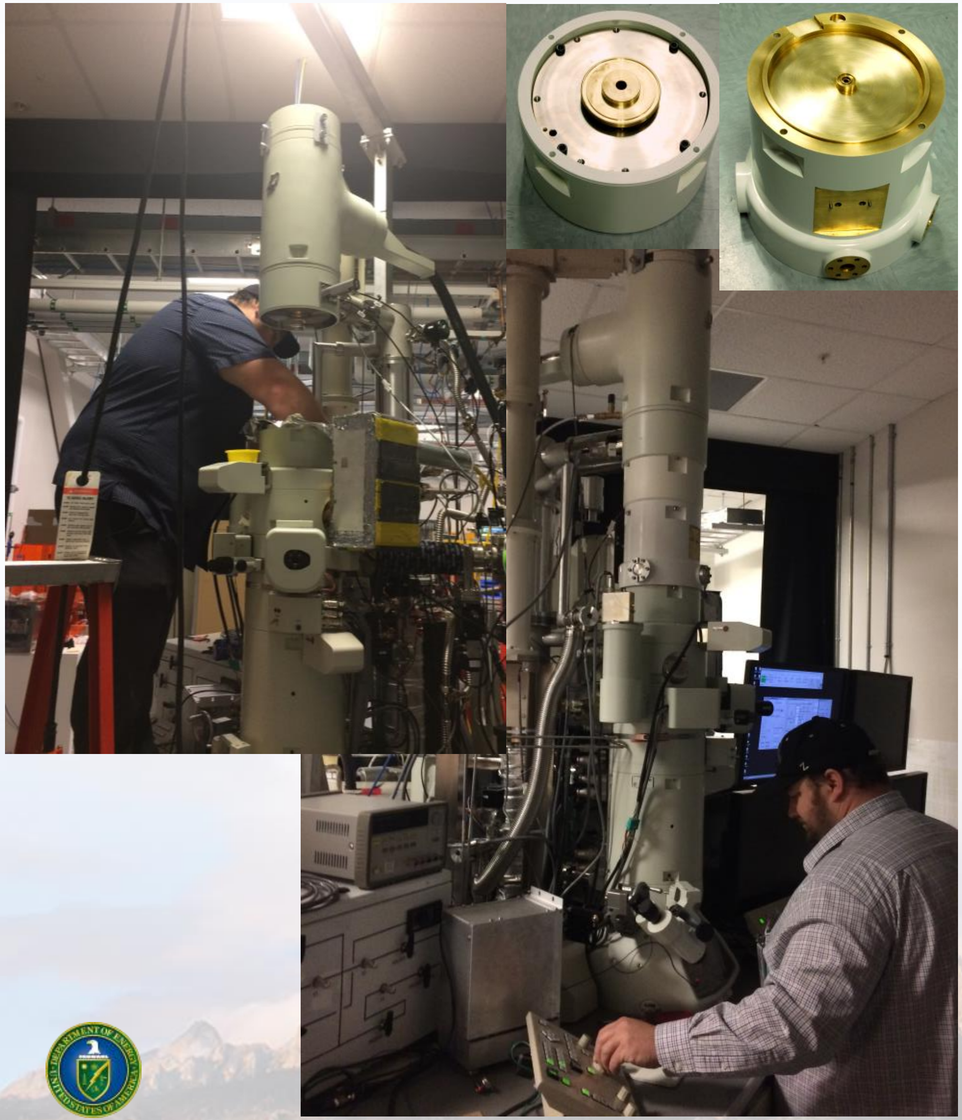
A standard LaB_6 TEM has on average 1 or 2 electrons in the column at a given time!

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

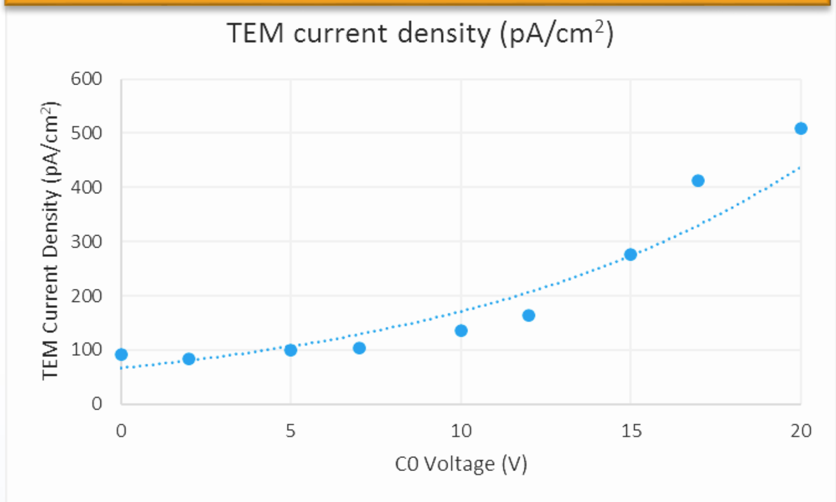
<u>DTEM Development</u>

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



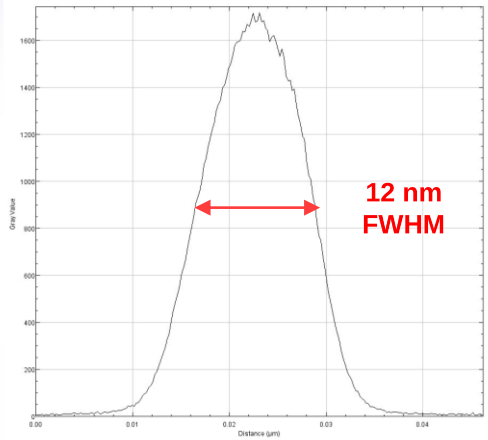
Addition of C_0 lens and drift section give a 5X increase in current density

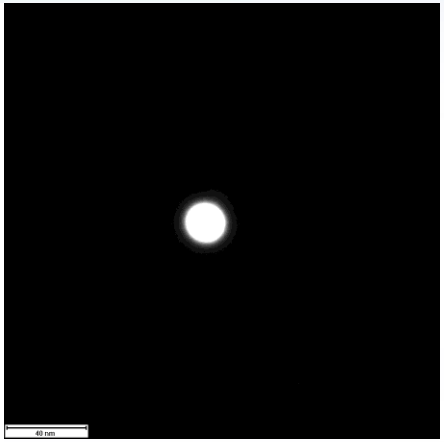


- **Disc cathode produces wider distribution of electrons than LaB_6**
- **Current density can be increased by adding C_0 lens to condense electrons to a smaller probe**
- **Strength of C_0 lens is controlled by external adjustable power supply**
  - Trade off between current density and resolution
-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

Tantalum Filament Exchange

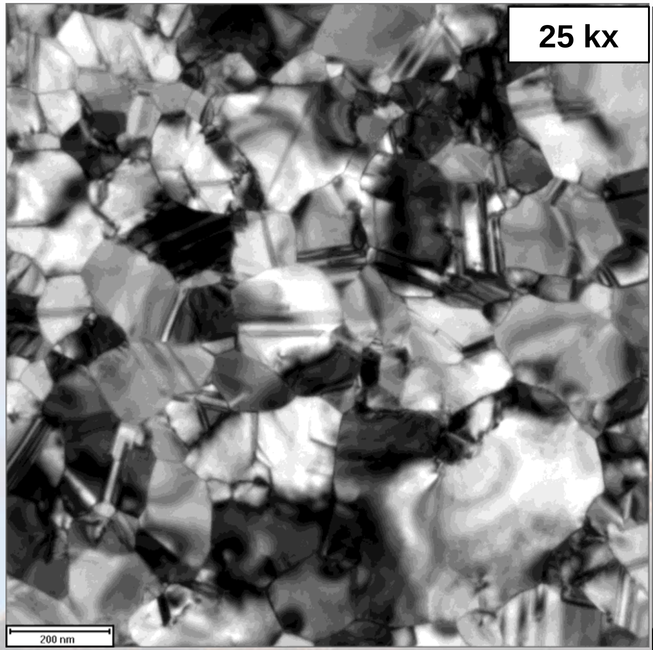
- **Changes with C_0 lens setting of 0V**
  - 50% reduction in electron
    - LaB_6 ~ 50 pA/cm^2
    - Ta ~ 30 pA/cm^2
  - Higher beam current at from source
    - LaB_6 ~ 7 μA
    - Ta ~ 23 μA
  - Increase in minimum spot size for precession
    - LaB_6 ~ 8 nm FWHM
    - Ta ~ 12 nm FWHM

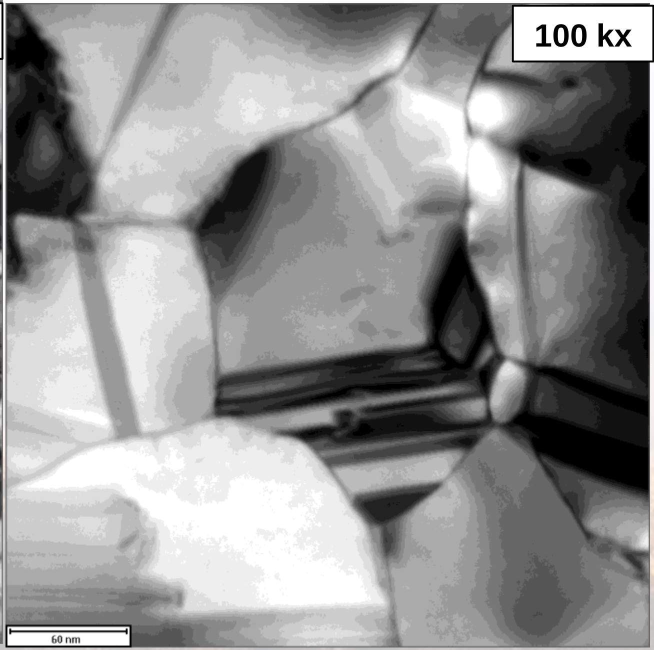




Above:

Smallest achievable spot size for precession with Ta cathode





Left:

Bright field TEM images of nanocrystalline gold taken with Ta filament

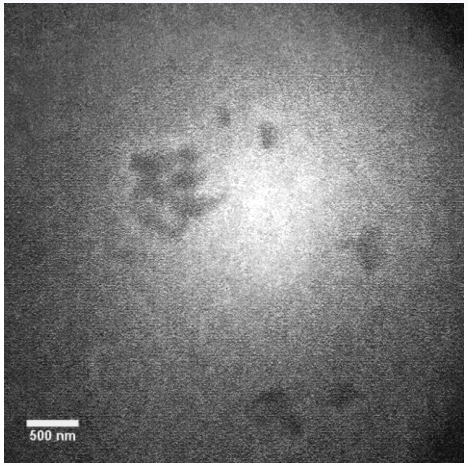
Easily switch between DTEM and thermal emission with good resolution in minutes!

Filament exchange in as little as 90 min.

Achieved Laser Induced Photoemission of Electrons

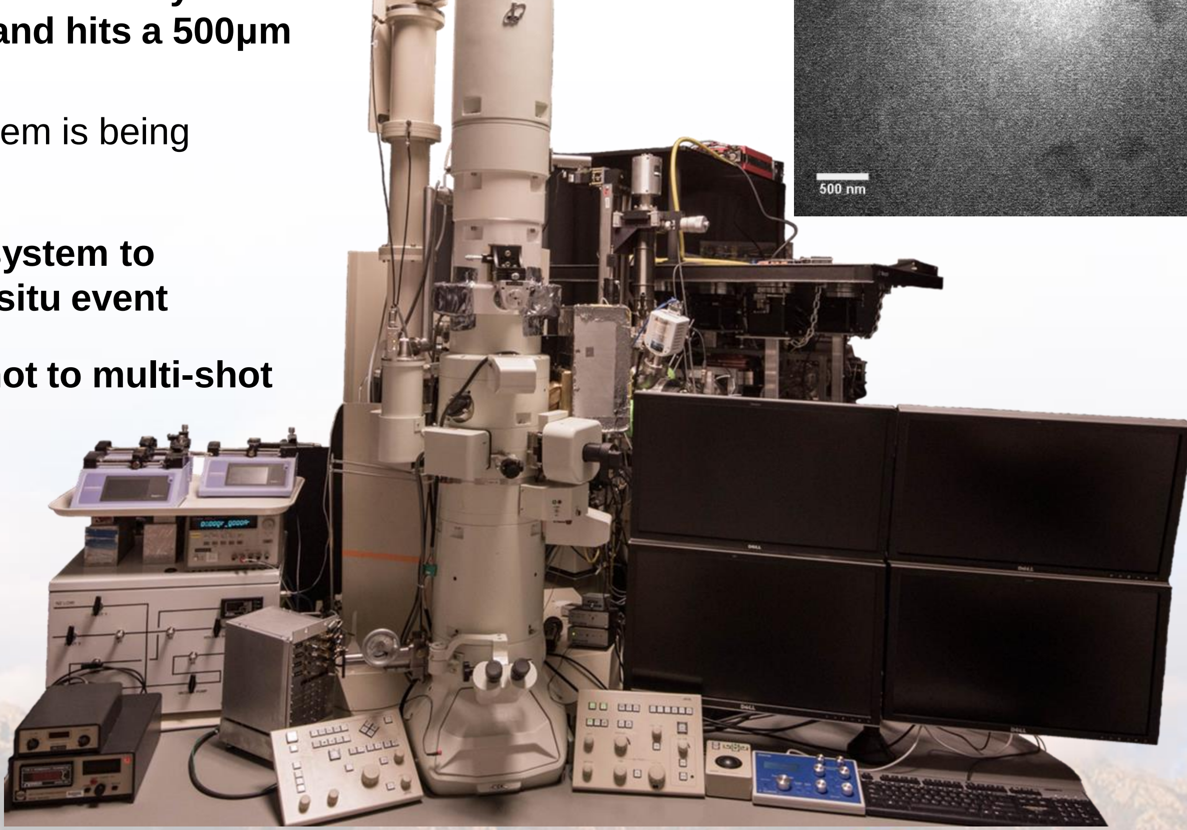
266 nm UV laser induced photoemission has been achieved!

6 ns single-shot DTEM image of P47

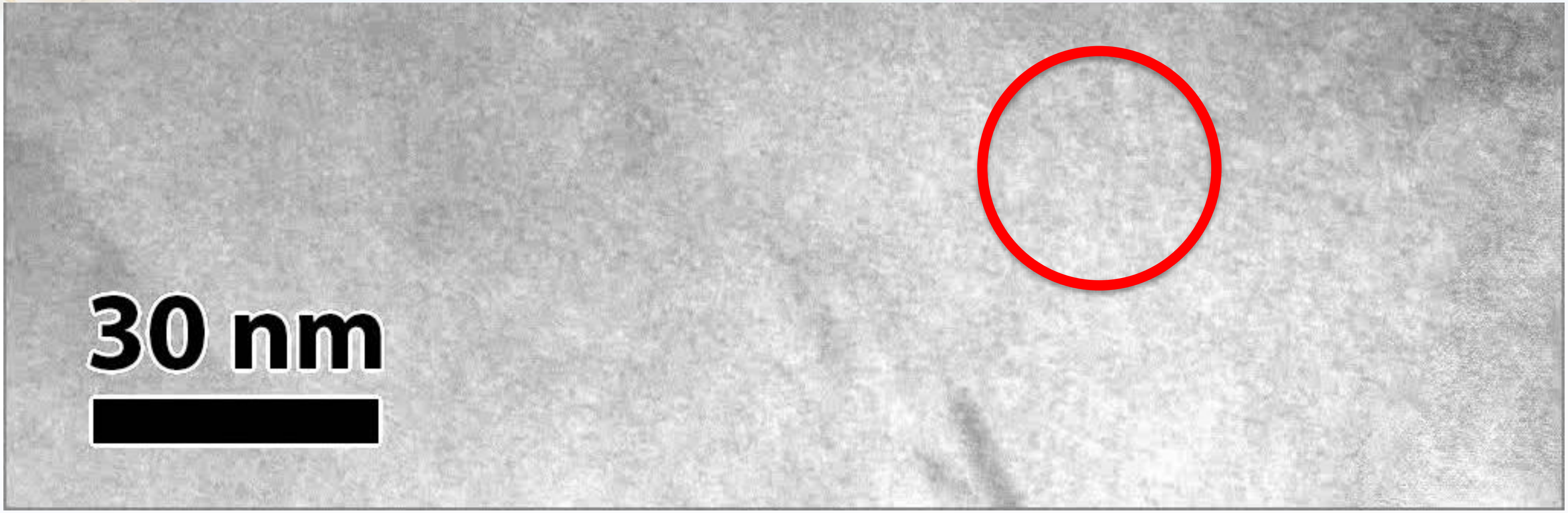


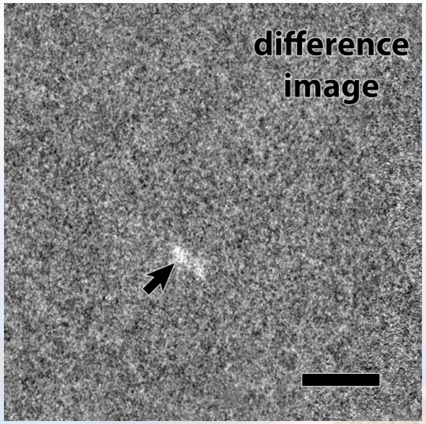
Future work

- **<u>Current Impediment:</u> Alignment of laser system is not trivial. Laser travels meters and hits a 500μm disc**
  - Next generation alignment system is being developed
- **Addition of photodiode timing system to synchronize laser pulse and in-situ event**
- **Converting laser from single-shot to multi-shot**
- **Increasing stability of laser**
- **Laser upgrade**



Motivation: In-Situ Single Ion Strikes in Si



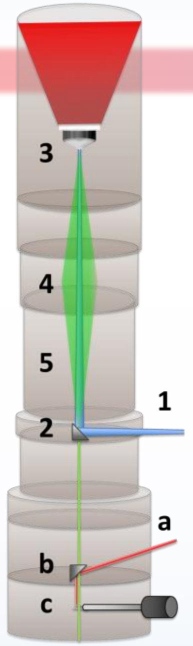


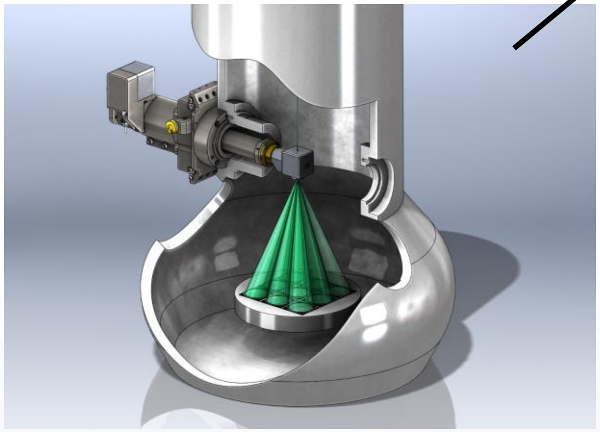
- 1.7 MeV Au into single crystal Si
- Single ion strikes can be observed in semiconductors
- Non-symmetric structure in contrast to the spherical approximation

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?

Increasing Temporal Resolution

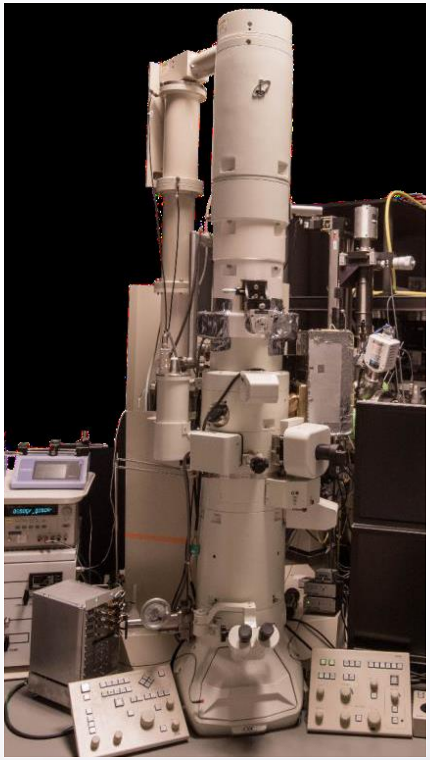


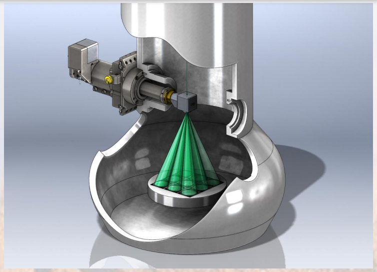


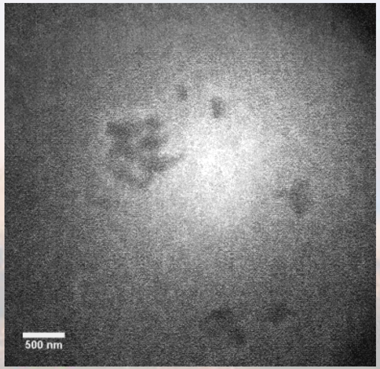
- **DTEM**
  - Laser induced photoemission of electrons is needed to achieve sufficient current density to produce an image
  - Provides nanosecond imaging of irreversible process
- **Deflector System**
  - Multiple images acquired on single frame
  - Microsecond imaging possible
  - Virtually no missing 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

Summary

- Fundamental TEM modifications for DTEM, movie mode, and compressive sensing have been completed.
- Maintained functionality of the TEM using LaB_6 thermal emission with minimal impact
- Ability to witch between DTEM and Ta cathode thermal emission in minutes with good spatial resolution
- Possible to switch between LaB_6 and DTEM in just a few hours
- Movie Mode/Compressive sensing can achieve microsecond temporal resolution
- Laser induced photoemission of electrons has been achieved with in 6 ns temporal resolution
- DTEM and movie mode functionality on the I^3TEM combined with the wide range of available in-situ holders will provide the unique capability to observe materials and ion irradiation processes in-situ with nano and microsecond temporal resolution







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