

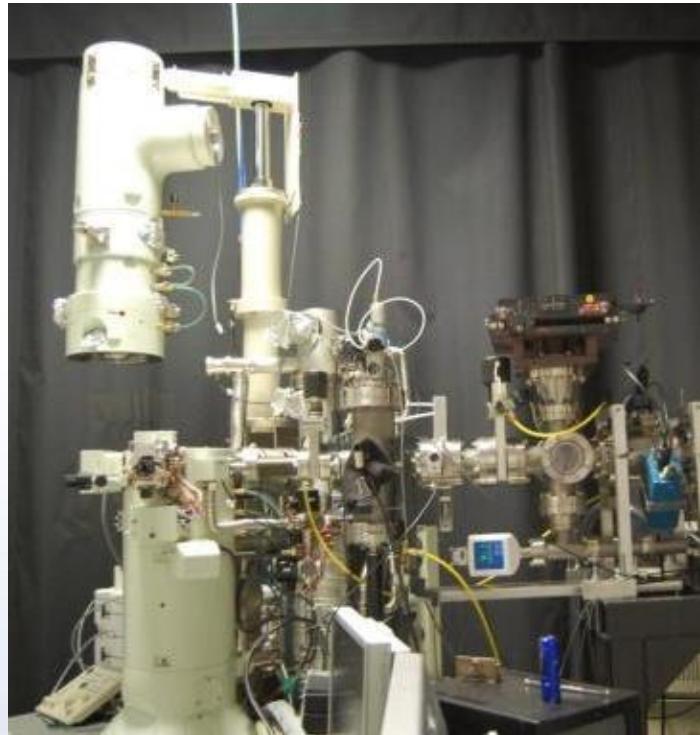
Can Single Ion Strikes be Directly Observed in Relevant Time and Length Scales?

SAND2016-12728C

P. Price, D. Bufford, R. Sisson, M. Abere, D. Adams, & K. Hattar
S.T. Park, B. Reed, & D. Masiel

Sandia National Labs
IDES Inc.

December 15, 2016



1. Single ion strikes can be observed in relevant length scales utilizing Sandia's I³TEM facility.
2. Work is underway to directly couple multiscale models and key experimental results
3. Can relevant time scales be achieved is still an outstanding question.

Collaborators:

- Sandia: [M. Franco](#), [M. Blair](#), [D. Buller](#), [G. Vizkelethy](#), [B.L. Doyle](#), [M.T. Marshall](#), [D. Saiz](#), [J.A. Scott](#), [B.R. Muntifering](#), [W. Wampler](#), [R. Dingreville](#), & [S. Foiles](#)

External: [T. La Grange](#) [A. Lang](#), [M. Taheri](#), [W. Ji](#), [A. Schleife](#)

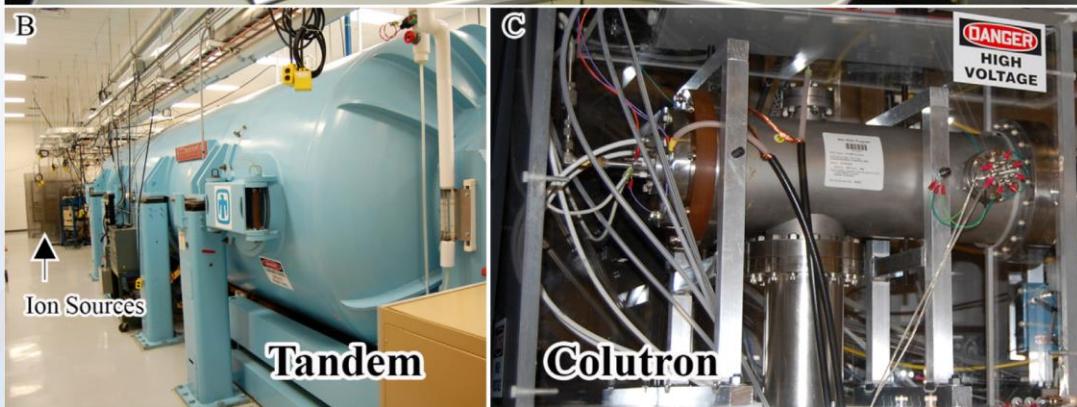
This work was supported by the US Department of Energy, Office of Basic Energy Sciences.

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Radiation Characterization with Nanometer Resolution in Real Time



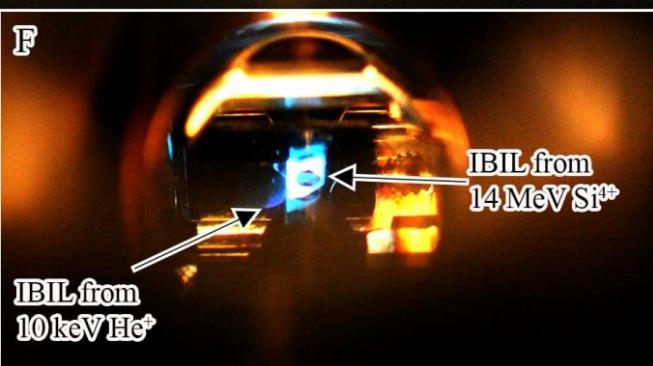
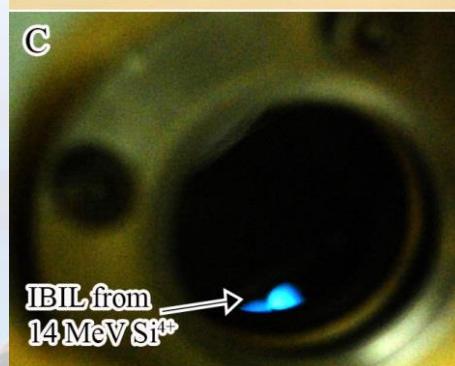
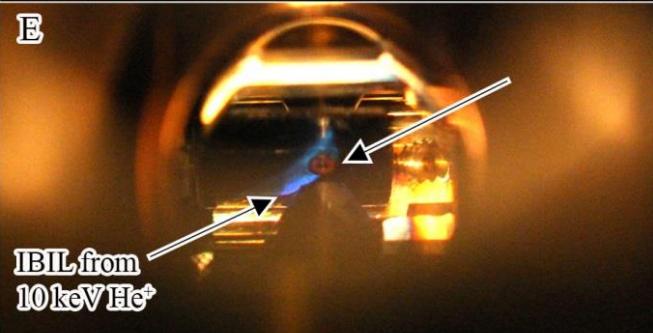
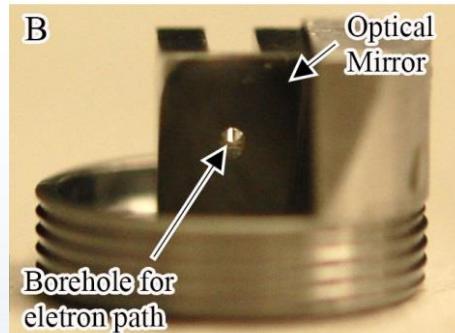
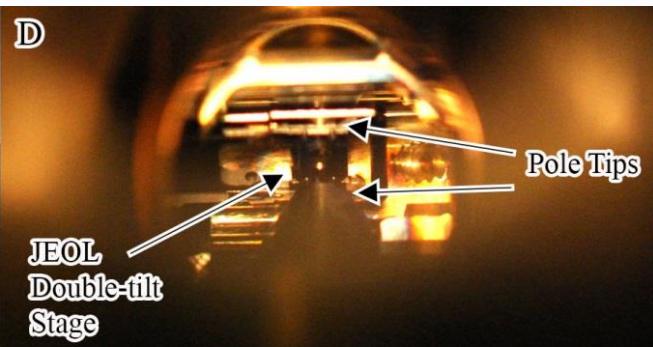
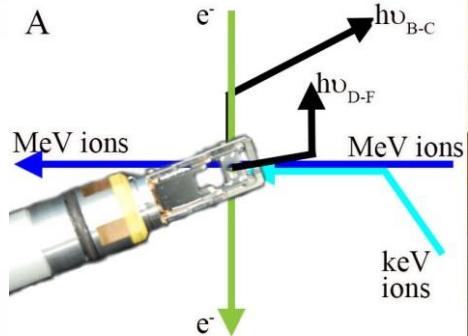
Hattar, K., D. C. Bufford, & D. L. Buller. "Concurrent in situ ion irradiation transmission electron microscope." *NIM:B* **338**, 56-65 (2014).



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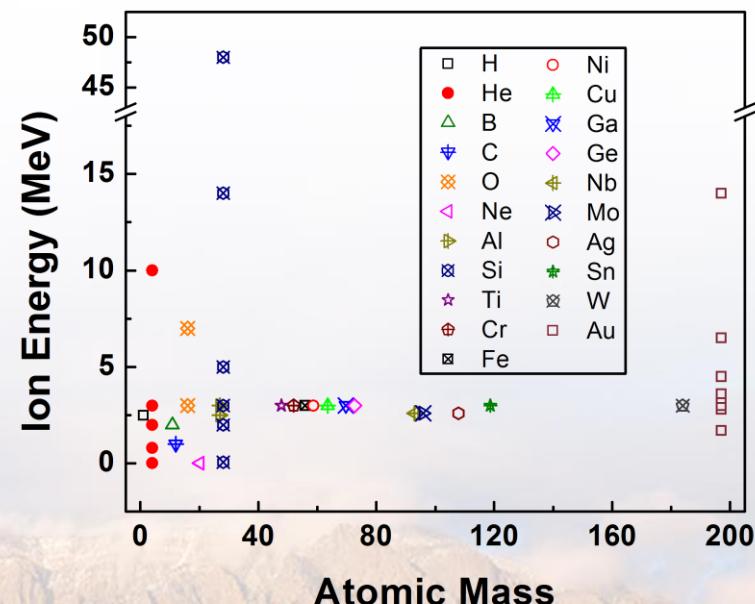
Visualizing Ion Beams in the TEM

IBIL from a quartz stage inside the TEM



Ion beam alignment at the sample confirmed using ion beam-induced luminescence

Ion species & energy introduced into the TEM

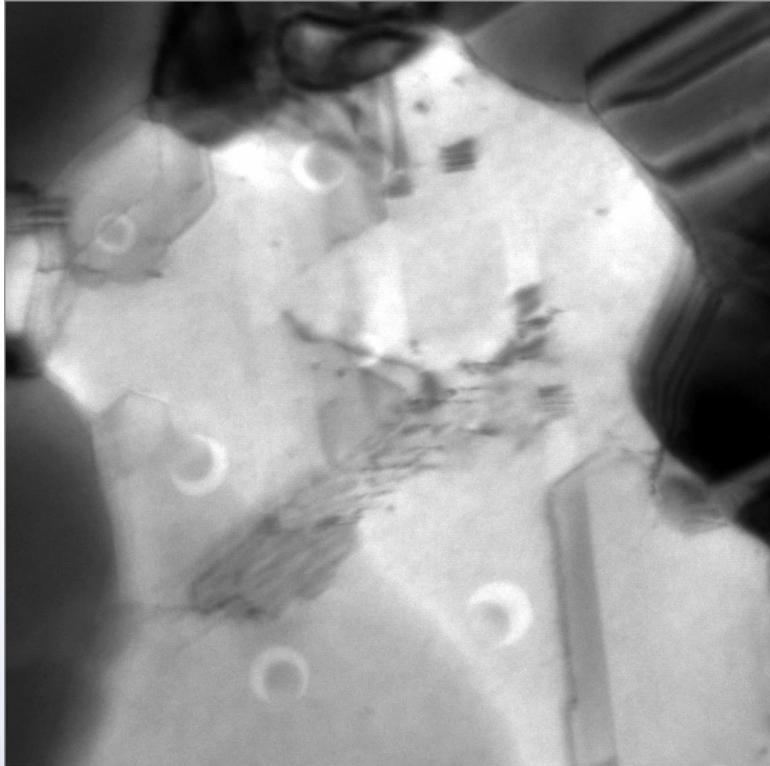


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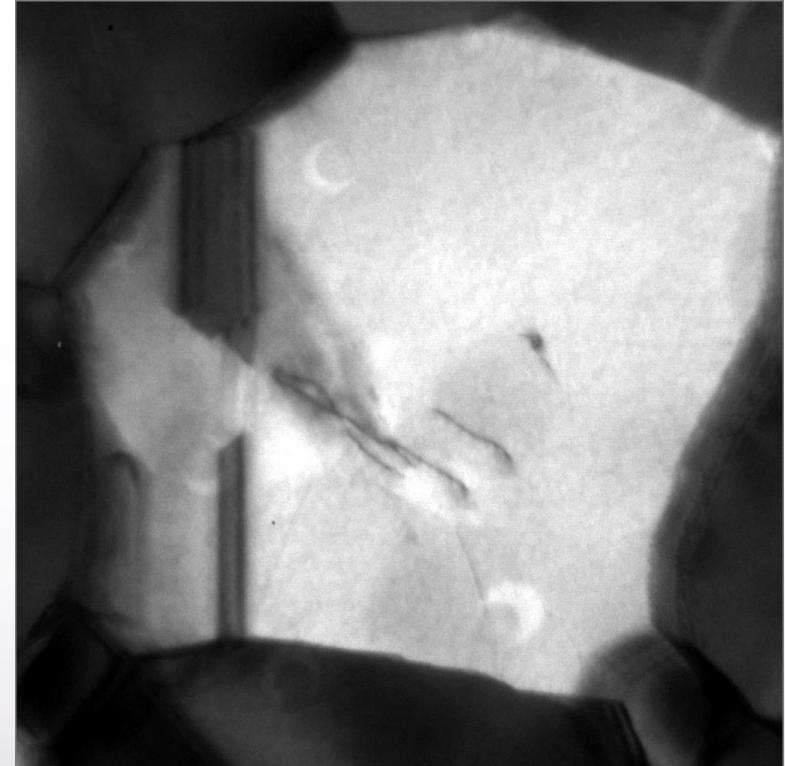
Overlapping Cascades vs. Single Ion Strikes

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

7.9×10^9 ions/cm²/s



6.7×10^7 ions/cm²/s



VS.

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

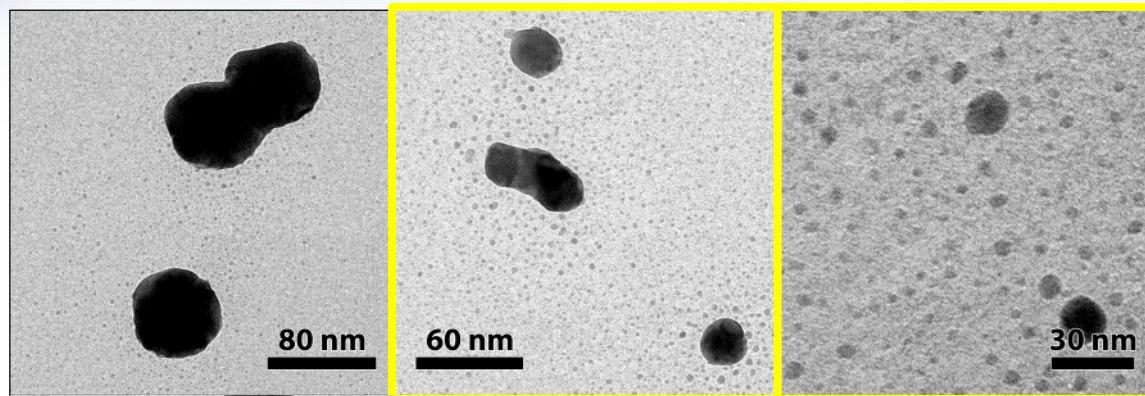


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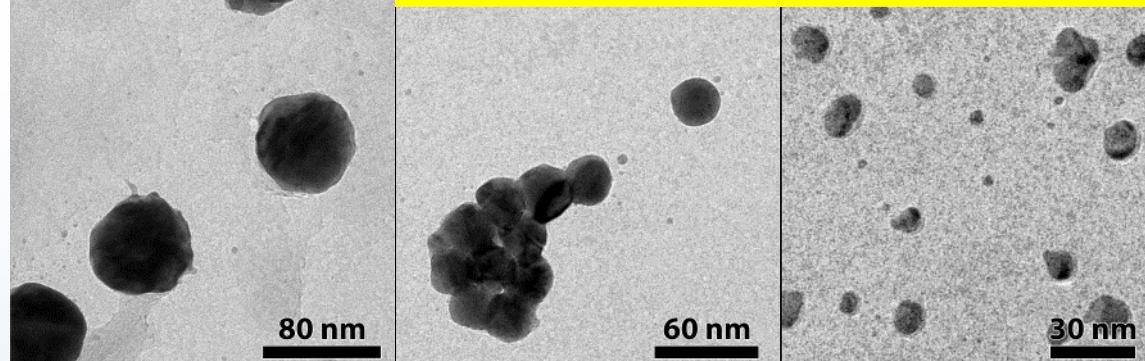
Cumulative Effects of Ion Irradiation as a Function of Ion Energy and Au Particle Size

Collaborator: D.C. Bufford

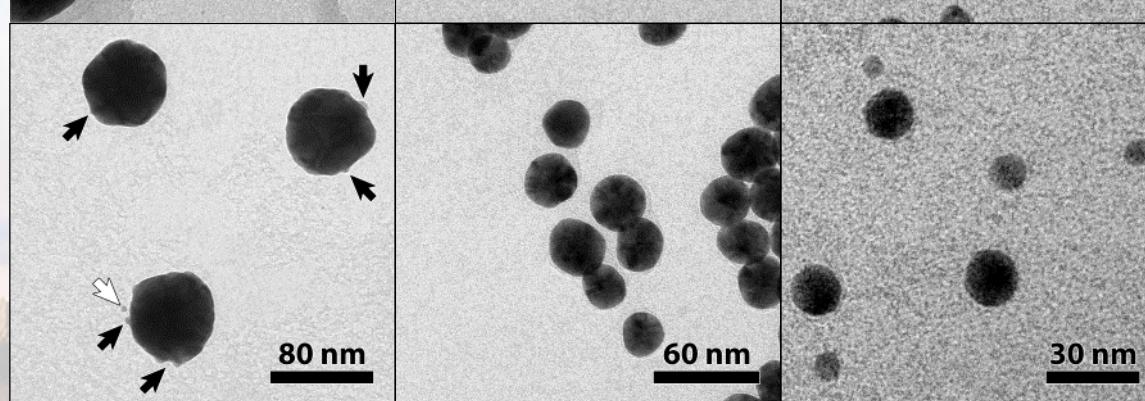
46 keV Au¹⁻
 $3.4 \times 10^{14} /cm^2$



2.8 MeV Au⁴⁺
 $4 \times 10^{13} /cm^2$



10 MeV Au⁸⁺
 $1.3 \times 10^{12} /cm^2$



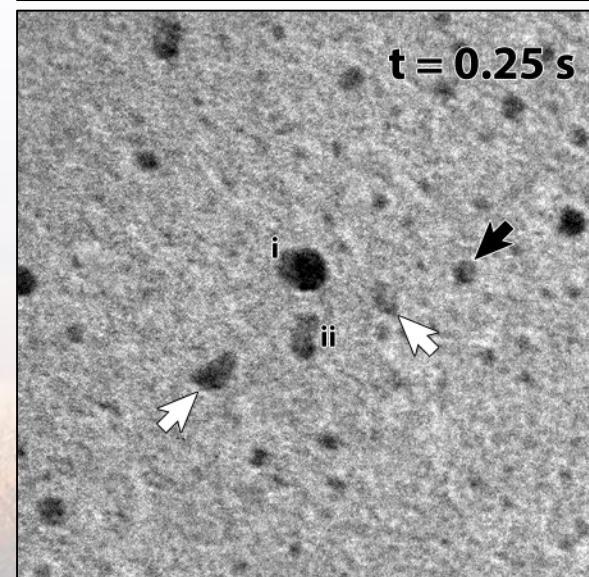
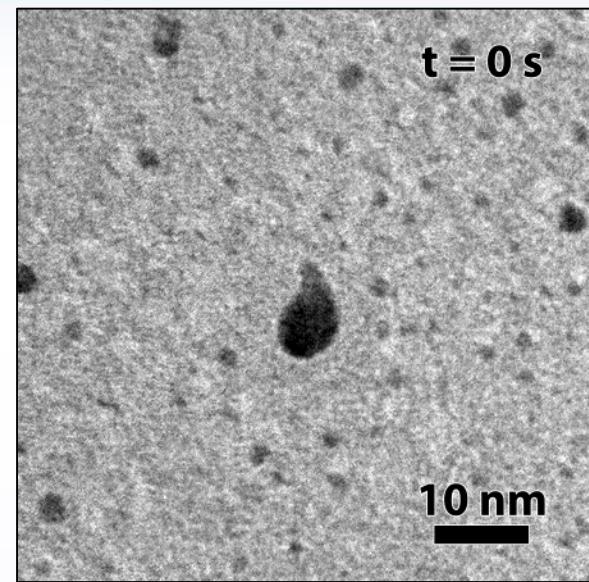
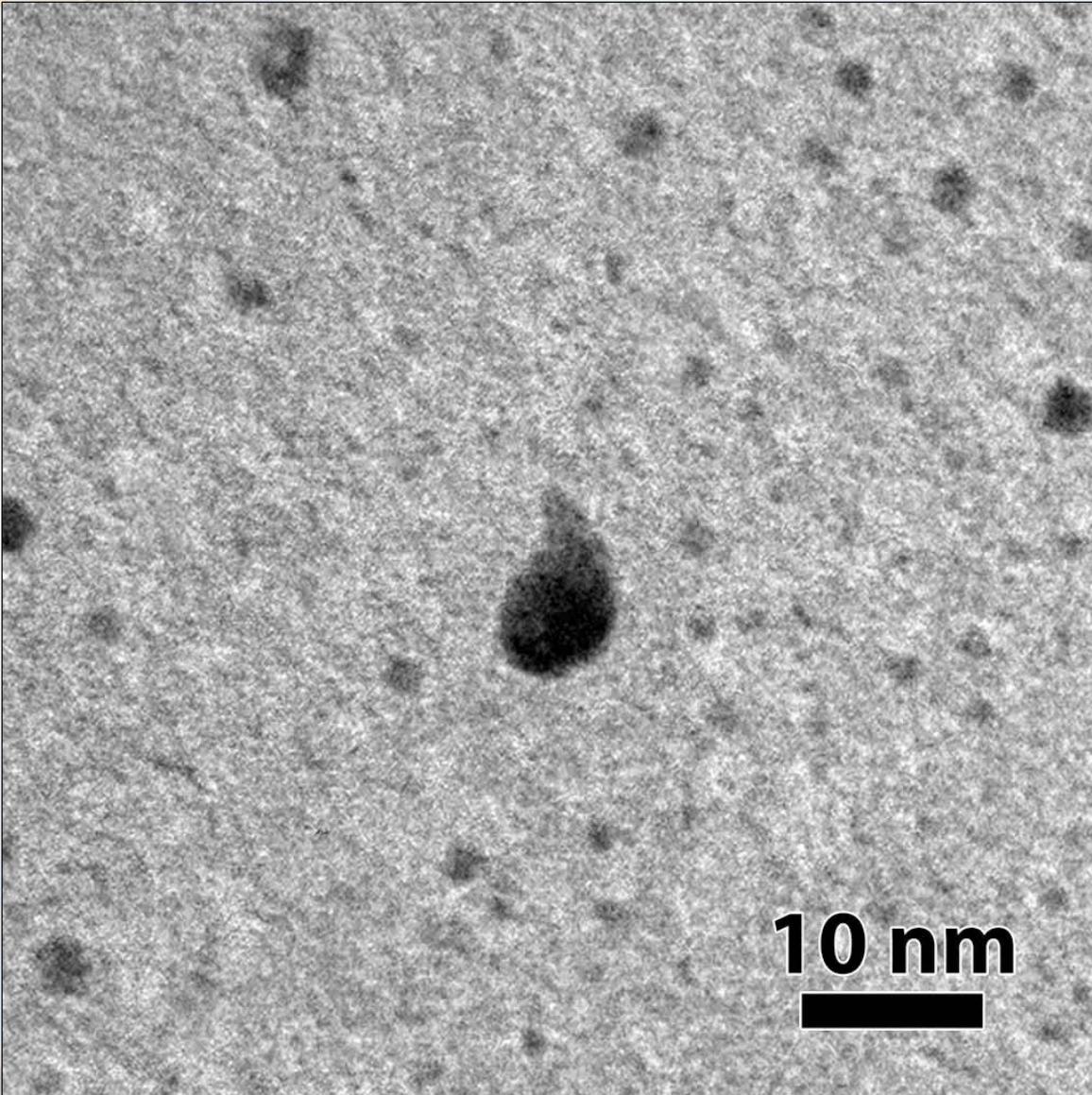
Particle and ion energy dictate the ratio of sputtering, particle motion, particle agglomeration, and other active mechanisms



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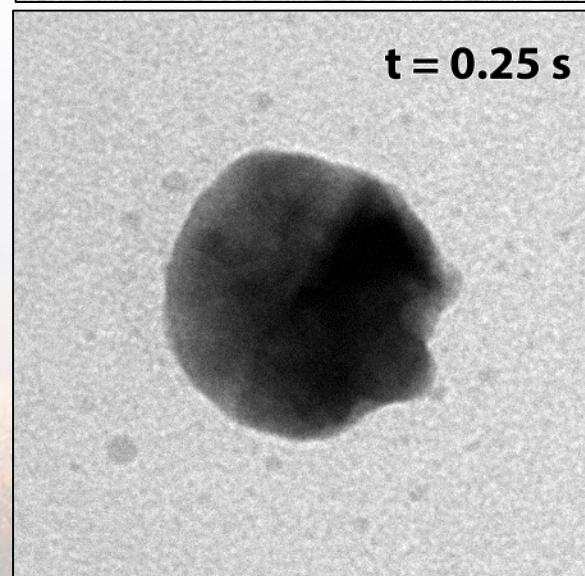
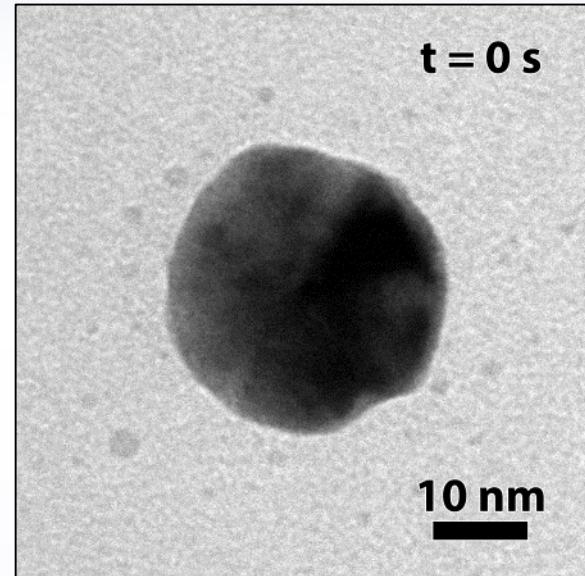
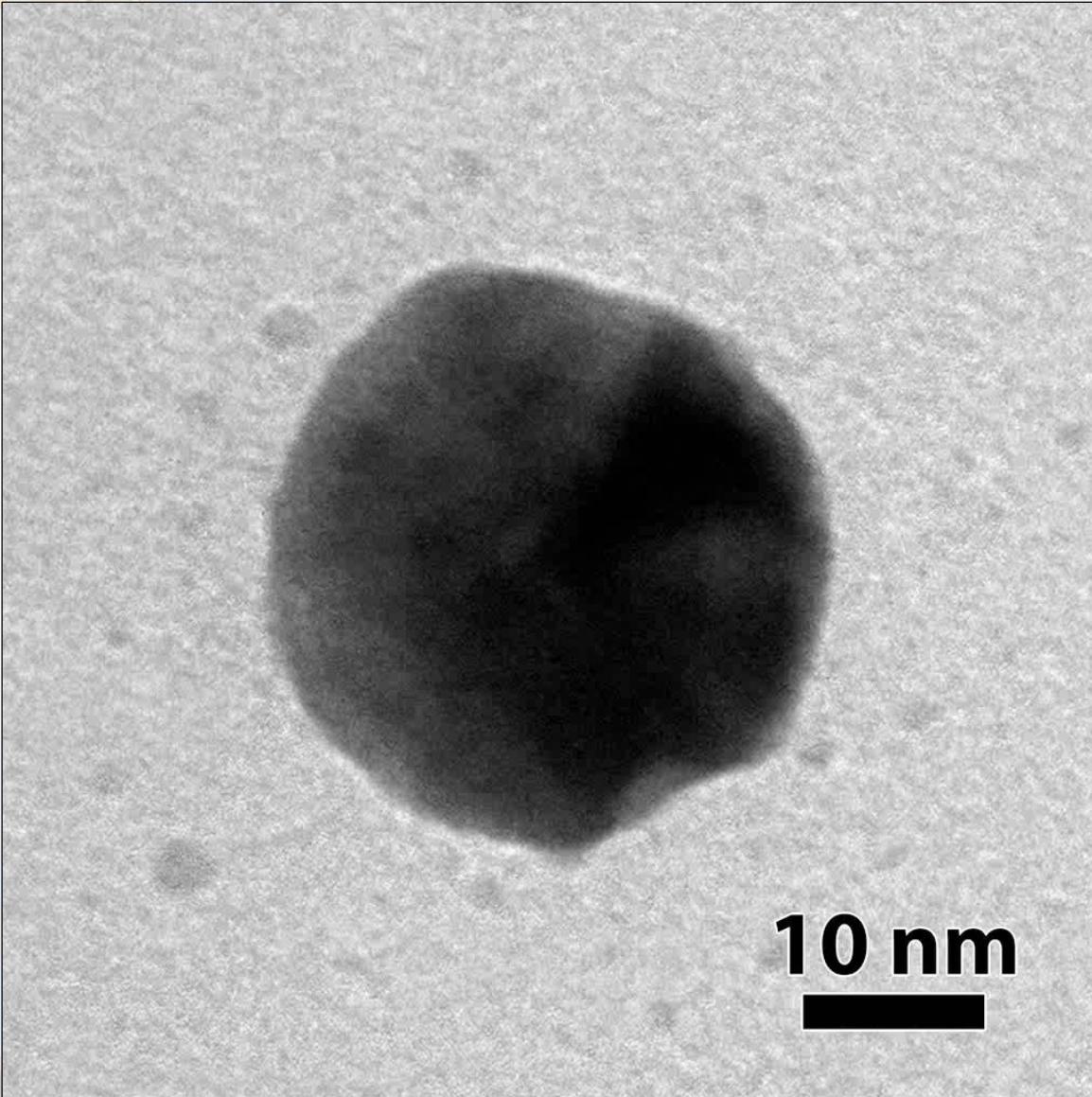
Single Ion Effects with 46 keV Au¹⁻ ions: 5 nm

Collaborator: D.C. Bufford



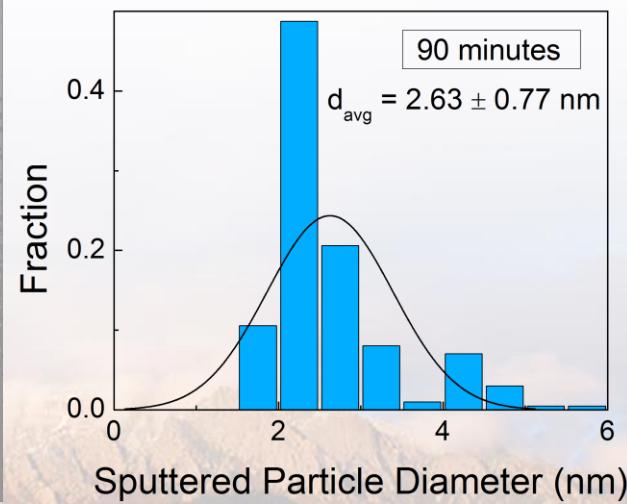
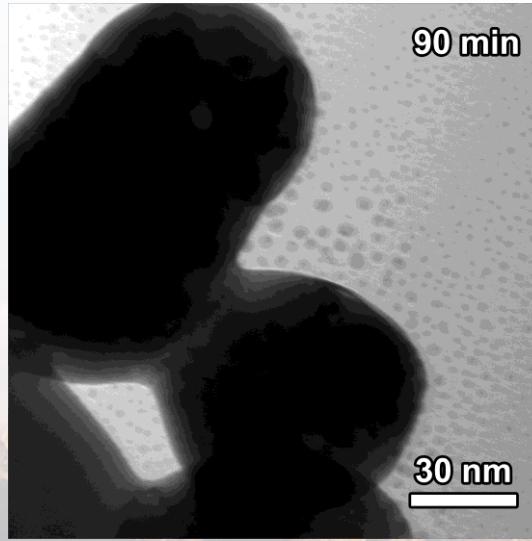
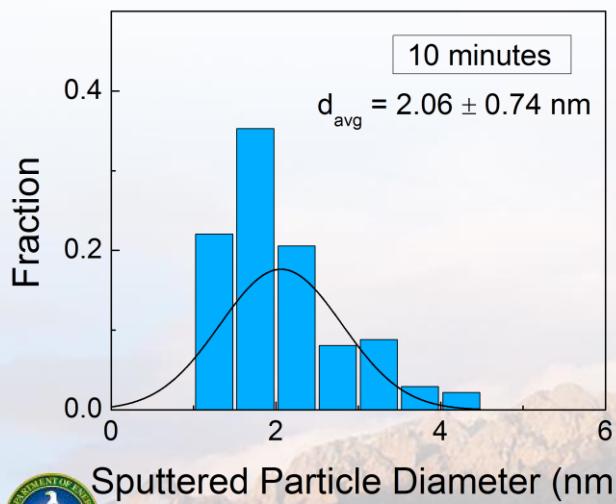
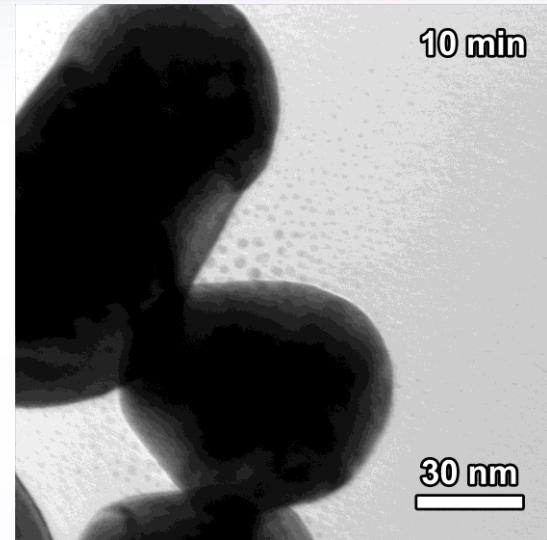
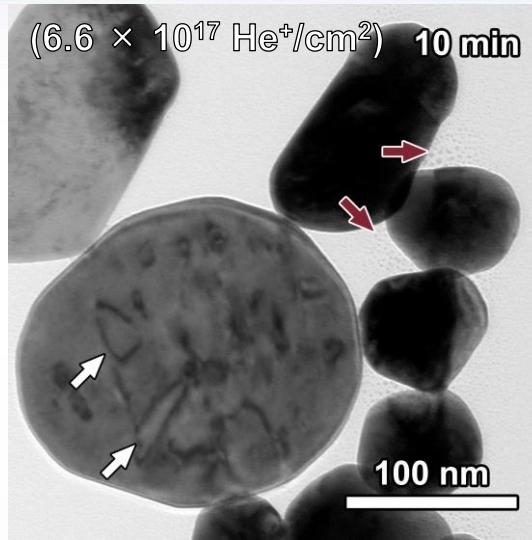
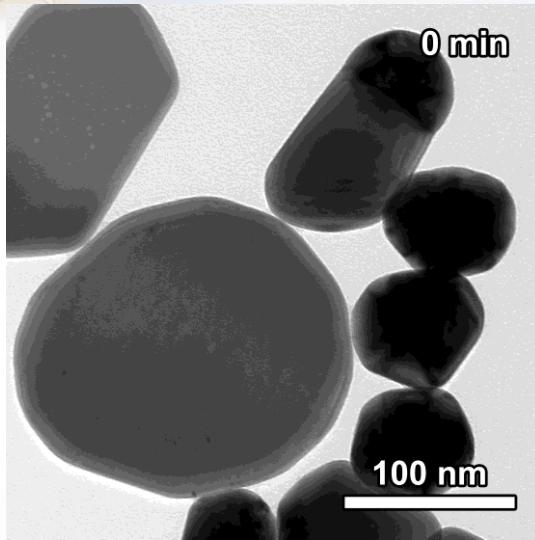
Single Ion Effects with 46 keV Au¹⁻ ions: 20 nm

Collaborator: D.C. Bufford



Formation of Dislocation Loops & Sputtered Particles due to He implantation

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



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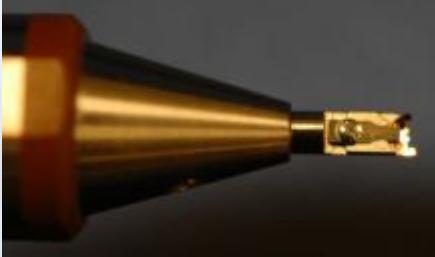
Advanced Microscopy Techniques Applied to Nanoparticles in Radiation Environments

Collaborators: S.H. Pratt & T.J. Boyle

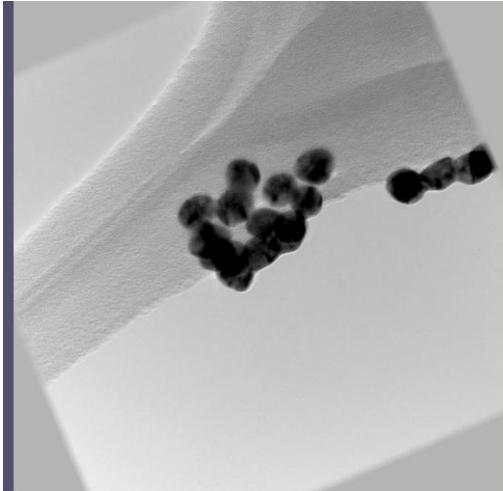
In situ Ion Irradiation TEM (I³TEM)



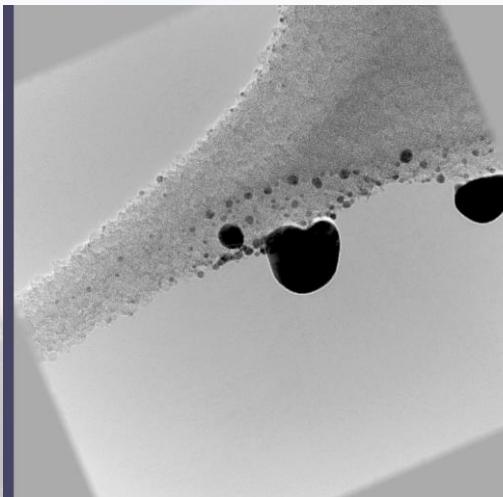
Hummingbird
tomography stage



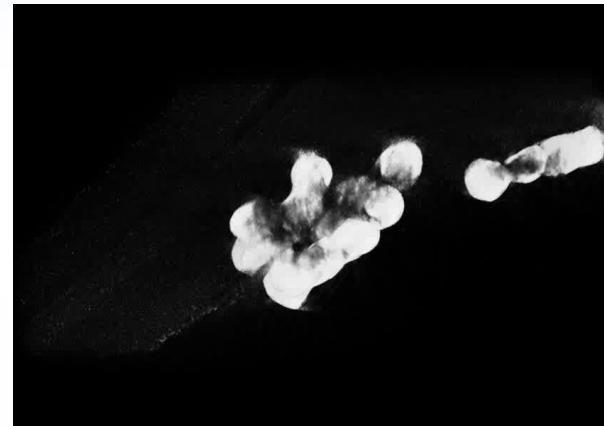
Aligned Au NP tilt series - unirradiated



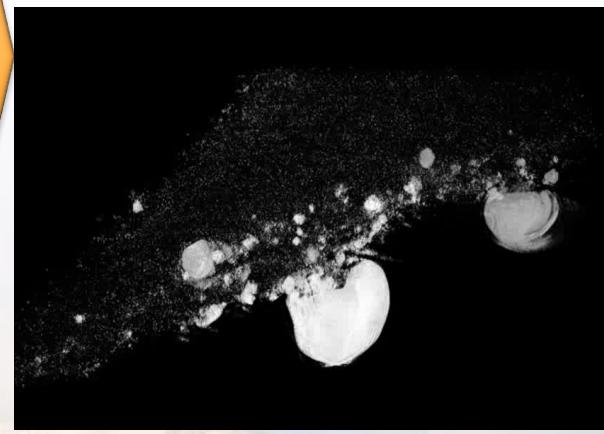
Aligned Au NP tilt series - irradiated



Unirradiated Au NP model



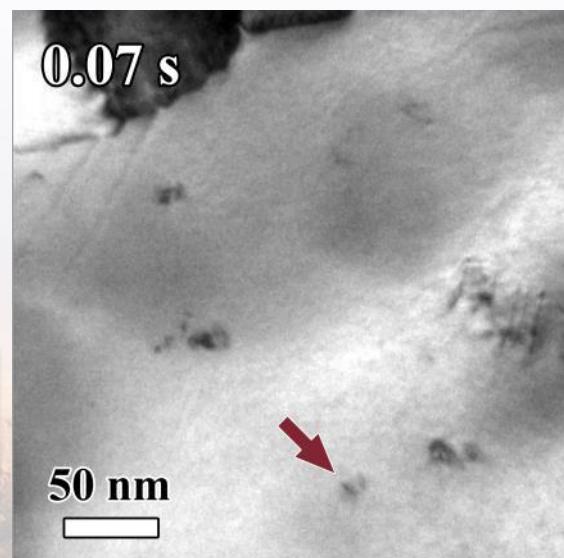
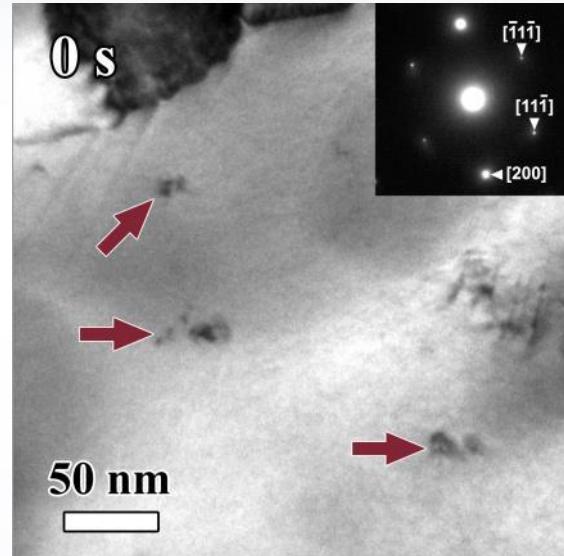
Irradiated Au NP model



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



Single 48 MeV Si Strikes into Au Thin Foil



Sped up 8x

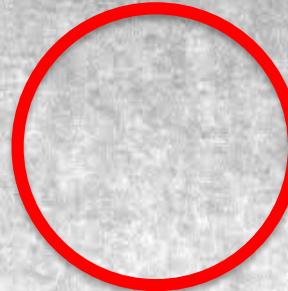
The majority of expected Si ion strikes are not observed. Those observed result in significantly smaller defect structures.



Demonstration of Single Ion Strikes in Si

30 nm

difference
image



- 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

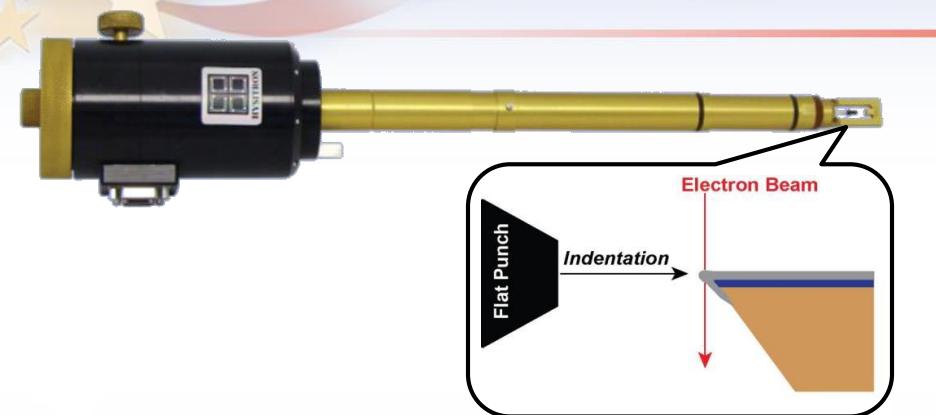
Can we go beyond this to observe:

- the important aspects of structural evolution as a function of time (ns to hrs.)?
- the evolution in more complex systems (GaAs)?
- Directly correlate it to key model parameters?



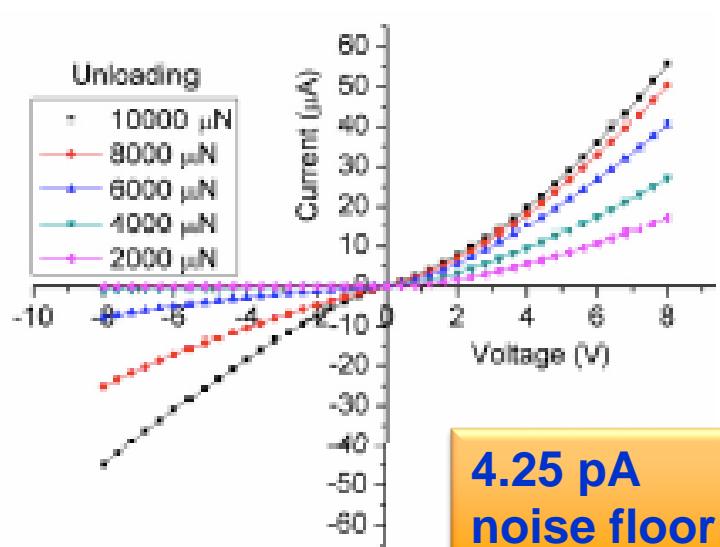
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Controlling the Sample through TEM Stage Upgrades



Range of Mechanical Testing Techniques

- Indentation
- Compression
- Tension
- Wear
- Fatigue
- Creep

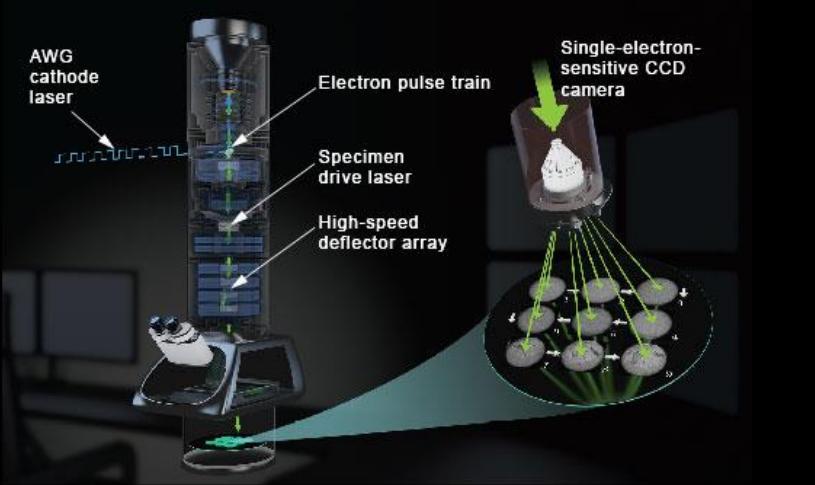


- Electromechanical upgrade to Hysitron PI-95
- Gatan double-tilt liquid nitrogen stage



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Can I³TEM and DTEM systems be combined?



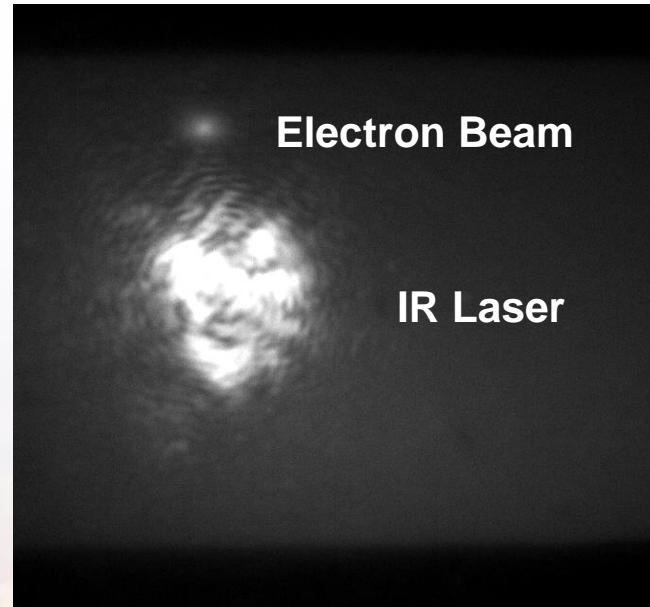
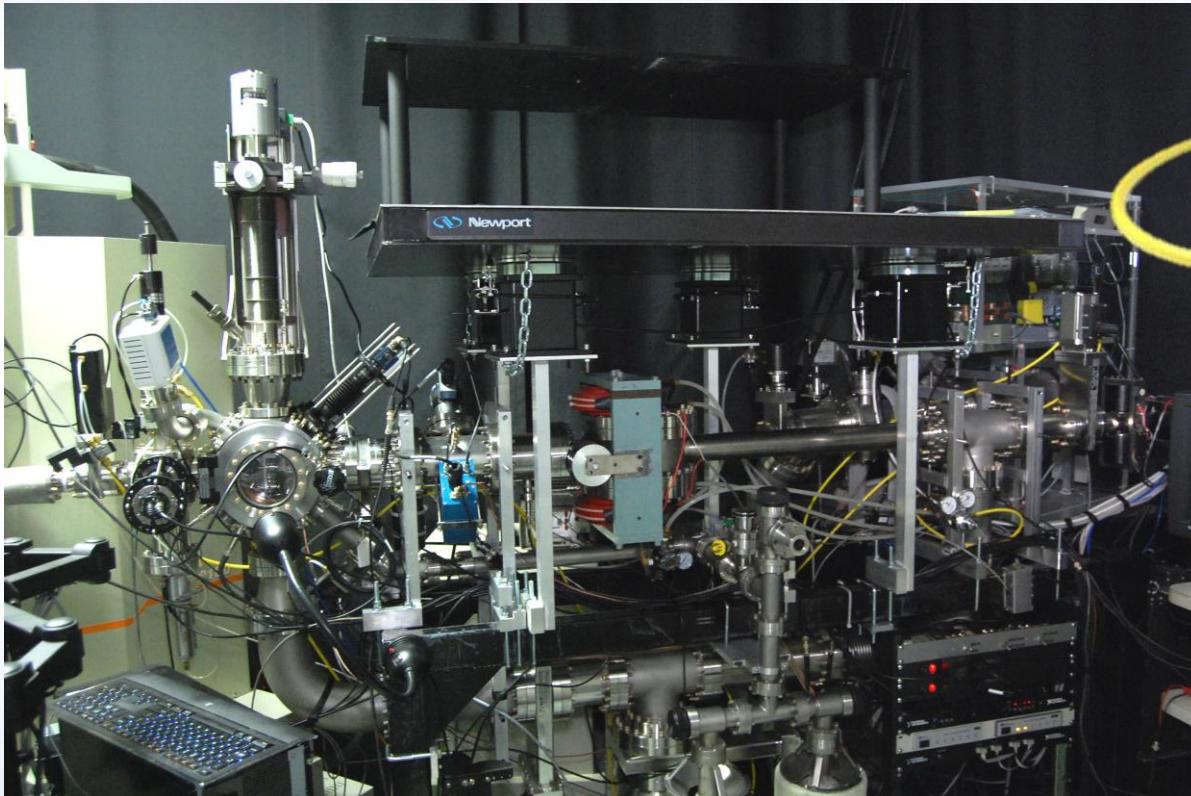
Goal:

To combine the state-of-the-art in microscopy developed at LLNL and SNL to elucidate the response of single radiation events and other extreme overlapping environments with adequate spatial and temporal resolution.



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Instillation and Operation of IR laser within the TEM



Both IR and UV lasers have been purchased, the optics are designed, the safety modifications and paperwork is complete, IR Laser is installed and operational for sample heating

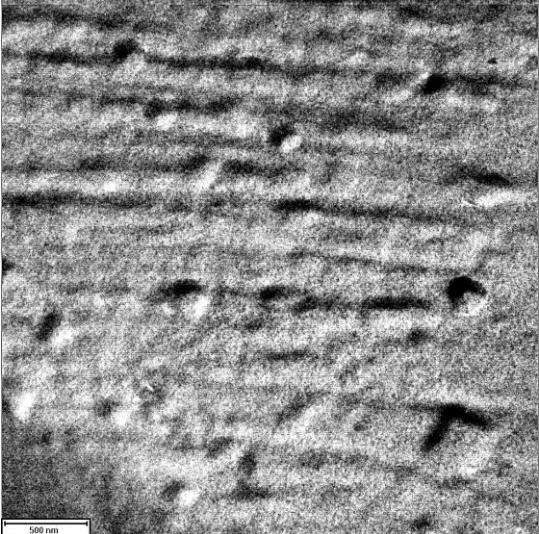


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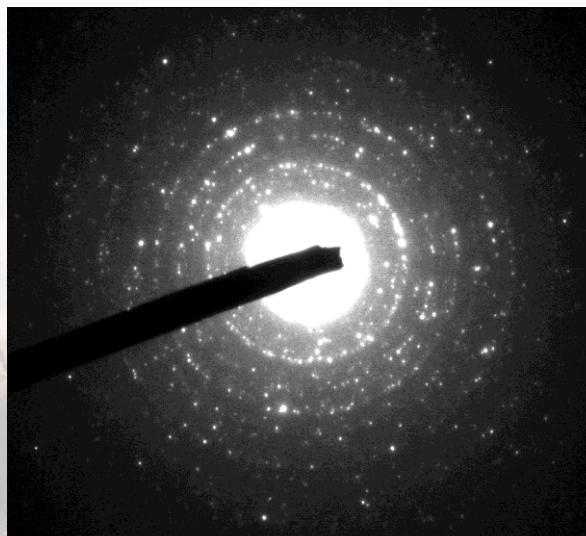
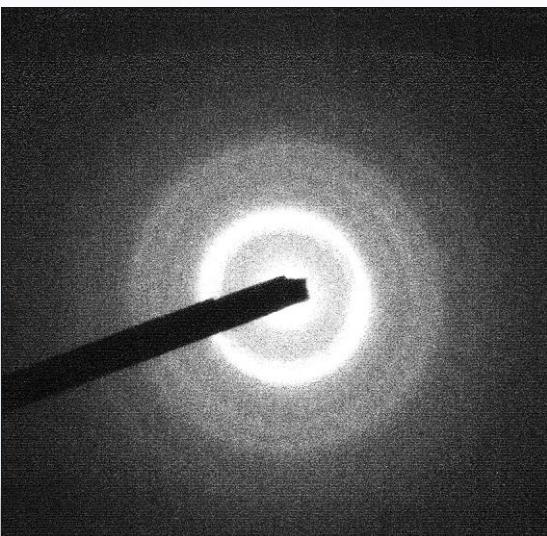
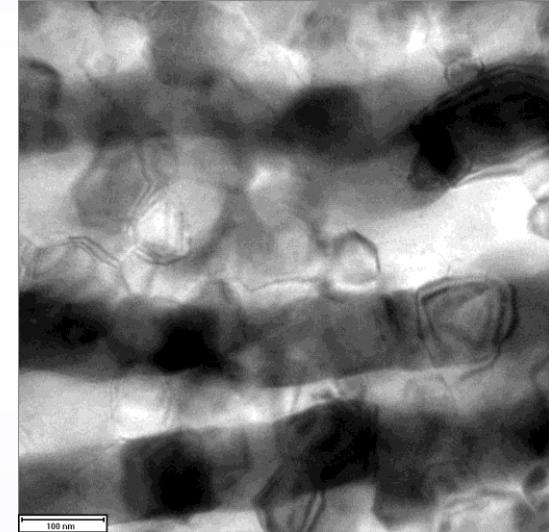
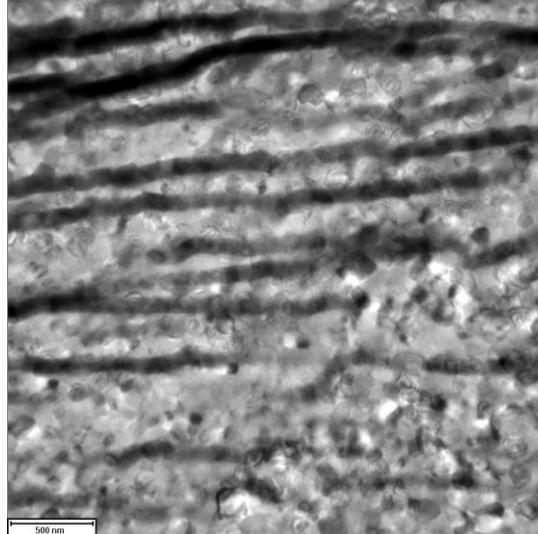
Laser Initiation of Multilayer Reactive Foils

Collaborator: for work on this slide

As Received Co/Al Multilayers



Foil After Laser Initiated Reaction



Laser Heating has been demonstrated 12/7/2016

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



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A Movie-Mode deflector to a conventional TEM can give you μ s-scale resolution with a standard camera

fs

ps

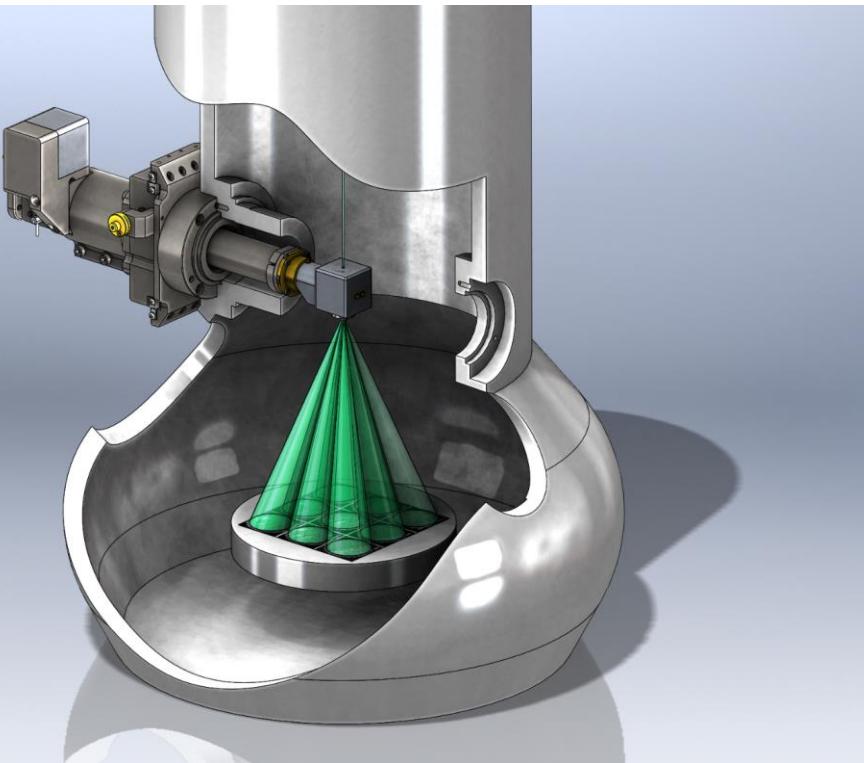
ns

μ s

ms

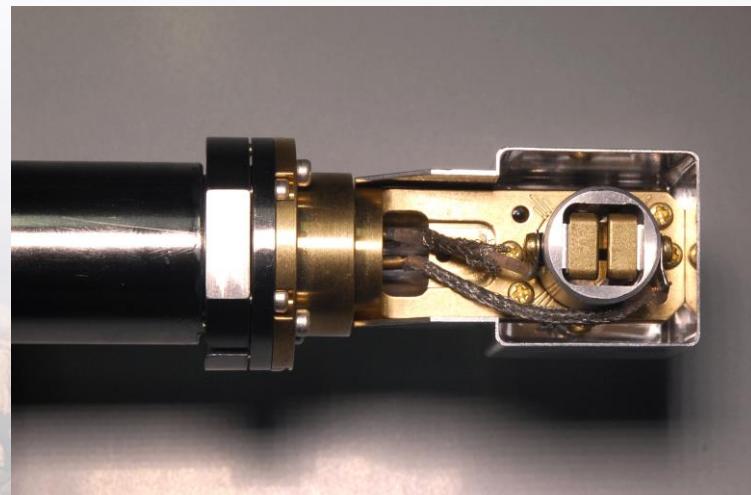
s

16 to ∞ Images/Event

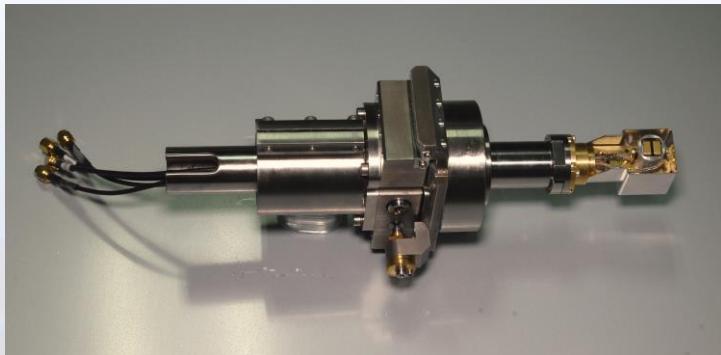
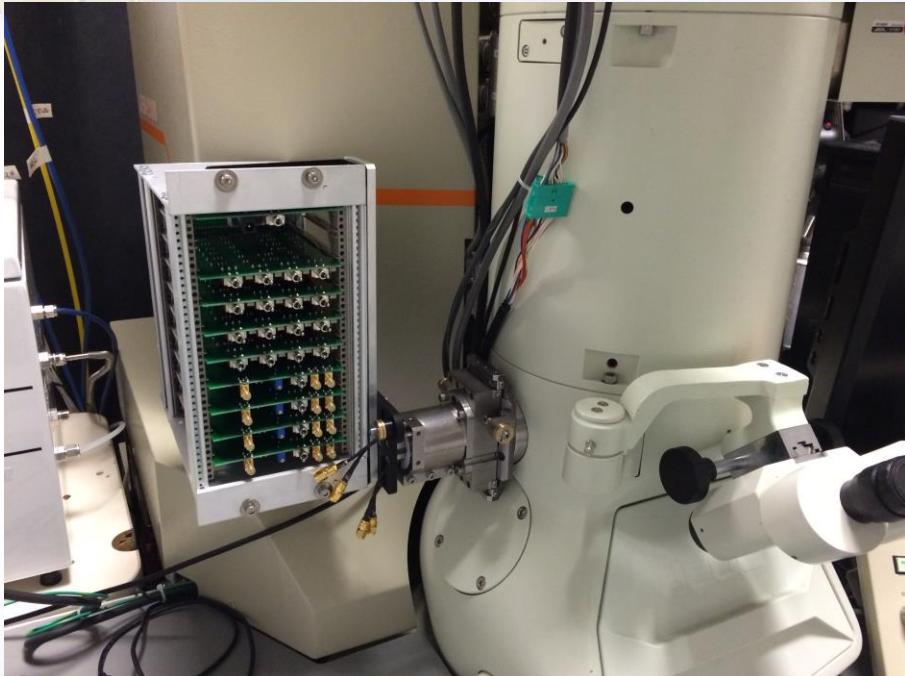


Can both Movie-mode and Photoemission source be done at the same time to further enhance the time resolution?

- 9-16 images per frame, spread over a large camera
- Any duty cycle up to the limits of the camera
- Ultimate limit is beam current/brightness



Status of the Movie-Mode Deflectors at Sandia



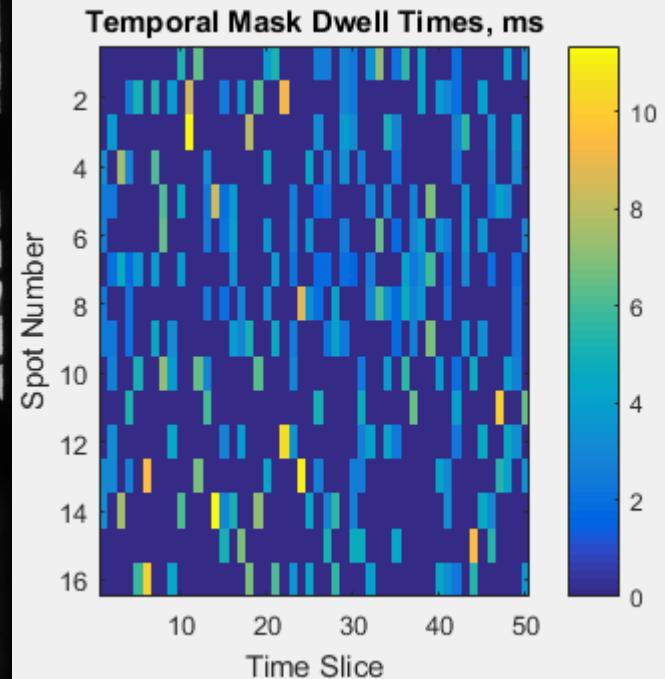
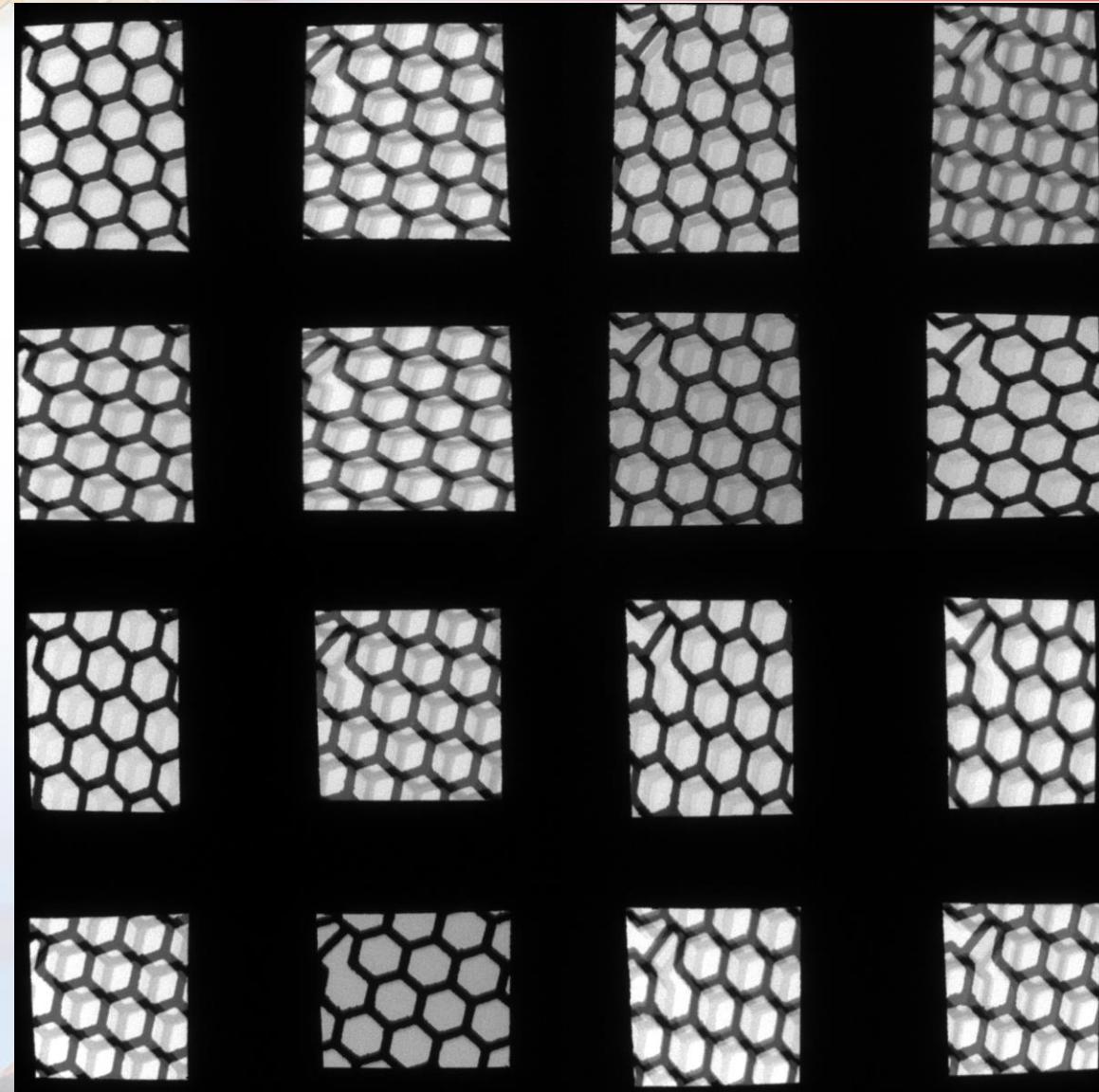
**Temporal compressive sensing works!
54 frames of 500 Hz TEM video from a
single camera acquisition**

This system was installed and functional at Sandia in July 2016.



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Demonstration of Movie Mode Deflectors

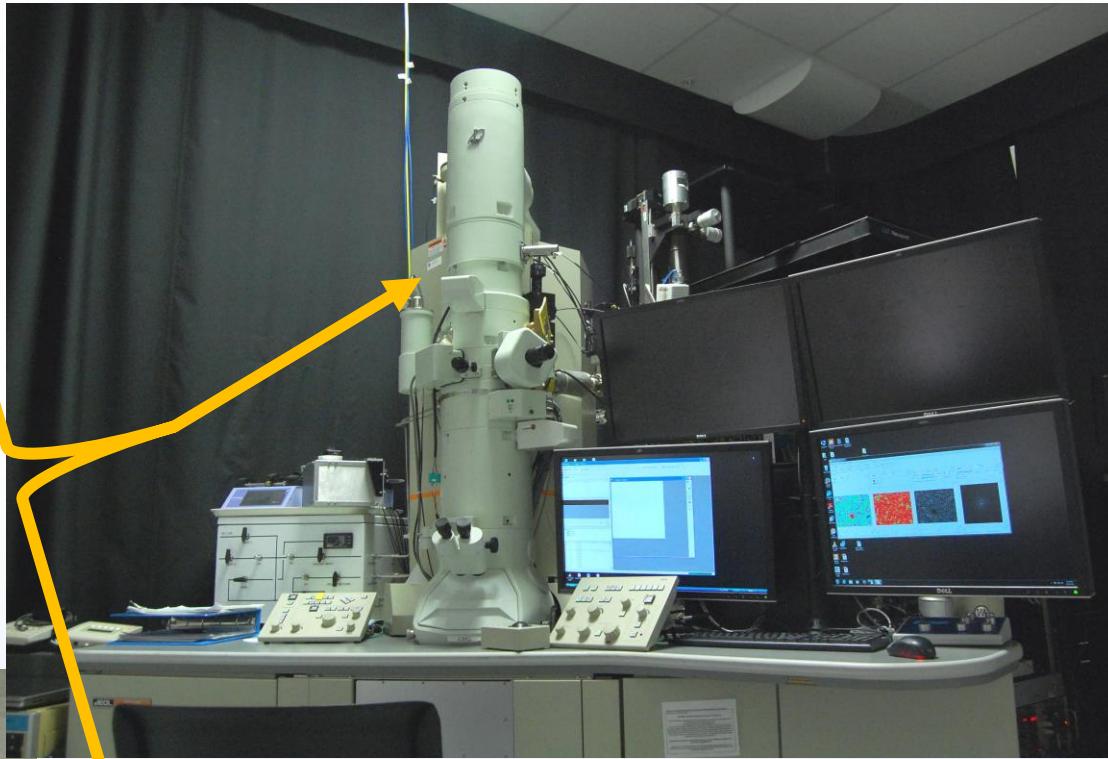
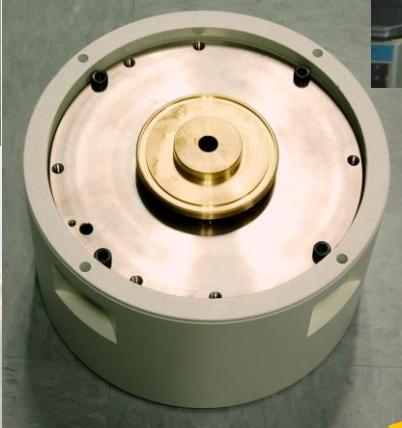


Demonstration of Relativity done
12/14/2016.



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Next step: Plans to Increase the TEM Brightness



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

An laser port, additional condenser lens, and robust cathode must be added to increase electron current.

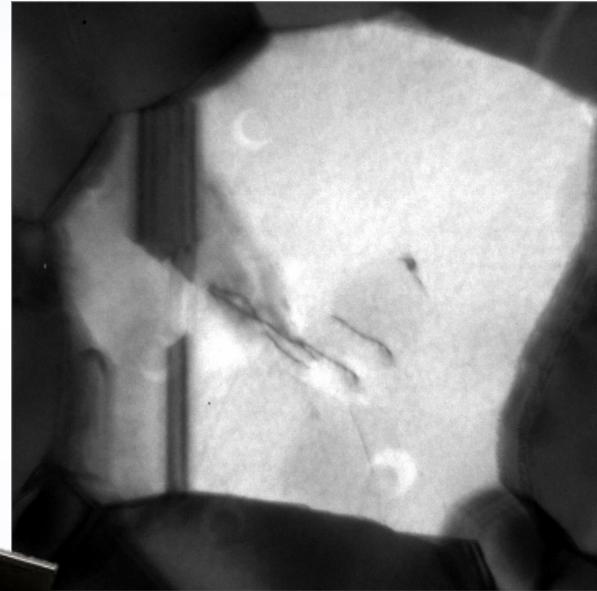
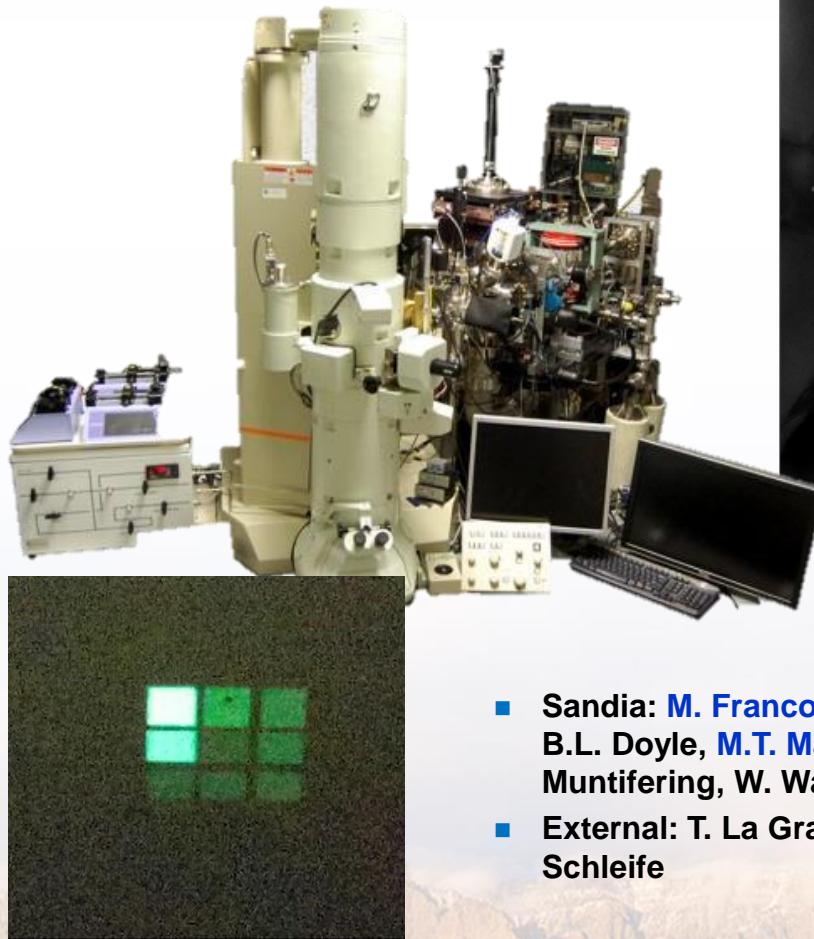


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Summary

1. Single ion strikes can be observed in relevant length scales utilizing I³TEM in:
 1. Range of metals
 2. Si
2. Relativity and IR laser heating systems are operational, but still needs to be optimized
3. C0 lens and Cathode laser are purchased and installs are planned for next year.
4. Can relevant time scales be achieved is still an outstanding question.



Collaborators:

- Sandia: [M. Franco](#), [M. Blair](#), [D. Buller](#), [G. Vizkelethy](#), [B.L. Doyle](#), [M.T. Marshall](#), [D. Saiz](#), [J.A. Scott](#), [B.R. Muntifering](#), [W. Wampler](#), [R. Dingreville](#), & [S. Foiles](#)
- External: [T. La Grange](#) [A. Lang](#), [M. Taheri](#), [W. Ji](#), [A. Schleife](#)



This work was partially funded by the Division of Materials Science and Engineering, Office of Basic Energy Sciences, U.S. Department of Energy. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.



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