SAND2019-9167C

Sandia National Laboratories

Initiation of Grain Growth Observed Using Ultrafast TEM Deflectors

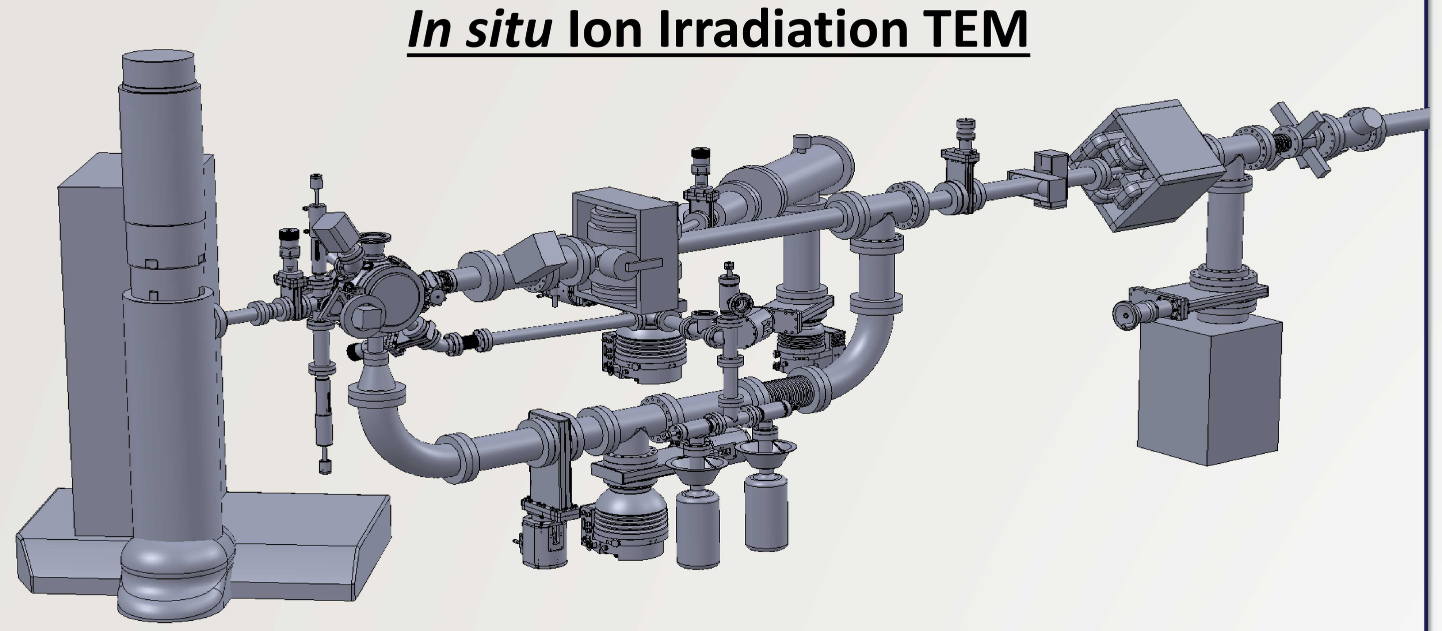
B.W. Reed[1], A.M. Monterrosa[2], A.A. Moghadam[1], R.S. Bloom[1], S.T. Park[1], S.A. Briggs[2,3], P.M. Price[2], S. Tutt[2], C.M. Barr[2], J.T. McKeown[4], D.J. Masiel[1], K. Hattar[2*]

[1]Integrated Dynamic Electron Solutions, Inc., [2] Sandia National Laboratories, [3]Oregon State University, [4]Lawrence Livermore National Laboratory

IDES INTEGRATED DYNAMIC ELECTRON SOLUTIONS

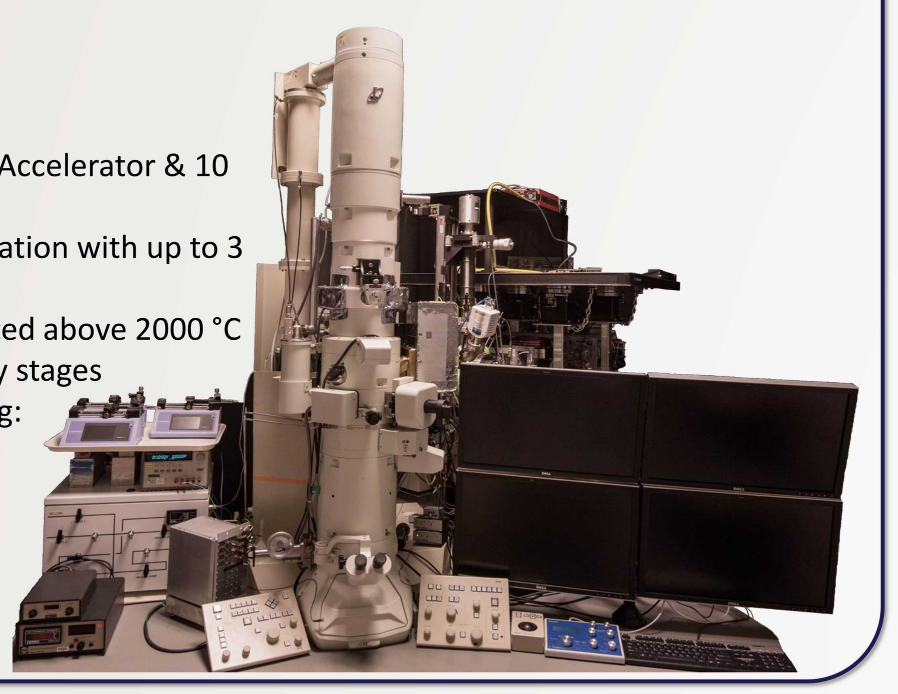
I[3]TEM Facility

In situ Ion Irradiation TEM



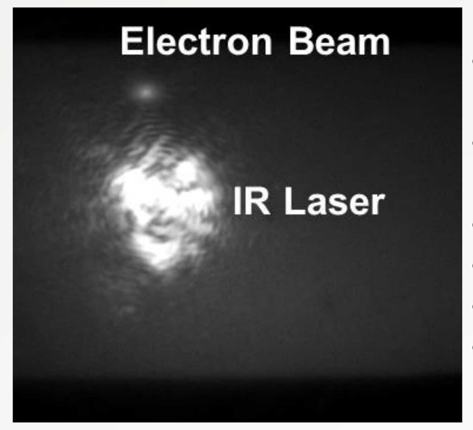
Capabilities

- A highly modified JEOL 2100 TEM
- Connected to a 6 MV HVE Tandem Accelerator & 10 kV Colutron
- Permits ion implantation and irradiation with up to 3 ion species at once
- Laser heating has been demonstrated above 2000 °C
- ±81° tilt and 4 electron tomography stages
- Over 15 in situ TEM stages including:
  - Quantitative Mechanical (PI-95)
  - Heating straining
  - Flowing liquid cell
  - Gas Heating
  - Cryogenic (77 K)
  - Heating (800 °C)



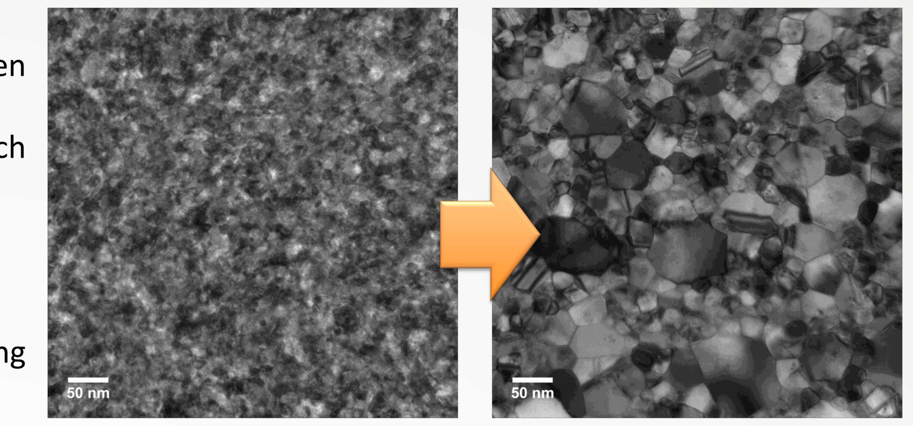
Grain Growth: Beyond the Temporal Limit of the Camera

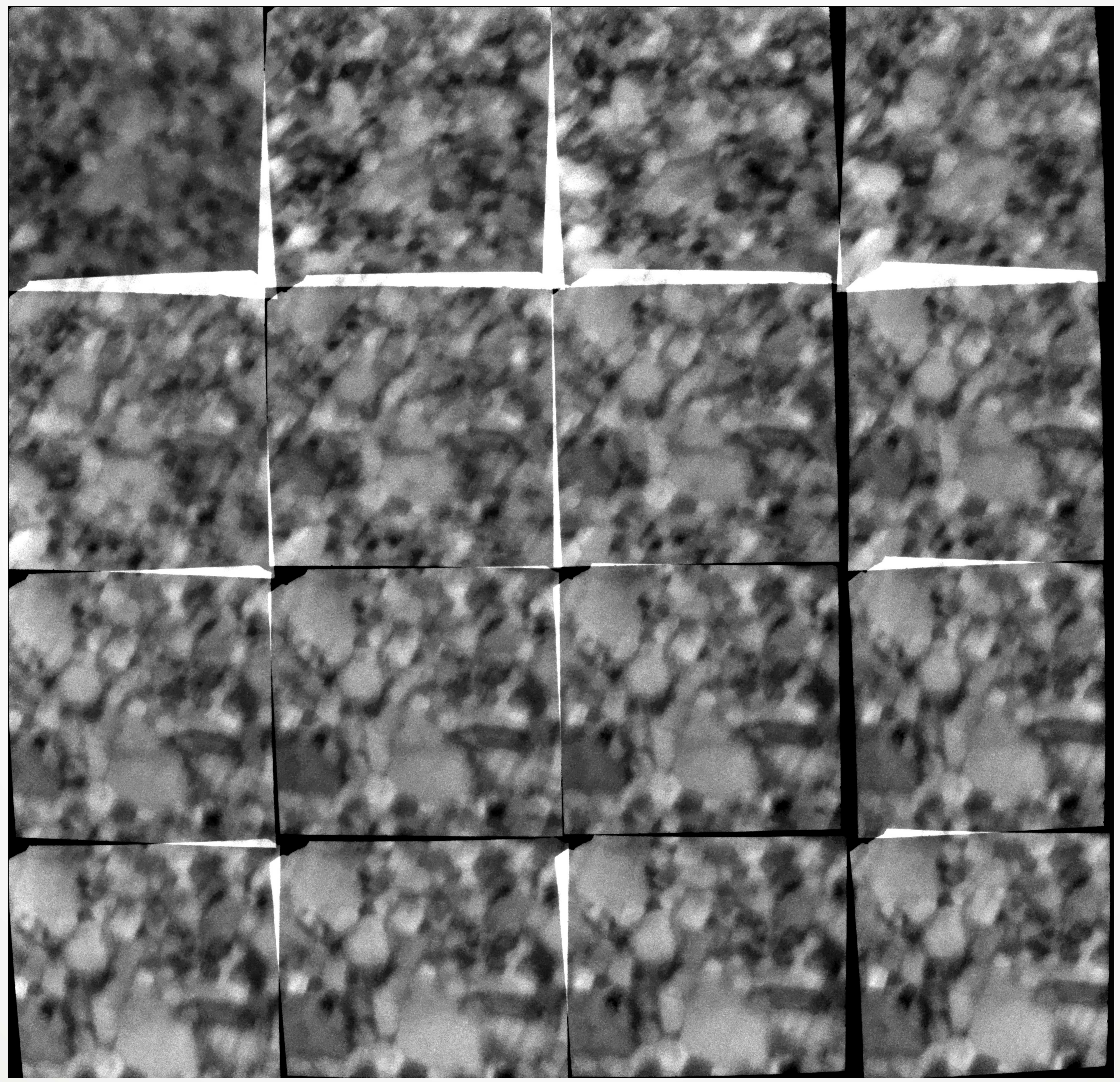
Laser Alignment



- Adjustable power 1064 nm infrared specimen (IR) drive laser
- IR laser is reflected directly onto the specimen with metal mirror
- 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

Before / After

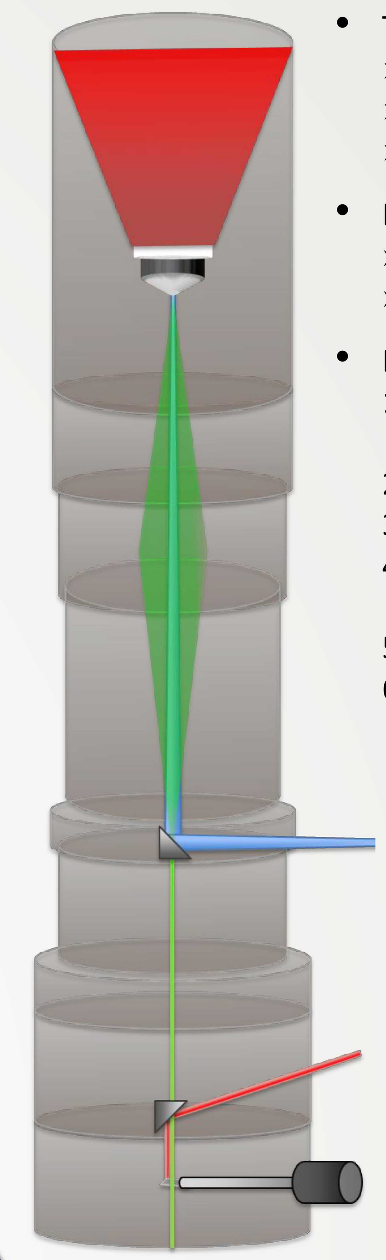


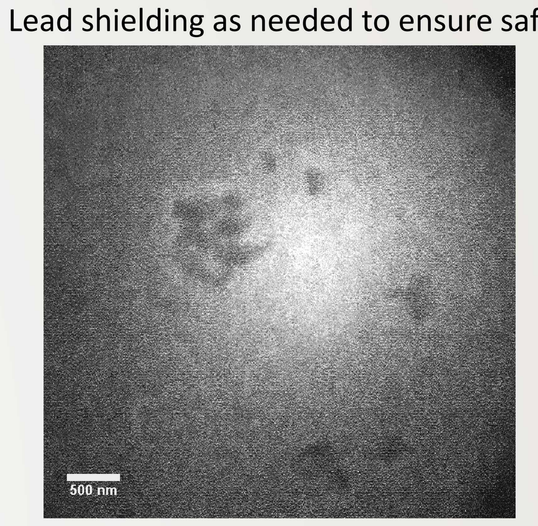


A single 4k x4k exposure → 16 images → 100 frame movie

DTEM Development

- Thermal emission microscope:
  - Not enough electron current for nanosecond imaging
  - Only a few electrons pass through sample in a nanosecond
  - 10^6 electrons are needed for image formation
- Dynamic transmission electron microscope (DTEM):
  - Utilizes a laser to achieve photoemission of electrons
  - Much greater electron current than a traditional LaB_6 filament
- DTEM conversion components using a JEOL 2100 HT:
  1. Ultraviolet laser and optics system capable of producing nanosecond pulses
  2. Adjustable molybdenum mirror to reflect the UV laser up the column
  3. Tantalum cathode disc filament
  4. Addition of a C_0 lens 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





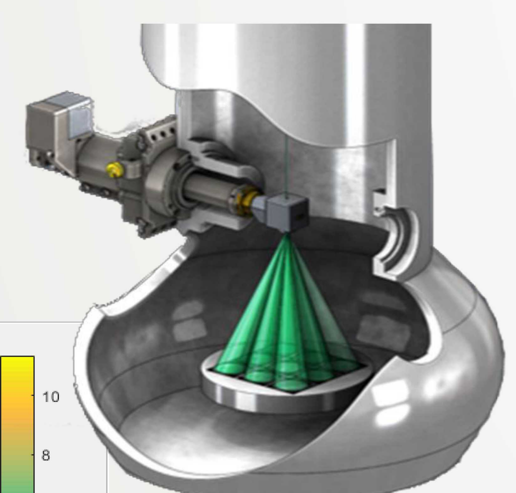
DTEM image of P47 particles with 6 ns pulsed UV laser

Our First DTEM Shot July 28, 2017!

Electrostatic Subframing

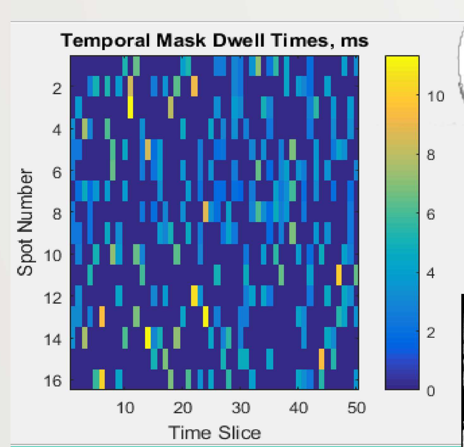
Hardware

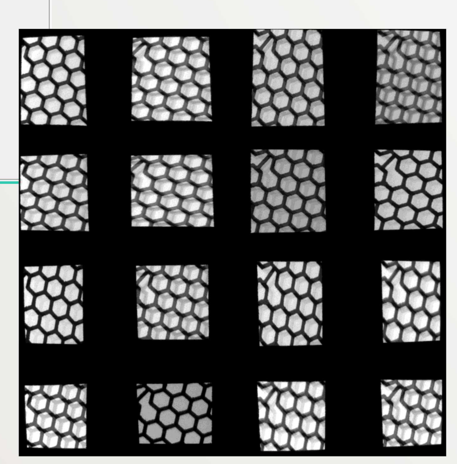
- Fast 2D electrostatic lens
  - Inserted below the projector lens
  - Retractable
  - Square limiting aperture



Software

- Can operate in 1-to-1 mode
- Compressive Sensing:
  - Digitally segmented and aligned
  - One camera pixel in overlapped region informs two or more output video voxels
  - Up to 15 frames per camera acquisition





Combined this converts the classical green screen into a programable disco floor!

* Ask to see video examples

Future Directions

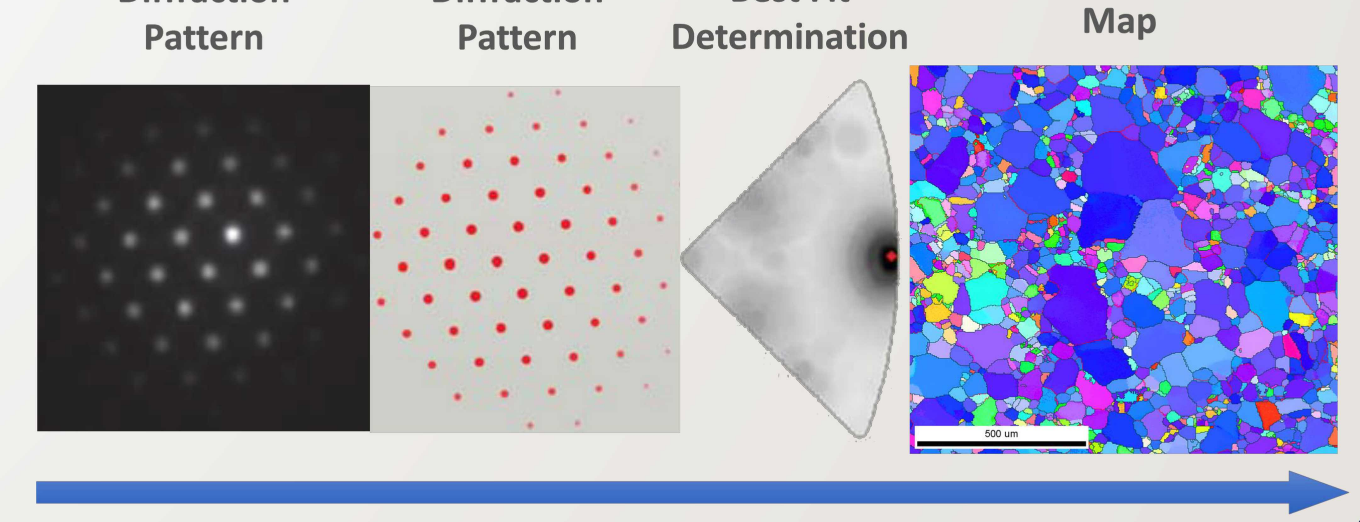
Couple to Other Experiments

- Utilize Electrostatic Subframing for:
  - Single-ion strike experiments
  - Radiation defect-defect interactions
  - High cycle fatigue tensile testing
- Couple with:
  - Automated Crystal Orientation Mapping
  - Electron Tomography
  - EDS and EELS chemical mapping

Couple to Models

- Couple to simulated electron diffraction patterns and micrographs
  - Subsequent simulated ACOM and virtual BF and DF maps
  - Directly relate to Molecular Dynamic or Mesoscale modeling
  - Approaching the spatial and temporal limits of MD simulations

Experimental Diffraction Pattern | Calculated Diffraction Pattern | Statistical Best Fit Determination | Crystal Orientation Map



NSUF User Facility

Access to the I[3]TEM can be granted through the Nuclear Science User Facilities rapid turn around proposal process. https://nsuf.inl.gov/Page/calls

This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. DOE's National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.

CINT User Facility

Access to the I[3]TEM can be granted through the Center for Integrated Nanotechnologies proposal process. Proposal submissions are open twice a year in the spring (April 1st) and in the fall (September 1st). http://cint.lanl.gov