

Accelerating Interface Science Through NREL's New Electron Microscopy Suite

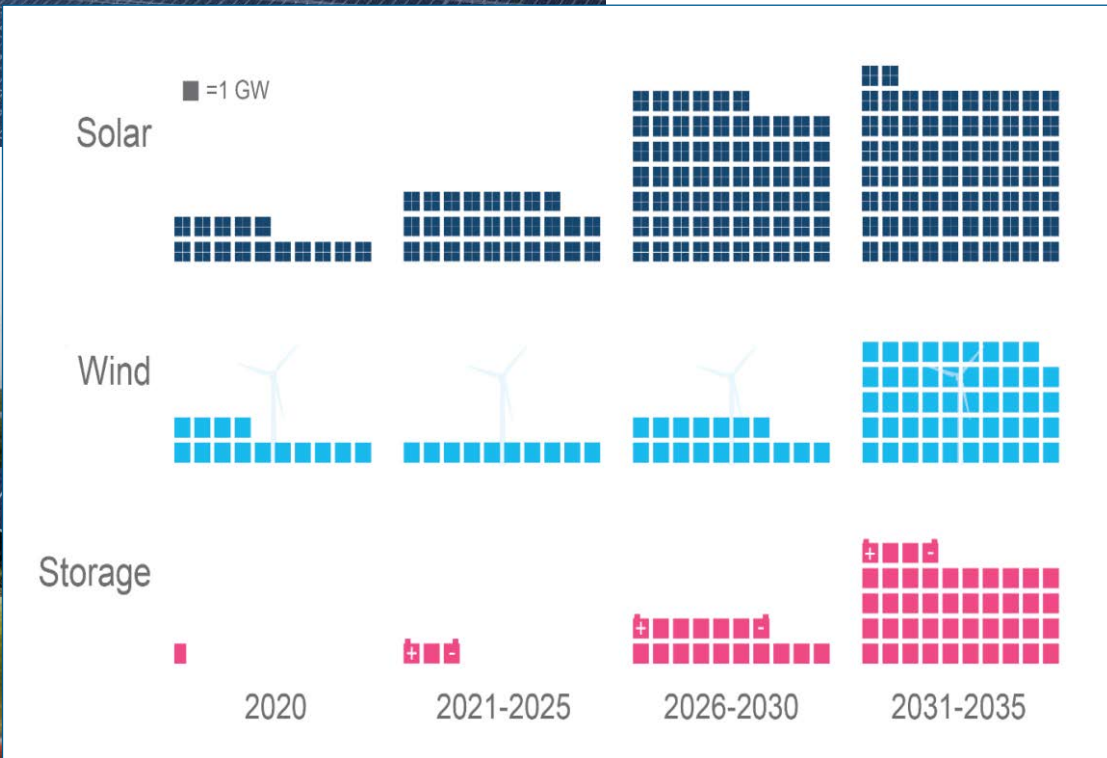
Steven R. Spurgeon

Senior Materials Data Scientist
Analytical Microscopy and Imaging Sciences

July 28th, 2024

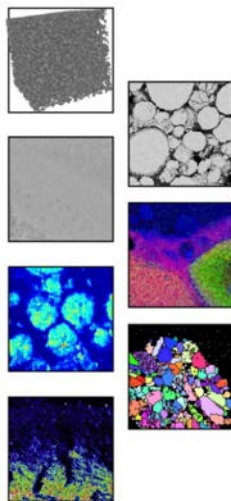


Our Mission: Clean Energy Generation & Storage

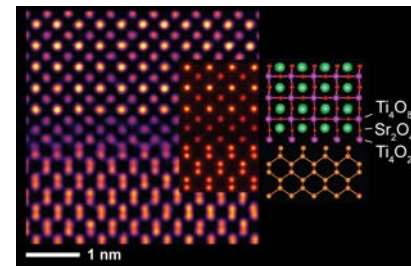
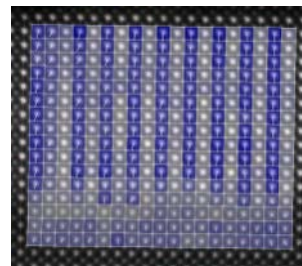
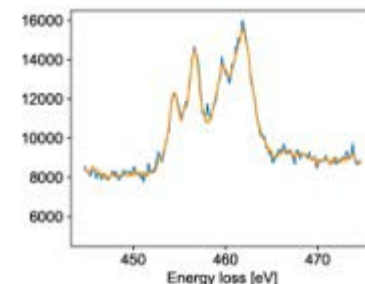
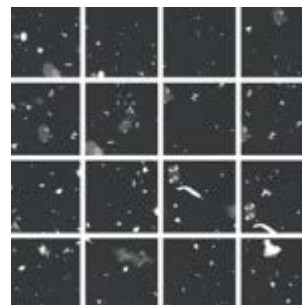




New Capabilities for Multiscale Electron Microscopy

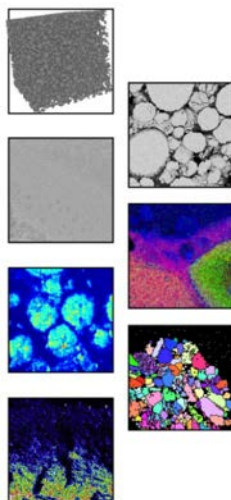


Toward Artificial Intelligence Guided Interface Science

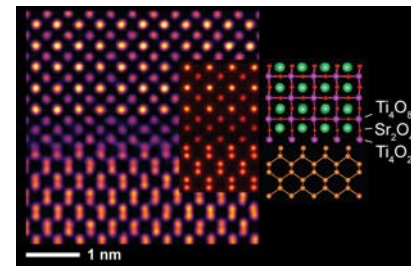
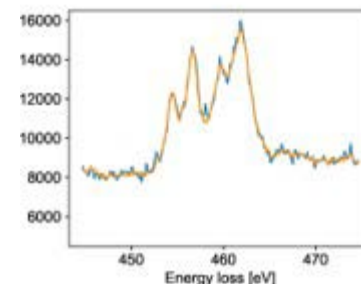
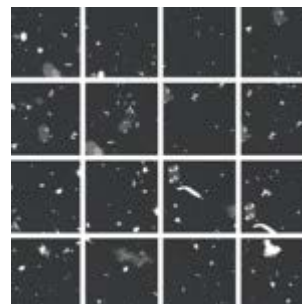




New Capabilities for Multiscale Electron Microscopy

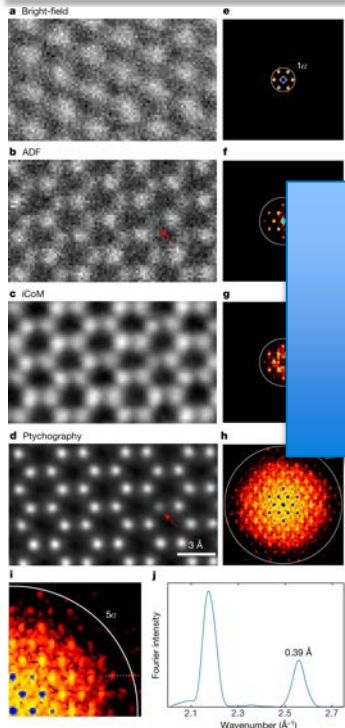


Toward Artificial Intelligence Guided Interface Science



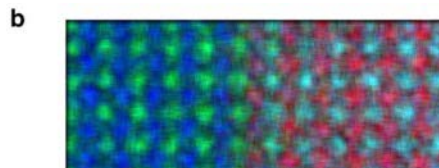


Imaging @ 0.39 Å

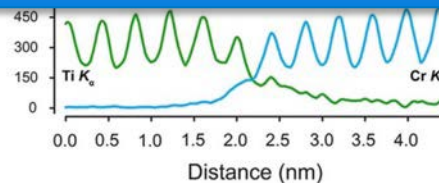


Jiang et al. Nature 559, 343 (2018)

Composition @ Atomic

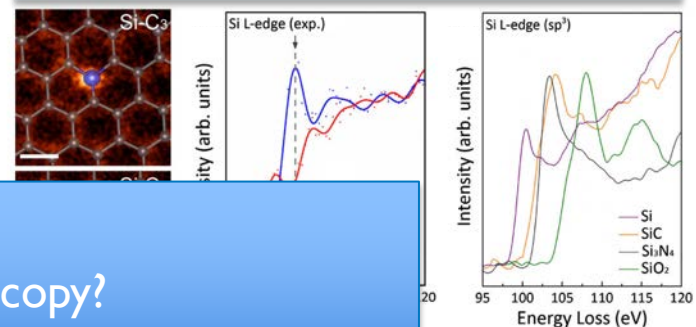


Why electron microscopy?
Local, direct analysis down to the atomic scale



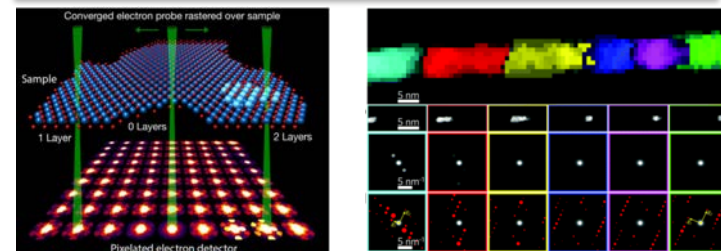
Spurgeon et al. Microsc. Microanal. 23, 513 (2017)

Bonding @ near Atomic Resolution



Phys. Rev. Lett. 109, 206803 (2012)

Imaging @ 2 nm

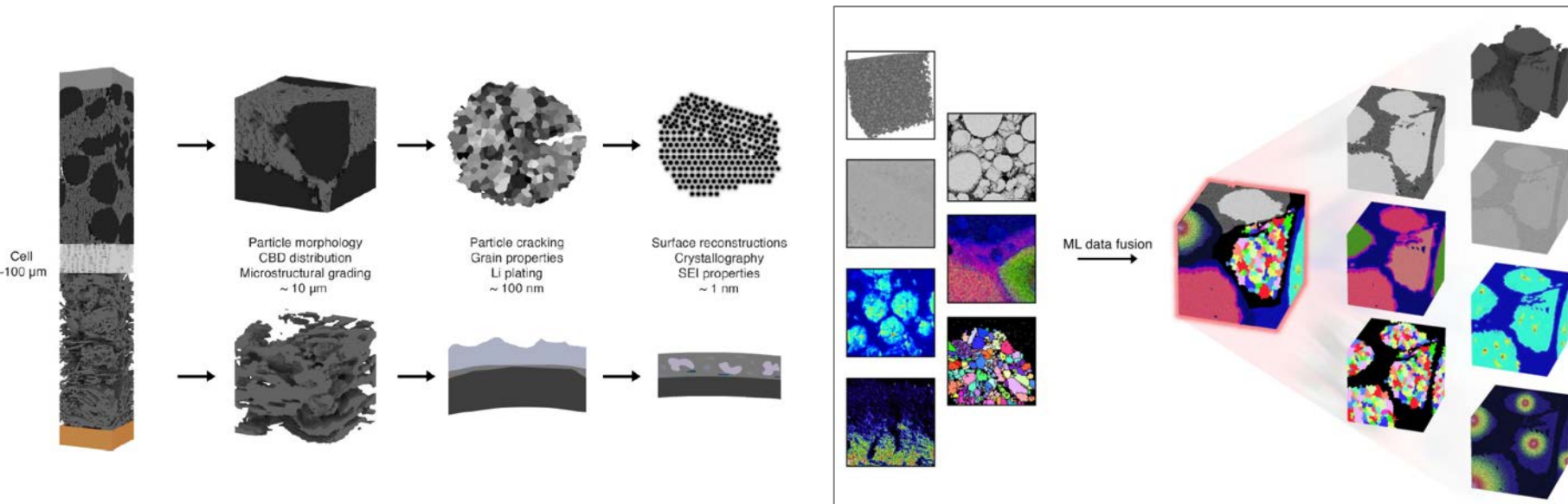


Ophus et al. Microsc. Microanal. 25, 563 (2019) Londoño-Calderon et al. Nanoscale. 13, 9606 (2021)





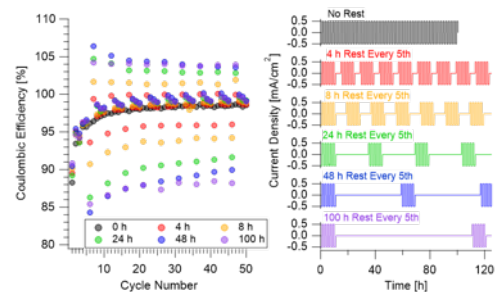
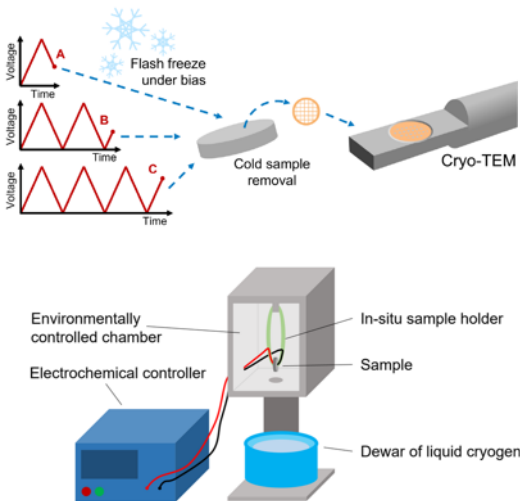
Multimodal Microscopy for 3D Understanding of Systems/Materials



Finegan et al., ACS Energy Lett. 7 (12), 4368 (2022)



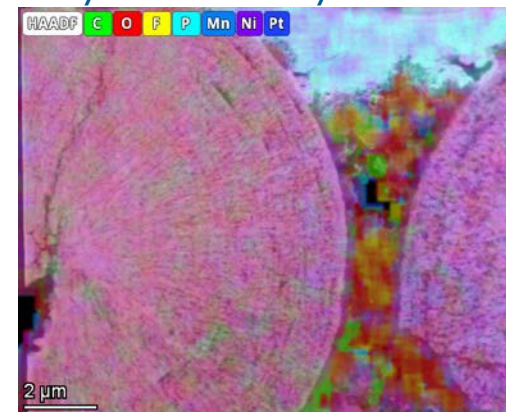
Operando Electrochemical Freezing Cryo EM



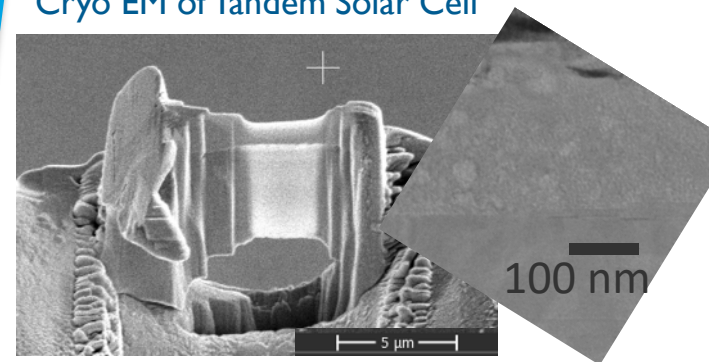
Unique Value

- Freeze-in 'active' biased states for interfacial analysis
- Cryogenic state enables high-resolution structural and chemical mapping of beam sensitive interfaces
- First demonstration from system-level materials, of an entire coin cell

Cryo EM of Battery Electrodes



Cryo EM of Tandem Solar Cell

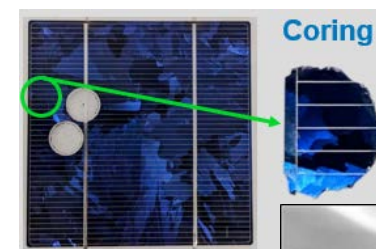




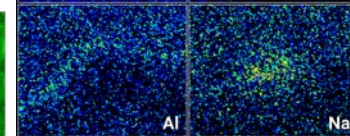
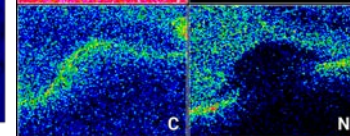
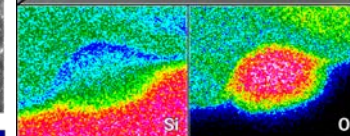
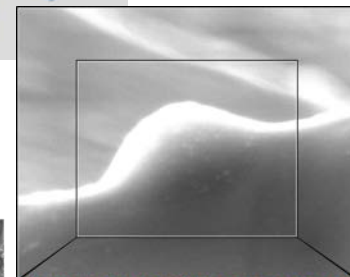
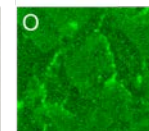
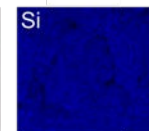
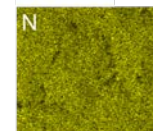
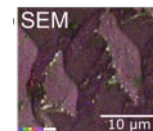
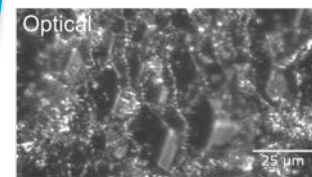
Tescan Solaris Ga-FIB
10's μm cross-sectional milling

Unique Value

- Precision milling with ion beam for site-specific cross-sectional imaging and mapping
- Cryogenic state enables sample preparation of beam sensitive interfaces
- Cryogenic preparation of samples for scanning transmission electron microscopy



X-ray Mapping of Degradation on Solar Cells





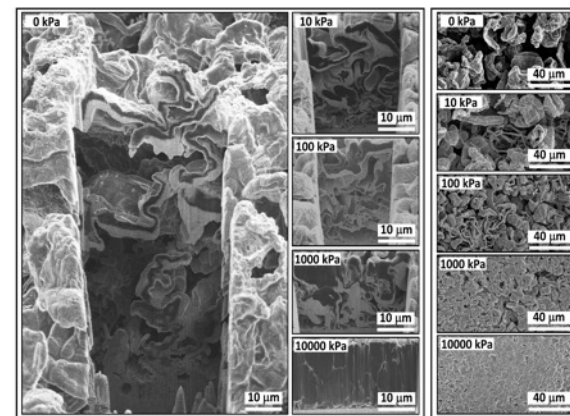
Unique Value

- Large-area milling with various ion beams for site-specific cross-sectional imaging and mapping
- Cryogenic state enables sample preparation of beam sensitive interfaces for scanning transmission electron microscopy
- Slice-n-view allows 3D reconstruction over large-areas of composites



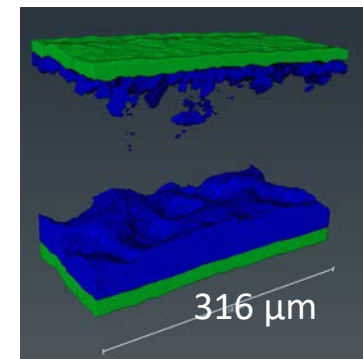
Helios Plasma FIB

100's μm cross-sectional milling

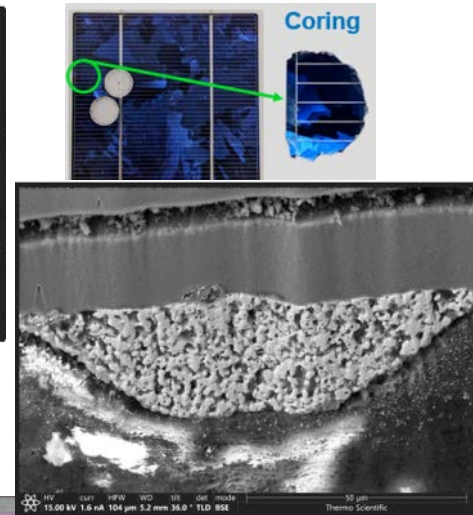
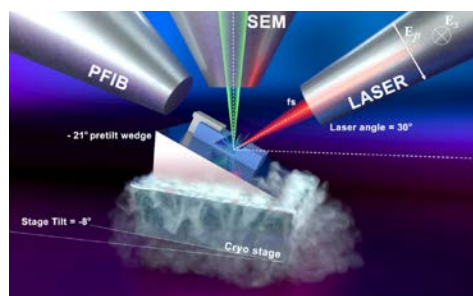
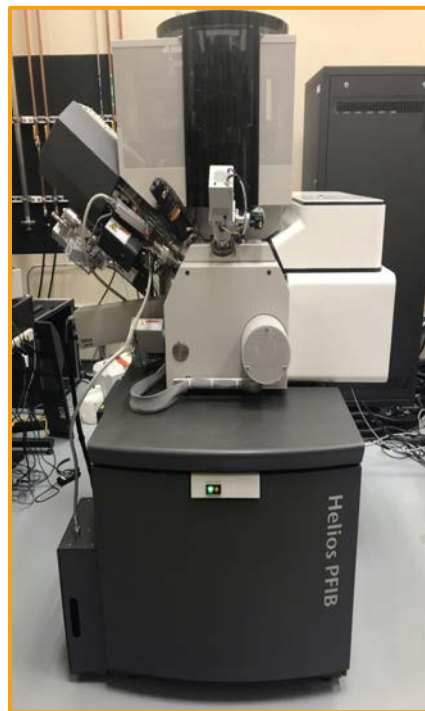


Harrison et al., ACS Appl. Mater. Interfaces 13, 31668 (2021)

Li Metal Morphology for Batteries

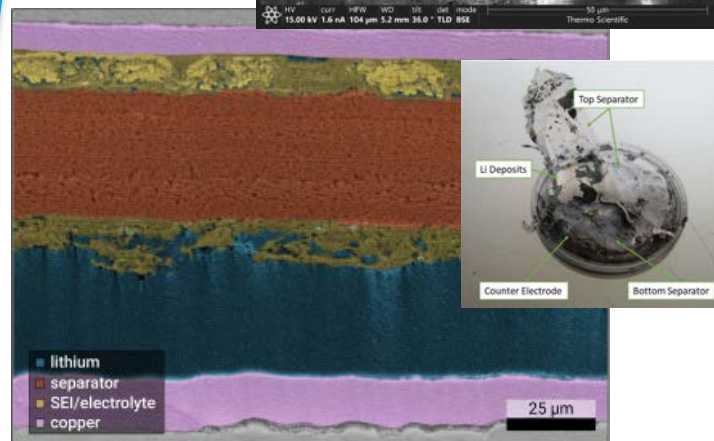


R. Gannon et al., (2024) In Preparation



Unique Value

- System-level milling fs-laser and ion beams for site-specific cross-sectional imaging, compositional mapping, and 3D reconstructions
- Cryogenic state enables sample preparation of beam sensitive materials

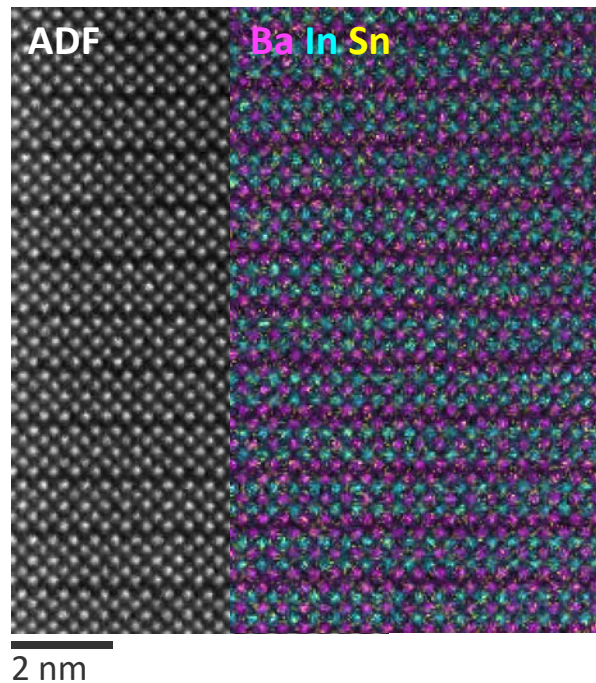


Helios Laser Plasma FIB
10 – 4,000 μm cross-sectional milling
fs laser mills 15,000x faster than Ga-ion FIB

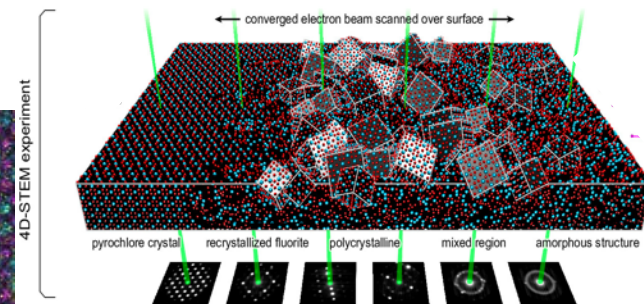


Thermo Fisher Spectra 200
Atomic-scale imaging,
compositional, and strain analysis

Sn Doped $BaIn_2O_4$

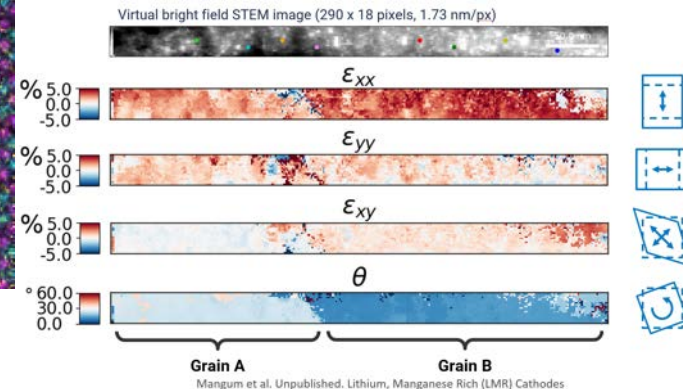


4D – Scanning Diffraction Analysis



Savitzky et al. *Microscopy and Microanalysis*, 27, 4 (2021)

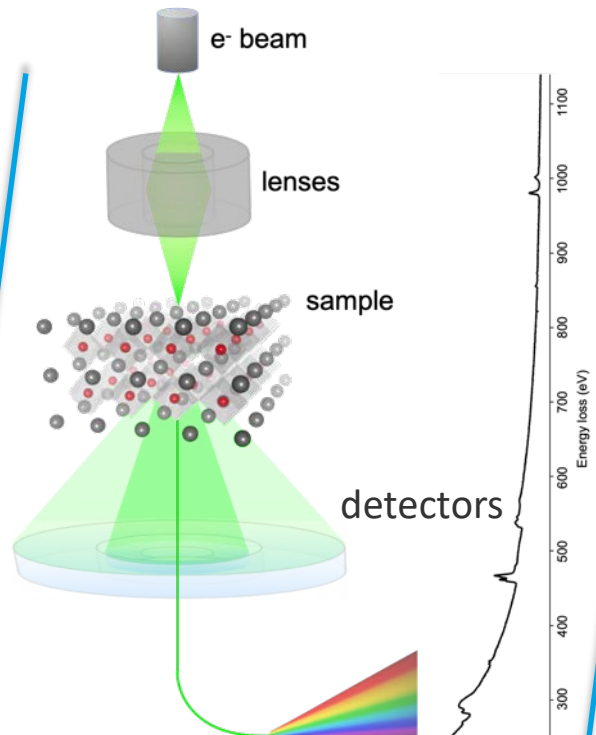
Nanoscale Strain Map Across a Grain Boundary



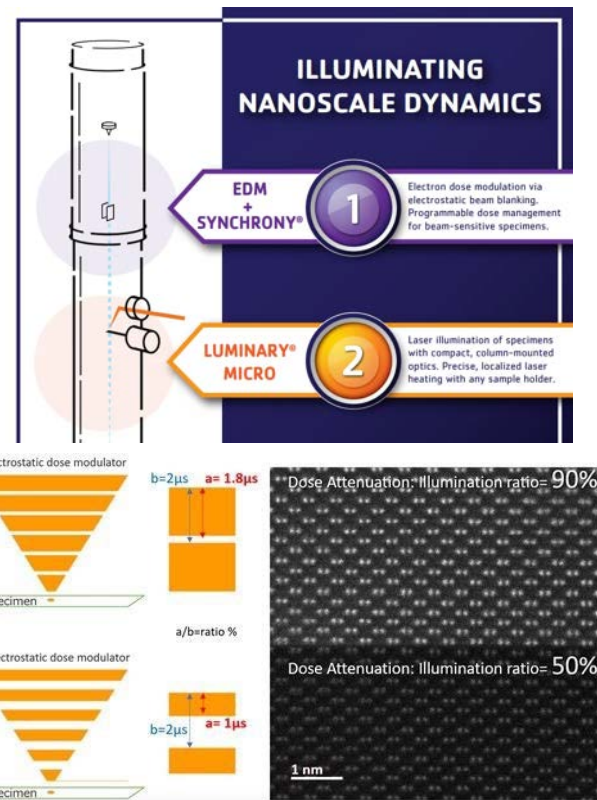
Mangum et al. Unpublished. Lithium, Manganese Rich (LMR) Cathodes



JEOL GrandARM 2
*Atomic-scale Imaging and
In-situ Reaction Monitoring*



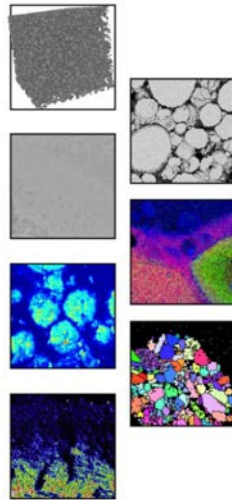
- High-speed imaging and in-situ spectroscopy (valence states & bonding)



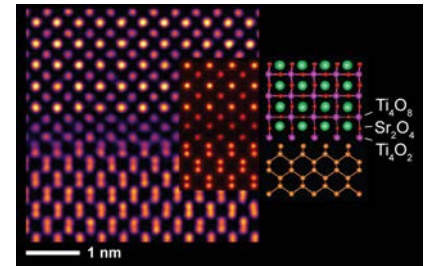
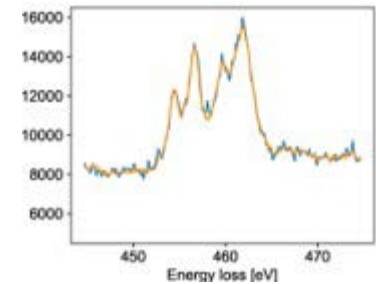
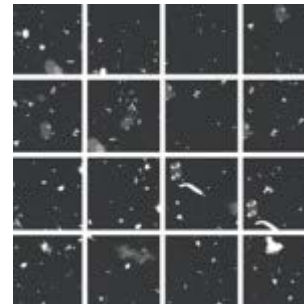
- Dose control for beam sensitive materials & light-induced reactions



New Capabilities for Multiscale Electron Microscopy

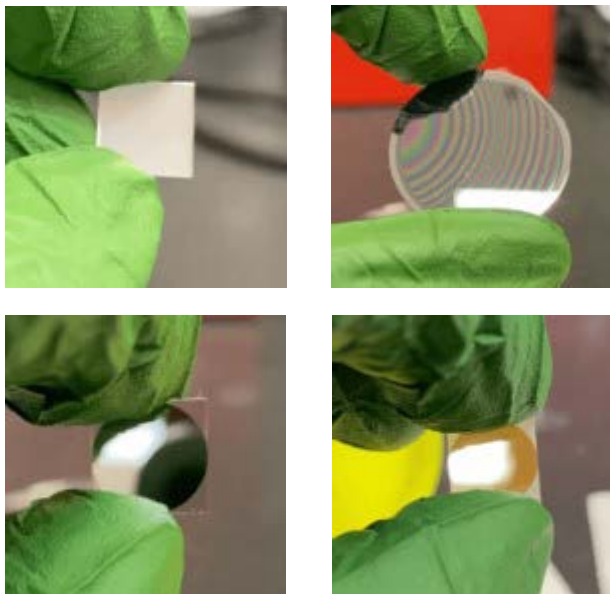


Toward Artificial Intelligence Guided Interface Science

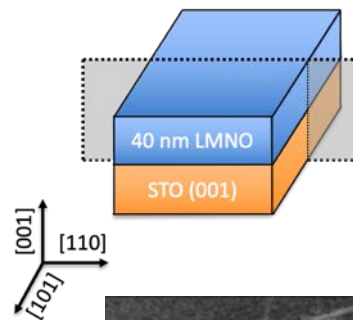


Understanding synthesis products requires direct local probes of structure and chemistry.

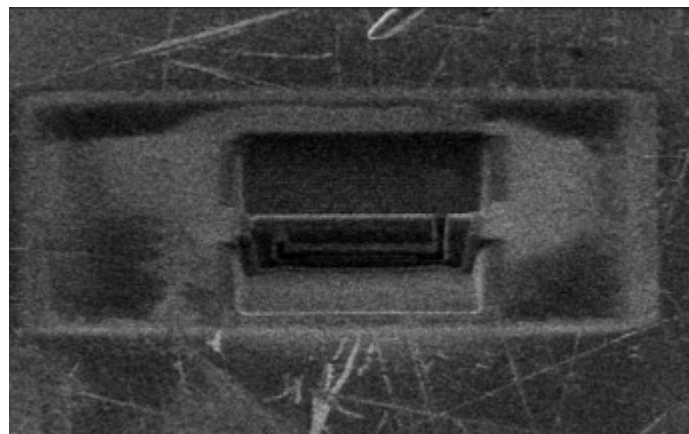
Functional Thin Films



~1 cm



Site Specific
Metrology

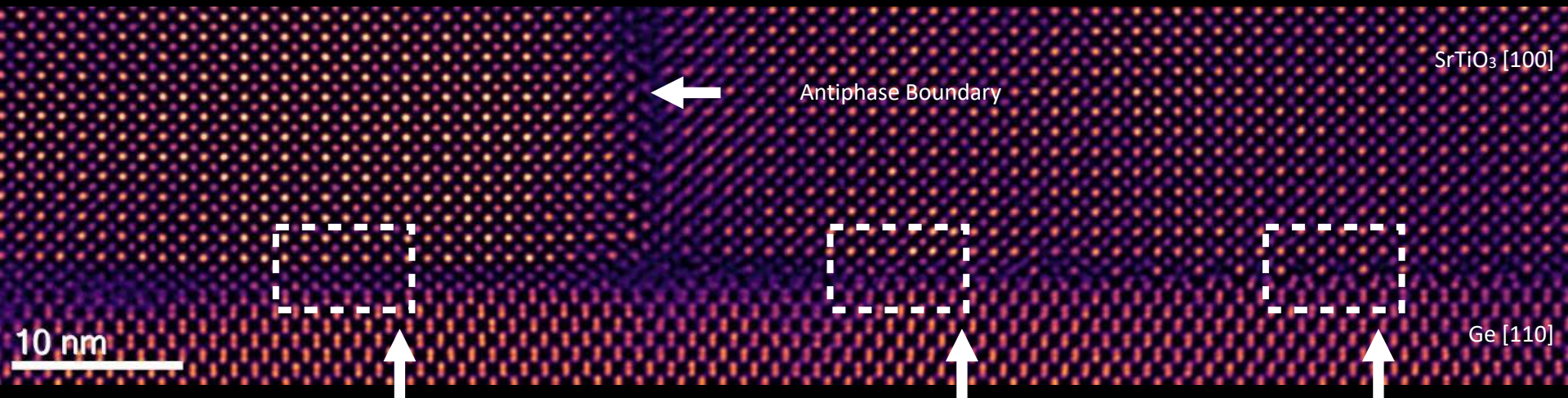


Focused Ion Beam



STEM

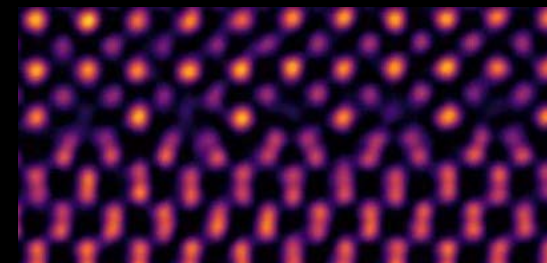
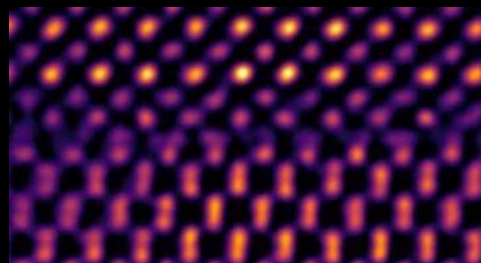
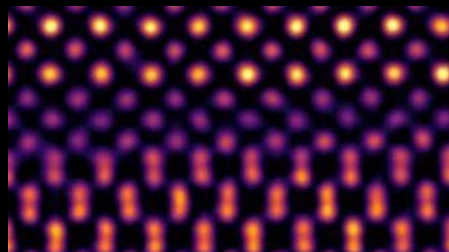
We can directly resolve interfaces in epitaxially integrated oxides and semiconductors.



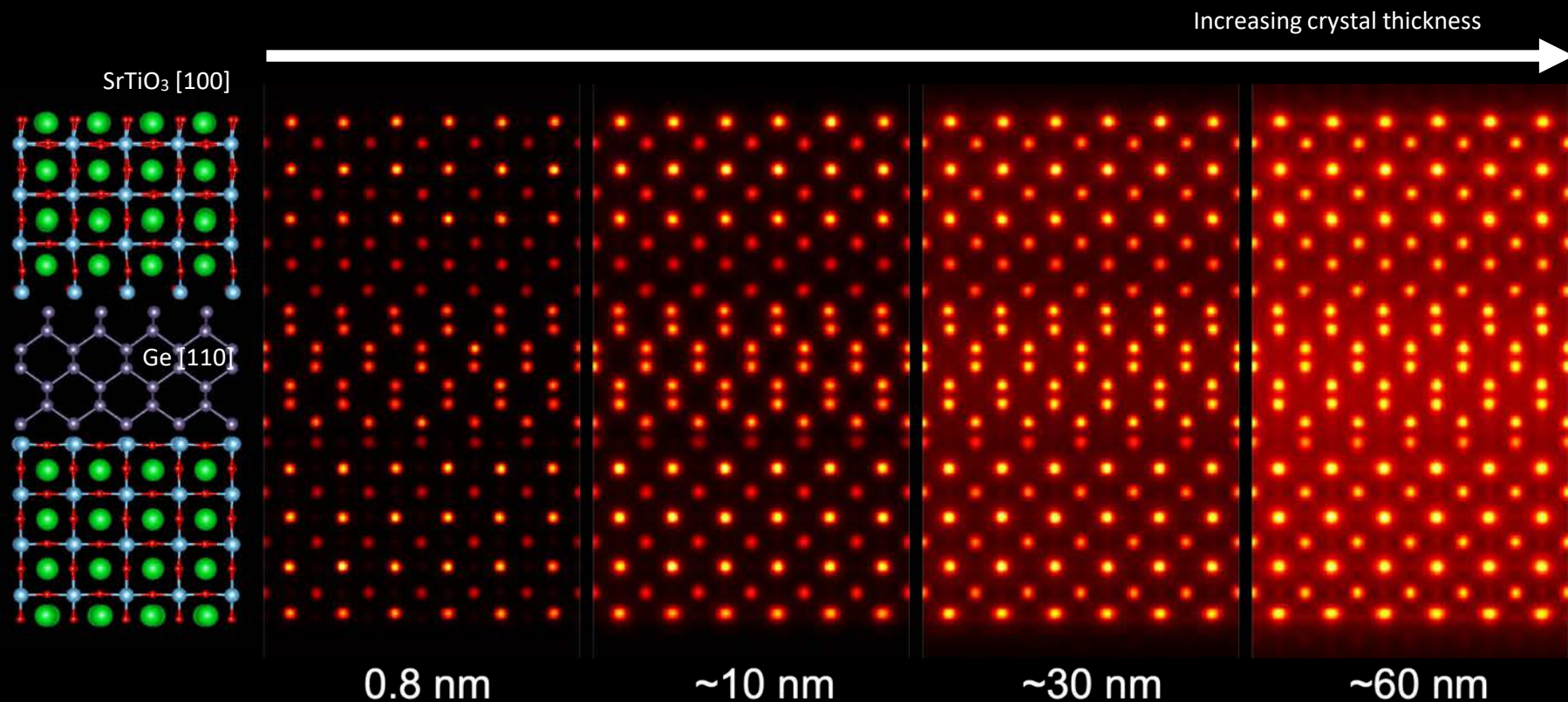
1 x 1 Reconstruction

1 x 1 "Twisted" Reconstruction

2 x 1 Reconstruction

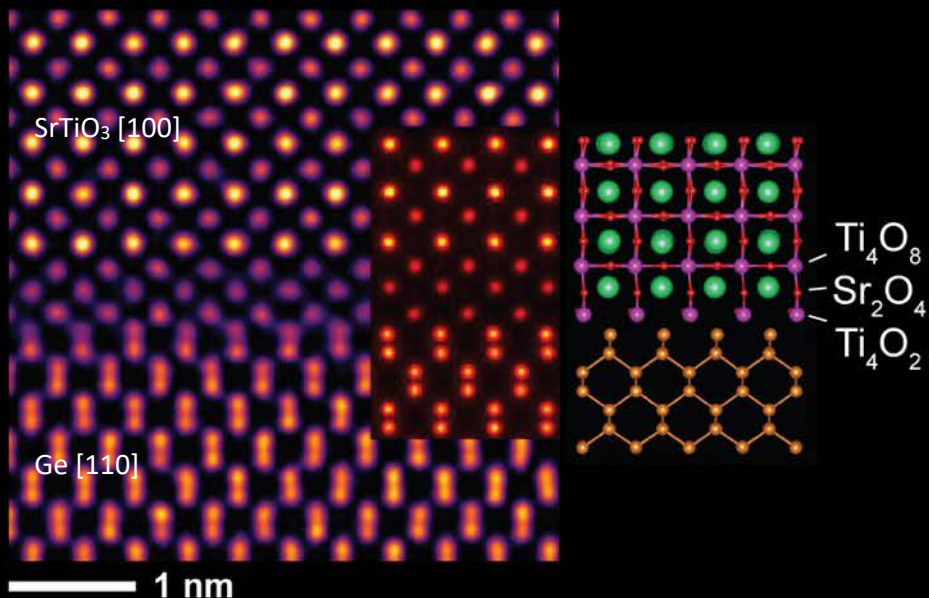


We can interpret such interfaces through image simulations based on atomistic models.

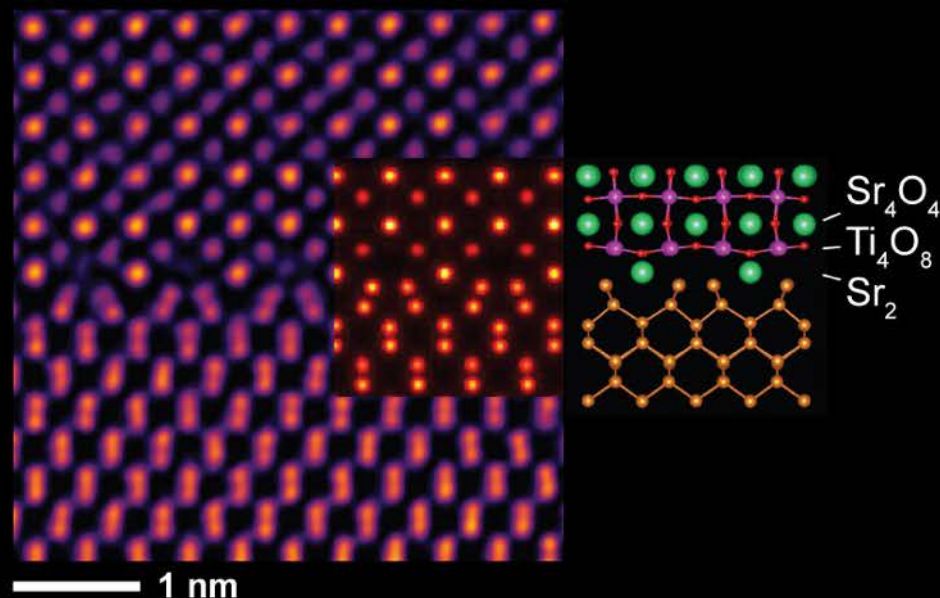


This approach allows us to better understand interface reconstructions, defects, and intermixing that affect properties and performance.

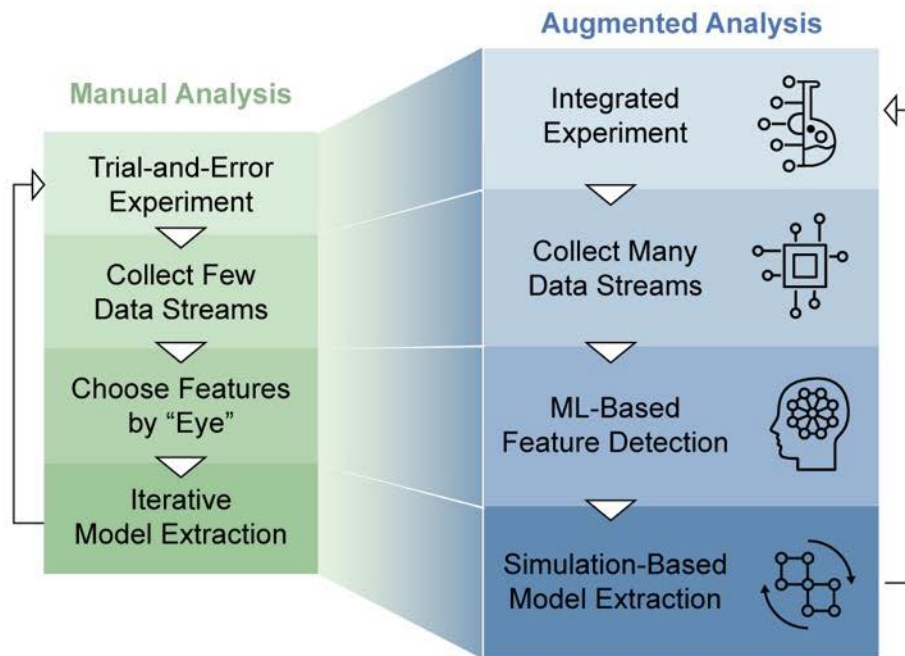
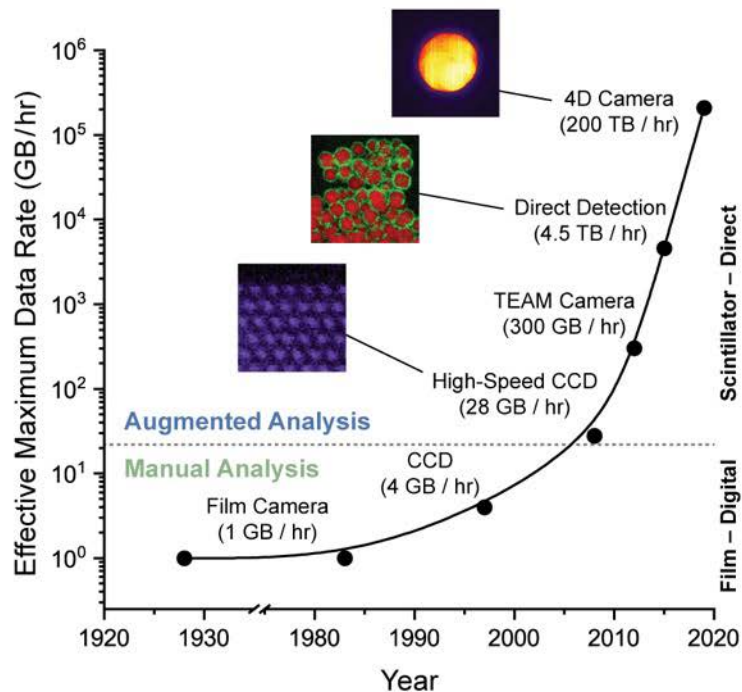
1 x 1 Reconstruction



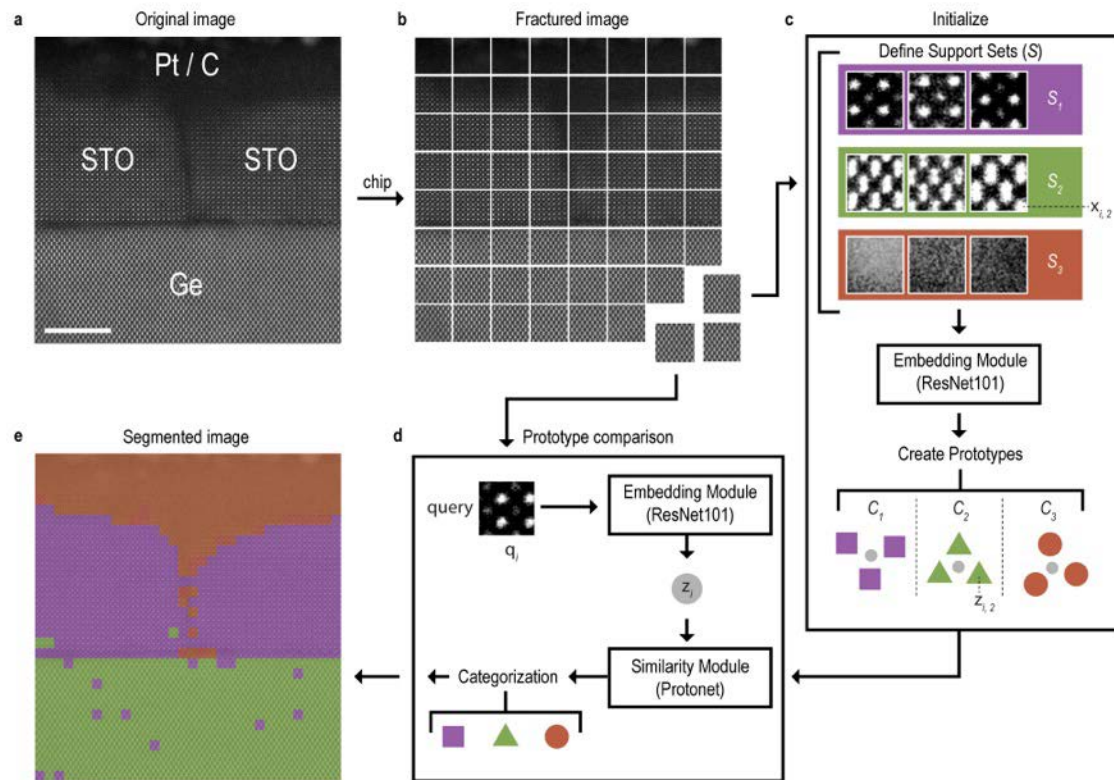
2 x 1 Reconstruction



However, to build even more accurate models for synthesis and degradation we must make sense of large, multi-modal data.



We have developed few-shot machine learning models that allow us to rapidly build statistical models for atomic motifs and defects.



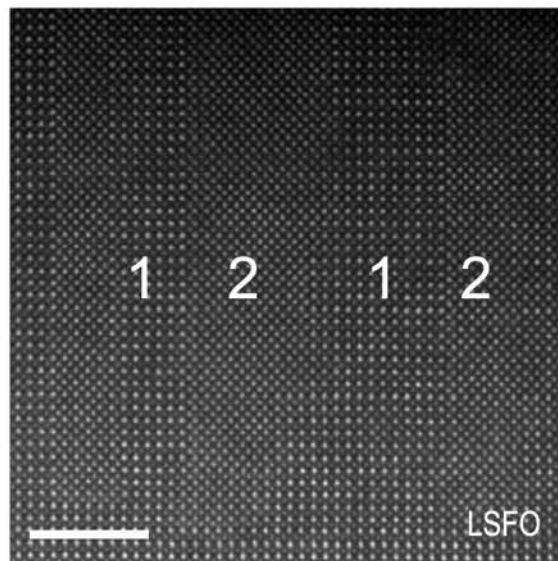
Scientists can rapidly train an ML model through simple graphical user interfaces.

The screenshot shows the GitHub repository page for 'pnnl/pychip_gui'. The repository is public and has 19 stars and 2 forks. The main branch is 'master' with 1 branch and 0 tags. The repository contains several files and folders, including 'Videos', 'docs', 'manuscript_images', 'prototype', '.Rhistory', '.gitignore', '.gitlab-ci.yml', 'DISCLAIMER', 'LICENSE', and 'README.md'. The 'About' section describes pyCHIP as a tool for segmentation and feature classification in transmission electron microscopy (TEM) images. The 'Releases' section shows no releases published.

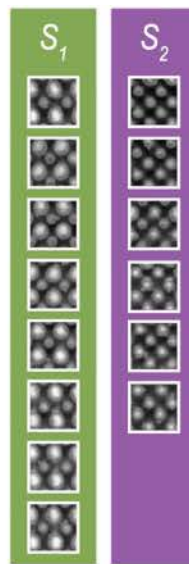
| File/Folder | Commit Message | Commit Date | Commit Type |
|-------------------|---------------------|-----------------------|-------------|
| stevenspurgeon | Update README.md | 7711277 - 2 years ago | 11 Commits |
| Videos | add in whole gui | 3 years ago | |
| docs | add in whole gui | 3 years ago | |
| manuscript_images | add in whole gui | 3 years ago | |
| prototype | update requirements | 3 years ago | |
| .Rhistory | add in whole gui | 3 years ago | |
| .gitignore | add in whole gui | 3 years ago | |
| .gitlab-ci.yml | add in whole gui | 3 years ago | |
| DISCLAIMER | add in whole gui | 3 years ago | |
| LICENSE | add in whole gui | 3 years ago | |
| README.md | Update README.md | 2 years ago | |

We classify complex synthesis products and degradation of materials microstructures.

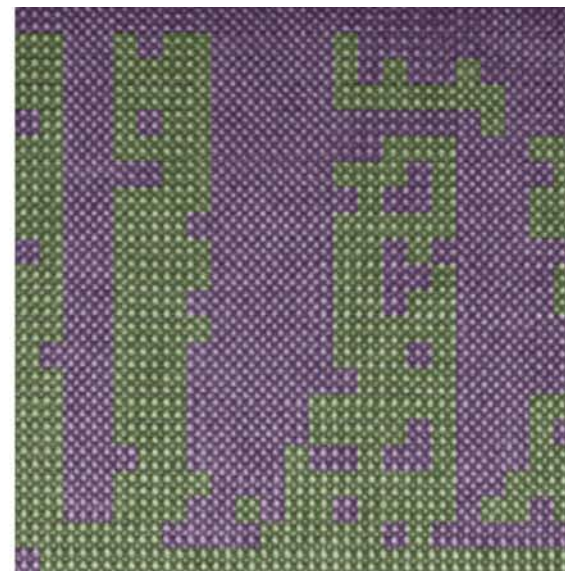
Original HAADF Image



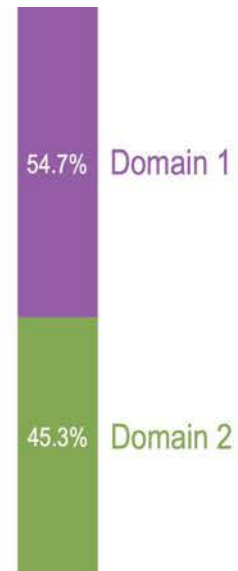
Support Sets



Segmented Image

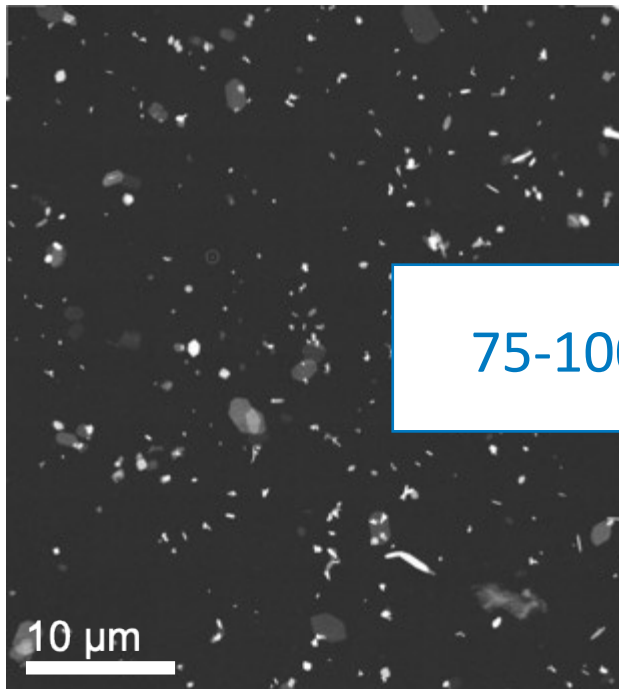


Pixel Fraction



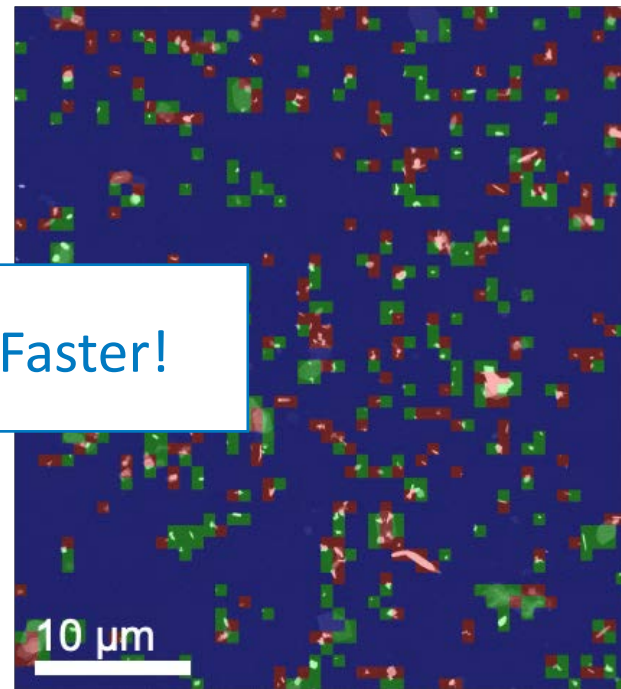
We can quickly and reproducibly extract microstructural descriptors by task.

MoO₃ Organic Photovoltaic Precursor



Manual Analysis
10 minutes

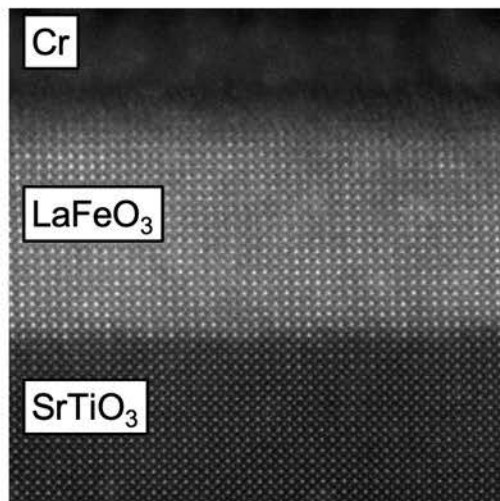
75-100x Faster!



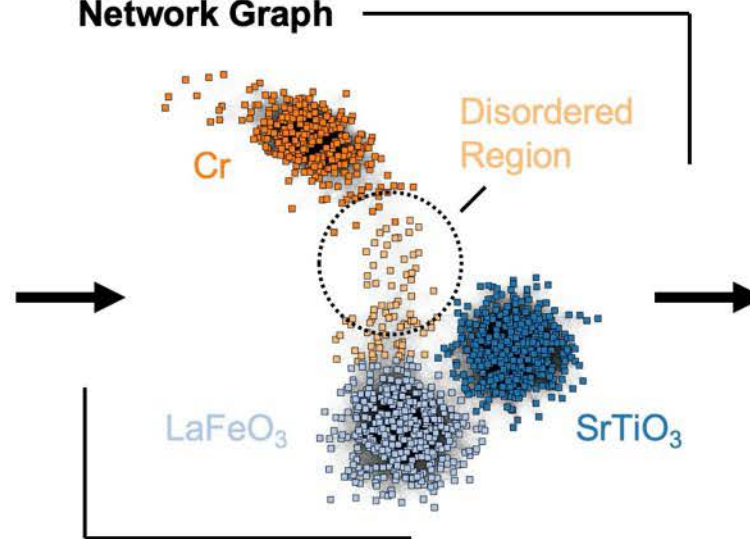
Few-Shot
Task 2
8 seconds

We can describe degradation signatures in an unsupervised manner using graph neural network models, informing more accurate statistical models.

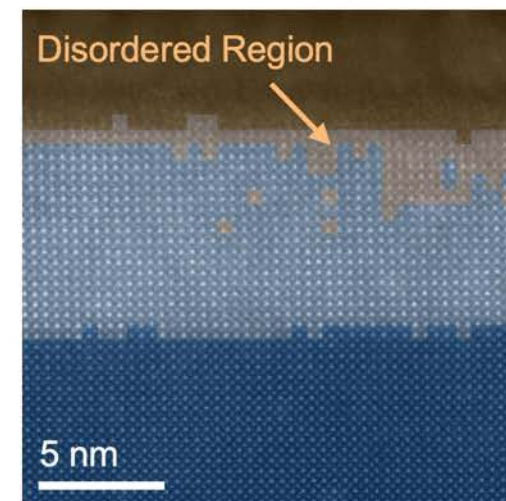
Raw HAADF Image



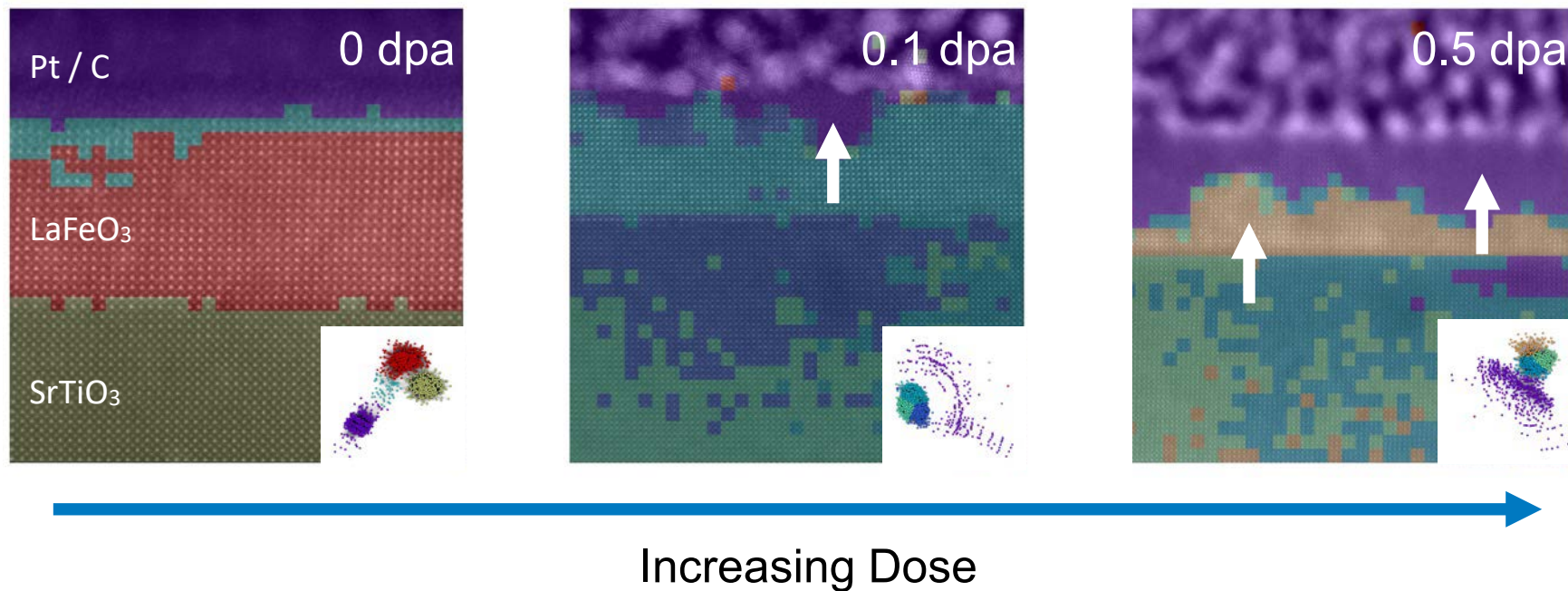
Network Graph



Cluster Analysis



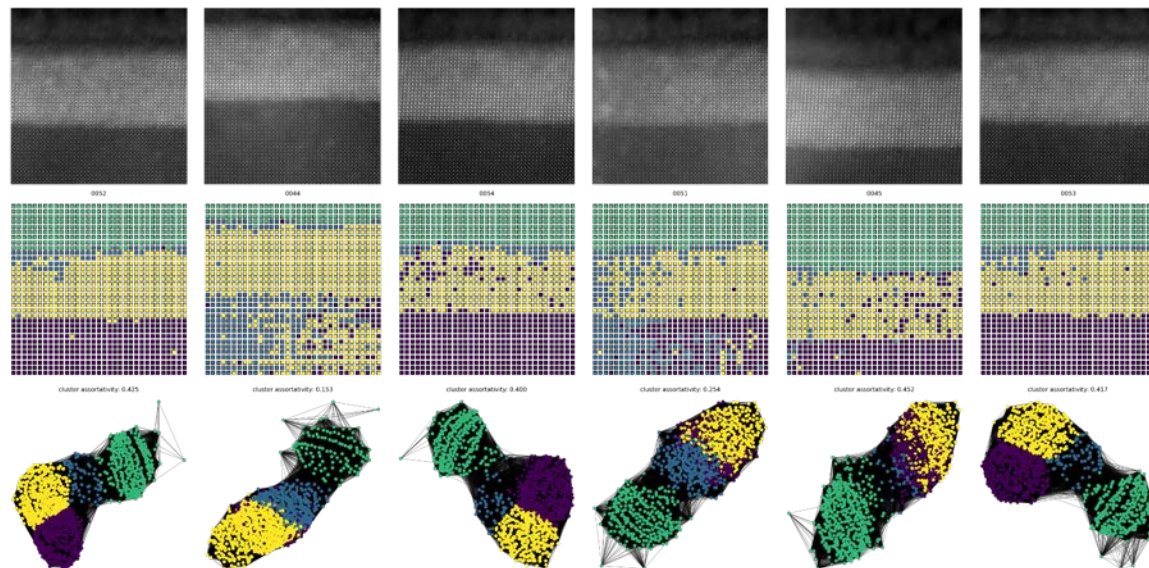
We can describe degradation signatures in an unsupervised manner using graph neural network models, informing more accurate statistical models.





**ARTIFICIAL INTELLIGENCE-GUIDED
TRANSMISSION ELECTRON MICROSCOPE
(AUTOEM)**

Through these approaches,
we can curate large libraries
of statistical defect
information across length
scales to inform properties
and performance.



NREL is advancing PV interface science through new world-class instrumentation and the development of AI-guided materials science workflows.

**For more information, visit:
<https://tinyurl.com/z8ryk4y3>**



NREL/PR-5K00-90723

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