

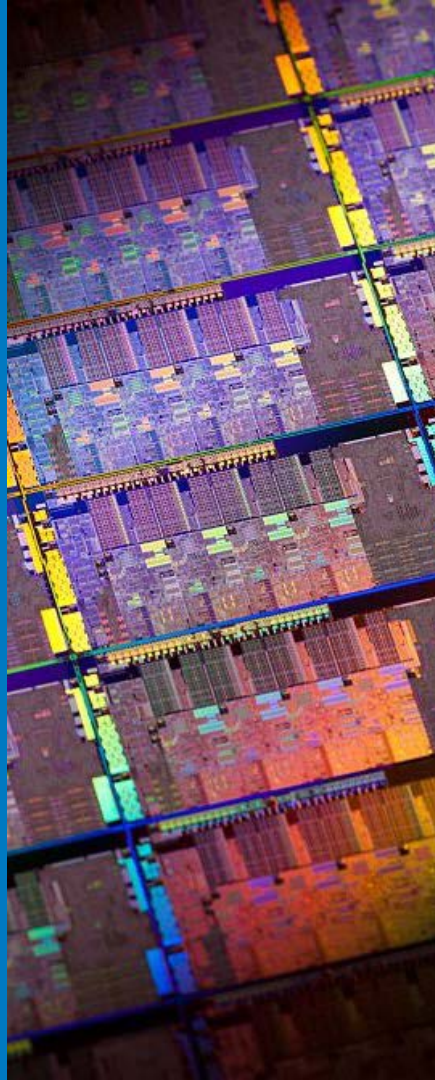


University of Colorado **Boulder**

The Rise of Intelligent Materials Science
*Unleashing the Power of Machine
Intelligence in Characterization*

Steven R. Spurgeon
*CINT Annual User Meeting
September 16, 2024*

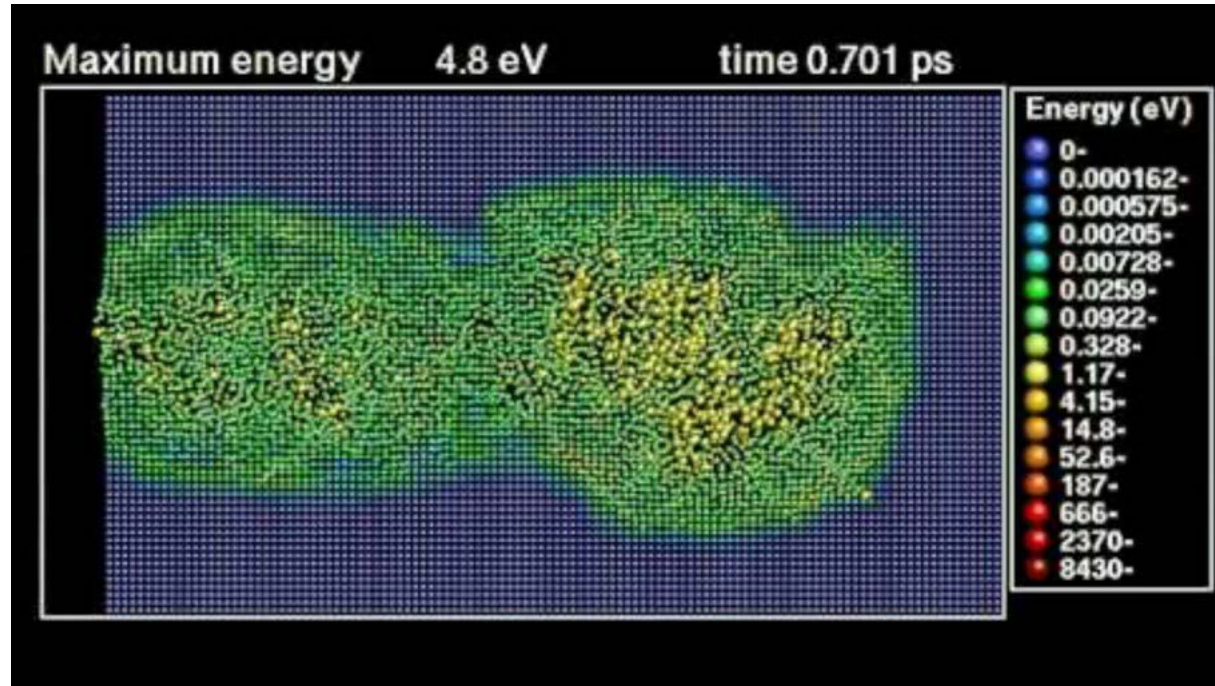
Everyday
technologies are
critically reliant on
mastering materials
lifecycles



Such mastery depends on rich understanding of materials synthesis pathways...



...and degradation
behavior in
extremes



Averback and Diaz de la Rubia, *Solid State Physics*, edited by Ehrenfest and Spaepen 51
(Academic Press, New York 1998) 281.

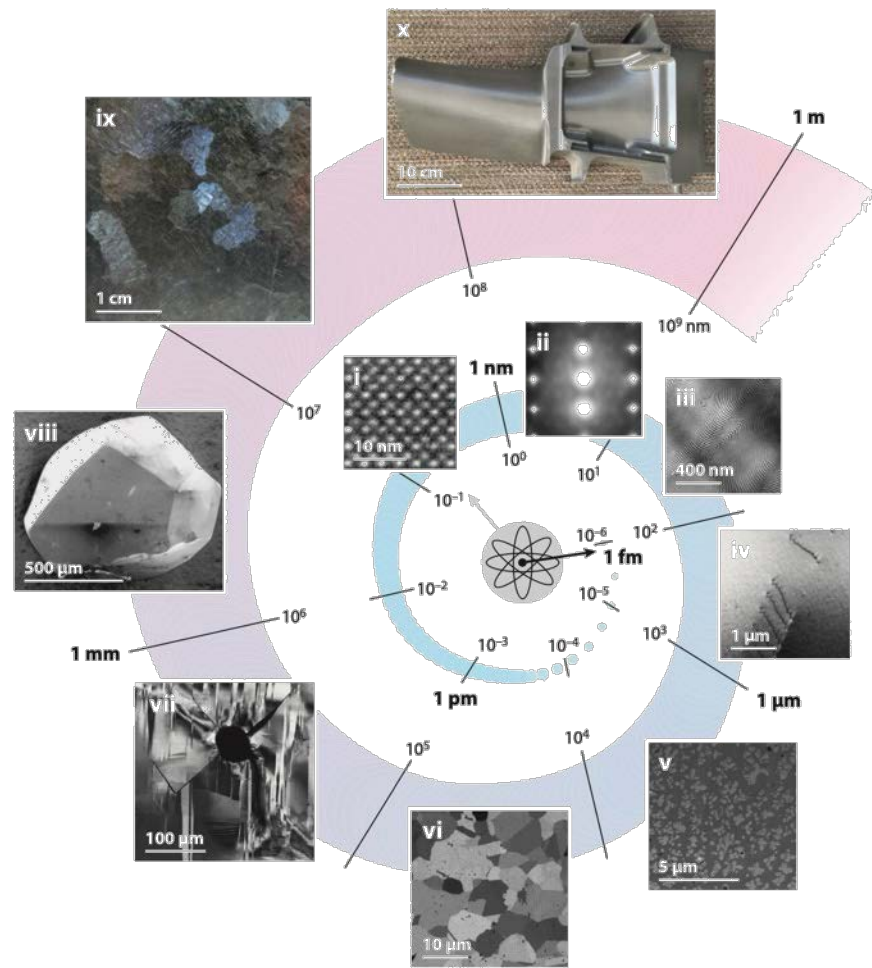
<https://www.youtube.com/watch?v=TyYBlj-A9tY>

Our aim is an ontology of the materials lifecycle:

A systematic mapping of data to meaningful semantic concepts...across spatial and temporal scales

Palantir Blog

<https://blog.palantir.com/ontology-finding-meaning-in-data-palantir-rfx-blog-series-1-399bd1a5971b>

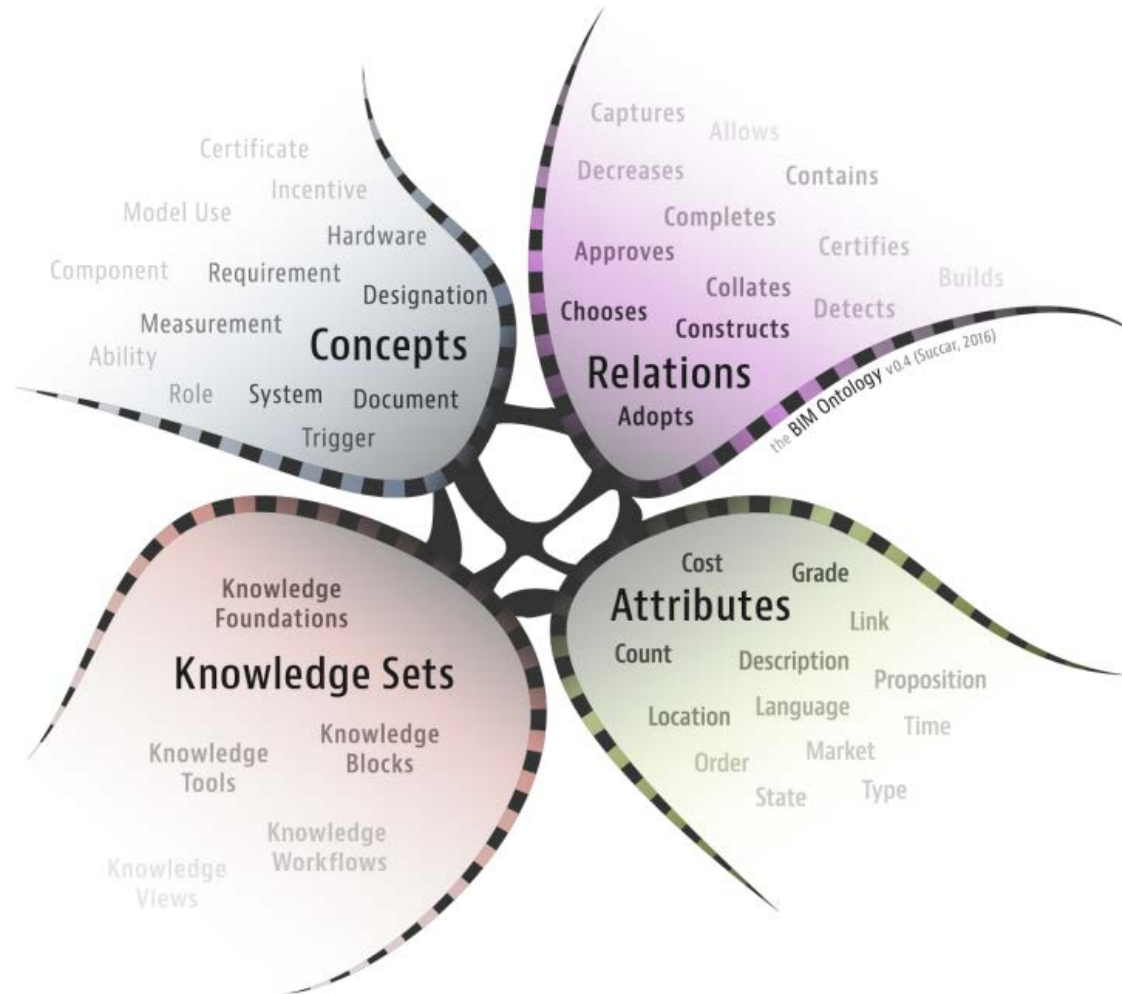


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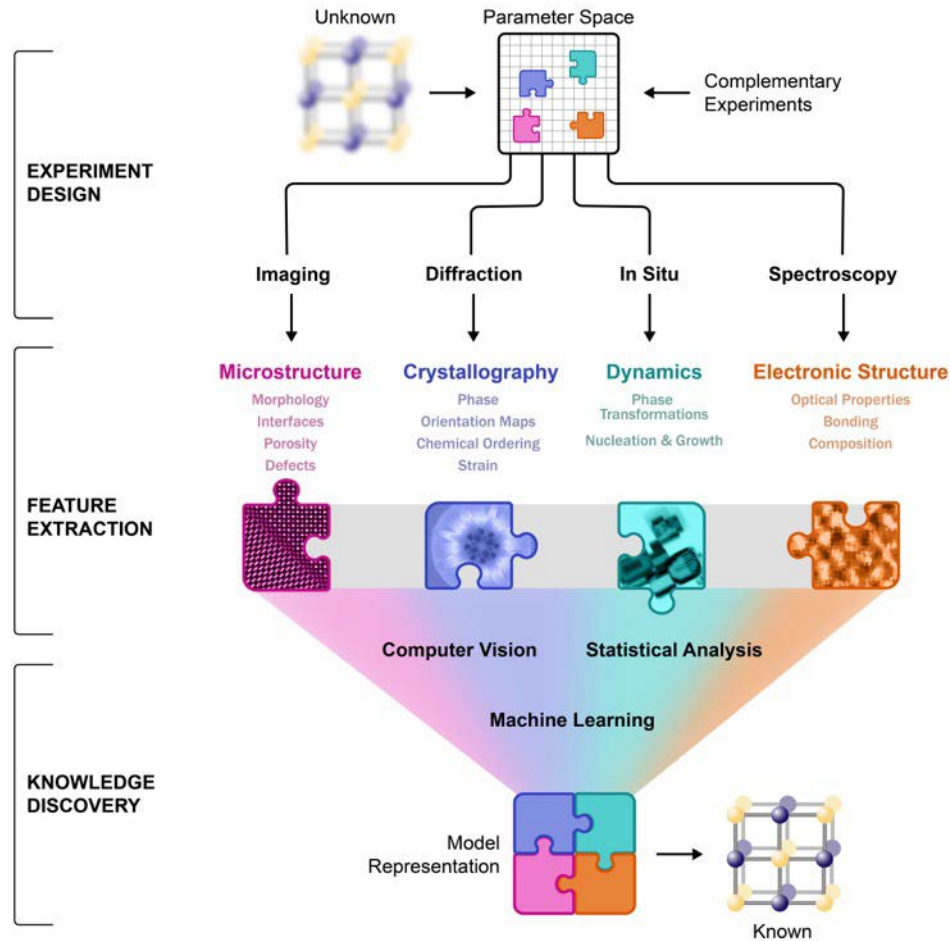
Palantir Blog

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<https://www.bimframework.info/2015/08/bim-ontology.html>

Such an ontology will transform raw multi-modal data into predictive physical models



Understanding Materials Synthesis

Epitaxial integration of semiconductors and oxides is a challenge for emerging devices

Complex oxide materials

- Sensors
- Energy harvesters
- Memory
- Transducers

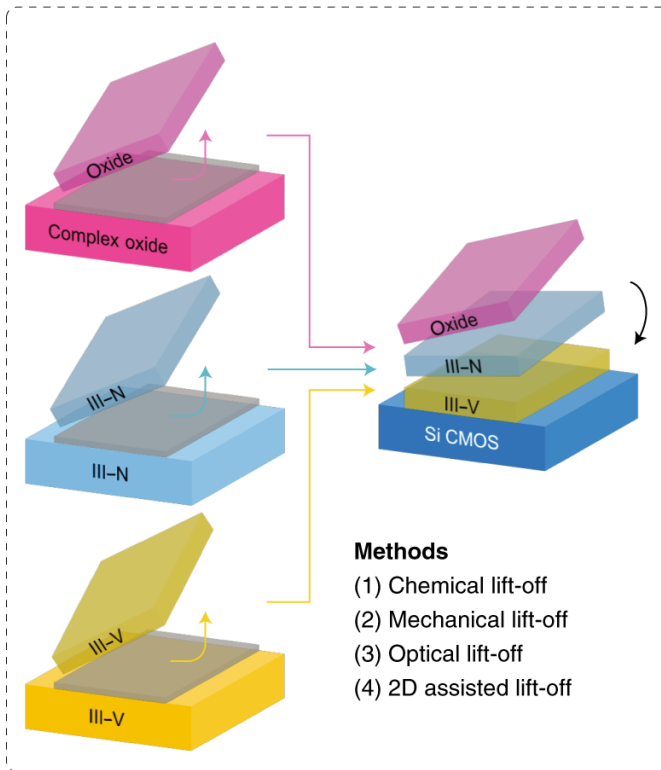
III-N materials

- High-power devices
- Ultraviolet–visible photonics
- Radio-frequency electronics

III-V materials

- High-speed FETs
- Infrared photonics
- Radio-frequency electronics
- Photovoltaics

Top-down epitaxial layer transfer



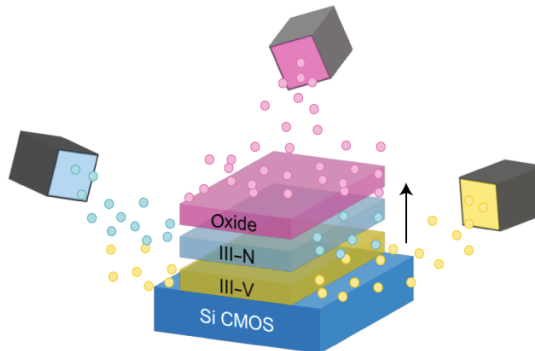
Methods

- (1) Chemical lift-off
- (2) Mechanical lift-off
- (3) Optical lift-off
- (4) 2D assisted lift-off

Bottom-up epitaxial growth

Tools

- (1) MOCVD
- (2) MBE
- (3) PLD

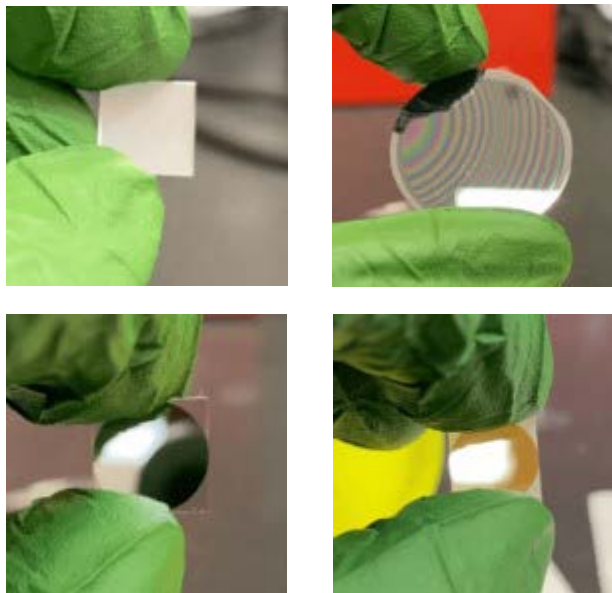


Techniques

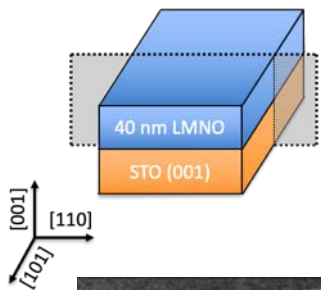
- (1) Metamorphic growth
- (2) Lateral overgrowth
- (3) Geometrically defined growth
- (4) vdW and remote epitaxial growth

Tailored materials design requires direct local probes of structure and chemistry

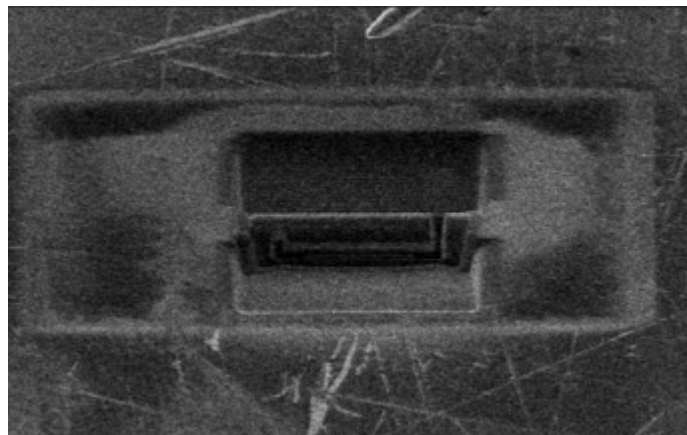
Functional Thin Films for Energy Applications



~1 cm



Site Specific
Metrology



Focused Ion Beam



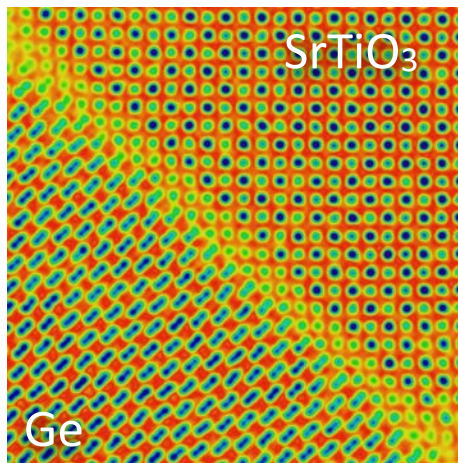
STEM

Electron microscopy can richly inform lifecycle models to achieve predictive control

Structure



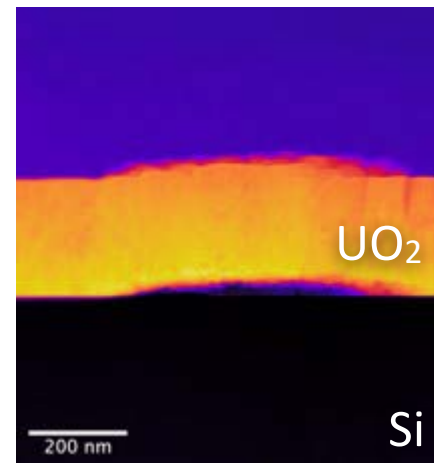
Chemistry



Imaging



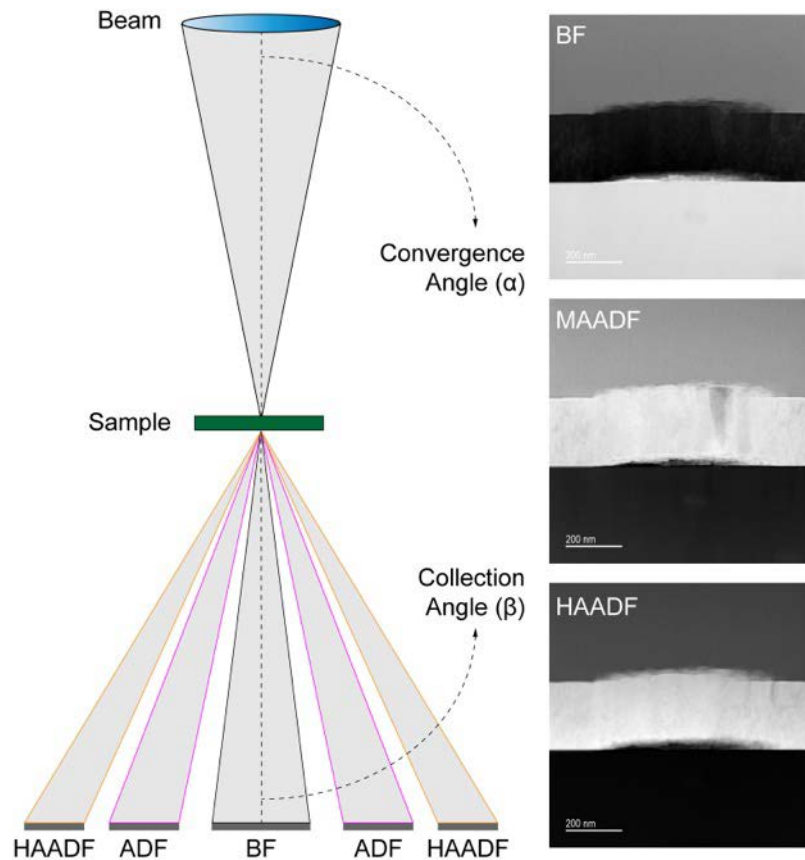
Diffraction



Spectroscopy

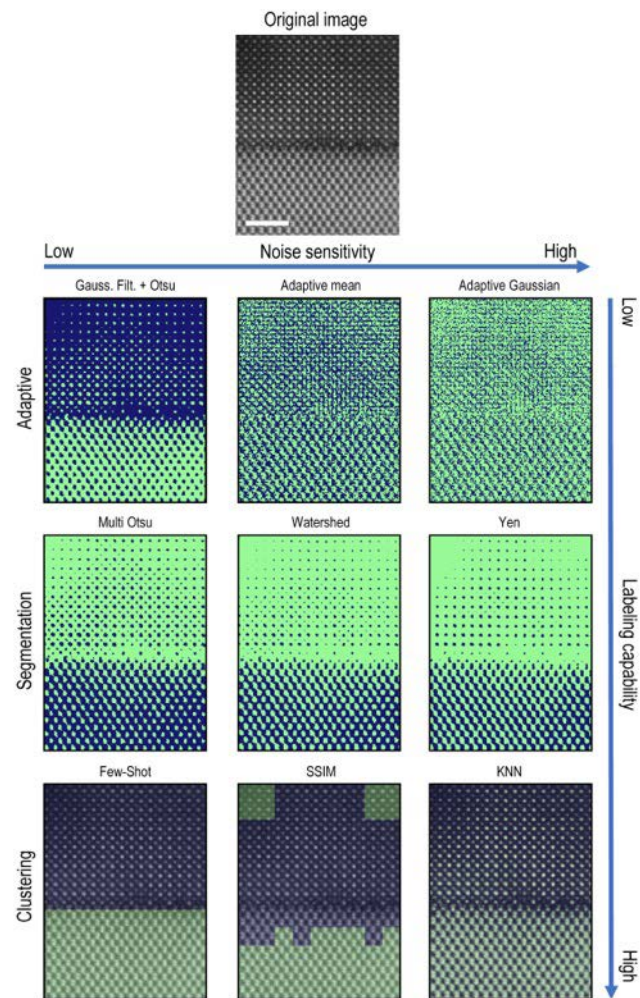
The Challenge

Imaging parameters strongly affect the representation of an object in data

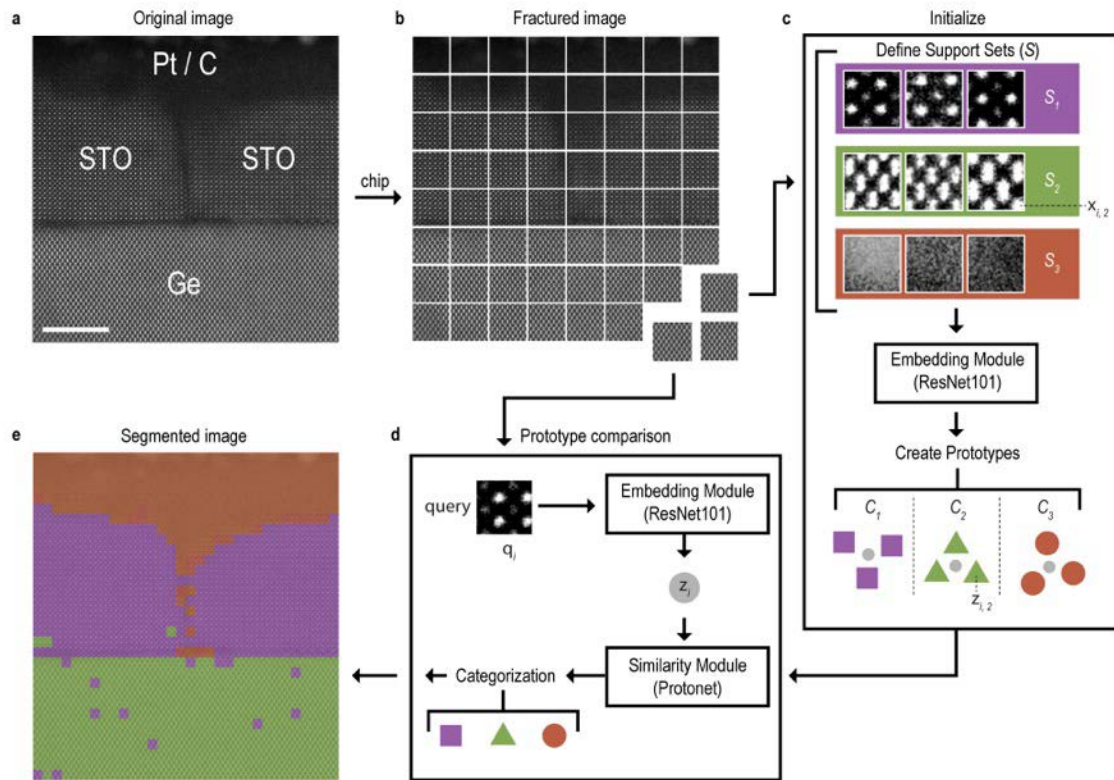


The Task

Classify
microstructural
features using
limited examples

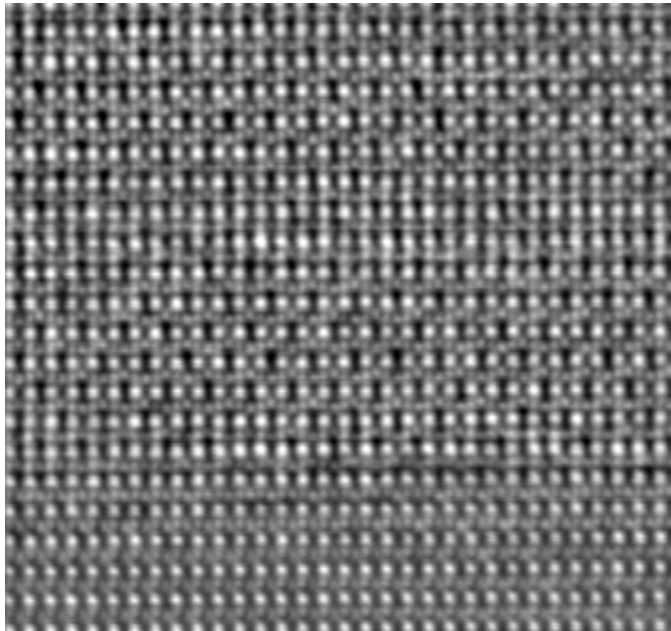


Few shot learning uses limited prior knowledge to classify features in discovery scenarios



Careful pre-processing is needed for best model performance

Without Contrast Leveling Adaptive Histogram Equalization (CLAHE)



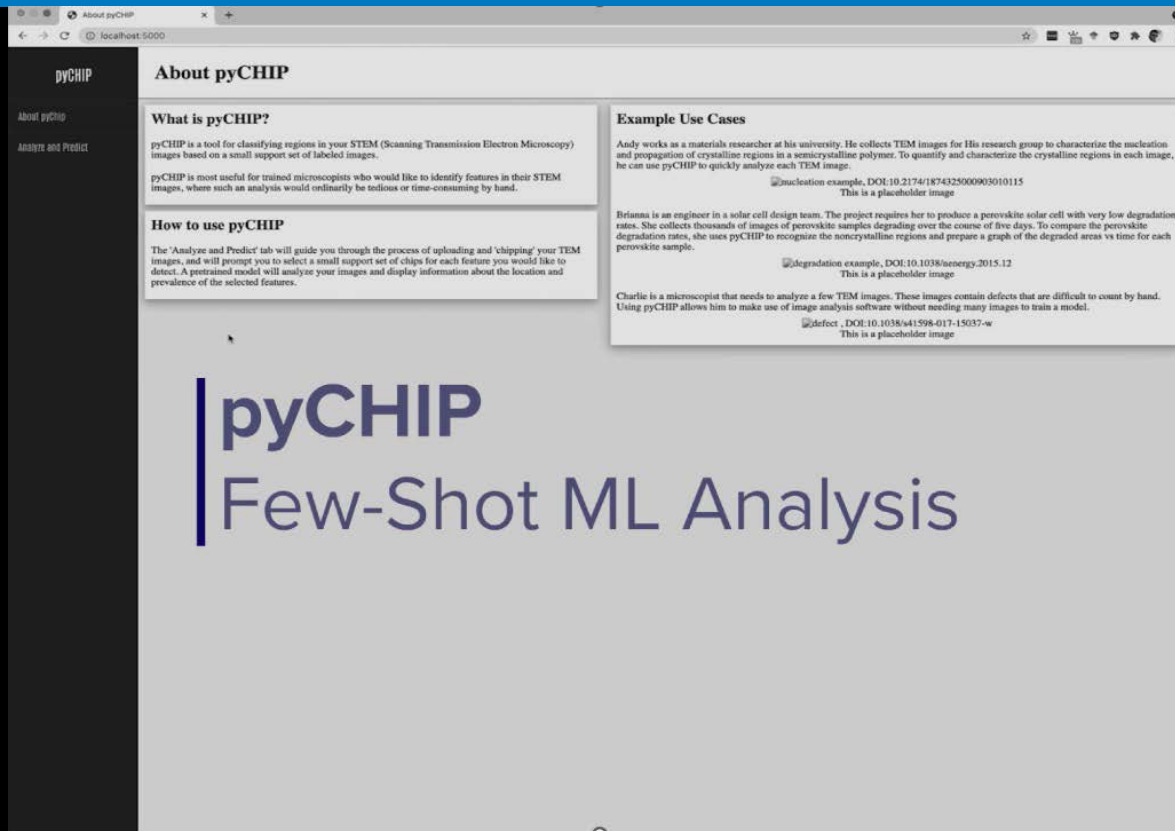
2 nm



With CLAHE



A simple GUI can interact with model parameters and support sets for different tasks



The screenshot shows a web browser displaying the pyCHIP application. The browser address bar shows 'localhost:5000'. The page has a dark sidebar on the left with the pyCHIP logo and navigation links for 'About pyCHIP' and 'ANALYZE AND PREDICT'. The main content area is titled 'About pyCHIP' and is divided into three columns. The first column, 'What is pyCHIP?', explains that it is a tool for classifying regions in STEM images. The second column, 'How to use pyCHIP', describes the 'Analyze and Predict' tab. The third column, 'Example Use Cases', lists three scenarios: a materials researcher using TEM images, a solar cell engineer using perovskite images, and a microscopist analyzing defects. Each use case includes a small image placeholder and a DOI link. At the bottom of the page, a large blue header reads 'pyCHIP Few-Shot ML Analysis'.

About pyCHIP

What is pyCHIP?

pyCHIP is a tool for classifying regions in your STEM (Scanning Transmission Electron Microscopy) images based on a small support set of labeled images.

pyCHIP is most useful for trained microscopists who would like to identify features in their STEM images, where such an analysis would ordinarily be tedious or time-consuming by hand.

How to use pyCHIP

The 'Analyze and Predict' tab will guide you through the process of uploading and 'chipping' your TEM images, and will prompt you to select a small support set of chips for each feature you would like to detect. A pretrained model will analyze your images and display information about the location and prevalence of the selected features.

Example Use Cases

Andy works as a materials researcher at his university. He collects TEM images for his research group to characterize the nucleation and propagation of crystalline regions in a semicrystalline polymer. To quantify and characterize the crystalline regions in each image, he can use pyCHIP to quickly analyze each TEM image.

[nucleation example, DOI:10.2174/1874325000903010115](#)
This is a placeholder image

Brianna is an engineer in a solar cell design team. The project requires her to produce a perovskite solar cell with very low degradation rates. She collects thousands of images of perovskite samples degrading over the course of five days. To compare the perovskite degradation rates, she uses pyCHIP to recognize the noncrystalline regions and prepare a graph of the degraded areas vs time for each perovskite sample.

[degradation example, DOI:10.1038/energy.2015.12](#)
This is a placeholder image

Charlie is a microscopist that needs to analyze a few TEM images. These images contain defects that are difficult to count by hand. Using pyCHIP allows him to make use of image analysis software without needing many images to train a model.

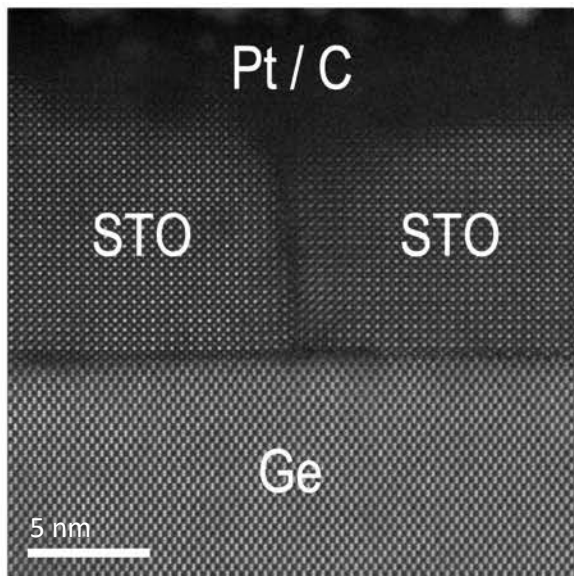
[defect, DOI:10.1038/s41598-017-15037-w](#)
This is a placeholder image

pyCHIP

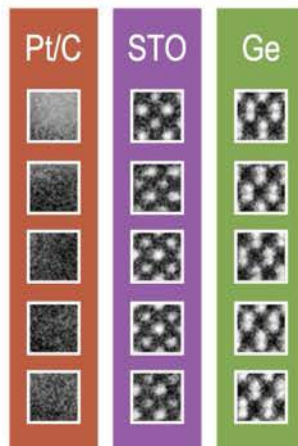
Few-Shot ML Analysis

We can rapidly classify atomic motifs in data to understand phase distributions

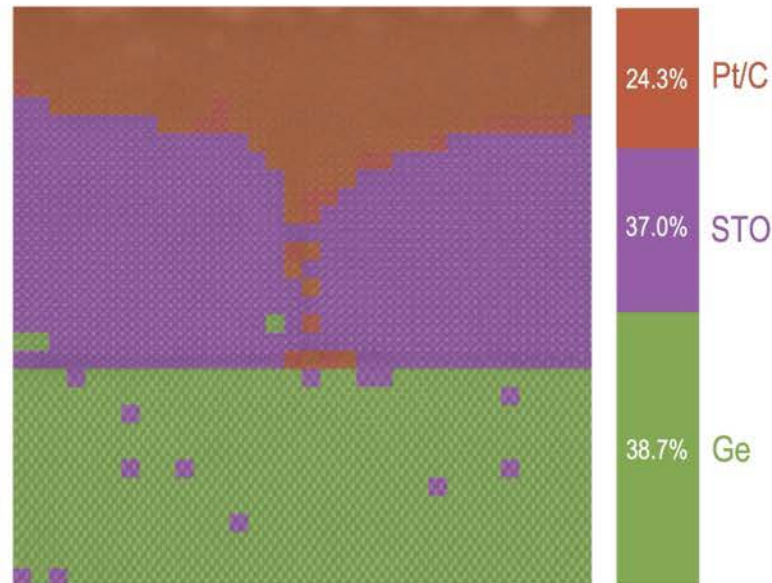
Original HAADF Image



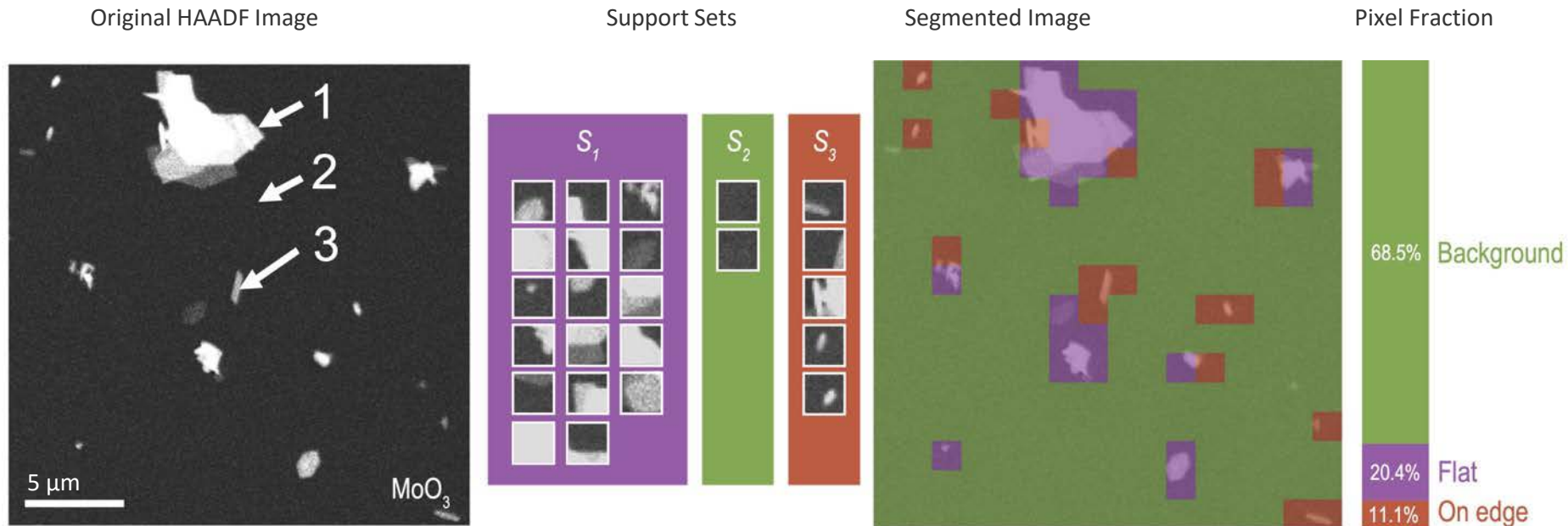
Support Sets



Segmented Image

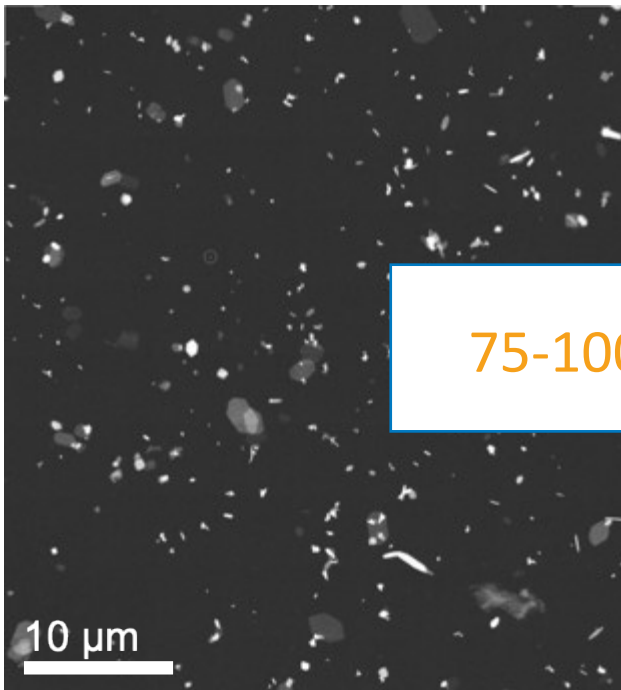


Such a model can easily be applied to different synthesis tasks



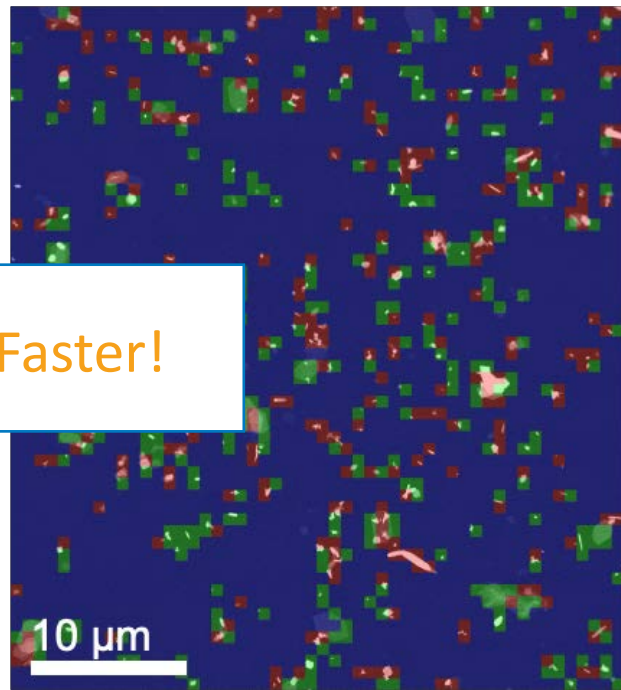
We can ultimately extract materials descriptors in a faster and more reproducible manner

MoO₃



Manual Analysis
10 minutes

Few-Shot
Task 1
8 seconds

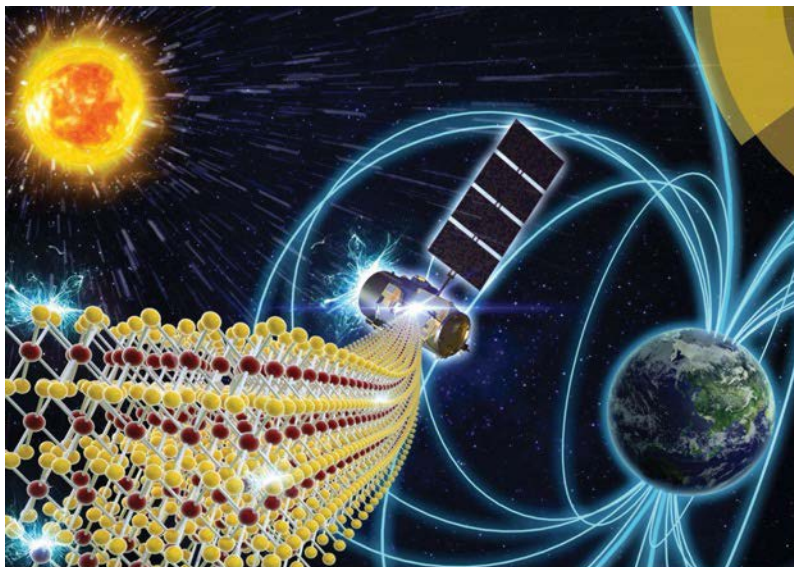


Few-Shot
Task 2
8 seconds

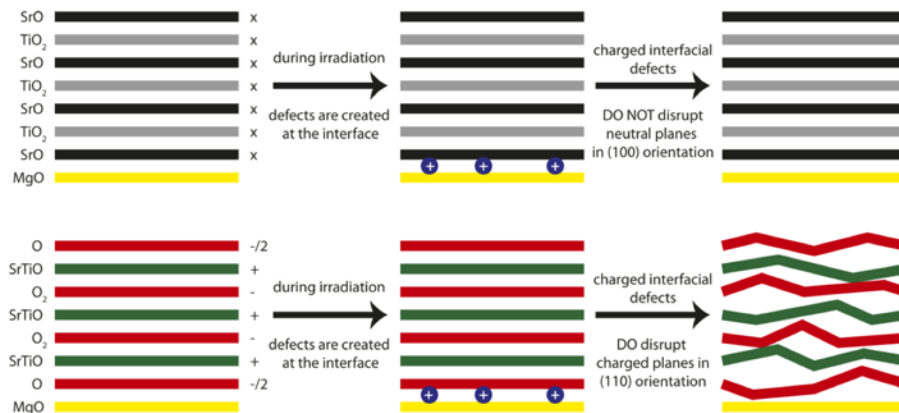
75-100x Faster!

Describing Disorder

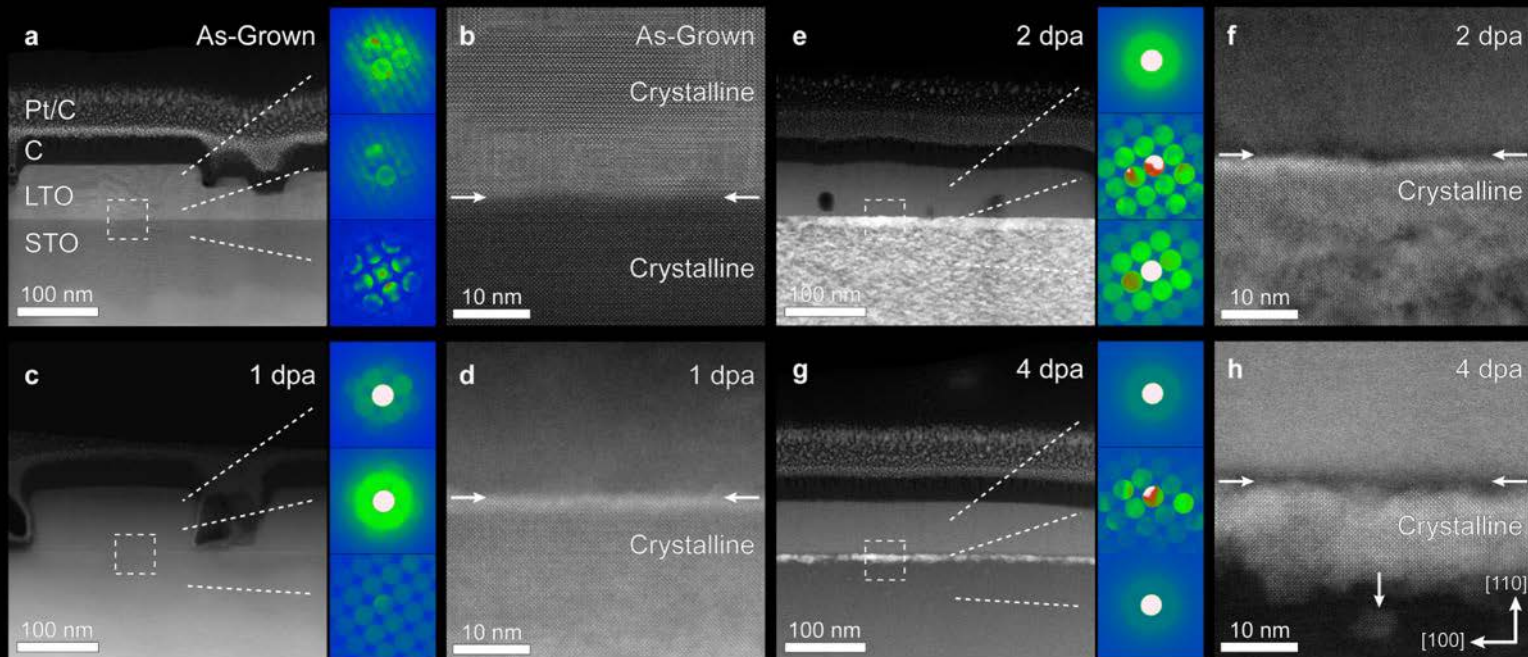
Controlling materials degradation is critical for electronics and sensors in extremes



Interface charge affects radiation-induced disorder



We can visualize damage buildup at these interfaces at stages of irradiation ex situ



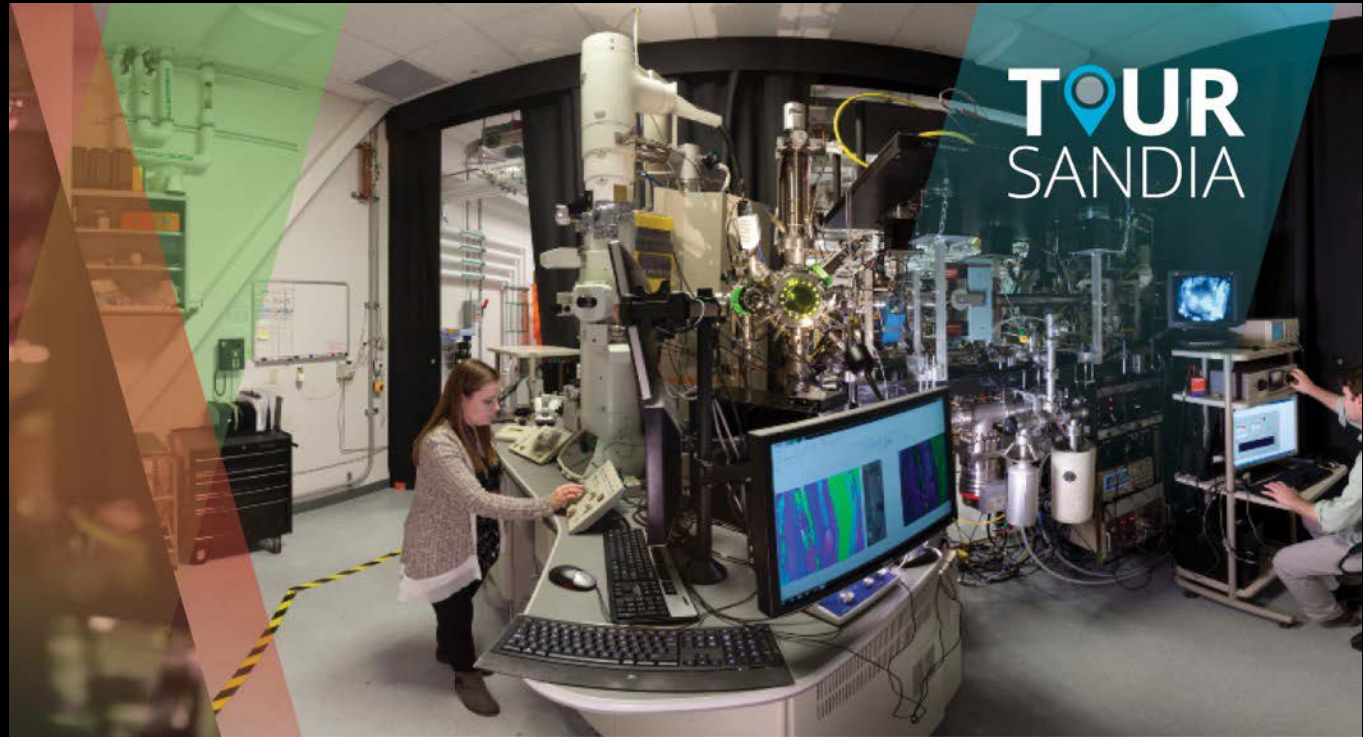
At CINT we took this one step further, visualizing materials breakdown in situ using the I³TEM



Khalid Hattar (UTK)

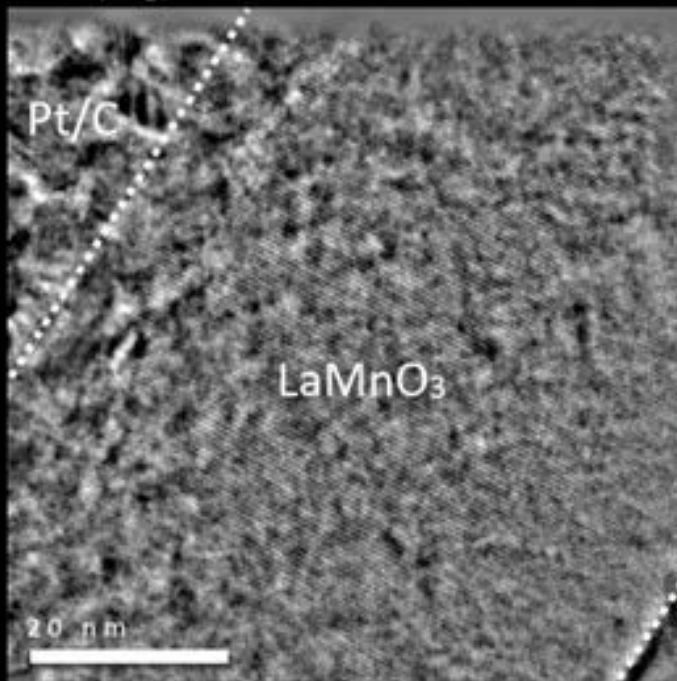


Chris Barr

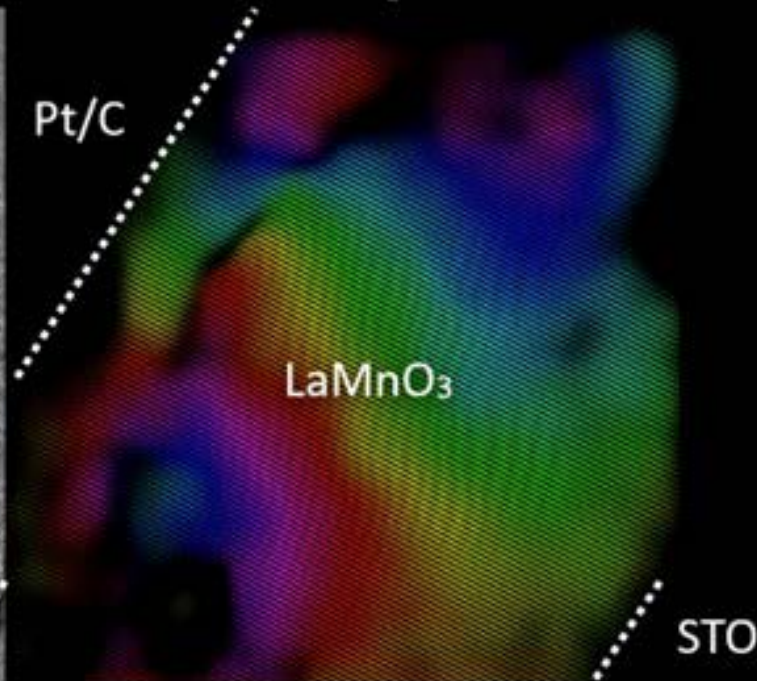


In situ TEM shows that disorder percolates through the material in a non-uniform manner

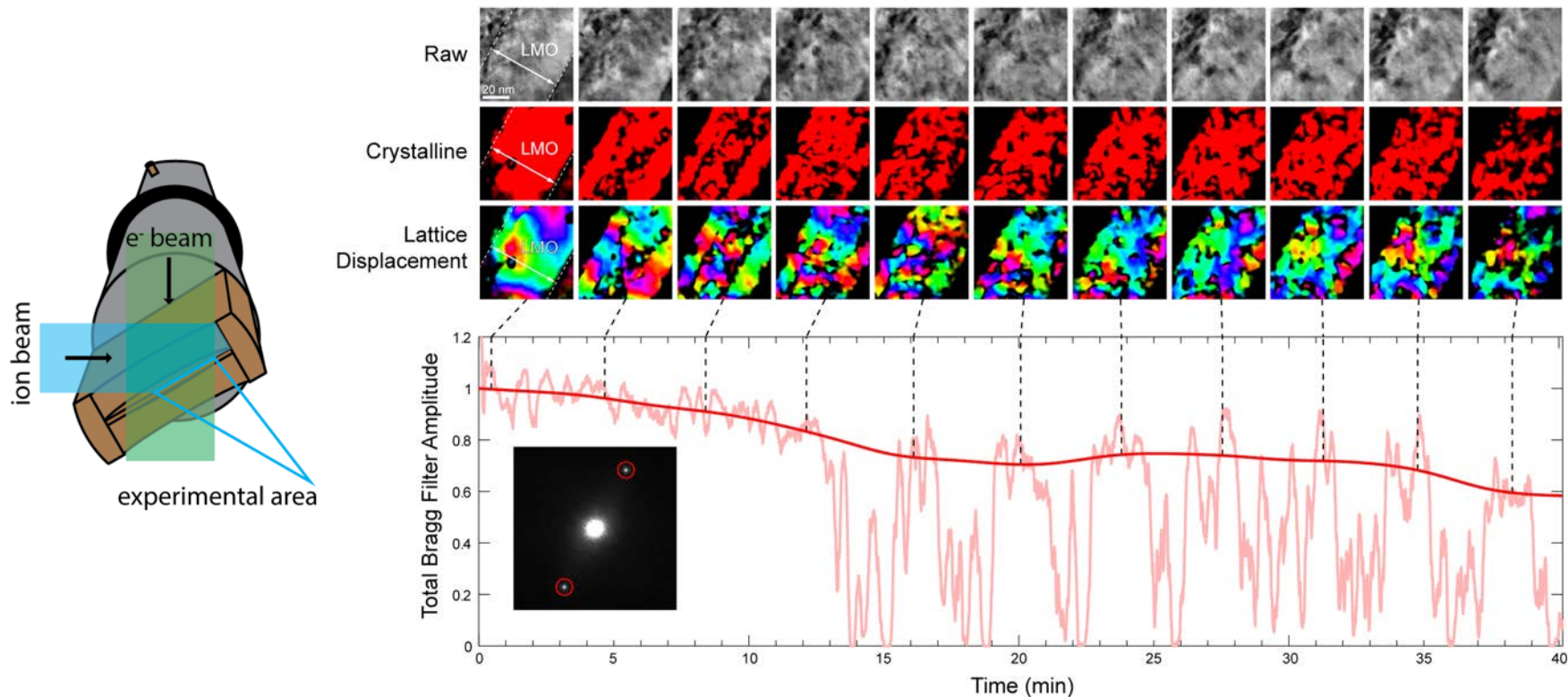
Filtered, Aligned HRTEM



Time-Resolved Fourier Filtering

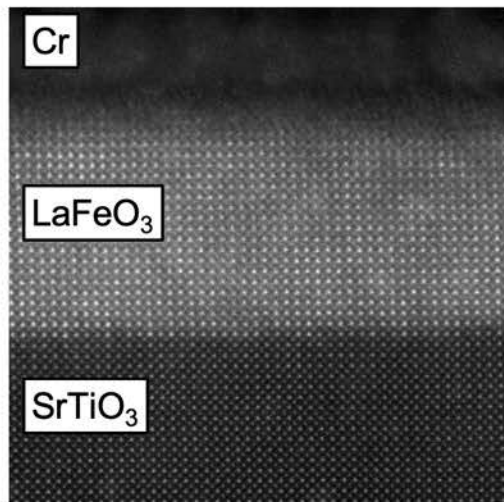


We can quantify the loss of crystallinity through statistical analysis of time series data

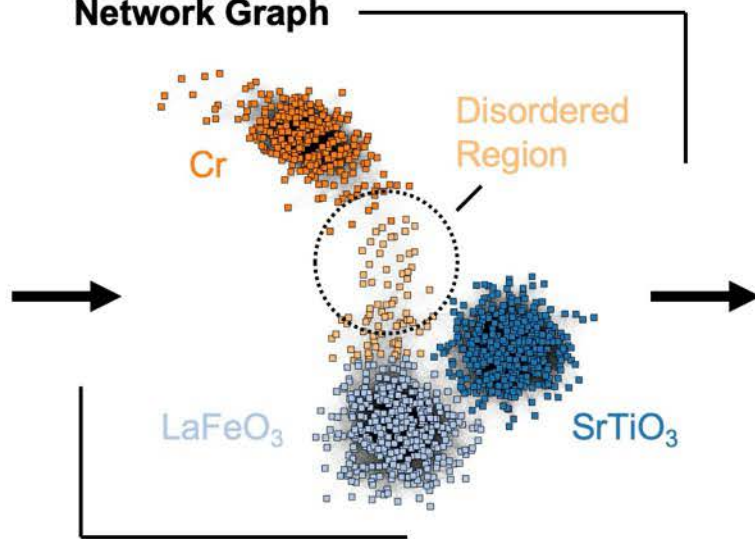


More recently, we have been developing unique order descriptors based on graph analytics

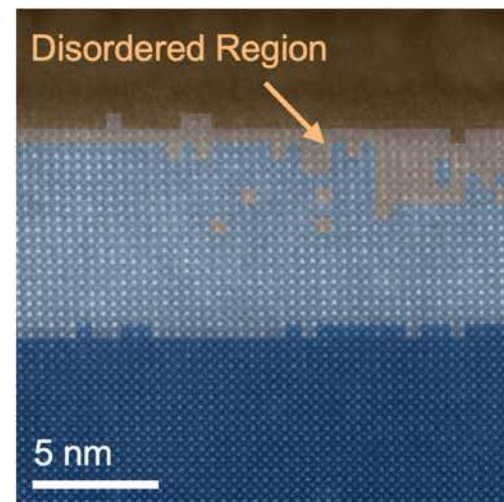
Raw HAADF Image



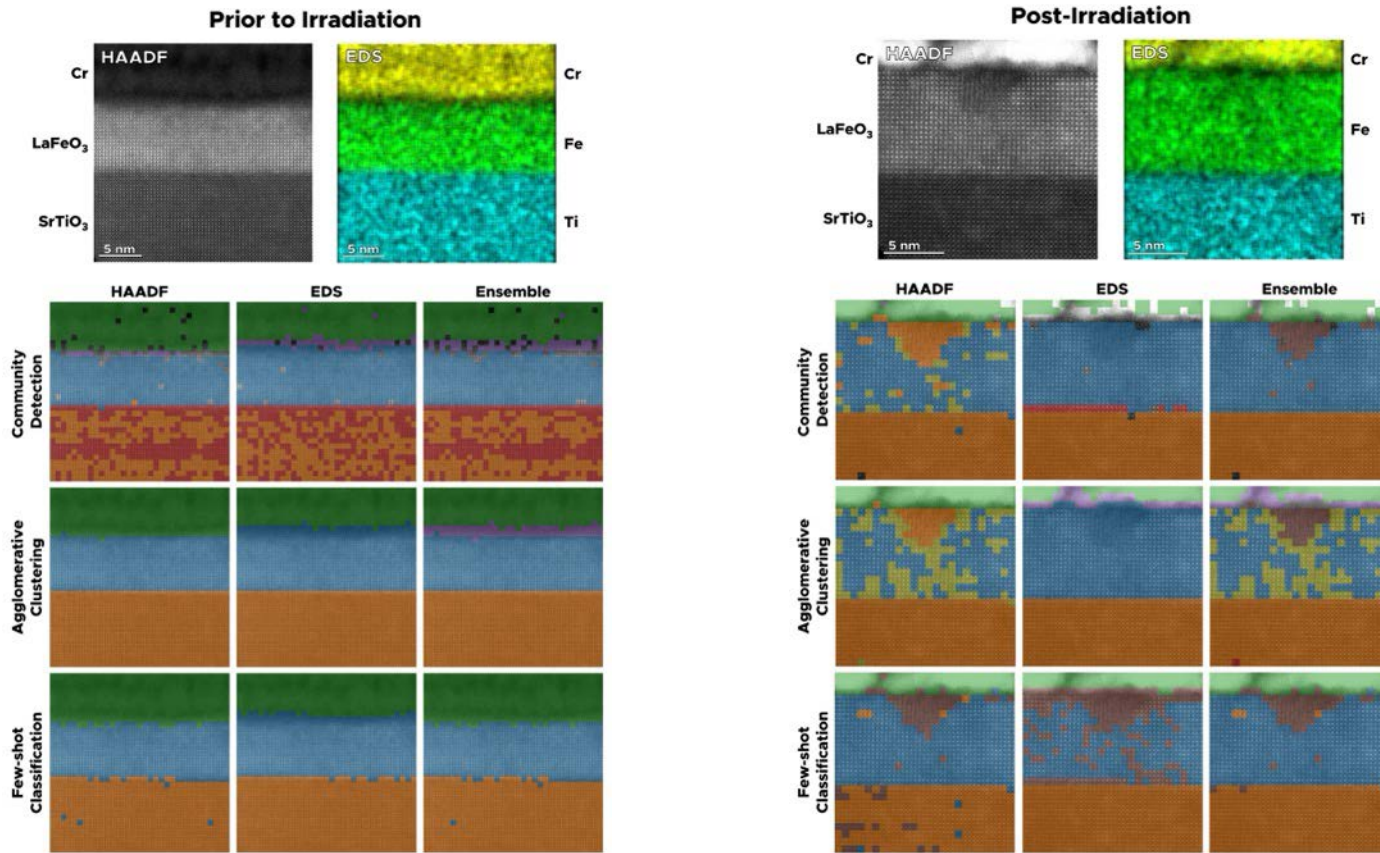
Network Graph



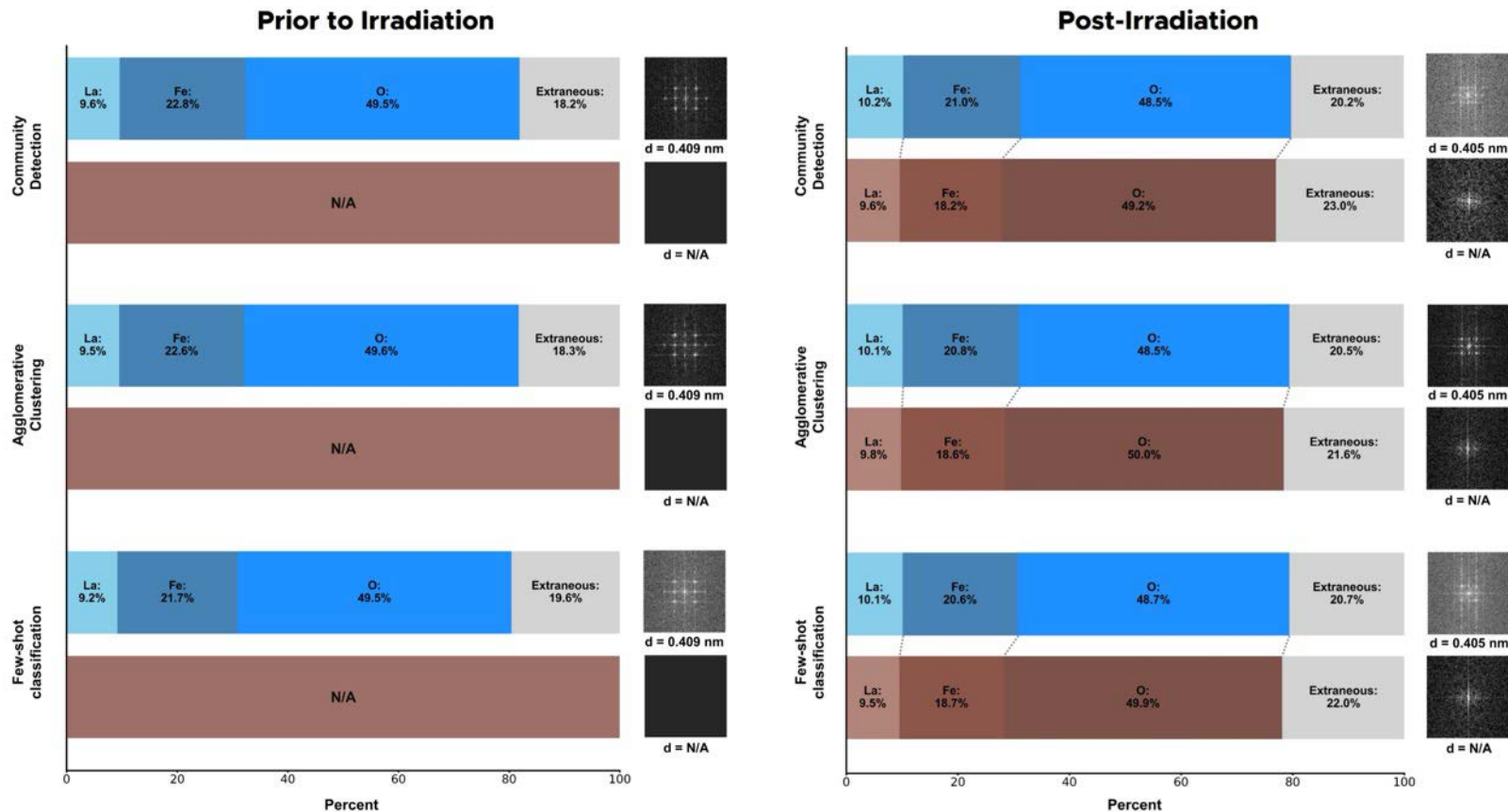
Cluster Analysis



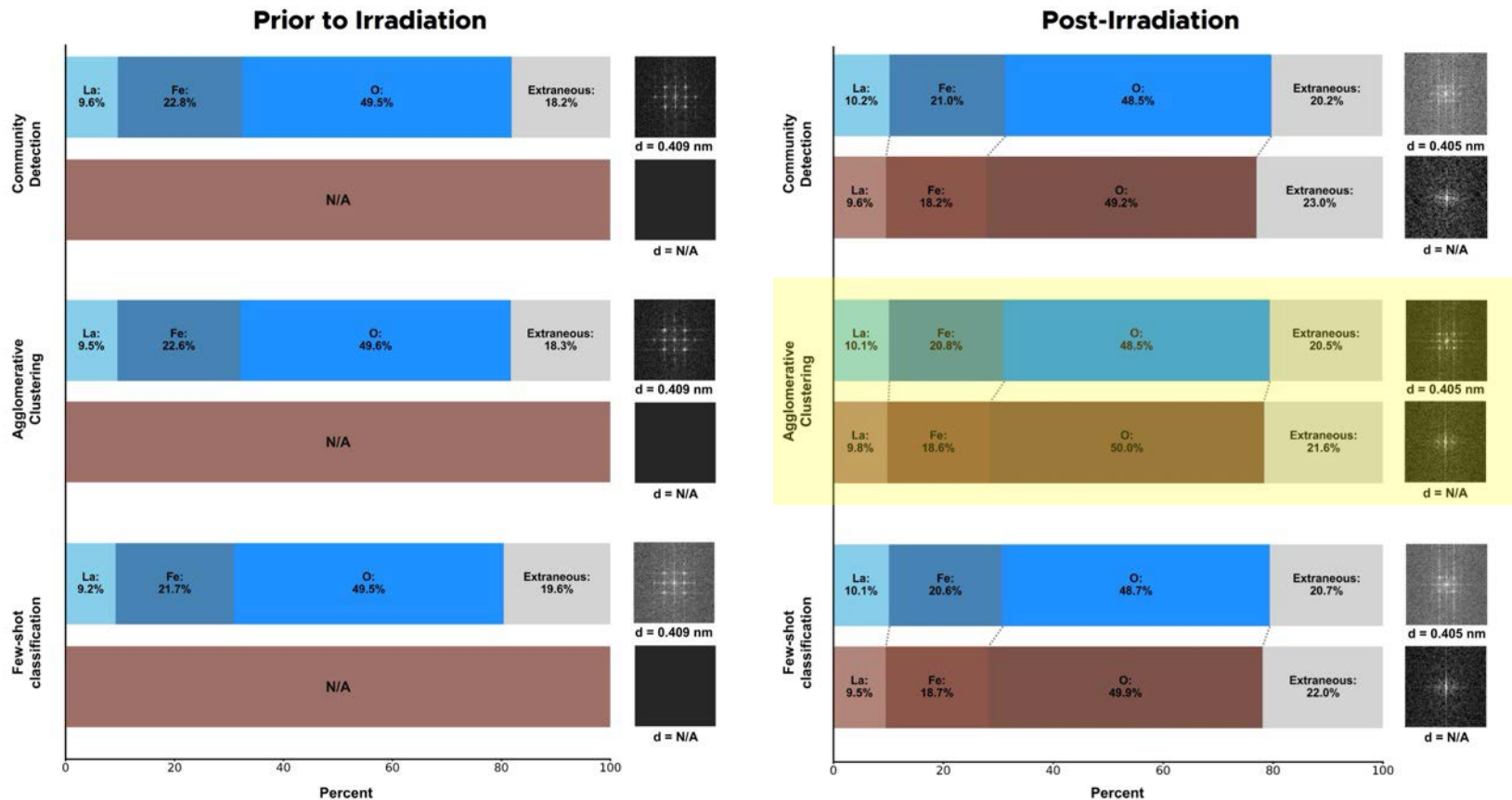
Multi-modal graphs effectively classify radiation damage signatures



Such models reveal changes in composition associated with irradiation

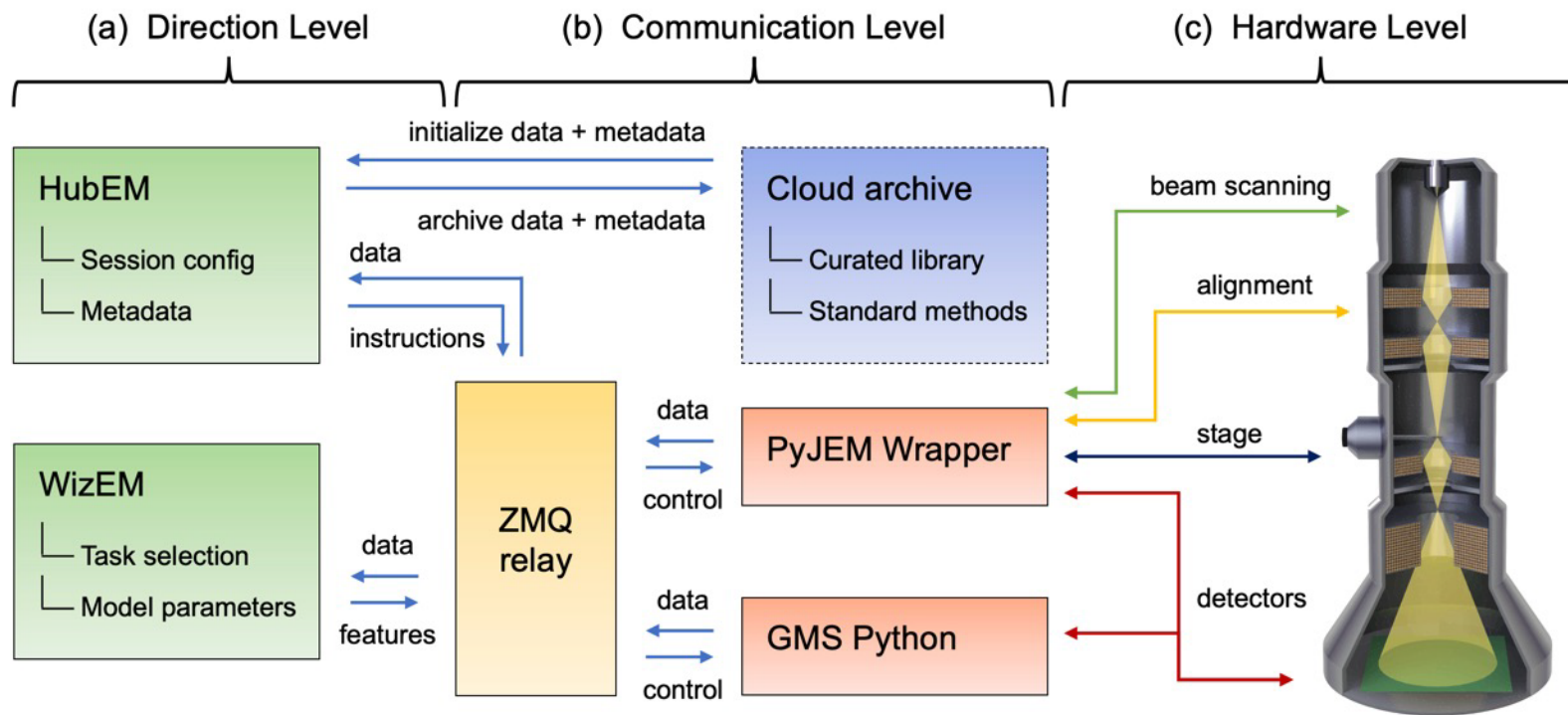


Such models reveal changes in composition associated with irradiation



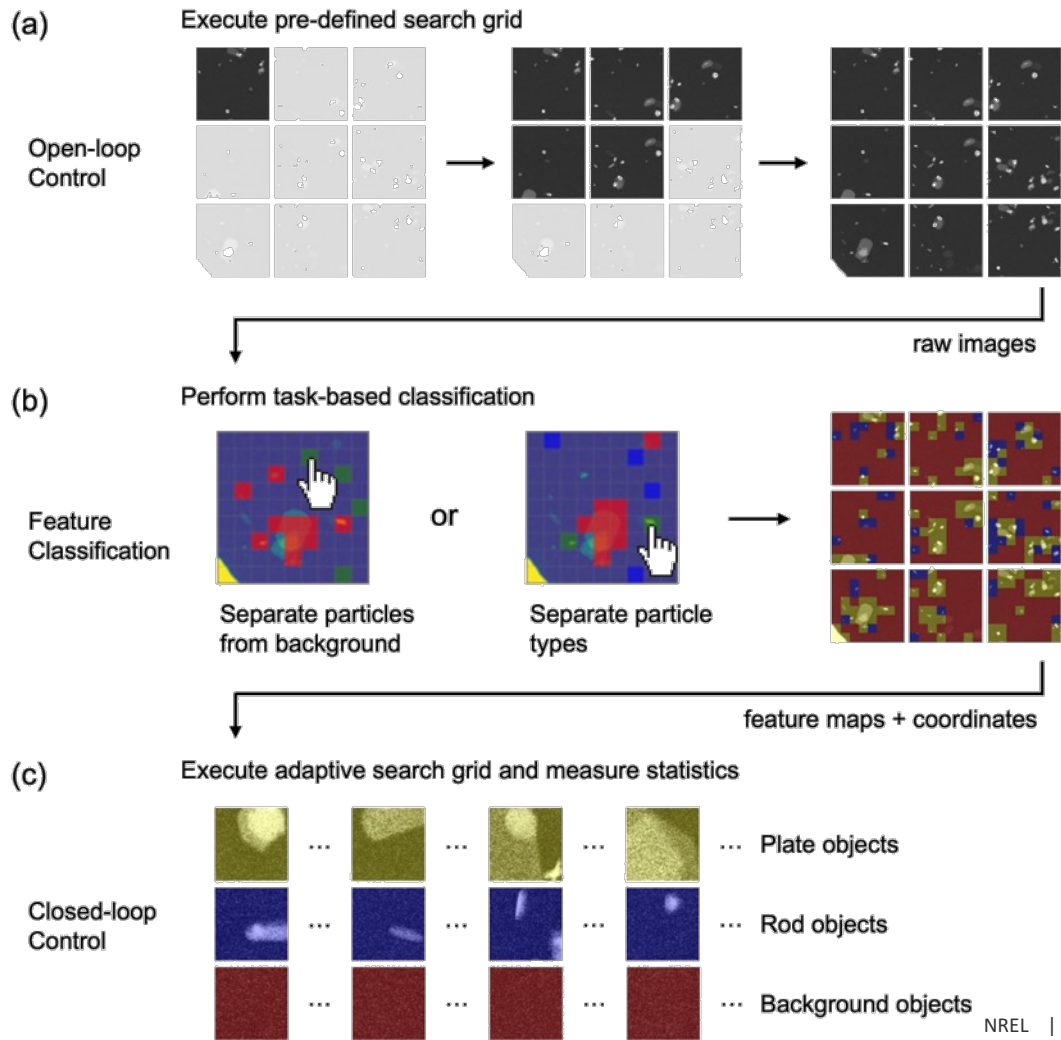
Toward Autonomous Experimentation

We have built an autonomous microscope platform based on few-shot and other ML models

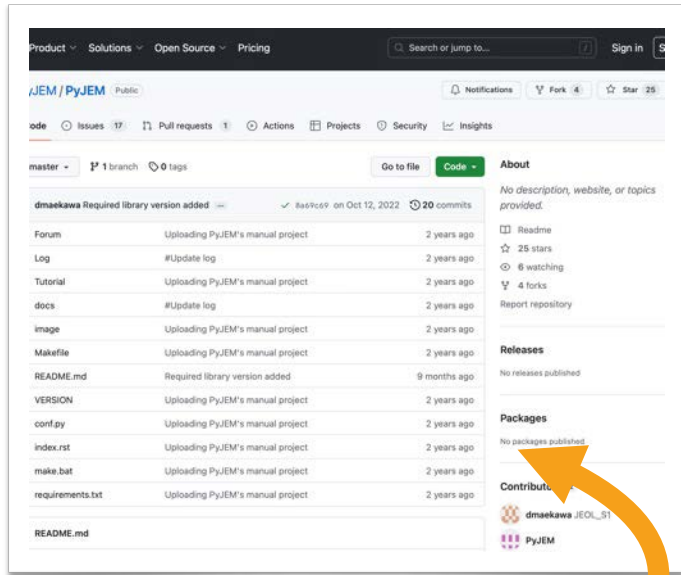


This platform enables intelligent closed-loop experiments and statistical analyses

Olszta et al. *Microscopy and Microanalysis*, 28 (5), 1611-1621. (2022).

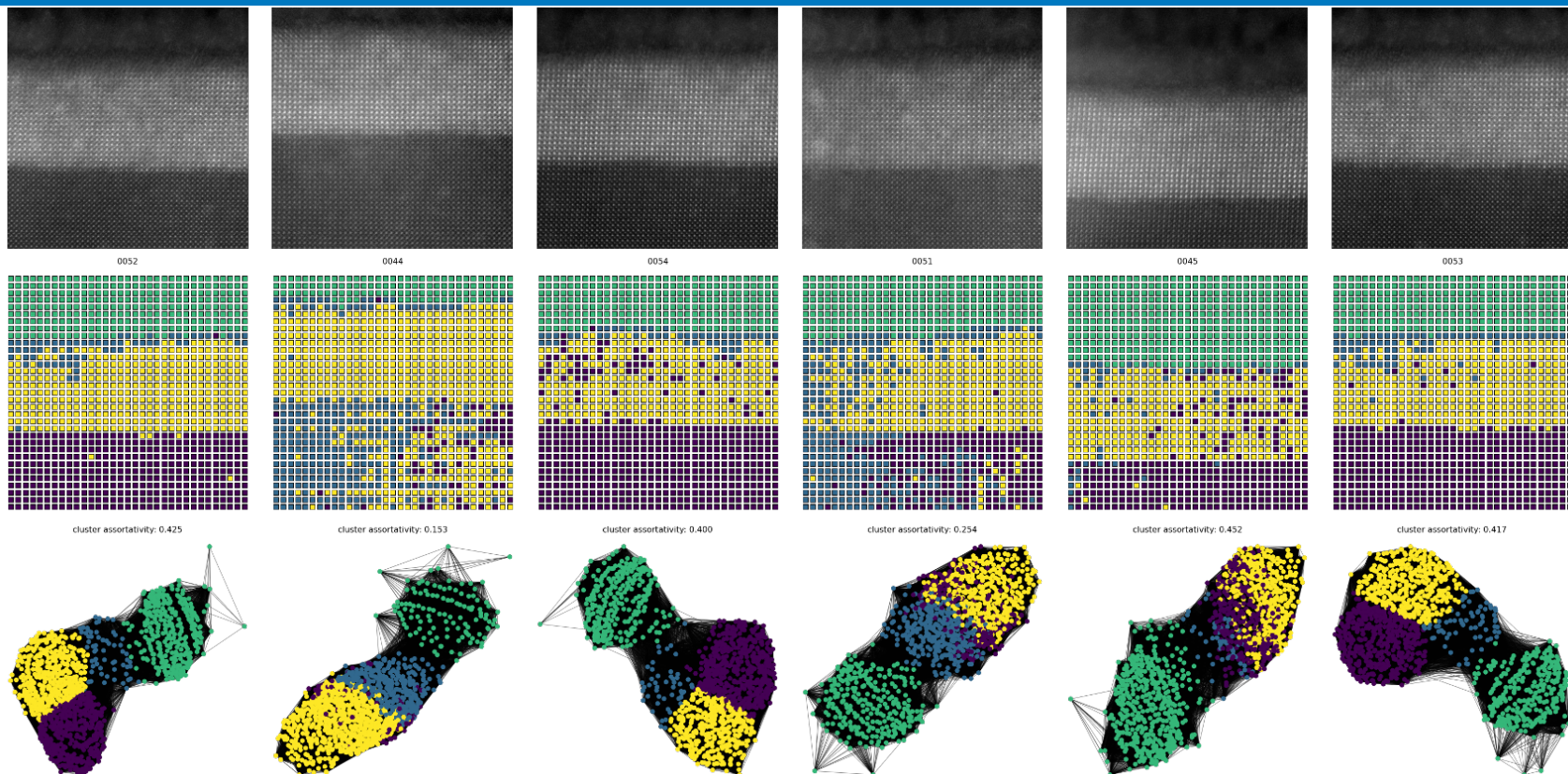


An Aside: Design of automated systems is challenging but it is getting easier



- **Electronic optical system control** : Beam control, detector In / Out, magnification change, brightness change, etc..
- **Stage control** : Absolute position movement, Relative position movement, Piezoelectric movement, etc.
- **Image acquisition** : STEM or TEM image acquisition, image storage type change, resolution specification, etc.
- **Auto function** : Auto Focus, Auto Contrast Brightness, Auto Stigmator, etc.

We can build large libraries of synthesis and degradation pathways



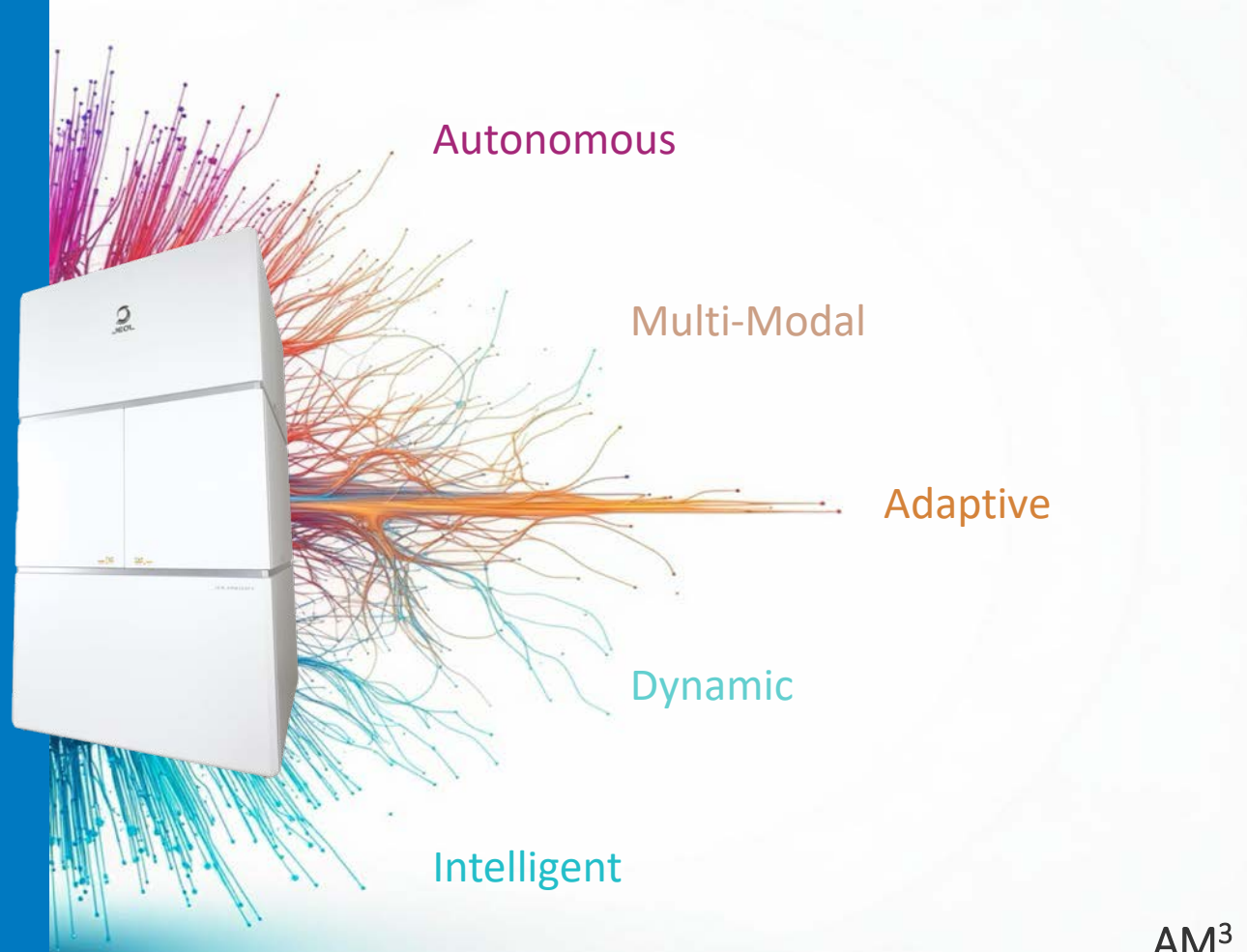
What is next?

NREL is leading a \$14M recapitalization of our electron microscopy center, with a focus on in situ and autonomous science



What is next?

NREL will be home to a new autonomous electron microscope platform built around dynamic and adaptive experiments



Autonomous

Multi-Modal

Adaptive

Dynamic

Intelligent

AM³

The Autonomous Multi-Modal Microscope

Autonomous science is revealing previously hidden materials lifecycles and transforming the design of clean energy systems

For more information on electron microscopy @ NREL, visit:
<https://tinyurl.com/z8ryk4y3>

NREL/PR-5K00-91333

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