NREL Transforming ENERGY

University of Colorado Boulder

The Rise of Intelligent Materials Science

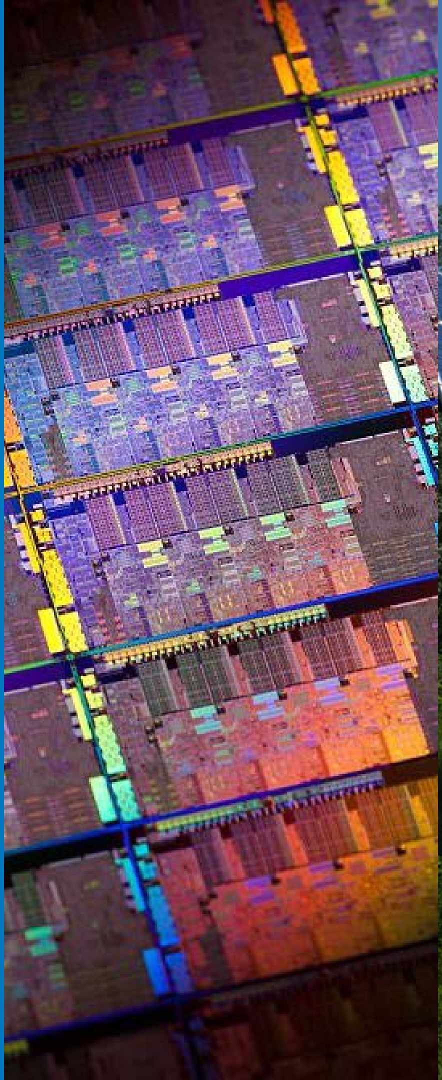
Unleashing the Power of Machine Intelligence in Characterization

Steven R. Spurgeon

CINT Annual User Meeting

September 16, 2024

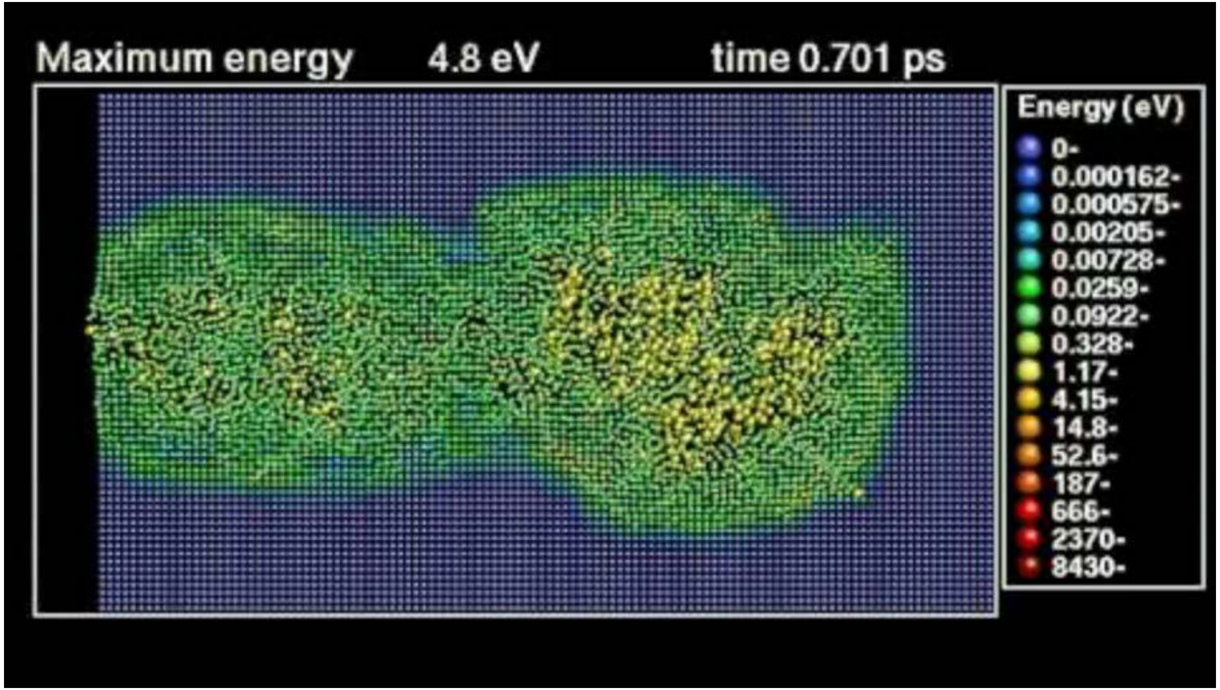
Everyday technologies are critically reliant on mastering materials lifecycles



Wafer: Intel Company
Array / SOFC: NREL

Such mastery depends on rich understanding of materials synthesis pathways…

…and degradation behavior in extremes



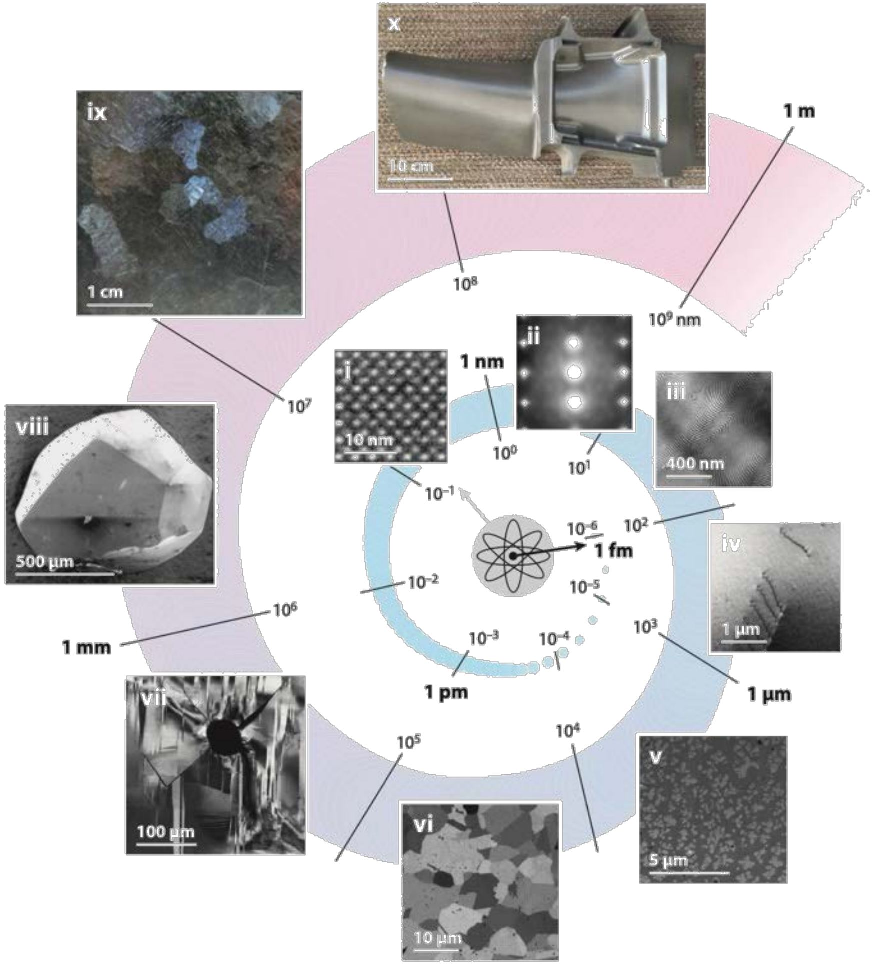
Averback and Diaz de la Rubia, Solid State Physics, edited by Ehrenfest and Spaepen 51 (Academic Press, New York 1998) 281.

https://www.youtube.com/watch?v=TyYBIj-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

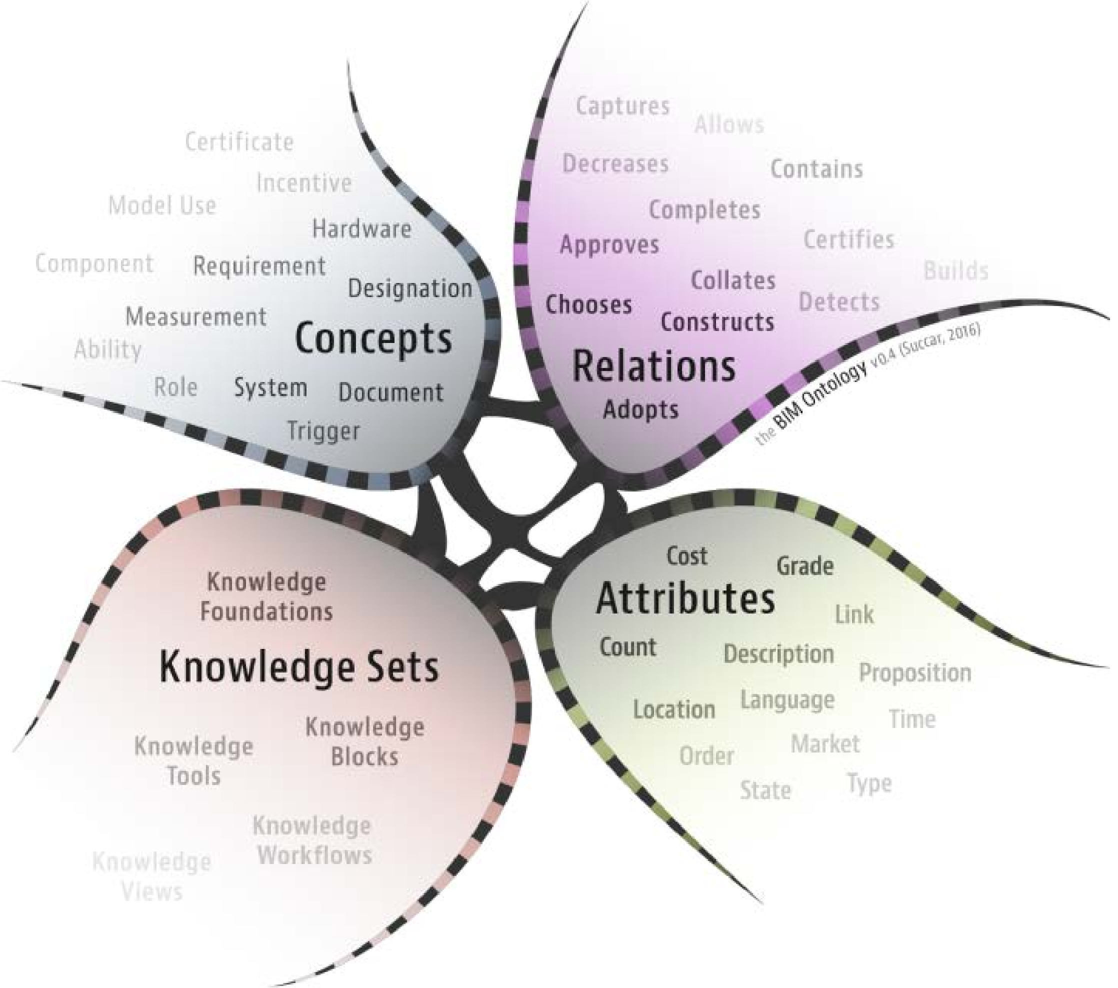


Kalidindi DOI:10.1146/annurev-matsci-070214-020844

Our aim is an ontology of the materials lifecycle:

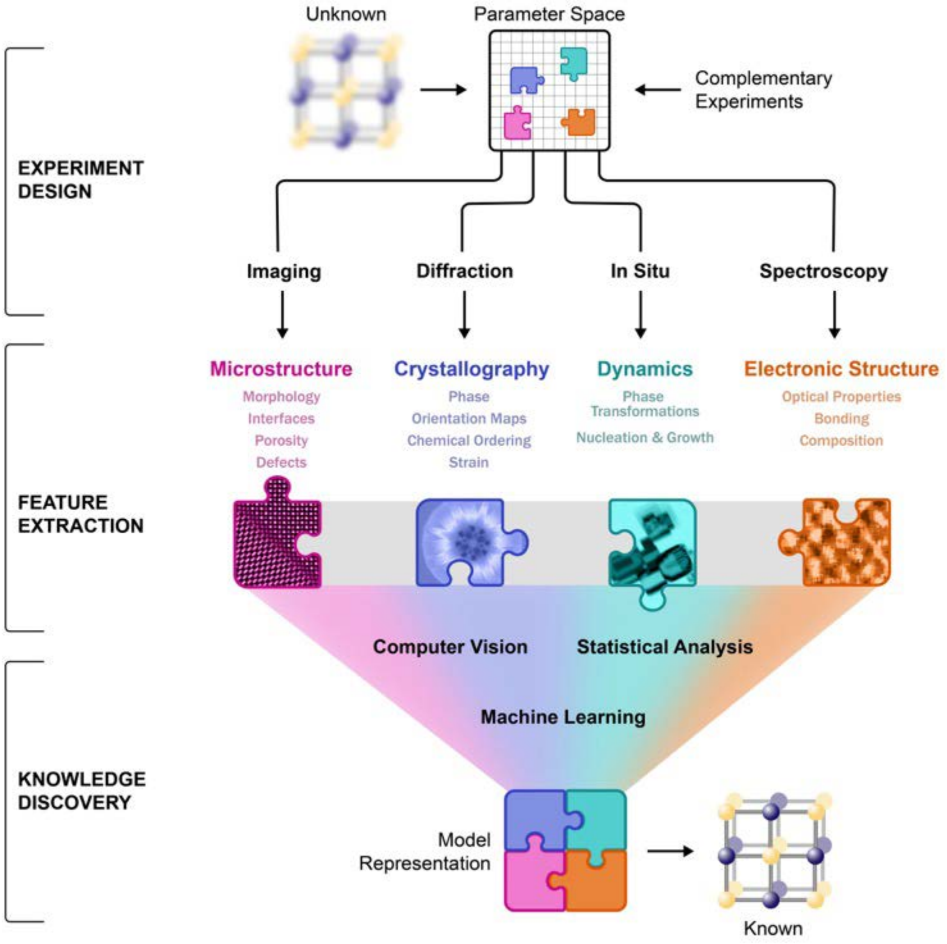
*A systematic mapping of data to meaningful semantic concepts…**across spatial and temporal scales***

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



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

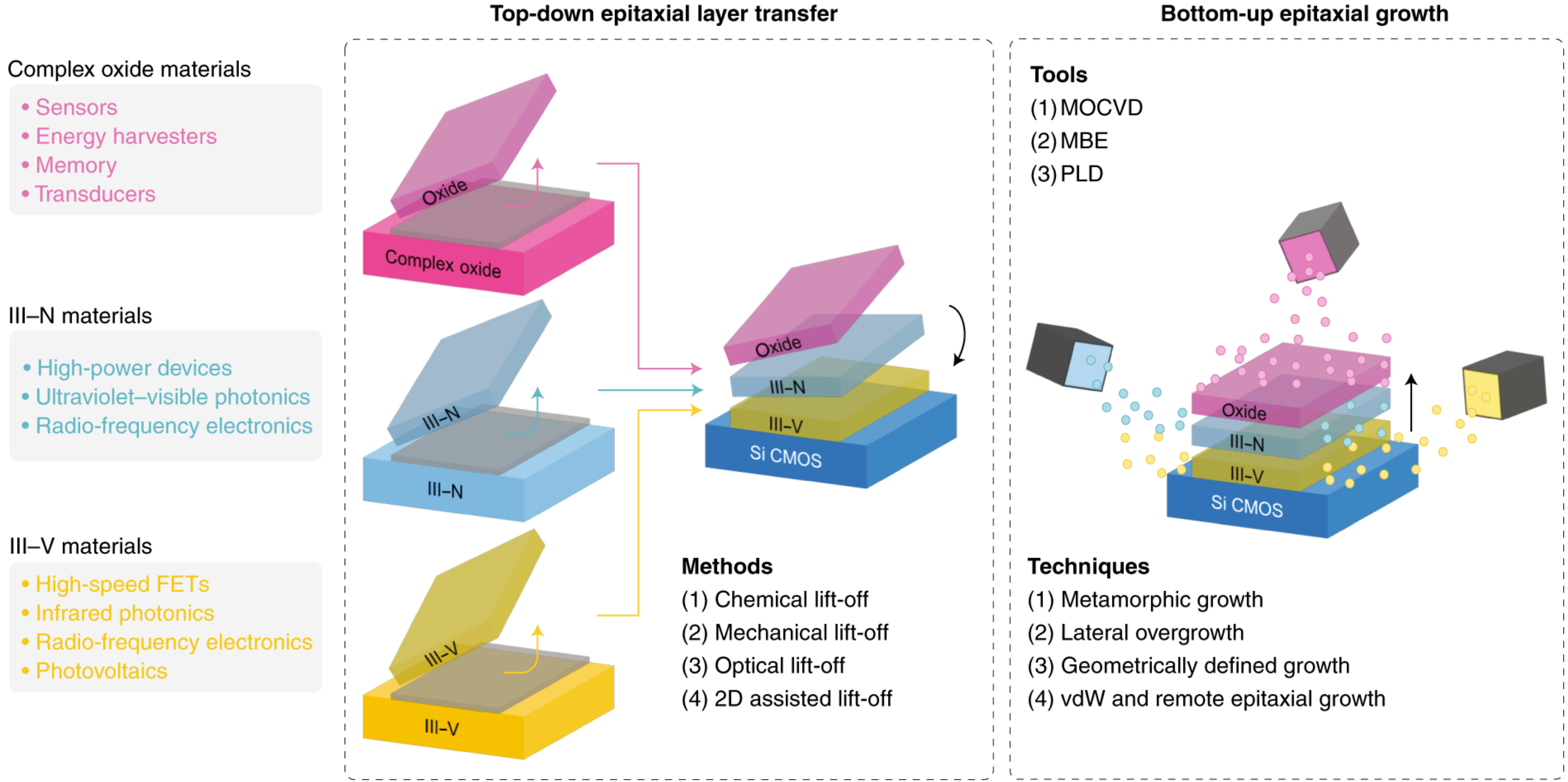
Such an ontology will transform raw multi-modal data into predictive physical models



Spurgeon et al. Nature mater, 20(3), 274–279. (2021).

Understanding Materials Synthesis

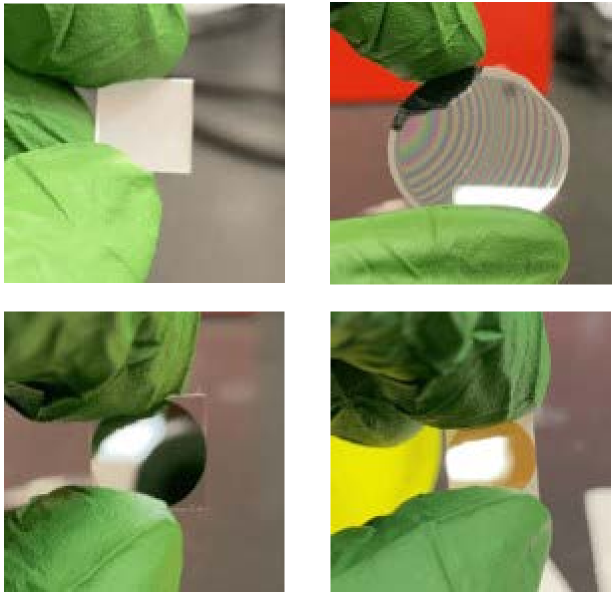
Epitaxial integration of semiconductors and oxides is a challenge for emerging devices



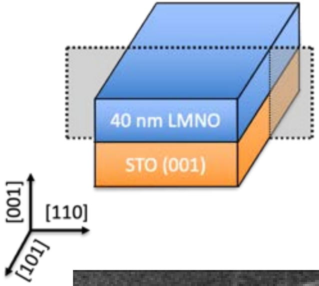
Kum et al. Nature Electron. 2, 439–450 (2019).

Tailored materials design requires direct local probes of structure and chemistry

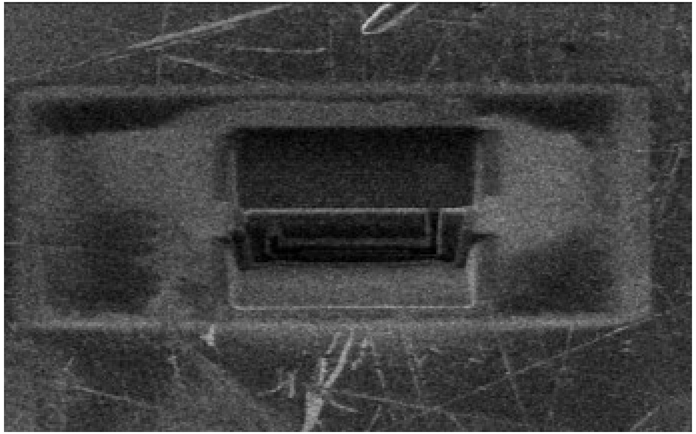
Functional Thin Films for Energy Applications



~1 cm



Site Specific Metrology



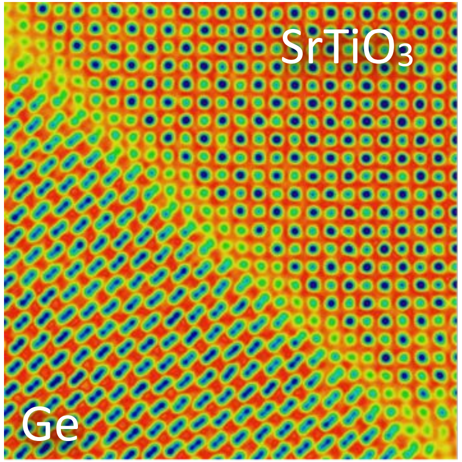
Focused Ion Beam

STEM

Spurgeon et al. Chem Mater, 28.11. 3814–3822. (2016).

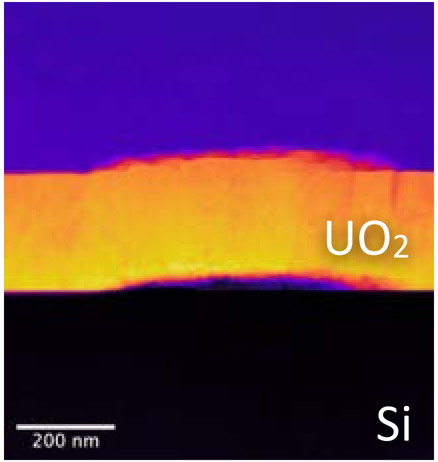
Electron microscopy can richly inform lifecycle models to achieve predictive control

Structure → **Chemistry**



Imaging

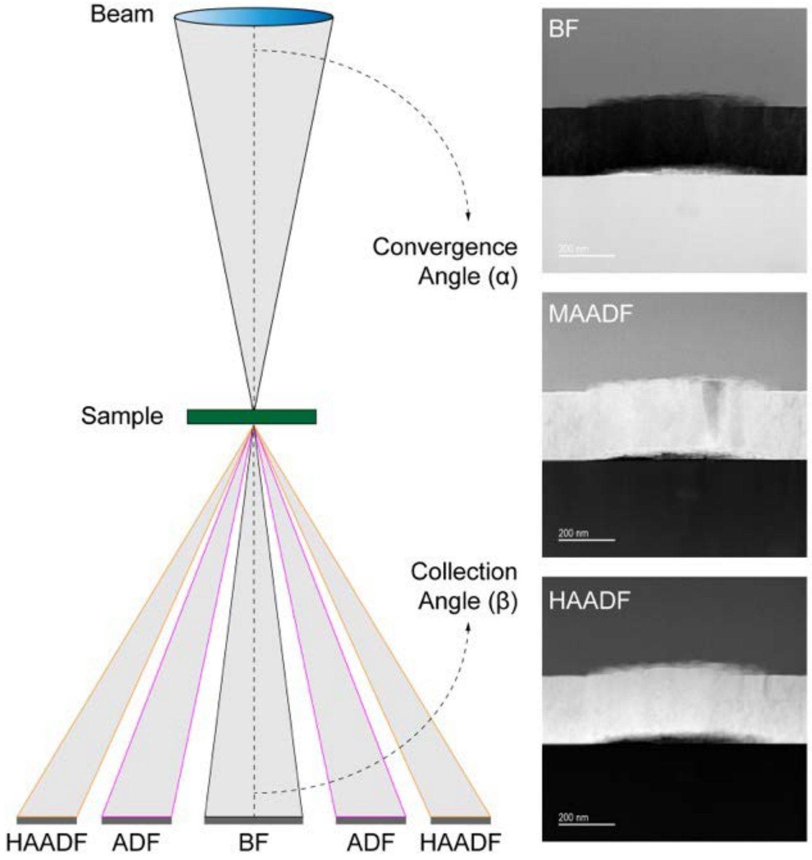
Diffraction



Spectroscopy

Du et al. Phys Rev B 2, 094602. (2018). | Popel et al. ACS Appl Mater Int. 12, 39781. (2020). | Spurgeon et al. PNAS 116, 17181. (2019).

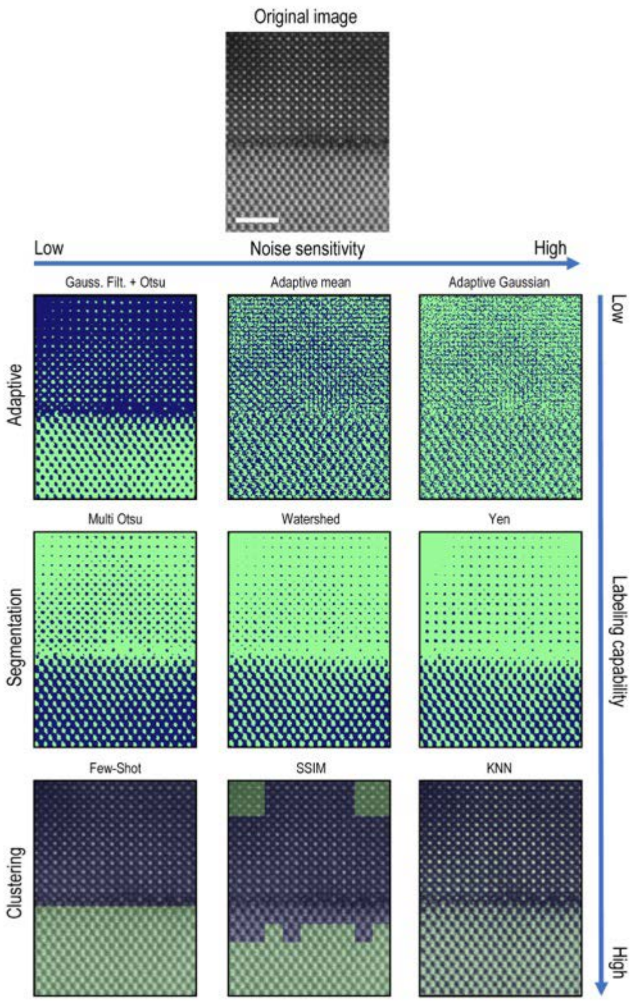
The Challenge
Imaging parameters strongly affect the representation of an object in data



Spurgeon. (2020). DOI:10.48550/arXiv.2001.00947

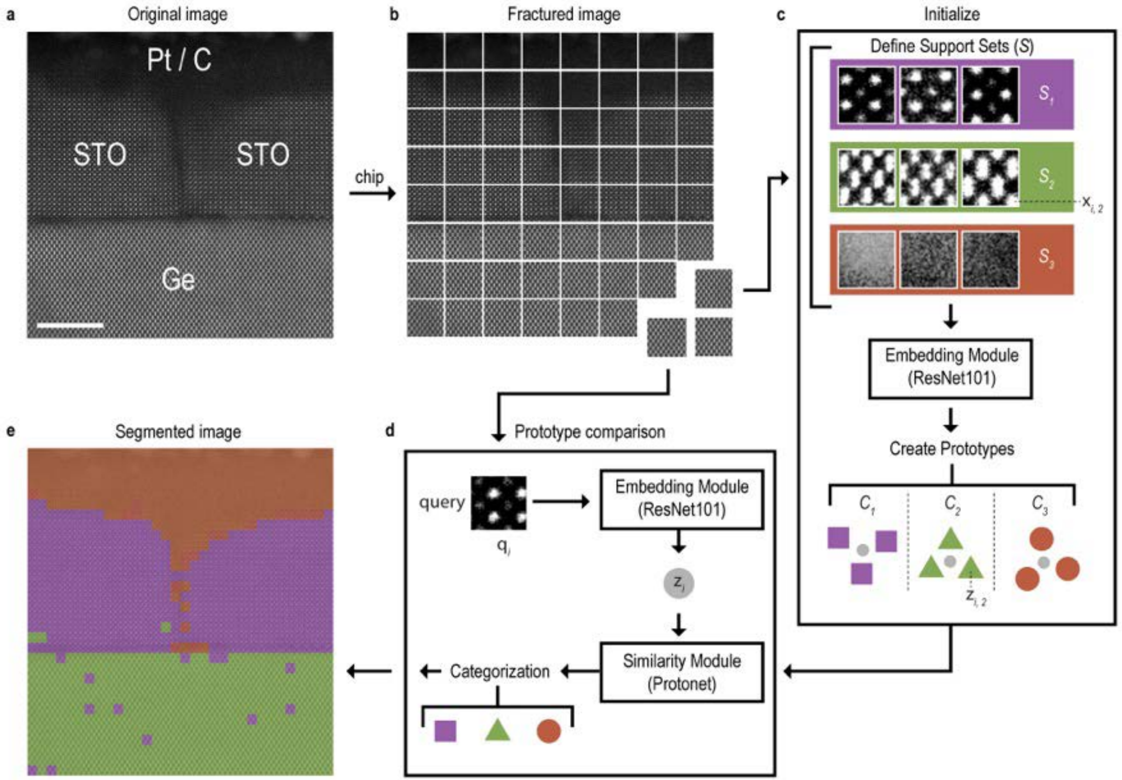
The Task

Classify microstructural features using limited examples



Akers et al. (2021). npj Computational Materials, 7(1), 187.

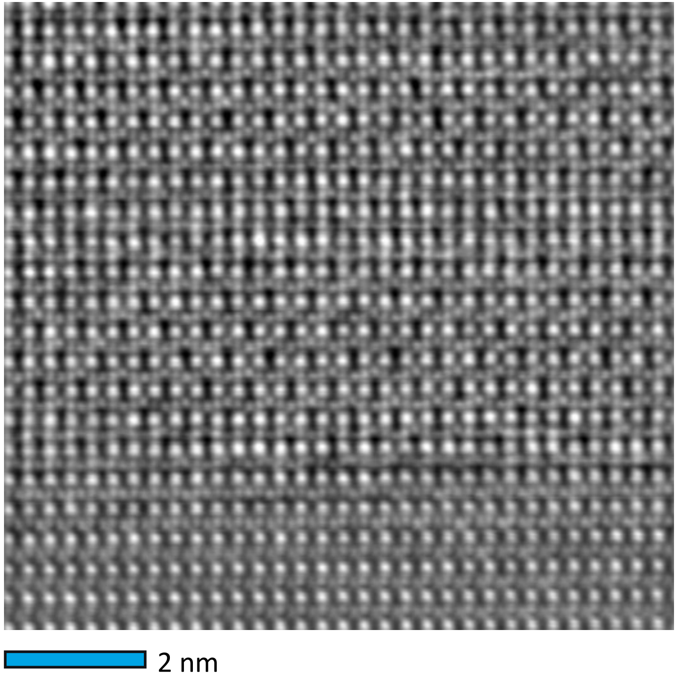
Few shot learning uses limited prior knowledge to classify features in discovery scenarios



Akers et al. (2021). npj Computational Materials, 7(1), 187.

Careful pre-processing is needed for best model performance

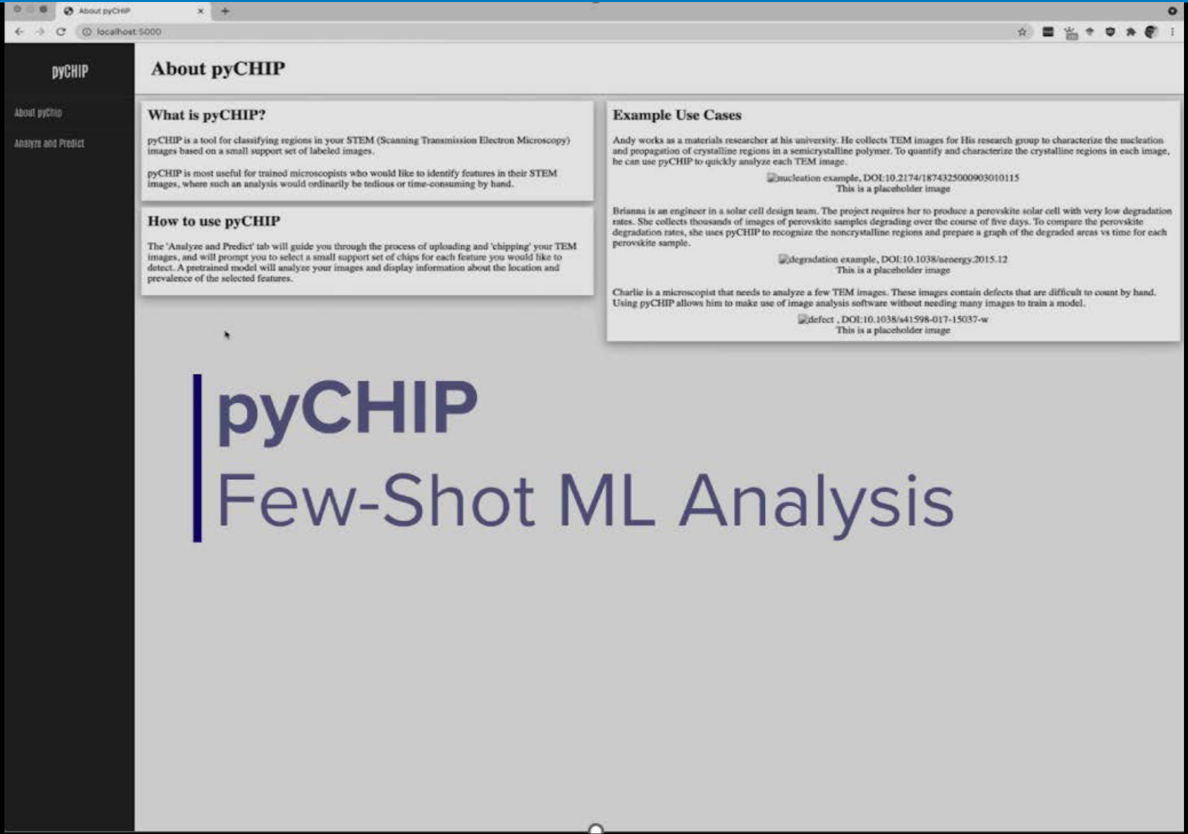
Without Contrast Leveling Adaptive Histogram Equalization (CLAHE)



With CLAHE

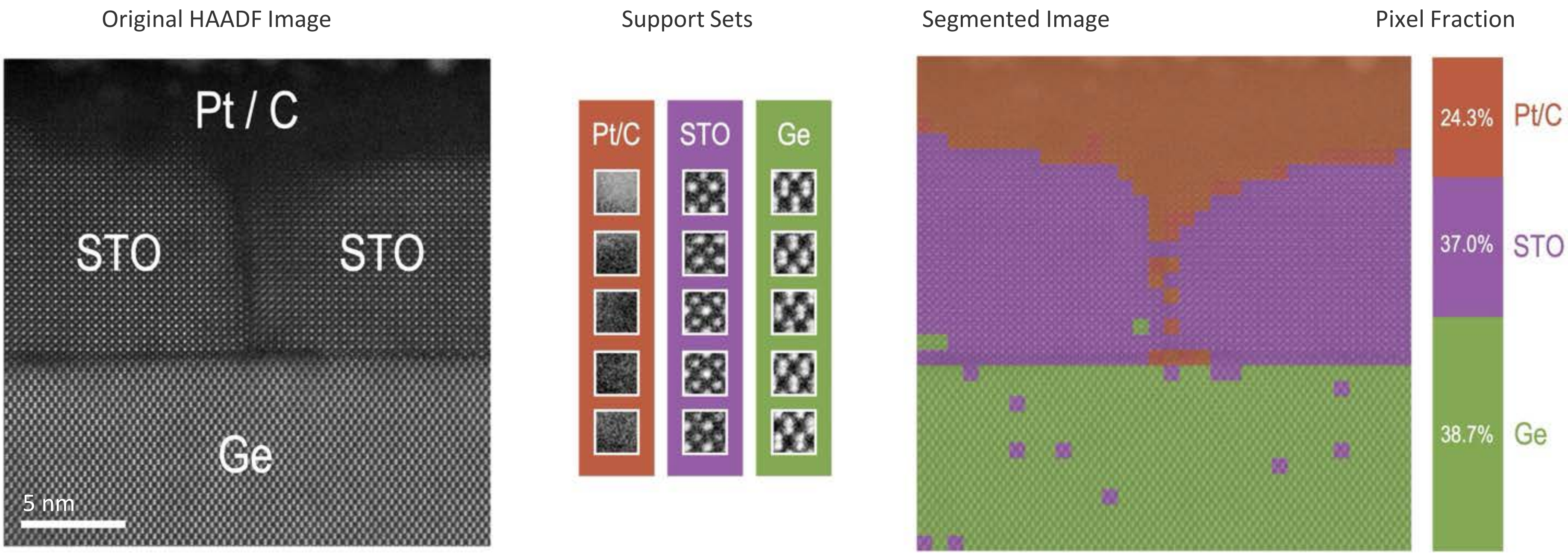
Akers et al. (2021). npj Computational Materials, 7(1), 187.

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



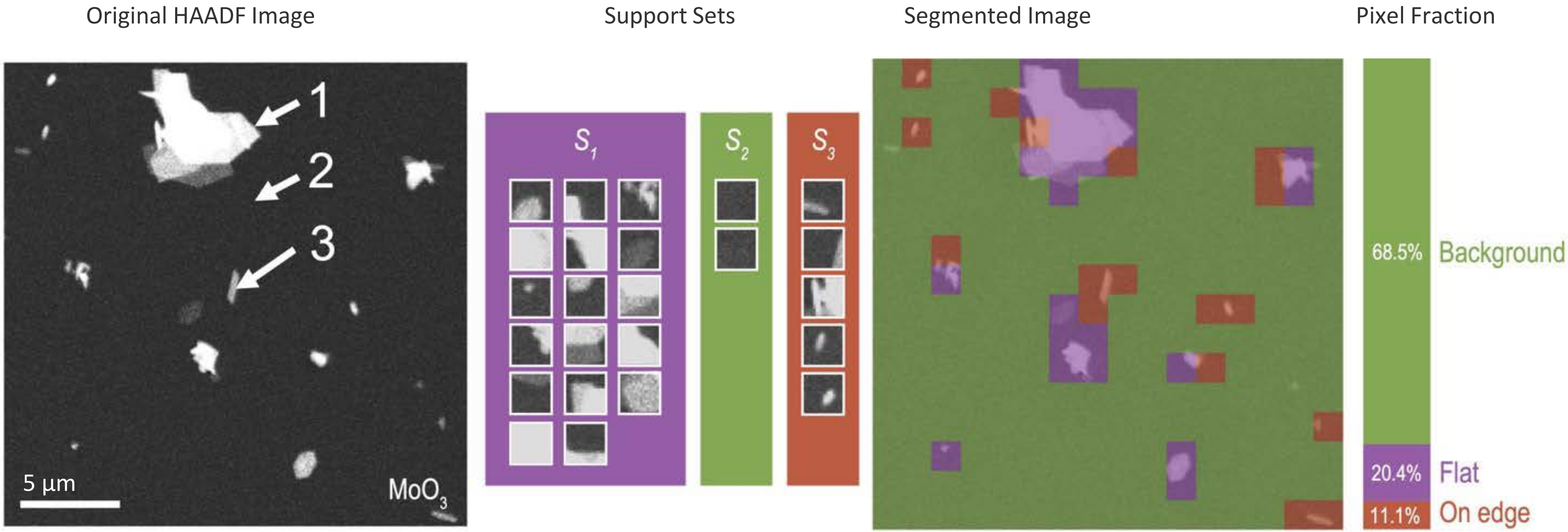
Doty et al. (2022). Computational Materials Science, 203, 111121.

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



Akers et al. (2021). npj Computational Materials, 7(1), 187.

Such a model can easily be applied to different synthesis tasks

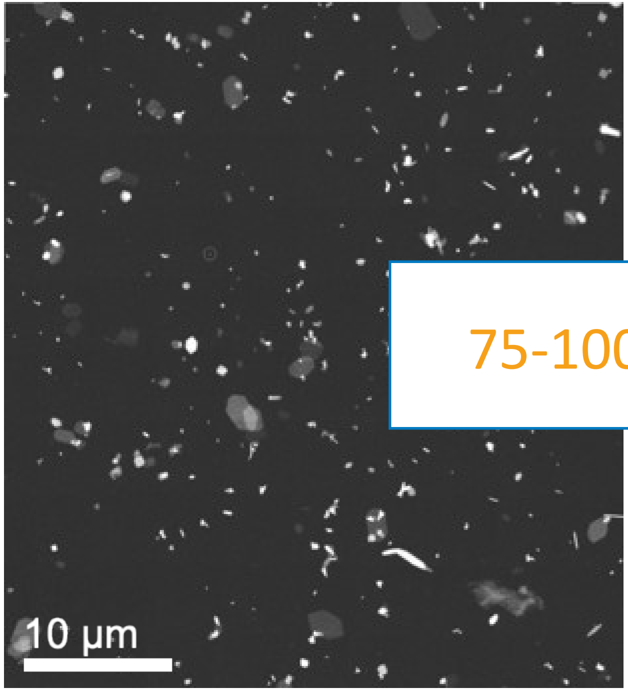


Akers et al. (2021). npj Computational Materials, 7(1), 187.

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

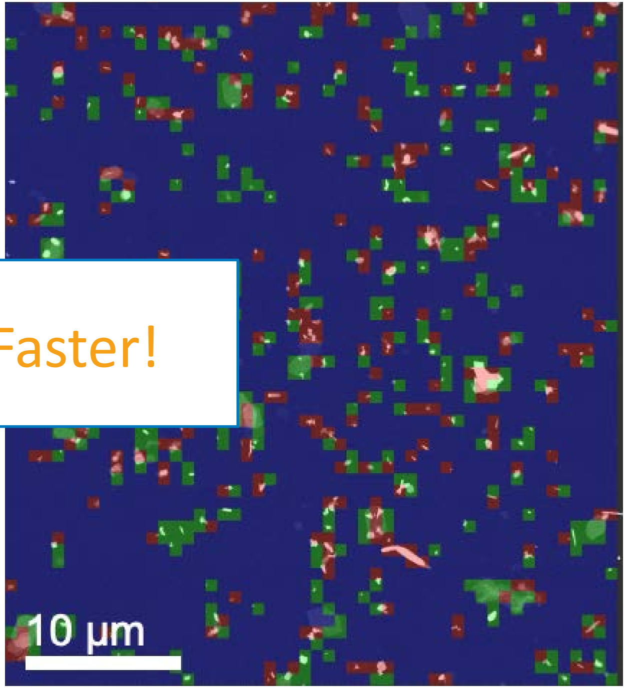
MoO_3

Manual Analysis
10 minutes



Few-Shot
Task 1
8 seconds

75-100x Faster!

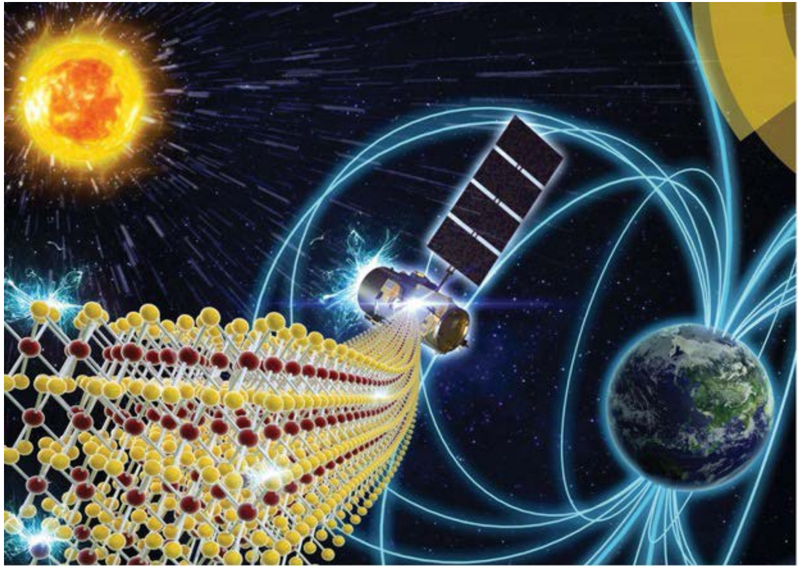


Few-Shot
Task 2
8 seconds

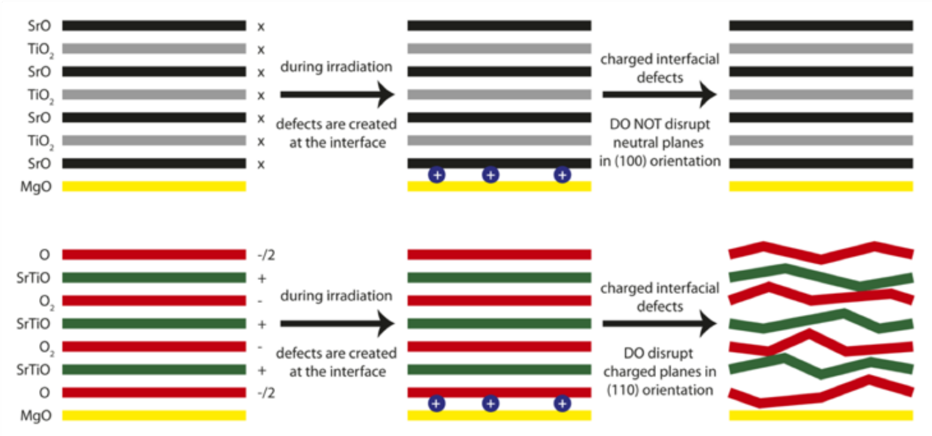
Doty et al. (2022). Computational Materials Science, 203, 111121. | Akers et al. (2021). npj Computational Materials, 7(1), 187.

Describing Disorder

Controlling materials degradation is critical for electronics and sensors in extremes

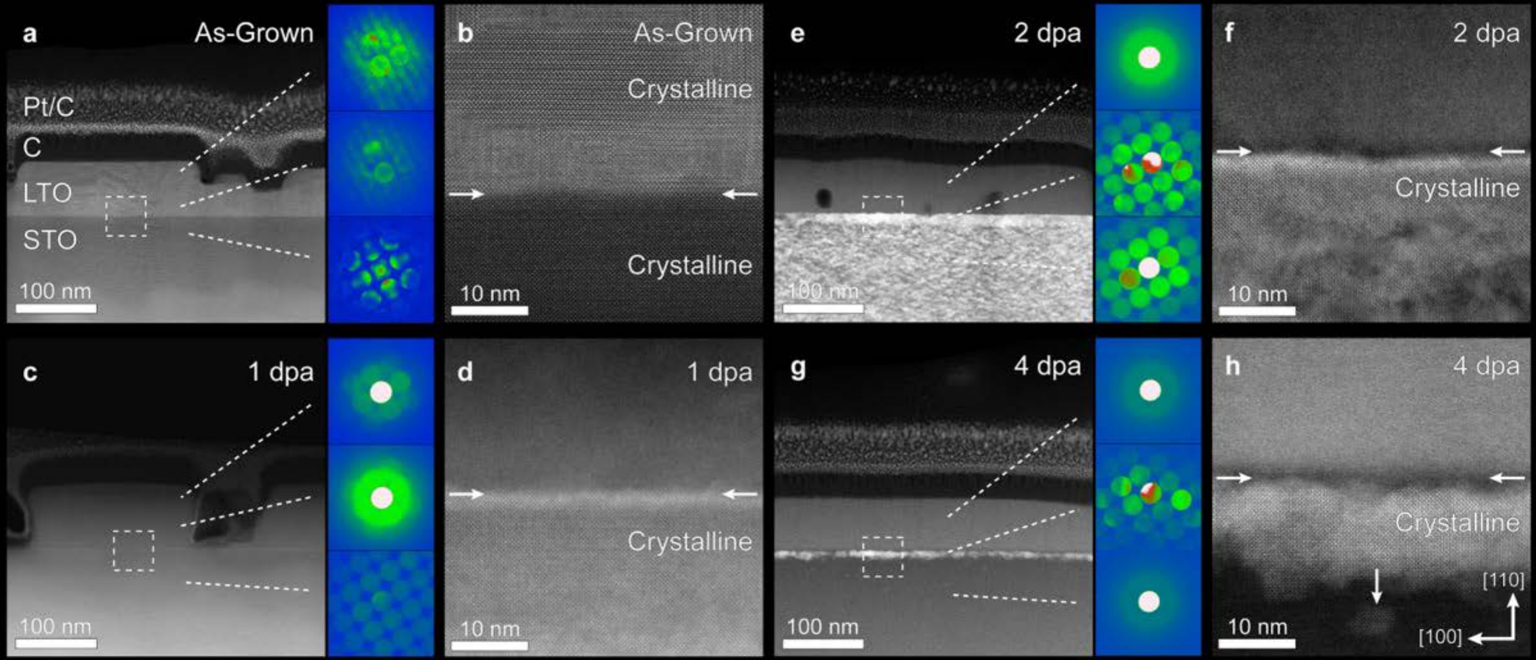


Interface charge affects radiation-induced disorder



Aguiar et al. J. Mater. Res. 29(16), 1699–1710 (2014). | Spurgeon. Curr. Opin. Solid. State Mater. Sci. 24(6), 100870 (2020). | Kim et al. J. Mater. Chem. C 7, 10–24 (2018).

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



Spurgeon, S. R. et al. Adv. Mater. Interfaces. 2020, 7(8). 1901901944.

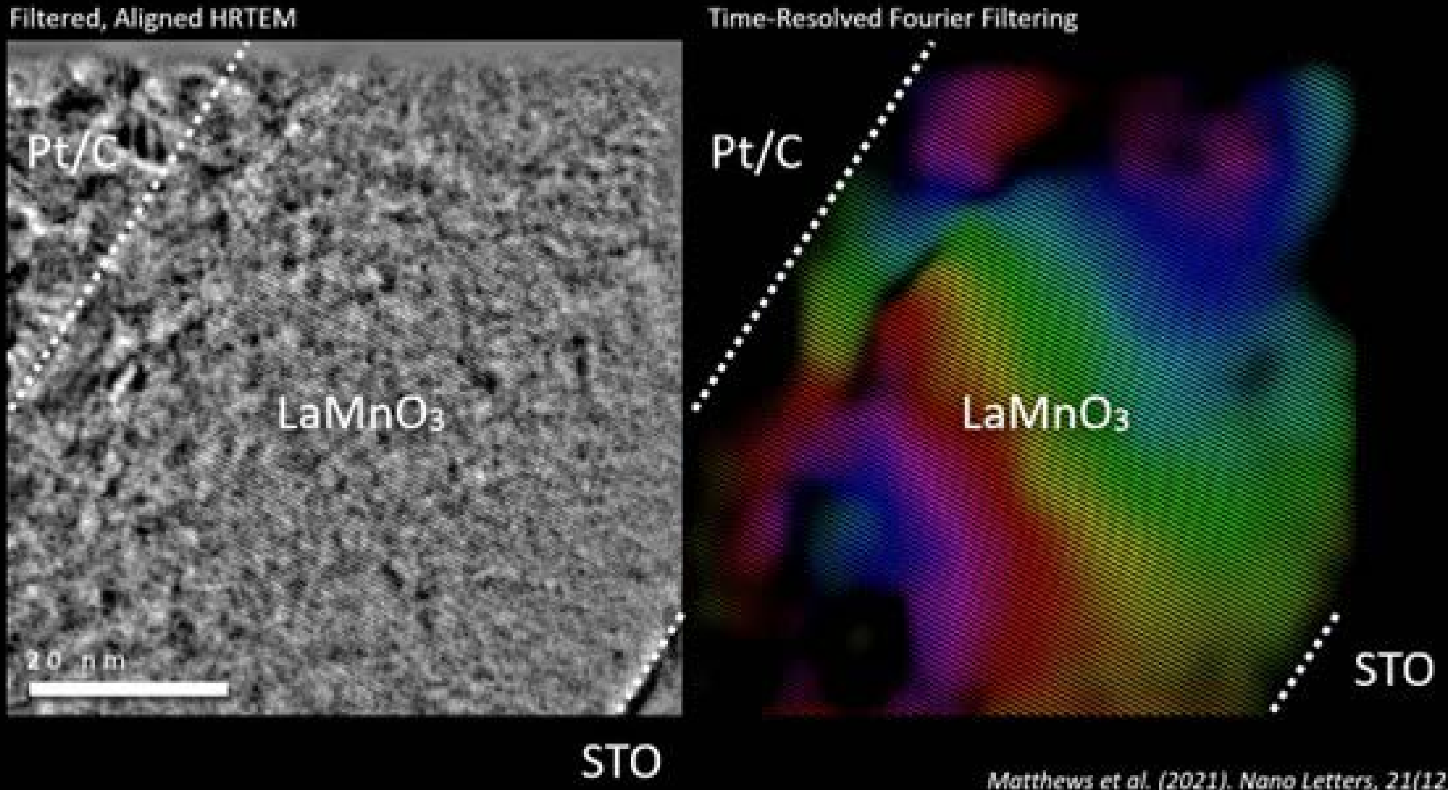
At CINT we took this one step further, visualizing materials breakdown in situ using the I^{3}TEM

Khalid Hattar (UTK)

Chris Barr

https://tours.sandia.gov/ibl_info.html

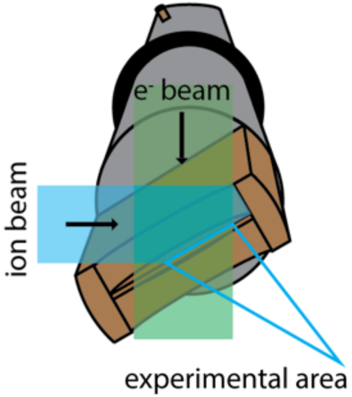
In situ TEM shows that disorder percolates through the material in a non-uniform manner

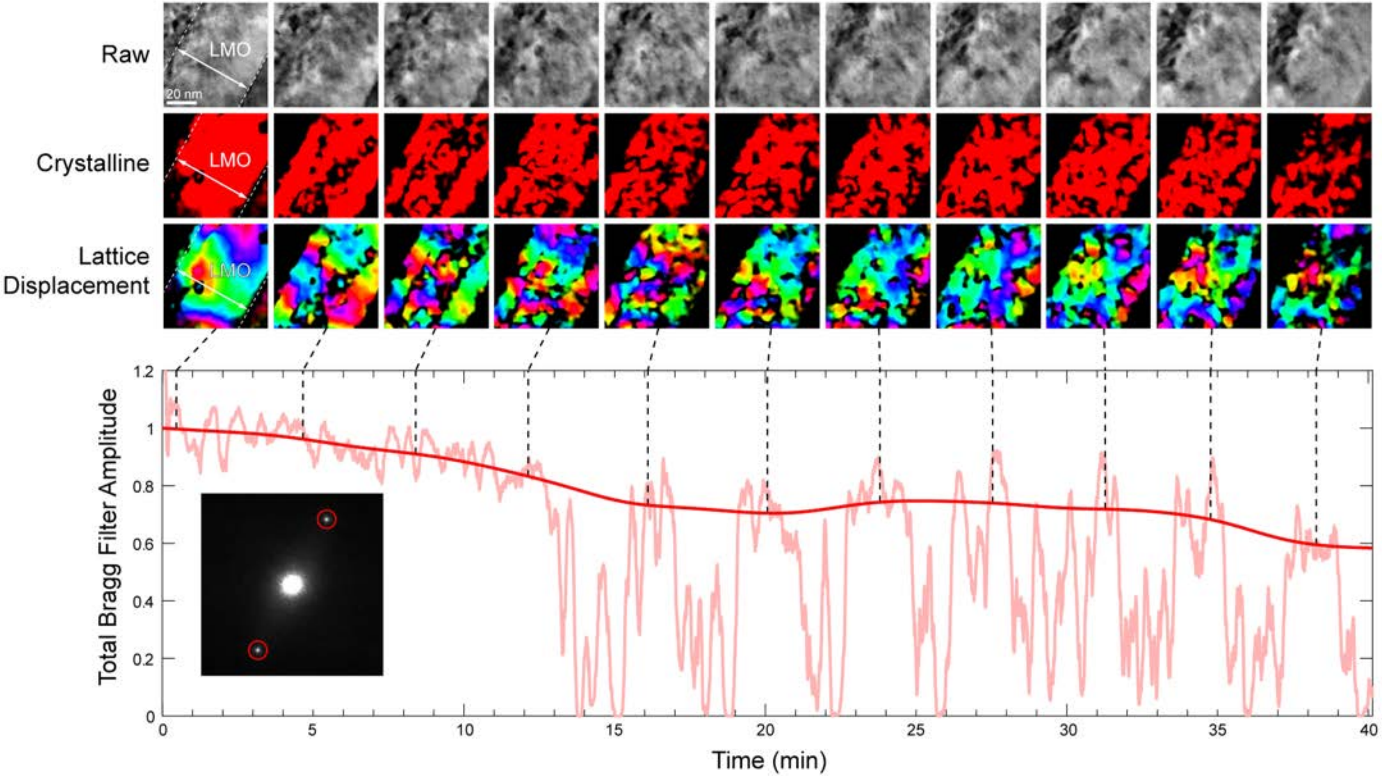


1 Hr Irradiation

Matthews et al. (2021). Nano Letters, 21(12), 5353–5359.

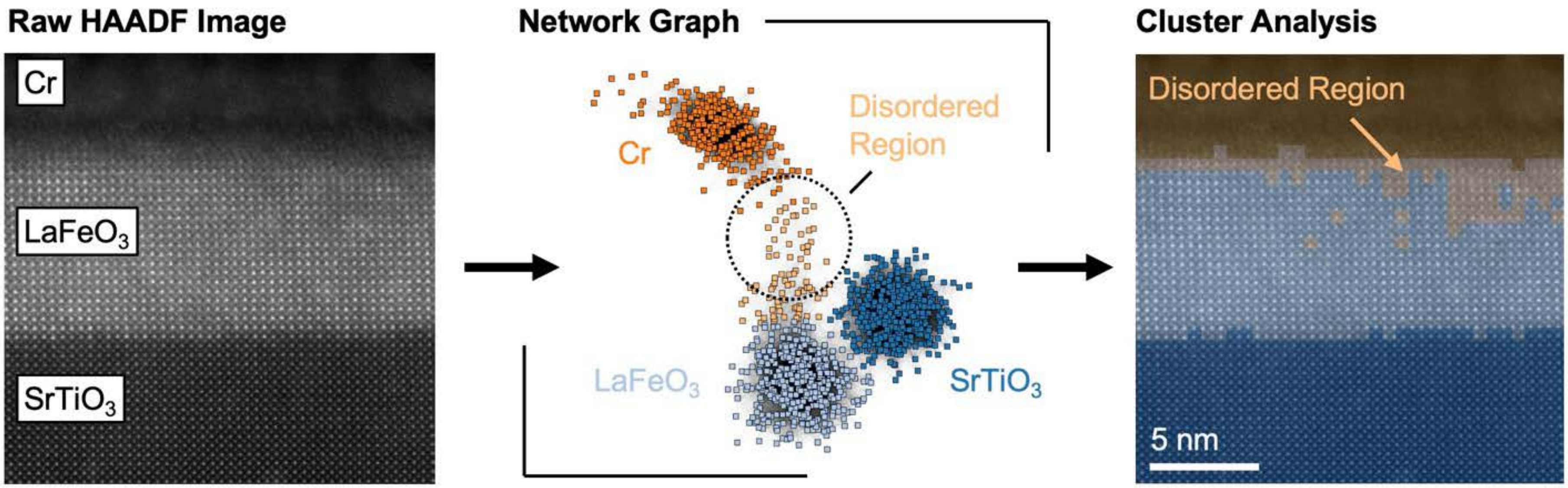
We can quantify the loss of crystallinity through statistical analysis of time series data





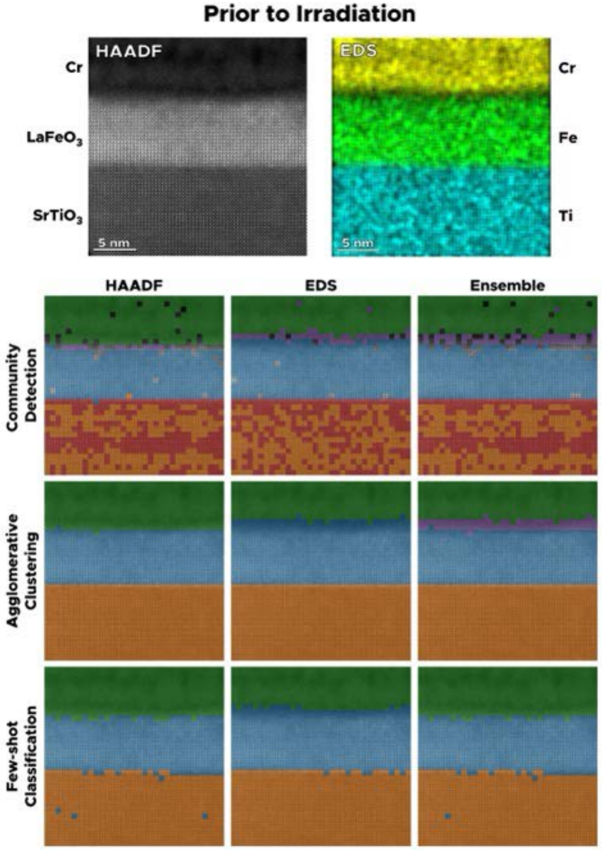
Matthews et al. (2021). Nano Letters, 21(12), 5353–5359.

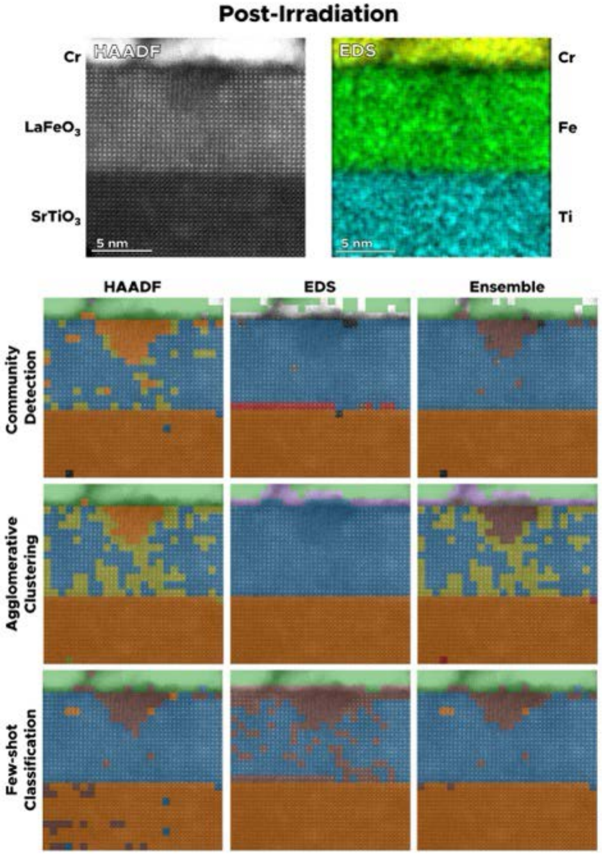
More recently, we have been developing unique order descriptors based on graph analytics



Ter-Petrosyan et al. Proc. Thirty-Seventh Conf. on Neural Information Processing Systems (NeurIPS). (2023). DOI:10.48550/arXiv.2311.08585

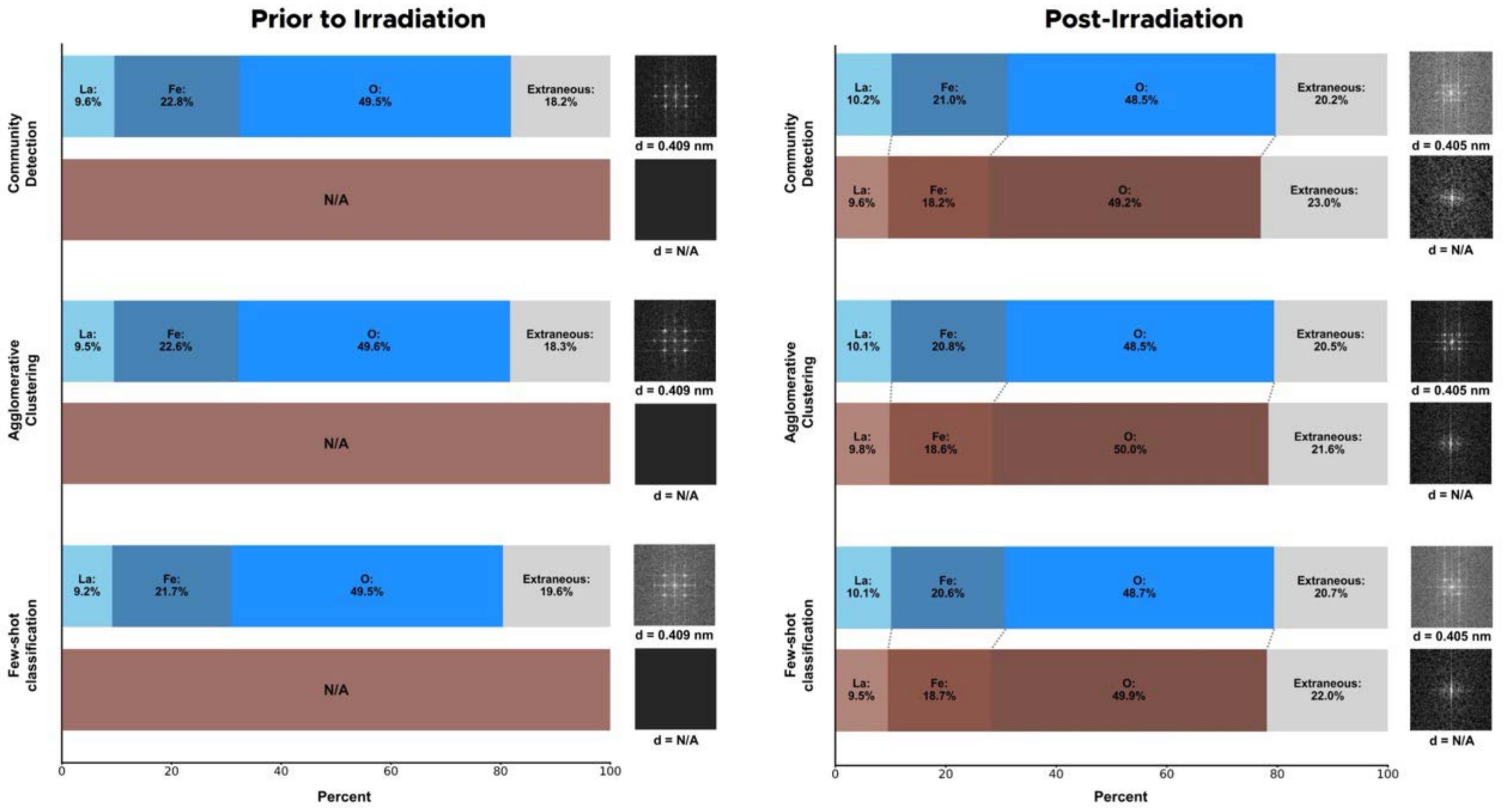
Multi-modal graphs effectively classify radiation damage signatures





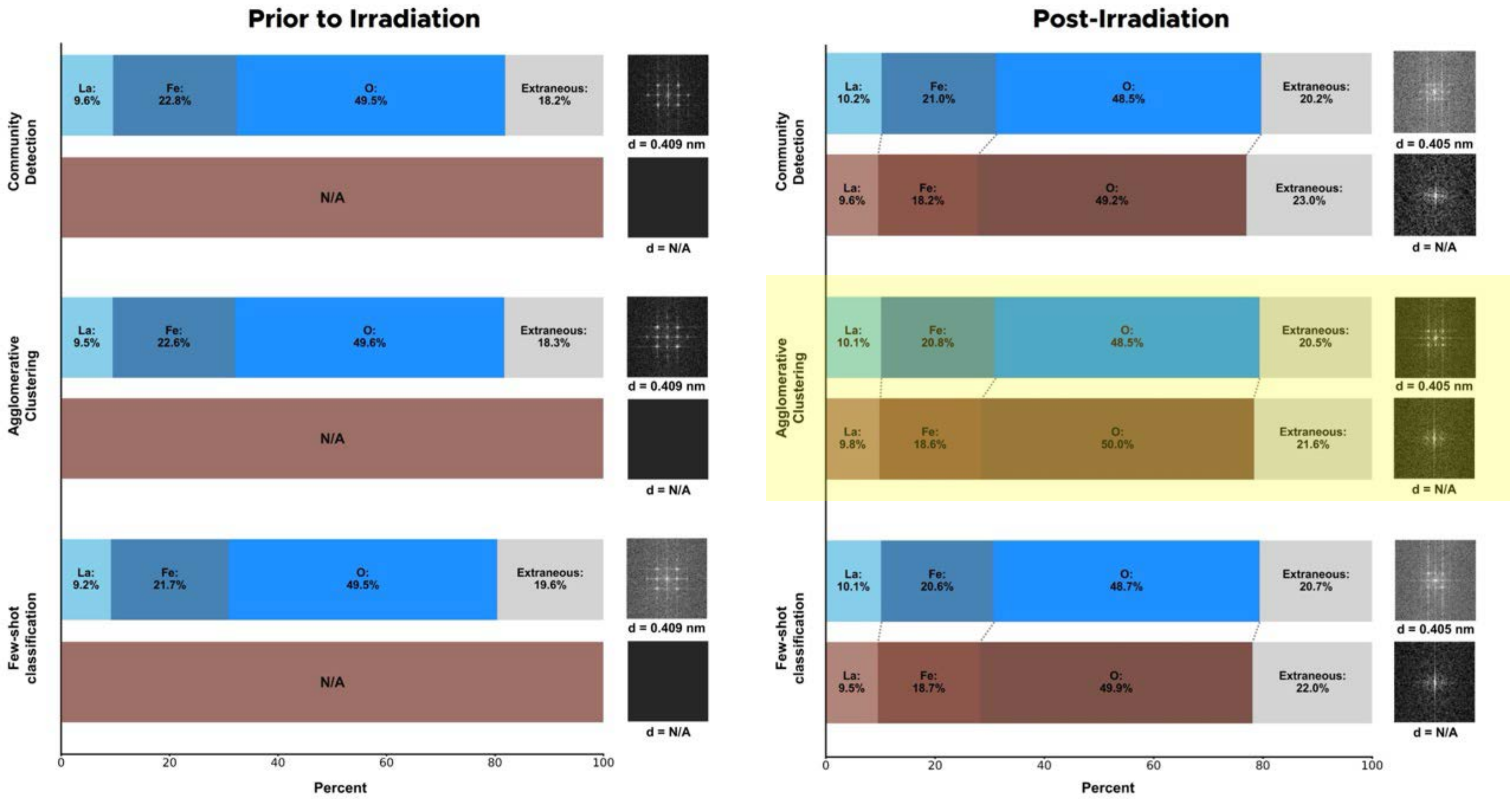
Ter-Petrosyan et al. In preparation.

Such models reveal changes in composition associated with irradiation



Ter-Petrosyan et al. In preparation.

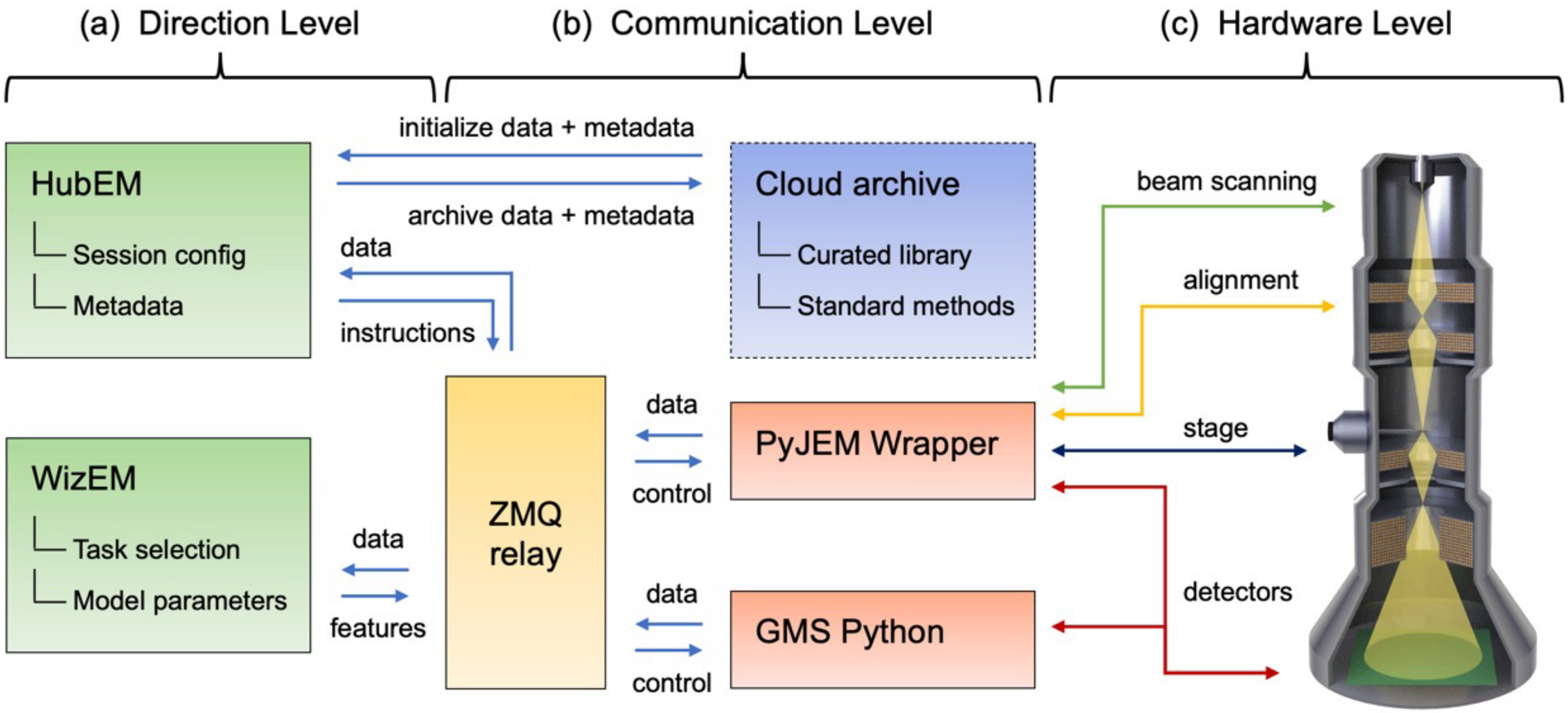
Such models reveal changes in composition associated with irradiation



Ter-Petrosyan et al. In preparation.

Toward Autonomous Experimentation

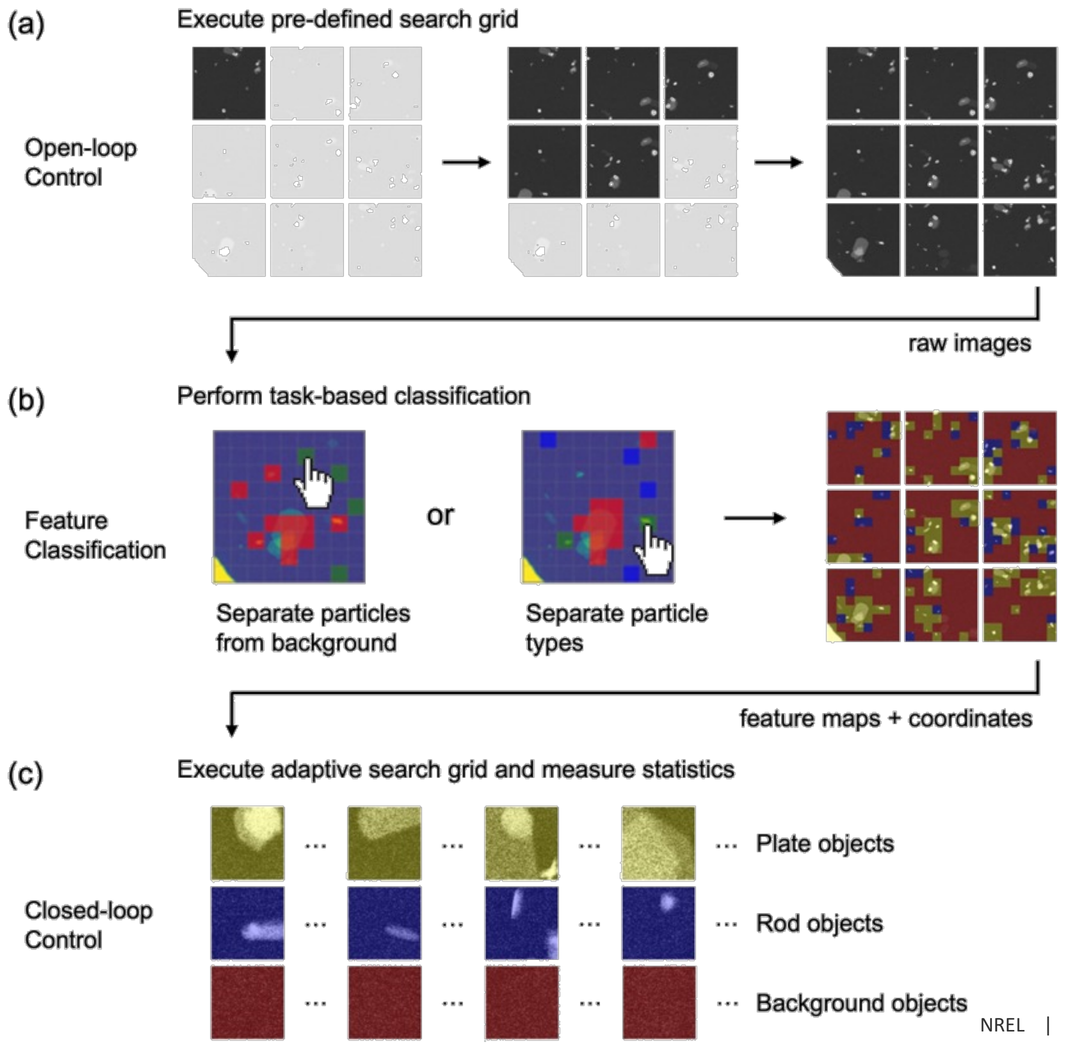
We have built an autonomous microscope platform based on few-shot and other ML models



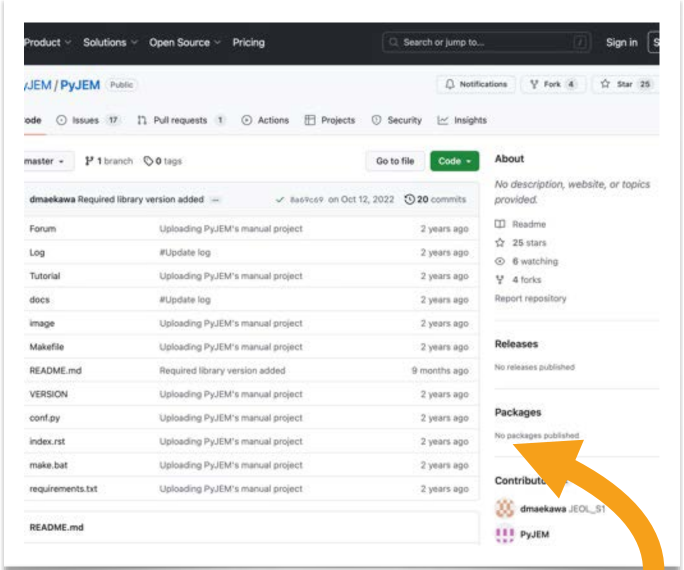
Olszta et al. Microscopy and Microanalysis, 28 (5), 1611-1621. (2022).

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

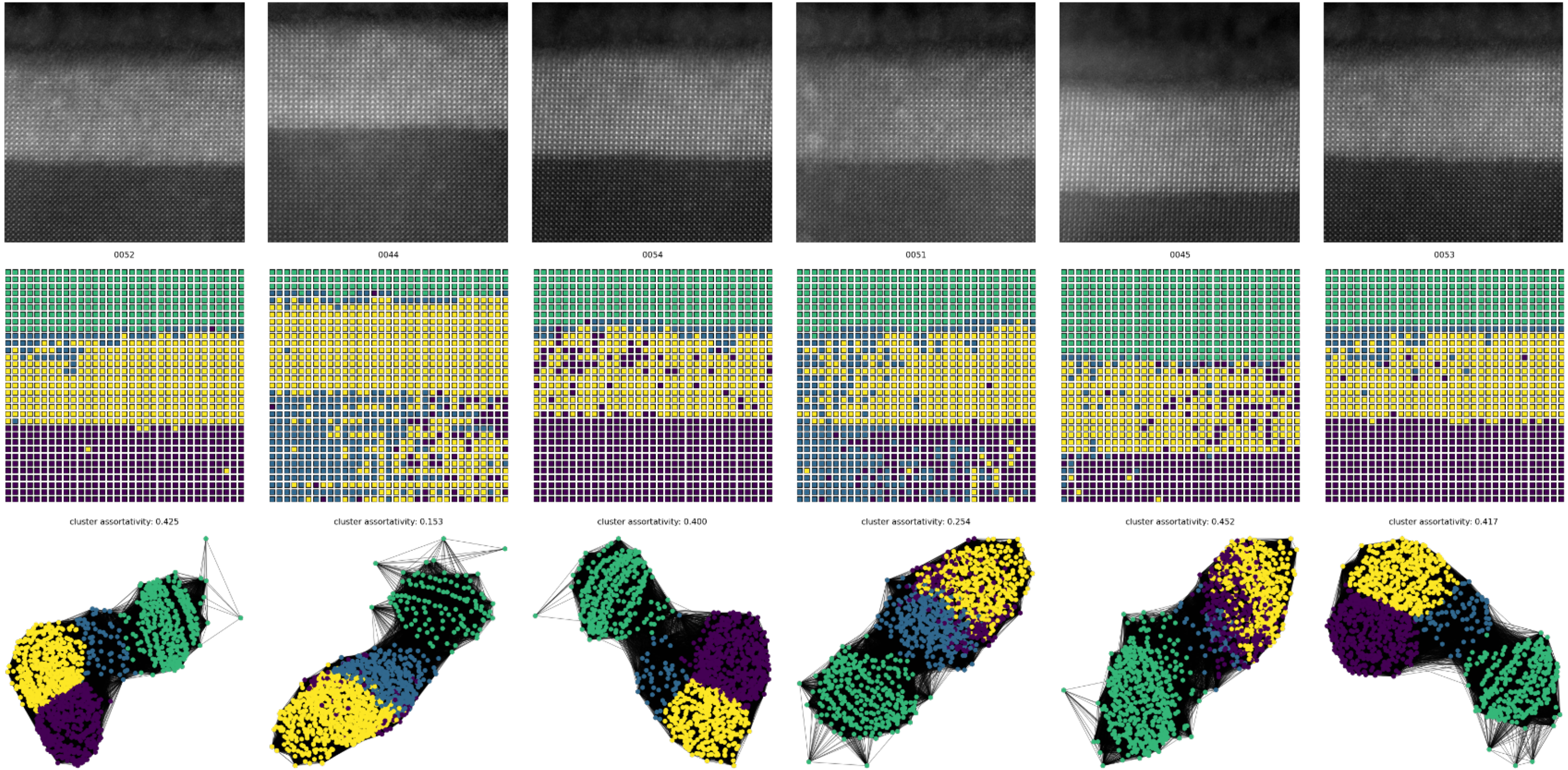


Get the PyJEM API!

- **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.

https://github.com/PyJEM/PyJEM

We can build large libraries of synthesis and degradation pathways



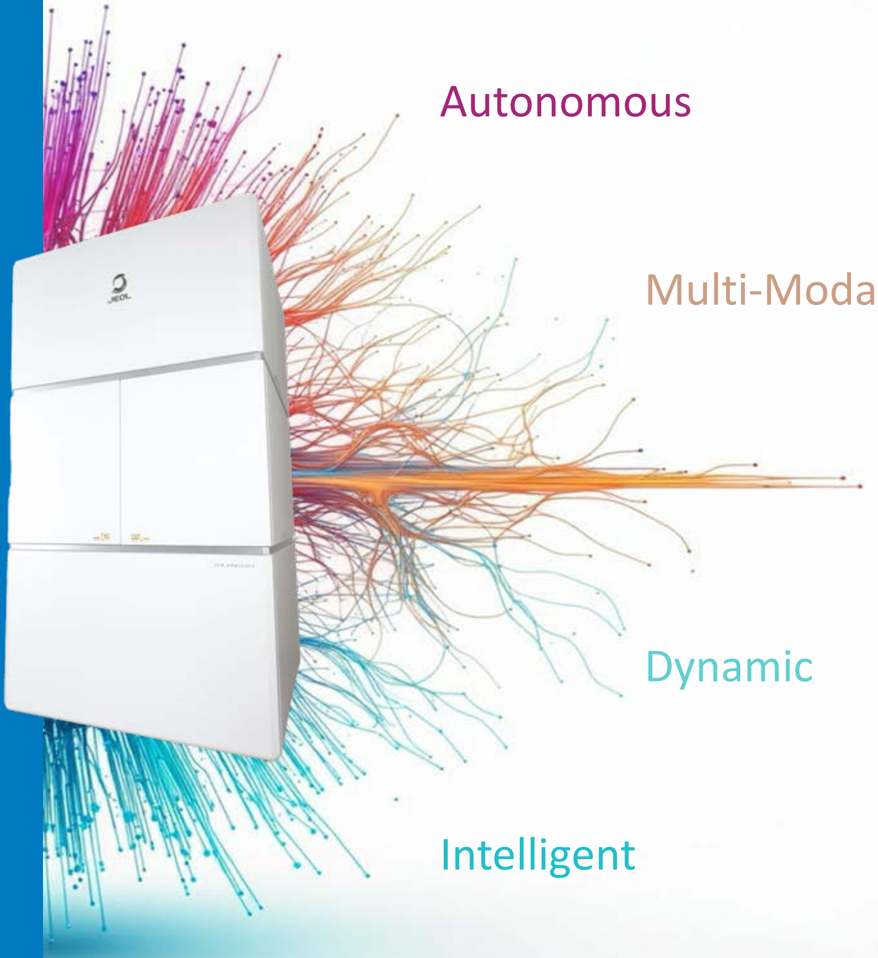
Ter-Petrosyan et al. Proc. Thirty-Seventh Conf. on Neural Information Processing Systems (NeurIPS). (2023). DOI:10.48550/arXiv.2311.08585

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

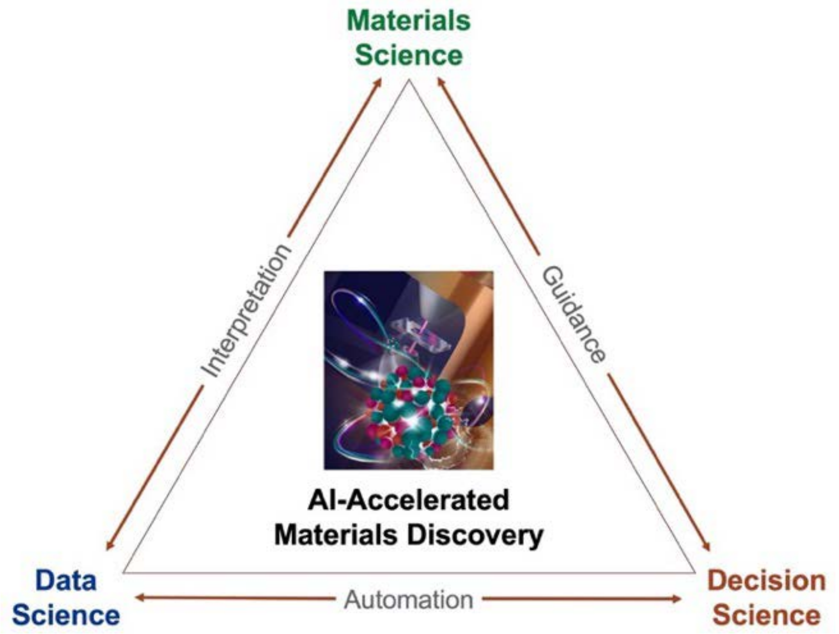
AM^3

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



This work was authored by the National Renewable Energy Laboratory, operated by Alliance for Sustainable Energy, LLC, for the U.S. Department of Energy (DOE) under Contract No. DE-AC36-08GO28308. The views expressed in the presentation do not necessarily represent the views of the DOE or the U.S. Government. The U.S. Government retains and the publisher, by accepting the article for publication, acknowledges that the U.S. Government retains a nonexclusive, paid-up, irrevocable, worldwide license to publish or reproduce the published form of this work, or allow others to do so, for U.S. Government purposes. The development of the few-shot model was supported by the Laboratory Directed Research and Development (LDRD) program at Pacific Northwest National Laboratory (PNNL). PNNL is a multiprogram national laboratory operated for the U.S. Department of Energy (DOE) by Battelle Memorial Institute under Contract No. DE-AC05-76RL0-1830. In situ ion irradiation work was performed at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. DOE. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology and 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. Work at the Molecular Foundry was supported by the Office of Science, Office of Basic Energy Sciences, of the U.S. DOE under Contract DE-AC02-05CH11231.